



Site Name: Wildfire Cleanup – Lahaina, Maui, Hawaii	Site	Conta	ct: Ted Brown			Tele	Telephone: 805.946.7413	
Location: Lahaina, Maui, Hawaii	Clier	t Con	tact: Marianne	Fuji Rossic	)	Tele	phone:	
EPA ID No.			By: Jonathan F	andozzi		Date	Prepared: 1.12.24 updated	9/8/24
Project No. TBD			<b>ctivities:</b> 2024-January 1	2, 2025		Eme	Emergency Response  Yes  No	
Objectives:		Site	Type: Check	as many as	applicable.			
Ambient community air monitoring for Hawaii Department of Health the wildfire cleanup response in Lahaina, Maui, Hawaii	during		Active		Landfill		Inner-City	
			Inactive		Railroad		Rural	
			Secured		Residential		Remote	
			Unsecured		Industrial		Other (specify)	
						_		
Project Scope of Work and Site Background  Tetra Tech will provide technical support to the HDOH Clean Air Branch (CAB) in efforts related to ambient community air monitoring during the wildfire response in the area of Lahaina, Maui, Hawaii. Tetra Tech will provide air monitoring field technicians and secure specialty laboratory and field equipmen subcontractors, uniquely qualified to undertake this task. Monitoring shall continue for the duration of cleanup efforts in the Lahaina area (anticipated to take months), unless requested to be lessened or extended.  Tetra Tech will conduct continuous air monitoring activities utilizing an E-BAM Mass Monitor (or equivalent). Tetra Tech personnel will conduct calibration and checks of air monitoring equipment daily. Community monitoring locations will monitor PM10 to allow for comparison to the National Ambient Air Quality S (NAAQS) and agreed upon regulatory standards for the State of Hawaii. Tetra Tech will collect ambient air samples in accordance with approved Standard C Procedures. Air sampling will occur 7 days each week.			ratory and field equipment su a area (anticipated to take up I will conduct calibration and sy onal Ambient Air Quality Stan with approved Standard Ope	ystem dards rating				
Health and Safety Approver Comments or Additional Instructio for any field personnel who desire a dust mask while performing air				toring activiti	ies is Level D. Vo	oluntar	y use of Dusk Masks are perm	itted
Health and Safety Plan Approver Signature: David Brow	/n				Da	ate:	9/9/2024	

Note: A minimum of two persons with appropriate training and medical surveillance must be on site for any fieldwork subject to Level 2 HASP requirements.

8 April 2014 Page 1 of 12



Initial Isolation and Protective Action Distances (for emergency response operations only): Use 2012 Emergency Response Guidebook (ERG) or delete Establishment of Work Zones; including exclusion, contamination reduction, and support zones; is required for ALL HAZWOPER projects. For heavy equipment (i.e. drilling operations), exclusions zone will be established around each equipment or drilling location based on site conditions and or noise levels (DCN 2-04, Hearing Conservation Program) at drilling operations (i.e. a circular exclusion zone based on noise levels >85 dbA from the drill rig or a minimum of 20 feet around the rig, whichever is greater). Work zones will be delineated using cones, barrier tape or similar visual indicators. ALL investigation-derived waste shall be drummed and remain onsite pending characterization for subsequent disposal. Spill control shall be conducted in accordance with the requirements of SWP 5-14, Spill and Discharge Control Practices. Wind Speed and Direction (approach from upwind) Probability of Weather Forecast Use www.weather.com or www.wunderground.com Temperature (°F) **Relative Humidity (%)** Precipitation (%) (such as partly cloudy, snow, etc.) Speed (mph): From Direction: Capture weather information daily on Tailgate Safety Briefing form or in site logbook Fire Extinguisher **On-Site Supplies:** First Aid Kit Air Horn Oral Thermometer Noise Dosimeter **Known or Anticipated Site Hazards or Concerns:** Work on active roadway Overhead utilities Energized electrical systems **Buried Utilities** Portable hand tool use Work over or near water Surface or underground storage tanks Portable electrical tool use Explosion or fire hazard Machine guarding Oxygen deficiency General slips, trips, falls Unknown or poorly characterized chemical hazards Portable fire extinguisher use Uneven, muddy, rugged terrain Inorganic chemicals Lift (man lift, cherry picker) use Driving personal vehicles Industrial truck (forklift) use Organic chemicals All-terrain vehicle use Asbestos Wood or metal ladder use Injury and Illness Prevention Program (California only) Dangerous goods shipped by air Respirable particulates Ergonomics (California only) Respirable silica Elevated work (over 6' high) Work in strip or shaft mines Heavy equipment use or operation Blasting and explosives Client-specific safety requirements (attach to HASP) Non-ionizing radiation (lasers, UV) Construction work Confined space entry and/or rescue Ionizing radiation (alpha, beta, gamma, etc.) Excavation or trenching Methamphetamine lab Heat stress Benching, shoring, bracing Biological hazards (i.e. ticks, snakes, poisonous plants) Scaffold use Cold stress Mold High noise Sun Exposure Other (insert) Medium **Explosion or Fire Potential:** High ⊠ Low Unknown

8 April 2014 Page 2 of 12



Chemical Products Tetra Tech EM Inc. Will Use or Store On Site: (Attach a Material	Safety Data Sheet [MSDS] for each item.)
Alconox or Liquinox Calibration gas (Methane)	Hydrogen gas Isopropyl alcohol
Hydrochloric acid (HCI)  Calibration gas (Isobutylene)	☐ Household bleach (NaOCI) ☐ HazCat Kit
☐ Nitric acid (HNO₃) ☐ Calibration gas (4-gas mixture)	☐ Sulfuric acid (H₂SO₄) ☐ Mark I Kits (number?)
☐ Sodium hydroxide (NaOH) ☐ Eyewash solution (potable water)	Hexane Other (specify)
WARNING: Eyewash solution shall be readily available on ALL projects where core	rosives (acids or bases) are used, including sample preservatives
Applicable Safety Programs and Safe Work Practices (SWP). Attach to HASP:	Tasks Performed At Job Site that are NOT Covered by SWPs
DCN 2-04 Hearing Conservation Program (always checked)	NOTE: Many AHA's can be found on the Health & Safety intranet site at:
DCN 4-05 Trenching and Excavation Safety	https://int.tetratech.com/sites/EMI/hs/Activity%20Hazard%20Analysi
DCN 4-08 Asbestos Protection Program	s%20Documents/Forms/AllItems.aspx Attach Activity Hazard Analysis (AHA) for each non-covered task
DCN 4-09 Haulage and Earth Moving	(non-covered task)
DCN 4-10 Lead Protection Program	(non-covered task)
SWP DCN 5-01 General Safe Work Practices	(non-covered task)
SWP DCN 5-02 General Safe Work Practices HAZWOPER	(non-covered task)
SWP DCN 5-03 Safe Work Practices for Office Employees	(non-covered task)
SWP DCN 5-04 Safe Drilling Practices	
SWP DCN 5-05 Safe Direct Push (GeoProbe) Practices	Tetra Tech Employee Training and Medical Requirements:
SWP DCN 5-06 Working Over or Near Water	Basic Training and Medical
SWP DCN 5-07 Use of Heavy Equipment	Initial 40 Hour Training
SWP DCN 5-08 Special Site Hazards (Firearms, Remote Sites, Mines, aircraft, etc.)	8-Hour Supervisor Training (one-time)
SWP DCN 5-09 Safe Electrical Work Practices	Current 8-Hour Refresher Training
SWP DCN 5-10 Fall Protection Practices	Current Medical Clearance (including respirator use)
SWP DCN 5-11 Portable Ladder Safety	Current First Aid Training and CPR Training
SWP DCN 5-12 Drum and Container Handling Practices  SWP DCN 5-13 Flammable Hazards and Ignition Sources	Current Respirator Fit-Test
SWP DCN 5-13 Flammable Flazards and ignition Sources  SWP DCN 5-14 Spill and Discharge Control Practices (always checked)	
SWP DCN 5-14 Spin and Discharge Control Fractices (always checked)	Other Specific Training and Medical Surveillance Requirements
SWP DCN 5-16 Cold Stress	Confined Space Training
SWP DCN 5-17 Biohazards	Level A Training
SWP DCN 5-18 Underground Storage Tank Removal Practices	Radiation Training
SWP DCN 5-19 Safe Lifting Procedures	OSHA 10-hour Construction Safety Training
SWP DCN 5-22 Hydrographic Data Collection	OSHA 30-hour Construction Safety Training
SWP DCN 5-23 Permit-Required Confined Space Entry Practices	☐ Asbestos Awareness Training ☐ Asbestos B-Reader X-Ray
SWP DCN 5-24 Non-Permit-Required Confined Space Entry Practices	Blood Lead Level and ZPP Pre, during and Post-Project
SWP DCN 5-26 Prevention of Sun Exposure	Urinary Arsenic Level Pre and Post-Project
SWP DCN 5-27 Respirator Cleaning Practices	Other
SWP DCN 5-28 Safe Use Practices for Use of Respirators	Other
SWP DCN 5-35 Underground Utilities, including 5-35F, Ground Disturbance Permit	
SWP DCN 5-36 Drill Rigs	

8 April 2014 Page 3 of 12



Materials Present or Suspected at Site	Highest Observed Concentration (specify units and sample medium)	Exposure Limit (specify ppm or mg/m³)	IDLH Level (specify ppm or mg/m³)	Primary Hazards of the Material (explosive, flammable, corrosive, toxic, volatile, radioactive, biohazard, oxidizer, or other)	Symptoms and Effects of Acute Exposure	Photoionization Potential (eV)
<b>Gasoline</b> - Liquid	NE	PEL = NE REL = NE TLV = NE [Skin] Hazard	NE	Flammable, Explosive	May cause irritation to respiratory system or eyes; lung fibrosis. Potential carcinogen.	NE
<b>Diesel Fuel</b> - Liquid	NE	PEL = NE REL = NE TLV = NE [Skin] Hazard	NE	Flammable, Explosive	Highly Corrosive. May cause irritation eyes, skin, nose, throat; pulmonary edema, bronchitis; emphysema; conjunctivitis; stomatis; dental erosion; eye, skin burns; dermatitis.	NE
<b>Lead</b> - Solid	NE	PEL = 0.05 mg/m3 REL = 0.05 mg/m3 TWA TLV = 0.05 mg/m3 TWA [Skin] Hazard	100 mg/m3	Toxic	Lassitude (weakness, exhaustion), insomnia; facial pallor; anorexia, weight loss, malnutrition; constipation, abdominal pain, colic; anemia; gingival lead line; tremor; paralysis wrist, ankles; encephalopathy; kidney disease; irritation eyes; hypertension.	NE
<b>Asbestos</b> - Solid	NE	PEL = 0.1 f/cc of Air (TWA) REL = Carcinogen; 0.1 f/cc of Air TWA TLV = 0.1 f/cc of Air TWA [Skin] Hazard	NA	Toxic	Sensitization dermatitis, allergic asthma, pneumonitis. Potential carcinogen.	NE
Crystalline Silica	NE	PEL = 50 µg/m³ TWA0.5 mg/m3 REL = 0.5 mg/m3 TWA TLV = 0.5 mg/m3 TWA [Skin] Hazard	Carcinogen; [25 mg/m³ (cristobalite, tridymite); 50 mg/m³ (quartz, tripoli)]	Other	Cough, dyspnea (breathing difficulty), wheezing; decreased pulmonary function, progressive resp symptoms (silicosis); irritation eyes. Potential carcinogen.	NE
Cobalt	NE	NIOSH REL TWA 0.05 mg/m3 OSHA PEL TWA 0.1 mg/m3 [Skin] Hazard	20 mg/m3	Toxic	Cough, dyspnea (breathing difficulty), wheezing, decreased pulmonary function; weight loss; dermatitis; diffuse nodular fibrosis; resp hypersensitivity, asthma	NE
Arsenic	NE	PEL = 0.01 mg/m3 TWA REL = 0.002 mg/m3 TWA TLV = 0.01 mg/m3 [Skin] Hazard	5 mg/m3	Toxic	Dermatitis, gastrointestinal disturbances, peripheral neuropathy, respiratory irritation, hyperpigmentation of skin. Potential carcinogen.	NE

Specify Information Sources: For example: NIOSH Pocket Guide to Hazardous Chemicals, September 2005 and American Conference of Governmental Industrial Hygienists (ACGIH). "Threshold Limit Values and Biological Exposure Indices for 2013."

Note: In the Exposure Limit column, include Ceiling (C) and Short-Term Exposure Limits (STEL) if they are available. Also, use the following short forms and abbreviations to complete the table above.

A = Air Ca = Carcinogenic eV = Electron volt U = Unknown IDLH = Immediately dangerous to life or health

mg/m³ = Milligram per cubic meter NA = Not available





NE = None established

PEL = Permissible exposure limit

ppm = Part per million

REL = Recommended exposure limit

S = Soil

TLV = Threshold limit value

GW = Groundwater

SW = Surface water

Sed = Sediment

8 April 2014 Page 5 of 12



Note: If no contingency level of protection is selected, all employ require upgrading PPE. Level A field work typically requires a Level					
Field Activities Covered Under this HASP:					
				rotection <sup>1</sup>	Date of
Task Description			Primary	Contingency	Activities
Ambient air monitoring and sample collection for asbestos, elemental meta	ls, and particulates (PM10)	A	BCXD	□ A □ B □ C ⊠ D	1.12.24 – 1.12.25
2		☐ A	B C D	□ A □ B □ C □ D	
3		ПА	BCDD	□ A □ B □ C □ D	
4		ПА	BCDD	□ A □ B □ C □ D	
5			BCDD	□ A □ B □ C □ D	
Site Personne	el and Responsibilities (incl	ude su	bcontractors):		
Employee Name and Office Code / Location	Task(s)			Responsibilities	
Eric Jensen Chelsea Saber		•	(SSC) aware of pertine communications with cl	ges the overall project, makes site nt project developments and plans ient as necessary. Additionally, Fo cutive week on-site, the PM is resp dit using Form AF-1.	, and maintains or projects lasting
Karysa Saldana	1	•	(SSC) aware of pertine	rects field activities, makes site sa nt project developments and plans e Project Manager and the client a	, and maintains
Karysa Saldana	1	•	protective equipment (F site personnel and sub- are or may be exposed enforces the HASP; ide communicates site haz	(SSC): Ensures that appropriate PPE) is available, enforces proper contractors; suspends investigative to an immediate health hazard; in entifies and controls site hazards wards to all personnel; and reports a ted conditions described in the hear representative.	use of PPE by on- e work if personnel aplements and hen possible; any deviations
Lena Diaz, Shaina Epstein, TBD	1		Field Personnel: Comp team leader, and SSC,	poordinator (if any) letes tasks as directed by the proje and follows the HASP and all SWI Tech, Inc., Health and Safety Ma	s and guidelines

### Note:

1. See next page for details on levels of protection

8 April 2014 Page 6 of 12



NOTE: Contingency level of protection section should be completed only if the upgraded level of protection is immediately available at the job site. If no contingency level of protection is denoted, all employees covered under this HASP must evacuate the immediate site area if air contaminant levels would require an upgrade of PPE.

Protective Equipment: (Indicate type or material as necessary for each task.)

Tas k	Primary Level of Protection (A,B,C,D)	PPE Component Description (Primary)	Contingency Level of Protection (A, B, C, D)	PPE Component Description (Contingency)
1	D (modified)	Respirator type: NA Cartridge type: NA CPC material: N/A Glove material(s): latex Boot material: steel/composite Other:	D (modified)	Respirator type: Dust Mask or N95 Mask Cartridge type (if applicable): N/A CPC material: N/A Glove material(s): latex Boot material: steel/composite Other:
2		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:
3		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:
4		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:
5		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:

#### Respirator Notes:

Respirator cartridges may only be used for a maximum time of 8 hours or one work shift, whichever is less, and must be discarded at that time. For job sites with organic vapors, respirator cartridges may be used as described in this note as long as the concentration is less than 200 parts per million (ppm), the boiling point is greater than 70 °Celsius, and the relative humidity is less than 85 percent. If any of these levels are exceeded, a site-specific respirator cartridge change-out schedule must be developed and included in the HASP using Tetra Tech Form RP-2 (Respiratory Hazard Assessment Form)

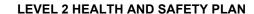
#### Notes

All levels of protection must include eye, head, and foot protection.

CPC = Chemical protective clothing

Thermoluminescent Dosimeter (TLD) Badges must be worn during all field activities on sites with radiation hazards. TLDs must be worn under CPC.

8 April 2014 Page 7 of 12





Monitoring Equipment: All monitoring equipment on site must be calibrated before and after each use and results recorded in the site logbook						
Instrument (Check all required)	Task	Instrument Reading	Action Guideline	Comments		
Combustible gas indicator model:	1 2	0 to 10% LEL	Monitor; evacuate if confined space			
	3	10 to 25% LEL	Potential explosion hazard; notify SSC			
	4 5	>25% LEL	Explosion hazard; interrupt task; evacuate site; notify SSC			
Oxygen meter model:	1 2	>23.5% Oxygen	Potential fire hazard; evacuate site			
	3	23.5 to 19.5% Oxygen	Oxygen level normal			
	4 5	<19.5% Oxygen	Oxygen deficiency; interrupt task; evacuate site; notify SSC			
Radiation survey meter model:	☐ 1 ☐ 2	Normal background	Proceed	Annual exposure not to exceed 1,250 mrem per quarter		
	3	Two to three times background	Notify SSC	Background reading must be taken in an area known to be free of radiation sources.		
	4 5	>Three times background	Radiological hazard; interrupt task; evacuate site; notify RSO			
Photoionization detector model:	1	Any response above background to 5 ppm above background	Level B is recommended Level C <sup>a</sup> may be acceptable	These action levels are for unknown gases or vapors. After the contaminants are identified, action levels should be based on the		
☐ 11.7 eV ☐ 10.6 eV ☐ 9.8 eV	2 3	> 5 to 500 ppm above background	Level B	specific contaminants involved.		
Other (specify):	☐ 4 ☐ 5	> 500 ppm above background	Level A			
Flame ionization detector model:	1 2	Any response above background to 5 ppm above background	Level B is recommended Level C <sup>a</sup> may be acceptable	These action levels are for unknown gases or vapors. After the contaminants are identified, action levels should be based on the		
	3	>5 to 500 ppm above background	Level B	specific contaminants involved.		
	4 5	>500 above background	Level A			
Detector tube models:	1 2 3 4 5 5	Specify: < 1/2 the PEL > 1/2 the PEL	Specify:	The action level for upgrading the level of protection is one-half of the contaminant's PEL. If the PEL is reached, evacuate the site and notify a safety specialist		
Other (specify): E-BAM	1 2 3 4 5 5	Specify: >PM25.35 ug/m3 TWA >PM 10 150 ug/m3 TWA	Specify: Monitor. Notify SSC			

Notes:

eV= electron volt LEL=Lower explosive limit mrem=Millirem PEL=Permissible exposure limit ppm=Part per million

a. Level B is required when chemical hazards are present but are uncharacterized. Level C may be acceptable for certain tasks in some situations. If you are uncertain, consult your Safety Manager.



## **LEVEL 2 HEALTH AND SAFETY PLAN**

Project-Specific Industrial Hygiene Requirements	Emergency Contacts:		Telephone No.
OSHA-Regulated Chemicals*:	WorkCare and Incident Intervention	on 888.449.	7787, or 800.455.6155
Check any present on the job site in any medium (air, water, soil)	Tetra Tech EMI 24-hour Anonymo	ous Hazard Reporting Line	866.383.8070
No chemicals below are located on the job site	U.S. Coast Guard National Respo	onse Center	800.424.8802
Friable Asbestos Silica, crystalline	InfoTrac		800.535.5053
Silica, crystalline	Poison Control		800.222.1222
alpha-Napthylamine	Fire department		911
alpha-Napthylamine Methyl chloromethyl ether 3,3'-Dichlorobenzidine (and its salts) bis-Chloromethyl ether beta-Napthylamine Benzidine 4-Aminodiphenyl Ethyleneimine beta-Propiolactone 2-Acetylaminoflourene 4-Dimethylaminoazobenzene	·		911
3,3'-Dichlorobenzidine (and its salts)	Police department		911
bis-Chloromethyl ether	Personnel Call-Down List:		
beta-Napthylamine	Job Title or Position:	Name	Cell Phone:
Benzidine	Safety Manager	Chalana Cahan	702 400 0074
4-Aminodiphenyl	Project Manager: Field Team Leader:	Chelsea Saber Ted Brown	703.489.2674 805.946.7413
Ethyleneimine	Site Safety Coordinator (SSC):	Ted Brown	805.946.7413
beta-Propiolactone	Subcontractor SSC:	NA	000.010.1110
2-Acetylaminoflourene			
	Medical and Site Emergencies:		
N-nitrosomethylamine   Vinyl chloride   Inorganic arsenic   Lead   Chromium (VI)   Cadmium   Benzene   Coke oven emissions   1,2-Dibromo-3-chloropropane   Acrylonitrile   Ethylene oxide   Formaldehyde	Signal a site or medical emergend	cy with three blasts of a loud horn	(car horn, fog horn, or
	similar device). Site personnel sh	nould evacuate to the area of safe	
Inorganic arsenic	the site map.		
	Hospital Name: Maui Men	norial Medical Center	
Chromium (VI)	Address: 221 Maha	alani St, Wailuku, HI 96793	
	General Phone:		+18082449056
☑ Benzene	Emergency Phone:		911
Coke oven emissions	Ambulance Phone:		911
1,2-Dibromo-3-chloropropane	Lloopital called to verify amorana	cy services are offered? YES	NO 🗆
Acrylonitrile	Hospital called to verify emergent	by services are offered? TES	NO 🗀
Ethylene oxide	Step-by-step Route to Hospital: (s	see Page 11 of 12 for route map)	
Methylenedianiline			
1,3-Butadiene			
Methylene chloride			
* NOTE: Many states, including California and New Jersey, have chemical-specific worker protection requirements and standards for many chemicals and known or suspected carcinogens.			

Note: This page must be posted on site.

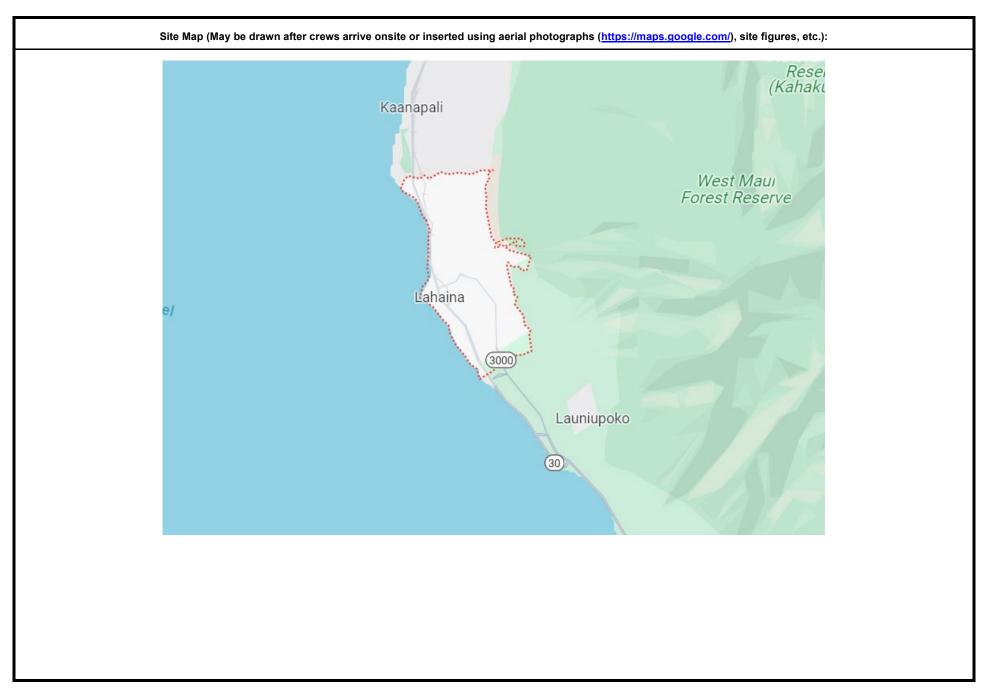
8 April 2014 Page 9 of 12



Decontaminati	on Procedures	Emergency Response Planning
The site safety coordinator overseas implementation of project decontamination procedures and is responsible for ensuring they are effective.		During the pre-work briefing and daily tailgate safety meetings, all on-site employees will be trained in the provisions of emergency response planning, site communication systems, and site evacuation routes.
Personnel Decontamination	Decontamination Equipment	In the event of an emergency that necessitates evacuation of a work task
Level D Decon - Wet Dry	Washtubs	<ul> <li>area or the site, the following procedures will take place.</li> <li>The Tetra Tech SSC will contact all nearby personnel using the on-site</li> </ul>
Level C Decon - Wet Dry	Buckets	communications to advise the personnel of the emergency.  The personnel will proceed along site roads to a safe distance upwind from
Level B Decon – Briefly outline the level B decontamination methods to be used on a	Scrub brushes	the hazard source.  The personnel will remain in that area until the SSC or an authorized
separate page attached to this HASP.	☐ Pressurized sprayer	individual provides further instructions.
Level A Decon – A Level 3 HASP is required. Notify your Safety Manager.	☐ Detergent [Type]	In the event of a severe spill or a leak, site personnel will follow the procedures listed below.
Equipment Decontamination	Solvent [Type]	Evacuate the affected area and relocate personnel to an upwind location.
	Household bleach solution	Inform the Tetra Tech SSC, a Tetra Tech office, and a site representative
All tools, equipment, and machinery from the Exclusion Zone (hot) or Contamination	Concentration/Dilution:	<ul> <li>immediately.</li> <li>Locate the source of the spill or leak, and stop the flow if it is safe to do so.</li> </ul>
Reduction Zone (warm) are decontaminated in the CRZ before they	☐ Deionized water	<ul> <li>Begin containment and recovery of spilled or leaked materials.</li> <li>Notify appropriate local, state, and federal agencies.</li> </ul>
are removed to the Support Zone (cold). Equipment decontamination procedures	Disposable sanitizer wipes	
are designed to minimize the potential for hazardous skin or inhalation exposure,	Dotable eyewash/drench/wash water	In the event of severe weather, site personnel will follow the procedures listed below.
cross-contamination, and chemical incompatibilities.	☐ Wire brush	<ul> <li>Site work shall not be conducted during severe weather, including high winds and lightning.</li> </ul>
Respirator Decontamination	Spray bottle	<ul> <li>In the event of severe weather, stop work, lower any equipment (drill rigs) and evacuate the affected area.</li> </ul>
Respirators are decontaminated in	Tubs / pools	Severe weather may cause heat or cold stress. Refer to SWPs 5-15 and 5- 16 for additional information.
compliance with SWP 5-27 and should be included with this HASP.	Banner/barrier tape	
Waste Handling for Decontamination	Plastic sheeting	All personnel working on Tetra Tech projects are expected to and responsible for reporting ANY unsafe conditions, behaviors or
Procedures for decontamination waste disposal meet all applicable local, state,	Tarps and poles	incidents including injuries, illnesses, fires, spills/releases, property damages and near-misses they face or encounter while performing
and federal regulations.	Trash bags	their work. According to TtEMI's reporting procedures, for non-emergency incidents you should:
	Trash cans	Notify WorkCare and Incident Intervention at 888.449.7787, or 800.455.6155
	Duct tape	<ul> <li>Notify your Office, Project or Safety Manager via phone immediately.</li> <li>Complete a "Tetra Tech Incident Report" (Form IR) within 24 hours and send</li> </ul>
	Paper towels	it to your Safety Manager. If an injury or illness has occurred, the Form IR-A must also be completed.
	Folding chairs	Additional reports may be necessary
	Other	

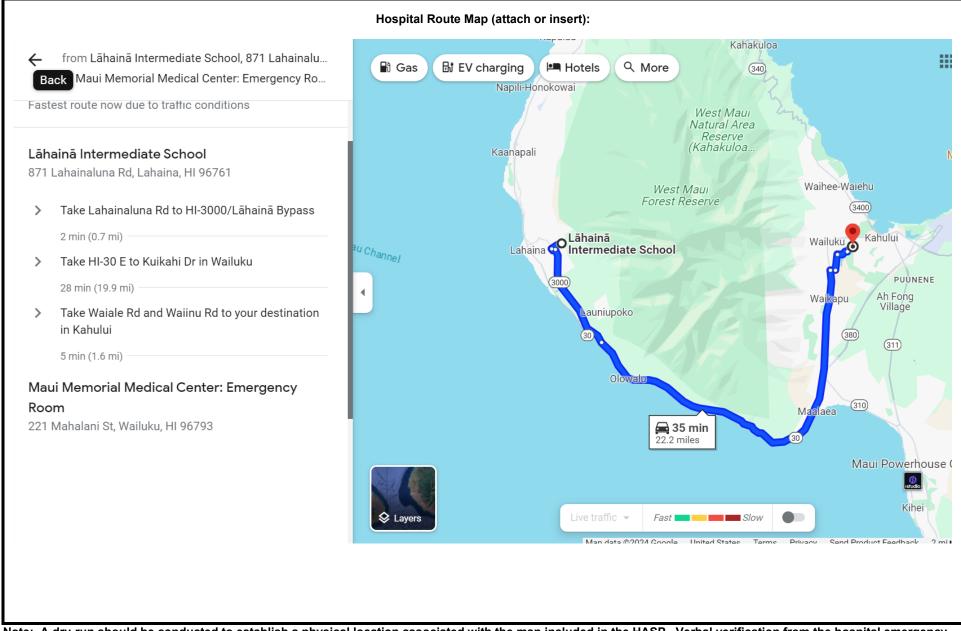
8 April 2014 Page 10 of 12





8 April 2014 Page 11 of 12





Note: A dry-run should be conducted to establish a physical location associated with the map included in the HASP. Verbal verification from the hospital emergency room should also be obtained to ensure that the hospital will accept chemically-contaminated patients.

8 April 2014 Page 12 of 12



	APPROVAL AND SIGN Project No:	-OFF FORM	
	rmation set forth in this Health and Safety Plan an	d will follow the direction of the Site Safety Coordi tand the training and medical requirements for co	
		ra Tech employees. Subcontractors, visitors, and c rafety program for specific information related to th	
Name	Company / Agency / Organization	Signature	Date
Tetra Tech, Inc., Health and Safety Manual.		n and enforce this HASP, as well as procedures ar ALL incidents and near-misses were reported at pi	
Name	Project-Specific Position	Signature	Date
	Project Manager		
	Field Team Leader		
	Site Safety Coordinator		
	Subcontractor SSC		
	Required Post-Project Field Team Check-In		
		ra Tech employees. Subcontractors, visitors, and o their safety program for specific information related	

Note: Use Additional sheets as necessary to ensure that all personnel sign and affirm this document.

8 April 2014 Page 13 of 12

#### **Emergency Contacts**

- WorkCare For issues requiring an Occupational Health Physician; assistance is available 24 hours per day, 7 days per week.
- **InfoTrac** For issues related to incidents involving the transportation of hazardous chemicals; this hotline provides accident assistance 24 hours per day, 7 days per week
- **U.S. Coast Guard National Response Center** For issues related to spill containment, cleanup, and damage assessment; this hotline will direct spill information to the appropriate state or region

Poison Control Center - For known or suspected poisoning.

#### Limitations:

#### The Level-Two HASP is not appropriate in some cases:

- Projects involving unexploded ordnance (UXO), radiation sources as the primary hazard, or known chemical/biological weapons site must employ the Level 3 HASP
- Projects of duration longer than 90 days may need a Level 3 HASP (consult your RSO)

#### **Decontamination:**

- Decontamination Solutions for Chemical and Biological Warfare Agents<sup>a</sup>: PPE and equipment can be decontaminated using 0.5 percent bleach (1 gallon laundry bleach to 9 gallons water) for biological agents (15 minutes of contact time for anthrax spores; 3 minutes for others) followed by water rinse for chemical and biological agents. In the absence of bleach, dry powders such as soap detergents, earth, and flour can be used. The powders should be applied and then wiped off using wet tissue paper. Finally, water and water/soap solutions can be used to physically remove or dilute chemical and biological agents. Do not use bleach solution on bare skin; use soap and water instead. Protect decontamination workers from exposure to bleach.
- **Decontamination for Radiological and Other Chemicals:** Primary decontamination should use Alconox and water unless otherwise specified in chemical specific information resources. The effectiveness of radiation decontamination should be checked using a radiation survey instrument. Decontamination procedures should be repeated until the radiation meter reads less than 100 counts per minute over a 100-square-centimeter area when the probe is held 1 centimeter from the surface and moving slower than 2.5 centimeters per second.
- **Decontamination Corridor:** The decontamination setup can be adjusted to meet the needs of the situation. The decontamination procedures can be altered to meet the needs of the specific situation when compoundand site-specific information is available.
- **Decontamination Waste:** All disposable equipment, clothing, and decontamination solutions will be double-bagged or containerized in an acceptable manner and disposed of with investigation-derived waste.
- **Decontamination Personnel:** Decontamination personnel should dress in the same level of PPE or one level below the entry team PPE level.
- All investigation-derived waste should be left on site with the permission of the property owner and the EPA on-scene coordinator. In some instances, another contractor will dispose of decontamination waste and investigation-derived waste. DO NOT place waste in regular trash. DO NOT dispose of waste until proper procedures are established.

#### Notes:

<sup>a</sup> Source: Jane's Information Group. 2002. Jane's Chem-Bio Handbook. Page 39.



# TETRA TECH, INC. DAILY TAILGATE SAFETY MEETING FORM

Date:	Time:		Project No.:
Client:		Site Location:	
Site Activities Planned for Toda	ıy:		
Weather Conditions:	· · · · · · · · · · · · · · · · · · ·		
	Safety To <sub>l</sub>	oics Discussed	
Protective clothing and equip	oment:		
Chemical and physical hazar	ds:		
Emergency procedures:			
Equipment hazards:			
Equipment nazarus.			
Other:			
	At	tendees	
Printed Nam	ne		Signature
Masting Cander to the			
Meeting Conducted by:			
Name	· · · · · · · · · · · · · · · · · · ·	Signature	



# TETRA TECH EM INC. HEALTH AND SAFETY PLAN AMENDMENT

Site Name:			_
Amendment Date:			
Purpose or Reason for A	Amendment:		_
Required Additional Saf	e Work Practices or Act	tivity Hazard Analyses:	
Required Changes in PF	PE:		
Action Level Changes:			
	AMENDME	NT APPROVAL	
DSO or Deciman			
RSO or Designee _	Name	Signature	Date
Site Safety _ Coordinator	Name	Signature	Date
Date presented during of	lailv site safetv meeting	:	



# TETRA TECH, INC. FIELD AUDIT CHECKLIST

Project Name: _	Project No.:	
Field Location: _	Completed by:	
Project Manager	Site Safety Coordinator:	

	In Compliance?			
Health	n and Safety Plan Requirements	Yes	No	NA
1	Approved health and safety plan (HASP) on site or available			
2	Names of on-site personnel recorded in field logbook or daily log			
3	HASP compliance agreement form signed by all on-site personnel			
4	Material Safety Data Sheets on site or available			
5	Designated site safety coordinator physically present on jobsite			
6	Daily tailgate safety meetings conducted and documented on Form HST-2			
7	Documentation available proving compliance with HASP requirements for medical examinations, fit testing, and training (including subcontractors)			
8	HASP onsite matches scope of work being conducted			
9	Emergency evacuation plan in place and hospital located			
10	Exclusion, decontamination, and support zones delineated and enforced			
11	HASP attachments present onsite (VPP sheet, audit checklist, AHA, etc.)			
12	Illness and injury prevention program reports completed (California only)			
Emer	gency Planning			
13	Emergency telephone numbers posted			
14	Emergency route to hospital posted			
15	Local emergency providers notified of site activities			
16	Adequate safety equipment inventory available			
17	First aid provider and supplies available			
18	Eyewash solution available when corrosive chemicals are present			
Air Mo	pnitoring			
19	Monitoring equipment specified in HASP available and in working order			
20	Monitoring equipment calibrated and calibration records available			
21	Personnel know how to operate monitoring equipment and equipment manuals available on site			
22	Environmental and personnel monitoring performed as specified in HASP			

Safety Items				In Compliance?			
Personal Protection				No	NA		
23	Splash suit, if required						
24	Chemical protective clothing, if required						
25	Safety glasses or goggles (always required)						
26	Gloves, if required						
27	Overboots, if required						
28	Hard hat (always required)						
29	High visibility vest, if required						
30	Hearing protection, if required						
31	Full-face respirator, if required						
Instru	ımentation						
32	Combustible gas meter and calibration notes						
33	Oxygen meter and calibration notes						
34	Organic vapor analyzer and calibration notes						
Supp	lies						
35	Decontamination equipment and supplies						
35	Fire extinguishers						
37	Spill cleanup supplies						
Corrective Action Taken During Audit:							
Note: NA = Not applicable							
Audit	Auditor's Signature Site Safety Coordinator's Signature						
Date							

						ACTIVITY HAZARD A	NALYSIS (AHA)
Tetra Tech.	lne.						Tetra Tech EM Inc.
	11151	(Insert Task Name Here)					
				Task Desc	ription		
	ial hazards po and training.	osed by each ma The hazard con	ajor step in this trols listed belo	task, lists proce	edures to control ha	ed by the Director of Health and Safet azards, and presents required equipme	
Below, go step by step (i.e. PPE, lock-out tagou						and describe the "actions" taken to elow.	control the hazard
T 101	Hazards			0 111 10 6		Actions	
Task Steps	<del> </del>	Potential Haz	zards_	Critical Safet	y Procedures and	Controls	
Insert additional rows a	s neeaea						
Equipment to be Used		Inspection R	<u>equirements</u>	Training Reg	<u>uirements</u>		
Assessed By	Nan	ne -	Signati	ure	Date	_	
Approved By						_	

Date

Signature

Name



Revision Date: 10/2021 Country: USA **Document Control Number** 02-04 Page 1 of 7

Industry:

\_X\_ General \_\_X\_ Construction \_\_

Mining \_\_X\_ Hazardous Materials

#### 1.0 **PURPOSE**

Tetra Tech, Inc. (Tetra Tech) has established a Hearing Conservation Program to protect employees from the harmful effects of noise exposure. This program is designed to comply with the Occupational Safety and Health Administration (OSHA) occupational noise exposure standard in Title 29 of the Code of Federal Regulations (CFR), Part 1910.95, as well as federal, state, local, and contractual requirements.

Electrical

#### 1.1 SCOPE

Tetra Tech's hearing conservation program applies to all employees who have potential exposure to noise above 85 dBA. This program will be made available to employees and their representatives. The Hearing Conservation Program will outline the following requirements:

- Roles and Responsibilities,
- Action levels,
- Monitoring,
- Employee notification,
- Audiometric testing,
- Hearing protection,
- Warning signs and information, and
- Training.

#### 2.0 **ROLES AND RESPONSIBLITIES**

Tetra Tech firmly believes protecting the health and safety of our employees is everyone's responsibility.

### 2.1 OU Health and Safety Representatives (HSR)



Revision Date: 10/2021

Country: USA

Document Control Number

O2-04

Page 2 of 7

Mining X Hazardous Materials

Industry: \_\_X\_ General \_\_X\_ Construction \_\_\_\_ Electrical \_\_\_ Mining \_\_X\_ Hazardous Materials

- Implementing and managing the hearing conservation program within their respective organizations.
- Maintaining records of all noise exposure measurements for at least two (2) years,
- Identifying employees to be included in the audiometric testing program and for scheduling audiometric exams through the Tetra Tech corporate medical provider.

## 2.2 Project Managers (PM), Managers, and Supervisors

 Ensuring compliance with hearing conservation controls and protection at their project sites.

### 2.3 Site Safety Coordinators (SSCs)

- Assisting project managers and the HSR with Implementing the hearing conservation controls within a project or on a site-specific basis.
- Identifying noise control areas and operations

### 2.4 Affected Employees

- Wearing appropriate hearing protection devices,
- Following hearing conservation procedures in noise control areas.
- Completing all required training

### 3.0 PROGRAM ELEMENTS

### **Permissible Exposure Limits**

The following table identifies OSHA permissible exposure limits for noise exposures. Whenever possible, administrative or engineering controls will be used to reduce sound levels. If controls are



dBA or greater.

Industry:

# TETRA TECH, INC. HEARING CONSERVATION PROGRAM

\_X\_ General \_\_X\_ Construction \_\_

Revision Date: 10/2021

Country: USA

Document Control Number

O2-04

Page 3 of 7

Mining X Hazardous Materials

not feasible or fail to reduce sound levels to below 85 dBA, hearing protection will be provided to employees to reduce sound exposures to below the 85 dBA limit. This Tetra Tech hearing conservation program *mandates* the use of hearing protection for 8-hour. TWA exposures of 85

Electrical

TABLE 1 - PERMISSIBLE NOISE EXPOSURES\*

Duration per day, hours	Sound level dBA slow response
8	90
6	92
4	95
3	97
2	100
1-1/2	102
1	105
1/2	110
1/4 or less	115

<sup>\*</sup> When the daily noise exposure is composed of two or more periods of noise exposure of different levels, their combined effect should be considered, rather than the individual effect of each. If the sum of the following fractions: C(1)/T(1) + C(2)/T(2) C(n)/T(n) exceeds unity, then, the mixed exposure should be considered to exceed the limit value. On indicates the total time of exposure at a specified noise level, and Tn indicates the total time of exposure permitted at that level. Exposure to impulsive or impact noise should not exceed 140 dB peak sound pressure level.

### Monitoring

In most instances, high noise levels at a project site are generated by heavy equipment, such as drill rigs and backhoes, or sources associated with the work site operations such as operating equipment and vehicles. Most common high-noise-level sources have been measured, and instances where hearing protection is required shall be indicated in the site-specific hazard assessment documents such as a health and safety plan (HASP), construction health and safety plan (C-HASP), job hazard analysis (JHA), job safety analysis (JSA), or permit.

When noise exposures at a work site are suspected to equal or exceed an 8-hour, TWA of 85 dBA resulting from noise sources not previously measured, a qualified person will conduct noise sampling/monitoring to characterize the noise sources and exposure levels.



Revision Date: 10/2021

Country: USA

Document Control Number

02-04

Page 4 of 7

Industry: \_\_X\_ General \_\_X\_ Construction \_\_\_ Electrical \_\_\_ Mining \_\_X\_ Hazardous Materials

A portable sound-level meter is recommended for surveying general work areas and for estimating noise exposure when the noise levels are relatively constant. Noise dosimeters are recommended for documenting full-shift noise exposures when noise sources fluctuate, are intermittent, or otherwise difficult to document with the sound-level meter. Monitoring for occupational noise exposure will be conducted for each representative task or job position identified during the hazard assessment process. The HSR shall assist with sound level monitoring and reporting as necessary.

All noise measurements will be taken in the hearing zone of the affected employee. The hearing zone is an area within a radius not to exceed 12 inches from the ear closest or in most direct proximity to the noise source.

Monitoring equipment must be in factory calibration and will be checked in the field with an appropriate field calibration check standard according to the equipment manufacturer's recommendation before and after each set of measurements. Documentation of test field calibration checks will be kept with the field data collected.

In some cases, such as on short-term projects, the SSC may forgo actual noise level measurements and use a simple rule-of-thumb test to determine if noise levels are in excess of 85 dBA. The test requires the SSC to determine how loud he or she must speak to be heard at arm's length from another person. If the SSC must raise his or her voice to be heard, average noise levels likely exceed 85 dBA.

### **Employee Notification**

The SSC is responsible for informing employees exposed at or above an 8-hour, TWA of 85 dBA of the results of the monitoring.

### Audiometric testing

Audiometric testing shall be conducted for all Tetra Tech employees potentially exposed to sounds levels greater than 85 dBA time weighted average (TWA). The audiometric testing program consists of baseline audiograms, annual audiograms, and termination audiograms.

Within 6 months of an employee's first exposure at or above the action level, Tetra Tech will establish a valid baseline audiogram against which future audiograms can be compared. When a



Revision Date: 10/2021					
Country: USA					
Document Control Number					
02-04					
Page 5 of 7					

Industry: \_\_X\_ General \_\_X\_ Construction \_\_\_\_ Electrical \_\_\_ Mining \_\_X\_ Hazardous Materials

mobile van is used, the baseline shall be established within 1 year. When employees are scheduled for a baseline audiogram, exposure to workplace noise for 14-hour proceeding the exam is prohibited. Operating units can meet this requirement with the use of hearing protection.

Employees who are exposed at or above the 8-hour TWA of 85 dBA will be required to completed annual audiogram testing. These tests will be compared to the employee's baseline audiogram to determine if a standard threshold shift has occurred. If a standard threshold shift does occur, the employee will be informed of this shift in writing, within 21 days of the determination.

When a standard shift is identified, unless determined by a physician that the shift is not work related or aggravated by occupational noise exposure, the PM, Site Supervisor and SSC shall ensure that the employee's hearing protection is refitted with hearing protection that offers greater attenuation (if necessary) and the employee is retrained in the use of hearing protection.

Employees will be informed of the results of these tests at the time of their examination. Audiometric test results will be retained for Tetra Tech by the corporate medical advisor and will become a part of each employee's permanent medical record. Medial records will be maintained as required by regulation and according to Tetra Tech's DCN 01-04 Recordkeeping and Reporting Requirements. Exposure and audiometric records will be made available to employees upon request.

### **Hearing Protection**

Hearing protection will be made available to employees exposed to an 8-hour time weighted average (TWA) of 85 dBA or more. The selection of hearing protection will consider the specific noise environments in which the hearing protection will be used and will provide sufficient attenuation to limit employee noise exposure to an 8-hour TWA of less than 85 dBA. Employees will be given an opportunity to select hearing protection from pre-approved options. Hearing protection will be provided to employees at no cost and replaced as necessary. The Site-Safety Coordinator will supervise the correct use of hearing protection at a work site and ensure proper fitting of hearing protection for employees. Employees will receive training in the use and care of all hearing protectors, including how to obtain a proper fit.

### Warning Signs and Information

The SSC will post "Hearing Protection Required" signs in areas where noise levels have been measured and determined to exceed the 85-dBA, TWA action level. Signs may also be posted in



\_\_X\_ General \_\_X\_\_ Construction \_\_

	Revision Date: 10/2021				
	Country: USA				
	<b>Document Control Number</b>				
М	02-04				
	Page 6 of 7				
Mining	X Hazardous Materials				

\_\_\_\_\_<del>-</del>\_\_\_\_<del>-</del>

Electrical

areas where monitoring has not been conducted but noise levels are expected to exceed the 85-dBA, TWA level based on similarity to past activities or on the judgment of the SSC.

For short-duration projects or where personnel exposure in the high-noise area is limited and controlled, the SSC may provide verbal notice of the need for hearing protection in place of the signs described above.

## 4.0 Training

Hearing conservation training may be conducted as a stand-alone course or may be included in HAZWOPER, construction safety, or other health and safety training. Hearing Conservation training will include the following:

- Effects of noise on hearing;
- The purpose of hearing protectors;
- The advantages, disadvantages, and attenuation of various types of hearing protection;
- Instruction on selection, fitting, use, and care of hearing protectors; and
- The purpose of audiometric testing and an explanation of the test procedure.

Employees within the Hearing Conservation Program are required to take training prior to initial work assignment and annually thereafter. Documentation of initial and refresher training will be through class attendance records and course agendas.

### **DEFINITIONS**

**Attenuation** —The reduction of sound level received by the ear through use of hearing protectors or engineering controls.

**Audiogram** —A chart, graph, or table that is derived from audiometric testing and that depicts an individual's hearing sensitivity. An audiogram shows hearing threshold level (HTL) measured in decibels (dB) as a function of frequency measured in hertz (Hz). A baseline audiogram is one against which future audiograms are compared.



Revision Date: 10/2021

Country: USA

Document Control Number

02-04

Page 7 of 7

Industry: \_\_X\_ General \_\_X\_ Construction \_\_\_\_ Electrical \_\_\_ Mining \_\_X\_ Hazardous Materials

**Decibel (dB)** —A unit on a logarithmic scale for measuring the relative intensity of sound levels detectable by the human ear. The value 1 represents the faintest audible sound; the threshold of pain is 140 dB. The abbreviation dBA indicates the A weighting scale, which reflects perceived loudness as opposed to actual sound intensity.

**Dosimeter** —A device that accumulates an individual's exposure to a substance over time; in the context of hearing conservation, an instrument that measures the amount of noise energy received by the employee over a time period compared with an allowable amount.

**Sound level meter (SLM)** —An instrument for measuring sound pressure levels in decibels. Standard Threshold Shift (STS)—A change in the hearing threshold, relative to the baseline audiogram, of an average of 10 dB or more in either ear at frequencies of 2000, 3000, and 4000 Hz.

**Time-weighted average (TWA)** —A value, expressed in dBA, that represents the average noise exposure measured over a typical workday (usually 8-hour or 12-hour).

#### **REFERENCES AND STANDARDS**

ANSI, S1.4-1983, "Specifications for Sound Level Meters"; S3.6-1969, "Specifications for Audiometers"; S1.32-1980, "Specifications for Personal Noise Dosimeters." OSHA, Department of Labor, 29 CFR 1910.95, "Occupational Noise Exposure."

## RELATED PROGRAMS, PROCEDURES AND FORMS

DCN 01-04 – Recordkeeping and Reporting Requirements DCN 02-25 - Medical Oversight Program

Revision Date		Revision Details			
Revision Date	Role	Name	Approval Date	Nevision Details	
10/2008	Author	Chris McClain	10/2008	Update from 1998 format	
10/2021	Author	Amber Bill	10/2021	Update format, Roles & Responsibilities,	
,	Sponsor	Chris McClain	10/2021	Training Requirements	



Revision Date: 2/2023

Country: USA

Safe Work Practice

04-08

Page 1 of 12

Industry:

X General X Construction \_\_\_\_ Electrical X Mining \_\_\_\_ Hazardous Materials

#### 1.0 PURPOSE

The purpose of this program is to provide guidance in the proper maintenance, handling, removal, and disposal of asbestos-containing materials (ACM) or presumed ACM (PACM). Proper control of asbestos fibers will minimize the potential for asbestos exposure-related illnesses such as lung cancer and mesothelioma. The requirements stated in this policy reflect EPA, OSHA (29 CFR 1910.1001 and 1926.1101), and DOT rules. In addition, Tetra Tech must follow applicable client requirements along with state and local regulations, which may be more stringent than federal regulations. Work on Mine Safety and Health Administration (MSHA) regulated sites will follow this program in addition to any MSHA specific requirements outlined in site specific procedures.

### 1.1 SCOPE

This program shall apply to any project or work activities that have the potential to disturb asbestos-containing materials (ACM), presumed asbestos-containing materials (PACM), or suspect materials are present that may exposure personnel to asbestos fibers.

Tetra Tech, Inc. does not participate in asbestos removal services, and, as such, shall employ qualified contractors for those instances where disturbance, repair, or removal of asbestos is necessary. The contractor shall assign a competent person, as defined in 29 CFR 1926.1101, to oversee asbestos related activities and provide information to Tetra Tech regarding progress of the work and safety precautions necessary when working near the asbestos related activities.

The following sections describe the requirements for job sites with asbestos-containing materials (ACM) or presumed asbestos-containing materials (PACM).

#### 2.0 ROLES AND RESPONSIBLITIES

### 2.1 Project Managers / Site-Supervisors

Project managers (PM)/supervisors are responsible for the following:

- Ensure asbestos containing materials are identified and properly removed prior to any remodeling, repair, refurbishing, or new construction that may disturb ACM, PACM, or suspect asbestos-containing material.
- Identify and hire appropriately certified contractors for the identification, handling, removal, and disposal of ACM. Contractors will be certified and trained to the Environmental



Revision Date: 2/2023

Country: USA

Safe Work Practice

04-08

Page 2 of 12

Industry: X General X Construction \_\_\_\_ Electrical X Mining \_\_\_\_ Hazardous Materials

Protection Agency (EPA) model accreditation plan training requirements as specified in 40 CFR part 763.

- When responsible to do so, notify employees, prospective contractors, multi-employer
  worksites, and building occupants of the locations where ACM is present on a worksite, the
  quantity of ACM they may encounter, and the precautions that must be taken to control
  airborne asbestos fibers.
- Ensure personnel involved in the project where the removal of ACM and asbestos containing materials has the appropriate level of documented training for the work they are to perform. (See section 4.0 for Class Level of Operations)
- When deemed as the general contractor on a construction project, exercise general supervisory authority over the work covered by this program.

## 2.2 Health and Safety Representatives

Health and Safety Representatives are responsible for the following:

- Maintain a register of preferred contractors for ACM removal.
- Determine the health surveillance monitoring and reporting requirements for relevant workers.
- Maintain a record of the health surveillance results and reasons for initiating health surveillance in the worker's medical file.
- Notify the worker of the results of health surveillance and any other related exposure information (e.g., exposure monitoring results).

### 2.3 Employees

Employees affected by this program are responsible for the following:

- Do not knowingly disturb ACM, PACM, or suspect asbestos-containing materials without proper training and controls in place.
- Report any disturbed or deteriorated ACM, PACM, or suspect asbestos-containing material to a supervisor or project manager.



Revision Date: 2/2023

Country: USA

Safe Work Practice

04-08

Page 3 of 12

Industry: X General X Construction \_\_\_\_ Electrical X Mining \_\_\_\_ Hazardous Materials

- Complete the appropriate level of asbestos training based on the work activities performed and exposure potential.
- Properly wear and maintain personal protective equipment issued.
- Abide by signage and barriers established within the work zone.

### 3.0 PROJECT HAZARD ASSESSMENT AND SURVEYS

Tetra Tech worksites are required to perform a project hazard assessment and will document the process using an Activity Hazard Analysis (AHA) or similar document. This will be done prior to starting work and when the scope of work changes. During the hazard assessment process if ACM or PACM is identified, it will be necessary to establish and implement appropriate safety procedures for the identification, removal, and disposal of ACM materials. This may include conducting or reviewing ACM surveys to properly assess for potential exposure to onsite employees.

When Tetra Tech is the general or primary contractor, Tetra Tech will coordinate ACM surveys to be conducted by an authorized and qualified person prior to any activity which involves demolition or removal of building materials. In the event operations involve only small disturbances, such as removal of vinyl asbestos tile to allow coring through a concrete floor, the survey scope may be limited to the affected area only. When required by the scope of the project, Tetra Tech will only hire contractors who meet local, state, and federal regulatory requirements for ACM removal and disposal. Tetra Tech may also be responsible for the notification and communication of onsite hazards (section 5.0).

When ACM or PACM is identified on a worksite where Tetra Tech is a subcontractor, it will be necessary to gather the appropriate information from the ACM survey performed to properly address employee safety procedures for the Tetra Tech employees. Based on Tetra Tech employee exposure and work to be performed, it may be necessary to provide employee training (section 7.0).

### 4.0 ASBESTOS REMOVAL REQUIREMENTS AND PROCEDURES

This section was written to serve as guidance to Project Managers, Health and Safety Representatives and Employees to better understand the general and class specific work requirements for ACM or PACM removal. Reviewing this document does not certify anyone as competent to perform this type of work.

### 4.1 General Requirements



Revision Date: 2/2023

Country: USA

Safe Work Practice

04-08

Page 4 of 12

Industry:

X General X Construction \_\_\_\_ Electrical X Mining \_\_\_\_ Hazardous Materials

- Removing and handling of ACM must comply with all applicable local, state, and federal regulations and the requirements of this program.
- ACM removal and repair shall be conducted by qualified contractors only.
- For Class 1, Class II, and Class III asbestos work, other employers at the site must be informed for the nature of the work and the measures that will be taken to prevent or control exposure to asbestos fibers (e.g., the use of wet methods and regulated areas).
- ACM work areas shall be restricted and demarcated in a manner that minimizes the number of persons within the area and protects persons outside the area from exposure to airborne asbestos.
- Contractors performing removal projects shall utilize containment techniques necessary, and required under regulation, to limit release of airborne asbestos.
- Contractors shall perform suitable decontamination of the removal or repair area and
  clearance monitoring necessary to ensure safe occupancy or use of the area following
  completion of the work. Removal methods must include impermeable drop cloths or plastic
  sheeting, wet methods and a HEPA vacuum to collect dust. Debris must be bagged at the end
  of the workday.
- No sanding, grinding, or mechanical chipping, cutting, sawing or dry sweeping of ACM is permitted.
- No use of compressed air to remove asbestos, or materials containing asbestos, unless the compressed air is used in conjunction with an enclosed ventilation system.
- Utilization of employee rotations as a means to reducing employee exposure is prohibited.
- Electrical circuits shall be deactivated, unless equipped with ground-fault circuit interrupters.
- Records of exposure monitoring, medical surveillance, training, and required notifications must be retained.

## 4.2 Class I and II Asbestos Work

 Class I and II asbestos work must be performed by a trained, licensed, and qualified asbestos removal contractor.



Revision Date: 2/2023

Country: USA

Safe Work Practice

04-08

Page 5 of 12

Industry: X General X Construction Electrical X Mining Hazardous Materials

- The work must be supervised and inspected frequently and regularly by a Competent Person trained in all aspects of asbestos removal and handling.
- Competent Persons are responsible for conducting initial exposure assessments and documenting a Negative Exposure Assessment, when applicable.
- If a Negative Exposure Assessment has not been made, the following additional requirements apply:
  - Daily personal exposure monitoring must be performed.
  - Demarcation signs must also include the following statement: "Respirators and Protective Clothing Are Required in This Area"
  - Workers must wear protective clothing. A decontamination area with HEPA vacuum and equipment-cleaning capabilities must be provided adjacent to the regulated area.
  - Workers must wear respiratory protection. A half-mask respirator with a HEPA cartridge is required at a minimum.
- Qualified persons are responsible for conducting daily exposure monitoring when a Negative Exposure Assessment is not documented. Daily exposure monitoring may be discontinued when measurements are below the allowable PELs and ELs.
- When alternative control methods are proposed for Class I asbestos work, the work must be certified by a CIH or PE who is also qualified as a project designer.
- Employee notification of monitoring results must occur as soon as possible, but no later than 5 working days after the receipt of the results. Results may be provided in writing or be posted in an appropriate area.
- Critical barriers shall be placed over all openings to the regulated area or other isolation method used to prevent migration of airborne asbestos.



Revision Date: 2/2023

Country: USA

Safe Work Practice

04-08

Page 6 of 12

Industry: X General X Construction E

Electrical X Mining Hazardous Materials

- The material shall be wetted prior to and during removal unless this presents a significant safety hazard.
- The material shall be removed in an intact state whenever possible.
- Cutting, abrading, or breaking ACM material shall be avoided unless it can be done with methods that control fiber release (e.g., wet methods, HEPA vacuum).
- The work must be performed within a regulated area demarcated with signs that read: "Danger, Asbestos, Cancer and Lung Disease Hazard, Authorized Personnel Only."

#### 4.3 Class III Asbestos Work

- Class III asbestos work is to be performed by workers who are trained in and qualified for Class III asbestos work.
- Competent Persons are responsible for conducting initial exposure assessments and documenting a Negative Exposure Assessment, when applicable.
- If a Negative Exposure Assessment has not been made, the following additional requirements apply:
  - Daily personal exposure monitoring must be performed.
  - Demarcation signs must also include the following statement: "Respirators and Protective Clothing Are Required in This Area"
  - Workers must wear protective clothing. A decontamination area with HEPA vacuum and equipment-cleaning capabilities must be provided adjacent to the regulated area.
  - Workers must wear respiratory protection. A half-mask respirator with a HEPA cartridge is required at a minimum.
- The work shall be performed using wet methods.
- The work must be performed within a regulated area demarcated with signs that read:
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Revision Date: 2/2023

Country: USA

Safe Work Practice

04-08

Page 7 of 12

Industry: X General X Construction \_\_\_\_ Electrical X Mining \_\_\_\_ Hazardous Materials

"Danger, Asbestos, Cancer and Lung Disease Hazard, Authorized Personnel Only."

- The work shall be performed using local exhaust, when feasible.
- When the work involves drilling, cutting, abrading, sanding, chipping, breaking, or sawing of thermal system insulation or surfacing material, the work area shall be isolated using minienclosures or glove bags and impermeable drop cloths.

### 4.4 Class IV Asbestos Work

- Class IV asbestos work shall be performed by workers who are trained to the asbestos awareness training program.
- The work shall be performed using wet methods, HEPA vacuums, and prompt clean up of debris containing ACM or PACM.
- Employees cleaning up debris and waste in a regulated area where respirators are required shall wear respirators which are selected, used, and fitted.

### 5.0 PERSONNEL NOTIFICATION AND HAZARD COMMUNICATION

Worksite requirements will include notifying and communicating the presence of ACM or PACM. The following will people need to be properly communicated regarding the worksite hazards:

- Potentially exposed employees,
- contractors, and
- building occupants

These work groups must be notified—verbally, in writing, or by posting (labeling, signage)—of the location and quantity of ACM and PACM they may encounter and the need to take precautions to contain potential airborne asbestos. Products and containers containing asbestos shall be labeled with a warning sign that reads:

 "Danger, Contains Asbestos Fibers, Avoid Creating Dust, Cancer and Lung Disease Hazard".

Regulated areas shall be marked with signs that read:



Revision Date: 2/2023
Country: USA
Safe Work Practice
04-08
Page 8 of 12

Industry: X G

X General X Construction \_\_\_\_ Electrical X Mining \_\_\_\_ Hazardous Materials

 "Danger, Asbestos, Cancer and Lung Disease Hazard, Authorized Personnel Only, Respirators and Protective Clothing Are Required in This Area".

#### 6.0 WASTE DISPOSAL

Tetra Tech does not perform ACM waste disposal procedures. ACM waste will be handled by a contractor approved to do this type of work. Disposal of asbestos-containing materials must comply with applicable local, state, and federal regulations. Disposal processing includes:

- Properly containing both friable and non-friable ACM into containers with appropriate labeling.
- Transporting ACM, including appropriately containerizing and labeling the vehicle based on the location of travel.
- Completing a hazardous waste manifest to accompany the waste during transport. This
  will be done by a competent person trained in Shipping of Hazardous Waste.

#### 7.0 TRAINING

Asbestos awareness training is required initially and annually thereafter for employees who do not perform asbestos abatement/removal activities but may be potentially exposed to asbestos at project sites or facilities through activities of the client facility and/or other contractors.

Contractors who perform Class I asbestos work must be qualified, licensed asbestos removal contractors who employ qualified personnel, including a Competent Person trained in the requirements of EPA 40 CFR 763, subpart E, appendix C and OSHA's asbestos standard 1926.1101. If the job involves the removal of RACM, the Competent Person must also be trained in the requirements of EPA asbestos regulations for demolition and renovation work (40 CFR 61.145); certification of such training must be posted on-site; and contractor personnel must also receive on-site individual training in asbestos demolition and renovation (40 CFR 61.145).

Personnel who perform Class II asbestos work must complete 8 hours of annual "hands-on" training that includes the following:

 General asbestos information (methods of recognizing asbestos, health effects, the relationship between smoking and asbestos exposure)



Revision Date: 2/2023

Country: USA

Safe Work Practice

04-08

Page 9 of 12

ndustry: X General X Construction Electrical X Mining Hazardous Materials

- Specific work practices and controls
- Nature of asbestos operations
- Respiratory protection
- Medical surveillance
- OSHA asbestos standards
- Smoking cessation programs
- Posting and labeling requirements

Personnel who perform Class III asbestos work must complete 16 hours of initial "hands-on" training and annual refresher training consistent with the training requirements specified by EPA for custodial and maintenance crews.

Personnel who perform Class IV asbestos work must complete at least 2 hours of asbestos awareness training that includes the following:

- Locations of identified or suspected asbestos-containing materials
- Recognition of damage, deterioration, and delamination of asbestos-containing building materials

Employee training records shall be maintained for one year beyond the last date of employment.

### 8.0 RECORDKEEPING

Worksite Objective and/or exposure measurement data, data to rebut PACM, and records of required notifications during the course of project completion will need to be maintained for 30 years past the date of work completion. Records required to be maintained will be made available to OSHA Inspector for examination and copying, upon written request, and will comply with record transfer requirements. Records impacting Tetra Tech employees for things like medical surveillance, exposure monitoring, or physician clearances, will be maintained according to the Tetra Tech DCN 01-04 Recordkeeping Requirements Program.

#### **Definitions**



### TETRA TECH, INC. ASBESTOS PROTECTION PROGRAM

Revision Date: 2/2023

Country: USA

Safe Work Practice

04-08

Page 10 of 12

Industry: X General X Construction Electrical X Mining Hazardous Materials

If a definition is not listed in this section, please contact your supervisor. If your supervisor is unaware of what the term means, please contact your Health and Safety Representative.

**Asbestos**: a naturally occurring fibrous mineral which is mined from the earth as one of the many varieties including chrysotile, amosite, crocidolite, tremolite asbestos, anthophyllite asbestos, actinolite asbestos, and any of these minerals that have been chemically treated and/or altered. This mineral features crystals that form long, thin, needle-like fibers.

Asbestos-containing material (ACM): means any material containing more than 1% asbestos. The Environmental Protection Agency (EPA) identifies three (3) basic categories: 1) Thermal systems insulations, 2) surface materials, and 3) Miscellaneous such as floor tile, transite, etc.

**Certified Industrial Hygienist (CIH):** means one certified in the practice of industrial hygiene by the American Board of Industrial Hygiene.

Class I Asbestos Work: includes the removal of asbestos-containing thermal system insulation (TSI) and sprayed-on or troweled-on surfacing materials. Thermal system insulation includes ACM applied to pipes, boilers, tanks, ducts, or other structural components to prevent heat loss or gain. Surfacing materials include decorative plaster on ceilings and walls; acoustical materials on decking, walls, and ceilings; and fireproofing on structural members.

Class II Asbestos Work: includes the removal of other types of ACM that are not thermal system insulation such as resilient flooring and roofing materials. Examples of Class II work include removal of asbestos-containing floor or ceiling tiles, siding, roofing, or transite panels.

**Class III Asbestos Work:** includes repair and maintenance operations, where ACM or PACM, including thermal system insulation and surfacing material, is likely to be disturbed.

Class IV Asbestos Work: Class IV of the OSHA asbestos standard is for cleanup activities. Workers remove dust and waste related to the Class I, II and III activities but do not actually remove asbestos. The work focuses on the identification of asbestos damage and ways to avoid exposure.

**Excursion limit for ACM:** The employer shall ensure that no employee is exposed to an airborne concentration of asbestos in excess of 1.0 fiber per cubic centimeter of air (1 f/cc) as averaged over a sampling period of thirty (30) minutes, as described in OSHA 29 CFR 1926.1101 Appendix A.

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### TETRA TECH, INC. ASBESTOS PROTECTION PROGRAM

Revision Date: 2/2023

Country: USA

Safe Work Practice

04-08

Page 11 of 12

Industry: X General X Construction \_\_\_\_ Electrical X Mining \_\_\_\_ Hazardous Materials

**Friable ACM:** means easily crumbled. Any material that is friable and containing more than 1% asbestos will be considered a Regulated Asbestos-Containing Material.

**High-efficiency particulate air (HEPA):** filter means a filter capable of trapping and retaining at least 99.97 percent of 0.3 micrometer diameter mono-disperse particles.

**Negative Exposure Assessment:** means a demonstration by the employer, which complies with the criteria outlined in OSHA 29 CFR 1926.1101 paragraph (f)(2)(iii), that employee exposure during an operation is expected to be consistently below the PELs.

**Non-Friable ACM:** means bonded asbestos, refers to when asbestos is firmly bound in with the material - such as cement.

**Presumed Asbestos-Containing Materials (PACM):** thermal system insulation and surfacing material found in buildings constructed no later than 1980. The designation of a material as "PACM" may be rebutted pursuant to paragraph (j)(8) of this section.

**Professional Engineer (PE):** an individual, who has fulfilled education and experience requirements and passed rigorous exams that, under State licensure laws, permits them to offer engineering services directly to the public.

**Regulated area**: means an area established by the employer to demarcate areas where airborne concentrations of asbestos exceed, or there is a reasonable possibility they may exceed, the permissible exposure limits.

**Time-weighted average limit (TWA) for ACM.** The employer shall ensure that no employee is exposed to an airborne concentration of asbestos in excess of 0.1 fiber per cubic centimeter of air as an eight (8) hour time-weighted average (TWA). as described in OSHA 29 CFR 1926.1101 Appendix A.

### **REFERENCES AND STANDARDS**

OSHA 29 CFR 1926.1101 - Construction Standard for Asbestos
OSHA 29 CFR 1926.1101 - Construction Standard for Asbestos - Appendix A
OSHA 29 CFR 1910.1001 - General Industry Standard for Asbestos
EPA 40 CFR 763, Subpart E, Appendix C - Asbestos Model Accreditation Plan
EPA 40 CFR 61.145 - Standard for Demolition and Renovation

### RELATED PROGRAMS, PROCEDURES AND FORMS

Tetra Tech DCN 01-04 Recordkeeping Requirements Program

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### TETRA TECH, INC. ASBESTOS PROTECTION PROGRAM

Revision Date: 2/2023

Country: USA

Safe Work Practice

04-08

Page 12 of 12

Industry: X General X Construction \_\_\_\_ Electrical X Mining \_\_\_\_ Hazardous Materials

Revision Date		Document Authorizer	Revision Details			
	Role	Name	Approval Date			
Oct 2008	Author/Sponsor	Chris McClain	Oct 2008	Updated from 1998 format		
	Author	Amber Bill	Jan 2023	Abatement Processes, including Hazard assessment process was		
Jan 2023	Sponsor	Chris McClain	Feb 2023	updated, included tetra tech roles and responsibilities, added training and recordkeeping requirements.		

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### TETRA TECH, INC. GENERAL SAFE WORK PRACTICES for HAZARDOUS WASTE SITE ACTIVITIES

Revision Date: 10/1/2008

Document Control Number:

SWP 5-2

Page 1 of 3

To prevent injuries and adverse health effects, the following general safe work practices (SWP) are to be followed when conducting work involving known and unknown site hazards on hazardous waste sites. These SWPs establish a pattern of general precautions and measures for reducing risks associated with hazardous site operations. This list is not inclusive and may be amended as necessary.

- Do not eat, drink, chew gum or tobacco, take medication, or smoke in contaminated or potentially contaminated areas or where the possibility for contact with site contamination exists.
- Wash hands and face thoroughly upon leaving a contaminated or suspected contaminated area. If a source of potable water is not available at the work site that can be used for hands-washing, the use of waterless hand cleaning products will be used, followed by actual hand-washing as soon as practicable upon exiting the site. A thorough shower and wash must be conducted as soon as possible if excessive skin contamination occurs.
- Avoid contact with potentially contaminated substances. Do not walk through puddles, pools, mud, or other such areas. Avoid, whenever possible, kneeling on the ground or leaning or sitting on drums, equipment, or the ground. Do not place monitoring equipment on potentially contaminated surfaces.
- Remove beards or facial hair that interferes with a satisfactory qualitative respirator fit test or routine pre-entry positive and negative pressure checks.
- Be familiar with and knowledgeable of and adhere to all instructions in the sitespecific health and safety plan (HASP). At a minimum, a safety meeting will be held at the start of each project to discuss the HASP. Additional meetings will be held, as necessary, to address new or continuing safety and health concerns.
- Be aware of the location of the nearest telephone and all emergency telephone numbers.
- Attend a briefing on the anticipated hazards, equipment requirements, SWPs, emergency procedures, and communication methods before going on site.
- Plan and delineate entrance, exit, and emergency escape routes.
- Rehearse unfamiliar operations prior to implementation.



### TETRA TECH, INC. GENERAL SAFE WORK PRACTICES for HAZARDOUS WASTE SITE ACTIVITIES

Revision Date: 10/1/2008

Document Control Number:

SWP 5-2

Page 2 of 3

- Use the "buddy system" whenever respiratory protection equipment is in use. Buddies should establish hand signals or other means of emergency communication in case radios break down or are unavailable.
- Buddies should maintain visual contact with each other and with other on-site team members by remaining in close proximity in order to assist each other in case of emergency.
- Minimize the number of personnel and equipment in contaminated areas (such as the exclusion zone). Nonessential vehicles and equipment should remain within the support zone.
- Establish appropriate support, contamination reduction, and exclusion zones.
- Establish appropriate decontamination procedures for leaving the site.
- Immediately report all injuries, illnesses, and unsafe conditions, practices, and equipment to the site safety coordinator (SSC).
- Maintain a portion of the site field logbook as a project safety log. The project safety log will be used to record the names, entry and exit dates, and times on site of all Tetra Tech personnel, subcontractor personnel, and project site visitors; air quality and personal exposure monitoring data; and other information related to safety matters. Form SSC-1, Daily Site Log, may be used to record names of on-site personnel.
- A portable eyewash station should be located in the support zone if chemical splashes to eyes are possible.
- Do not bring matches and lighters in the exclusion zone or contamination reduction zone. Flames and open fires are not permitted on site.
- Observe coworkers for signs of toxic exposure and heat or cold stress.
- Inform coworkers of nonvisual effects of illness if you experience them, such as headaches, dizziness, nausea, or blurred vision.

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### TETRA TECH, INC. GENERAL SAFE WORK PRACTICES for HAZARDOUS WASTE SITE ACTIVITIES

Revision Date: 10/1/2008

Document Control Number:

**SWP 5-2** 

Page 3 of 3

Revision Date	Docum	ent Authorizer	Revision Details			
	Name	Approval Date				
10/1/2008	Chris McClain		Update from 1998 format			
	Rick Lemmon					



### TETRA TECH, INC. SPILL AND DISCHARGE CONTROL PRACTICES

Revision Date: 10/1/2008

Document Control Number:

SWP 5-14

Page 1 of 3

This safe work practice (SWP) provides contingency measures for spills and unintentional discharges from handling and transporting hazardous materials. Spill and discharge control practices should follow specific procedures to ensure the safety of responders and bystanders and to limit environmental impacts.

### 1.0 GENERAL PROCEDURES

Immediate action should be taken to control and contain any spill following the general guidelines below:

- Unnecessary personnel should be kept away from the spill or discharge.
- The hazardous area should be isolated.
- If the spill or discharge creates a hazardous situation or results in injury or an environmental release, the emergency procedures of the HASP should be implemented. Emergency response telephone numbers, designated contacts, and special reporting procedures are presented in the HASP.
- Personnel should stay on the upwind side of the spill or discharge.
- Entry into a confined space or low area where liquids or vapors may accumulate should be avoided.
- Sources of ignition should be eliminated if the spill or discharge involves combustible materials.
- Drains, manholes, waterways, sewers, and the like should be identified and covered or protected.
- The spill should be controlled or absorbed using appropriate media or devices.
- When the spill or discharge is fully contained and under control, spill or discharge material should be collected.
- Following cleanup, the spill area should be evaluated by collecting soil samples and screening the area with air monitoring instruments.



### TETRA TECH, INC. SPILL AND DISCHARGE CONTROL PRACTICES

Revision Date: 10/1/2008

Document Control Number:

SWP 5-14

Page 2 of 3

### 2.0 SOLIDS

If the spill or discharge material is solid and nonreactive, the material should be scooped up and placed in a suitable and compatible container until the disposal method has been determined.

### 3.0 LIQUIDS

If liquid is discharged, the following general procedures apply:

- The point of discharge should be immediately identified and measures taken to eliminate further discharges by uprighting or patching containers, transferring contents, or other appropriate methods.
- Any discharged liquids or sludge should be removed or retrieved.
- Discharged materials should be cleaned up with absorbent materials or devices.
- Spent absorbent material should be placed into storage or disposal containers.

### 4.0 REPORTING

In some instances, a release may require reporting to government agencies. If a reportable quantity is released (this quantity is stated on the Material Safety Data Sheet) or human health or the environment is threatened, appropriate national, state, and local administering agency personnel should be notified. The timeframe for notification may vary from agency to agency. Notification may be required immediately or within 24 hours, depending on the type, location, and amount of released material. The appropriate agency to report spills to should be determined during HASP development.

All spills and chemical releases must be reported and investigated in accordance with DCN 2-2 Incident Reporting and Investigation Program. Details must be documented on the IR Form and IR – B.

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### TETRA TECH, INC. SPILL AND DISCHARGE CONTROL PRACTICES

Revision Date: 10/1/2008

Document Control Number:

SWP 5-14

Page 3 of 3

damages that result from unauthorized reuse of this SWP. Authorized users are responsible for obtaining proper training and qualification from their employer before performing operations described in this SWP.

Revision Date	Document Authorizer	Revision Details
10/1/2008	Chris McClain	Update from 1998 format



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 1 of 22

### 1.0 INTRODUCTION

This safe work practice (SWP) addresses situations during which heat illness is likely to occur and provides procedures for preventing and treating heat-related injuries and illnesses. This SWP is applicable to all Tetra Tech employees performing outdoor activities at both domestic and international project locations. This SWP incorporates safety regulations of the States of California and Washington to protect outdoor workers from heat-related illness. An "outdoor place" is an open area such as an agricultural field, forest, park, equipment and storage yard, outdoor utility installation, tarmac, and road. An outdoor workplace also can include a construction site at which no building shell has been completed, and areas of a construction site outside of any building shells that may be present.

Many factors contribute to heat illness and UV exposure, including personal protective equipment (PPE), ambient temperature and humidity, workload, sun exposure, and the physical condition of the employee, as well as predisposing medical conditions. However, the primary factors of heat illness are elevated ambient temperatures in combination with fluid loss. Because heat illness is one of the more common health concerns during field activities, employees must be familiar with the signs, symptoms, and various treatment methods of each form of heat illness. Health effects from heat illness may range from transient heat fatigue or rashes to serious illness or death. Tracking the weather is imperative during outdoor field projects because heat-related illness and fatalities occur primarily during heat waves.



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 2 of 22

### 2.0 Definitions

The following are typical terms and definitions associated with heat illness prevention and monitoring activities:

**Acclimatization** – Gradual adaptation of the body to work under temperature conditions to which it is exposed. Acclimatization peaks in most people within 4 to 14 days of regular work taking up at least 2 hours per day in the heat.

Ambient Temperature - Temperature of the surroundings.

**Electrolytic Sports Drink** – A beverage containing sodium and potassium salts that replenish the body's water and electrolyte levels after dehydration caused by physical activity.

**Environmental Risk Factors for Heat Illness** – Working conditions under which heat illness could occur. Environmental risk factors include air temperature, relative humidity, radiant heat from the sun and other sources, conductive heat sources such as the ground, air movement (or lack of), workload severity and duration, and protective clothing and PPE worn by employees.

**Heat Illness** – A serious medical condition resulting from the body's inability to cope with a particular heat load. Symptoms include heat cramps, heat exhaustion, and heat stroke (see Table 1).

**Heat Index** – An index that combines air temperature and relative humidity to indicate the human-perceived equivalent temperature (i.e., how hot it feels outdoors).

**Heavy Work** – Digging/hand-auguring, heavy lifting, cutting trees, using heavy hand tools, and similar tasks.

**Light Work** - Walking, writing notes, handling samples, and similar tasks.

**Medium Work** – Bailing wells, moving light equipment, driving nails, and similar tasks.



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 3 of 22

**Personal Risk Factors for Heat Illness** – Factors such as an individual's age, degree of acclimatization, health, water consumption, alcohol consumption, caffeine consumption, and use of prescription medications that affect the body's water retention or other physiological responses to heat.

**Preventive Cool Down and Recovery Period** – Period of time needed to recover from the heat in order to prevent heat illness.

**Relative Humidity** – The amount of water vapor that exists in a gaseous mixture of air and water vapor.

**Shade** – Blockage of direct sunlight. Canopies, umbrellas, and other temporary structures or devices may be used to provide shade. One indicator that blockage is sufficient is absence of a shadow of an object within the area of blocked sunlight. Shade is not adequate when heat in the area of shade defeats the purpose of shade, which is to allow the body to cool. For example, a car sitting in the sun does not provide acceptable shade to a person inside it unless the car is running with air conditioning.

**Wet Bulb Globe Temperature** (WBGT) - a measurement used to indicate heat stress. WBGT takes into account the effects of humidity



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 4 of 22

### 3.0 Employee Duties and Responsibilities

Written procedures help Project Managers (PM), Site Safety Coordinators (SSC), and field team members reduce the risk of heat-related illnesses, and ensure that emergency assistance is provided without delay to all Tetra Tech employees. The following are the duties and responsibilities of the Project Team for implementing and managing the Heat Illness Prevention and Monitoring SWP.

### 3.1 Project Management

The PM must understand and agree to the responsibility for implementing this SWP for worker safety. The PM will assure that all employees at the work site comply with this SWP.

- The PM must designate an appropriate field team member to serve as the SSC who will implement this SWP and who will perform and document necessary monitoring requirements for worker safety.
- The PM will ensure necessary resources required to implement this SWP and necessary monitoring resources for worker safety are acquired and present at the work site prior to initiation of project activities in hot environments.
- The PM will work with the Director of Health and Safety and identify at risk employees.
- The PM will ensure all field team members are trained in heat illness management and emergency response procedures prior to working outdoors.
- The PM and SSC will modify working hours to schedule work during the cooler hours of the day, when possible. When a modified or shorter work-shift is not possible, more water and rest breaks shall be provided.
- The PM and SSC will verify that the elements of this SWP are documented in the Health and Safety Plan, as necessary.



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 5 of 22

### 3.2 Site Safety Coordinator

- The SSC must understand and agree to the responsibility for implementing this SWP in the field and implement the necessary monitoring requirements for worker safety during outdoor activities.
- The SSC must have appropriate Occupational Safety and Health Administration (OSHA)-related training and experience to understand and implement this SWP, and to ensure required monitoring for worker safety during outdoor activities.
- The SSC must ensure that resources needed to implement this SWP and required monitoring for worker safety are acquired and present at the work site prior to initiation of project activities in hot environments.
- The SSC must maintain all necessary resources required under the SWP during project activities in hot environments.
- The SSC must ensure implementation and appropriate documentation of required monitoring for worker safety during site activities.
- The SSC must be familiar with and continuously monitor all employees and must remain alert for onset of heat-related symptoms.
- The SSC and co-workers are encouraged never to discount any signs or symptoms of heat-related illness shown by one or more project team members, and to immediately report these signs or symptoms.
- The SSC will carry a cell phone or other means of communication to ensure that
  emergency services can be contacted and will verify that these resources are
  functional at the worksite prior to each shift.

### 3.3 Field Team

• The field team will be able to recognize the hazards of working in warm environments.



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 6 of 22

- Co-workers will use a "buddy system" to monitor each other closely for discomfort or symptoms of heat illness.
- Every morning, workers must attend a daily tailgate safety meeting to be reminded of site-specific emergency procedures.
- A copy of site specific heat illness procedures shall be available for employee review.

### 4.0 Description and Requirements

### 4.1 Effects of Hot Weather

As the environment heats up, the body tends to warm up as well. The body's internal thermostat maintains a constant temperature by pumping more blood to the skin, which is cooled by evaporation from increasing perspiration production. In this way, the body increases the rate of heat loss to balance the heat burden created by a hot environment. Such situations generally do not cause harm, as long as the body is allowed to adjust to cope with the increasing heat.

In a very hot environment, however, the rate of heat gain exceeds the rate of heat loss. In this situation, the body's coping mechanisms can be overwhelmed, resulting in heat illness and leading to a range of serious and possibly fatal conditions.

### 4.2 Preparation for Hot Weather Work

The following list describes the process for preparing to work in hot weather conditions:

- Identify work that can pose a risk of heat stress and Ultraviolet (UV) exposure.
- · Identify at-risk employees.
- Identify possible controls:
  - Establish controls for hot weather situations
  - Determine mandatory work and rest regimens based on current conditions, workload, clothing requirements, temperature and humidity for Threshold Limit Value (TLV).
  - Identify required fluid and food replacement schedules.



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 7 of 22

- Provide a location to cool down during breaks.
- Establish requirements to address UV exposure.
- Monitor workers in extreme heat conditions.
- Establish emergency response procedures to be followed for heat-related emergency situations.
- Provide for first aid and establish the requirement that first aid be administered immediately to employees displaying symptoms of heat-related illness.
- Provide training to employees and verify training records about site legal and regulatory requirements and about the characteristics and effects of heat stress and the recognition and prevention of heat-related injuries (See Table 1).

### 5.0 Employee Training

Training is an important component of heat illness prevention. Employees are instructed to recognize and treat heat-related illnesses during 8-hour health and safety refresher and first aid training courses. The conditions, symptoms, and treatment for heat-related illnesses are listed below in Table 1.



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 8 of 22

### TABLE 1 HEAT ILLNESS CONDITIONS

Condition	Causes	Signs and Symptoms	Treatment
Heat cramps	Fluid loss and electrolyte imbalance from dehydration	<ul> <li>Painful muscle cramps, especially in legs and abdomen</li> <li>Faintness</li> <li>Profuse perspiration</li> </ul>	<ul> <li>Move affected worker to cool location</li> <li>Provide sips of liquid such as Gatorade®</li> <li>Stretch cramped muscles</li> <li>Transport affected worker to hospital if condition worsens</li> </ul>
Heat Exhaustion	Blood transport to skin to dissipate excessive body heat, resulting in blood pooling in the skin with inadequate return to the heart	<ul> <li>Weak pulse</li> <li>Rapid and shallow breathing</li> <li>General weakness</li> <li>Pale, clammy skin</li> <li>Profuse perspiration</li> <li>Dizziness</li> <li>Unconsciousness</li> </ul>	<ul> <li>Move affected worker to cool area</li> <li>Remove as much clothing as possible</li> <li>Provide sips of cool liquid or Gatorade® (only if conscious)</li> <li>Fan the person but do not overcool or chill</li> <li>Treat for shock</li> <li>Transport to hospital if condition worsens</li> </ul>
Heat Stroke**	Life threatening condition from profound disturbance of body's heat-regulating mechanism	<ul> <li>Dry, hot, and flushed skin</li> <li>Constricted pupils</li> <li>Early loss of consciousness</li> <li>Rapid pulse</li> <li>Deep breathing at first, and then shallow breathing</li> <li>Muscle twitching leading to convulsions</li> <li>Body temperature reaching 105 or 106 degrees Fahrenheit (°F) or higher</li> </ul>	<ul> <li>Immediately transport victim to medical facility</li> <li>Move victim to cool area</li> <li>Remove as much clothing as possible</li> <li>Reduce body heat promptly by dousing with water or wrapping in wet cloth</li> <li>Place ice packs under arms, around neck, at ankles, and wherever blood vessels are close to skin surface</li> <li>Protect patient during convulsions</li> </ul>

<sup>\*\*</sup> Any of these symptoms require immediate attention. If heat stroke is suspected, emergency medical personnel should be immediately contacted and on-site first aid provided.



Revision Date: 12/19/2018

Document Control Number:

**SWP 5-15** 

Page 9 of 22

Employee training procedures include, but are not limited to, the following:

- All employees (including and especially newly hired employees) will receive heat illness prevention training prior to working outdoors. This training will review the signs and symptoms of heat illness, detail the concept and importance of acclimatization and Tetra Tech's responsility to provide water, shade, cool-down rests and access to first aid. Training will also communicate the employees' right to exercise their rights without retaliation.
- SSCs will hold short tailgate meetings daily to review important heat illness and prevention information with all field team members. Information communicated in tailgate meetings will include a reminder of the importance of frequent consumption of small quantities of water, up to 4 cups per hour when the work environement is hot and employees are likely to be sweating more than usual.
- The expectation to immediately report any symptoms or signs of heat illness in themselves or in co-workers.
- All workers will be assigned a "buddy" or experienced coworker to ensure that they understood the training and follow the company procedures.
- Training will include a review of how emergency services will be provided if necessary, procedures for contacting emergency medical services and if necessary transporting employees to a point where they can be reached by emergency medical services.
- PMs and SSCs will be trained before assignment to supervise outdoor workers.
- 6.0 Heat Illness Prevention and Monitoring Requirements
- 6.1 Identification of Work Conditions



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 10 of 22

Hot weather is a condition that will be encountered during Tetra Tech operations. When work takes place outdoors during warm weather, working conditions shall be identified for both heat stress conditions and UV exposure.

### 6.2 Heat Index

The Heat Index (HI) can be used as a first indicator of thermal comfort. The HI can be obtained by directly measuring the dry bulb temperature and relative humidity. The dry bulb temperature and relative humidity forecast can be obtained by checking the local weather station information or measured by using a wet bulb thermometer. A direct reading of HI can be obtained by placing a heat stress monitor in full shade at the workplace.

The HI does not take into account acclimation, clothing or nature of work; therefore, if the HI is at 80°F (26.7°C) or above, further evaluation is required to adjust workload and clothing.

### 6.3 Heat Exposure Limits and Measurement

The TLV is a means of providing heat exposure limits and gauging potential heat impacts. To determine the TLV, the Wet Bulb Globe Temperature (WBGT) index is measured. The WBGT is calculated using a formula that takes into account air temperature, speed of air movement, radiant heat from hot objects, sunshine and body cooling due to sweat evaporation. WBGT direct reading meters, often called 'heat stress analyzers,' are also available. These meters give direct WBGT readings; no calculations are necessary.

A trained person shall take WBGT measurements. If a WBGT direct reading meter is not available, two different methods are used to calculate WBGT in the workplace: one for workplaces with direct sunlight, and the other for workplaces without direct sunlight. In addition, when conditions of the workplace fluctuate widely, time-weighted WBGT is often used. The WBGT calculation is used in determining heat stress exposure guidelines and heat stress and clothing guidelines. Table 2 presents approximate WBGT values.



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 11 of 22

Dry E	100000000000000000000000000000000000000																				
Temper																					
°C	°F	5%	10%	15%	20%	25%	30%	35%	40%	45%	50%	55%	60%	65%	70%	75%	80%	85%	90%	95%	100%
18.33	65	59	59	60	61	62	62	63	64	64	65	66	67	67	68	69	70	70	71	72	73
18.89	66	59	60	61	61	62	63	64	65	65	66	67	68	68	69	70	71	71	72	73	74
19.44	67	60	61	61	62	63	64	65	65	66	67	68	69	69	70	71	72	72	73	74	75
20.00	68	60	61	62	63	64	64	65	66	67	68	69	69	70	71	72	73	74	74	75	76
20.56	69	61	62	63	63	64	65	66	67	68	69	69	70	71	72	73	74	75	75	76	77
21.11	70	62	62	63	64	65	66	67	68	69	69	70	71	72	73	74	75	76	77	77	78
21.67	71	62	63	64	65	66	67	68	69	69	70	71	72	73	74	75	76	77	78	79	79
22.22	72	63	64	65	66	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81
22.78	73	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82
23.33	74	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83
23.89	75	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84
24.44	76	65	66	67	68	69	71	72	73	74	75	76	77	78	79	80	81	82	83	85	86
25.00	77	66	67	68	69	70	71	72	74	75	76	77	78	79	80	81	82	84	85	86	87
25.56	78	66	67	69	70	71	72	73	74	76	77	78	79	80	81	82	84	85	86	87	88
26.11	79	67	68	69	71	72	73	74	75	76	78	79	80	81	82	84	85	86	87	88	90
26.67	80	68	69	70	71	72	74	75	76	77	79	80	81	82	84	85	86	87	88	90	91
27.22	81	68	69	71	72	73	75	76	77	78	80	81	82	83	85	86	87	89	90	91	92
27.78	82	69	70	71	73	74	75	77	78	79	81	82	83	85	86	87	88	90	91	92	94
28.33	83	69	71	72	73	75	76	78	79	80	82	83	84	86	87	88	90	91	92	94	95
28.89	84	70	71	73	74	76	77	78	80	81	83	84	85	87	88	90	91	92	94	95	97
29.44	85	71	72	73	75	76	78	79	81	82	84	85	87	88	89	91	92	94	95	97	98
30.00	86	71	73	74	76	77	79	80	82	83	85	86	88	89	91	92	94	95	97	98	100
30.56	87	72	73	75	76	78	80	81	83	84	86	87	89	90	92	93	95	97	98	100	101
31.11	88	72	74	76	77	79	80	82	84	85	87	88	90	92	93	95	96	98	100	101	103
31.67	89	73	75	76	78	80	81	83	85	86	88	90	91	93	94	96	98	99	101	103	104
32.22	90	74	75	77	79	80	82	84	86	87	89	91	92	94	96	97	99	101	103	104	106
32.78	91	74	76	78	80	81	83	85	87	88	90	92	94	95	97	99	101	102	104	106	108
33.33	92	75	77	79	80	82	84	86	88	89	91	93	95	97	98	100	102	104	106	107	109
33.89	93	76	77	79	81	83	85	87	89	90	92	94	96	98	100	102	103	105	107	109	111
34.44	94	76	78	80	82	84	86	88	90	92	93	95	97	99	101	103	105	107	109	111	113
35.00	95	77	79	81	83	85	87	89	91	93	95	97	99	101	103	105	107	108	110	112	114
35.56	96	77	79	82	84	86	88	90	92	94	96	98	100	102	104	106	108	110	112	114	116
36.11	97	78	80	82	84	86	89	91	93	95	97	99	101	103	105	108	110	112	114	116	118
36.67	98	79	81	83	85	87	90	92	94	96	98	100	103	105	107	109	111	113	116	118	120
37.22	99	79	82	84	86	88	91	93	95	97	99	102	104	106	108	111	113	115	117	120	122
37.78	100	80	82	85	87	89	91	94	96	98	101	103	105	108	110	112	115	117	119	121	124
38.33	101	81	83	85	88	90	92	95	97	100	102	104	107	109	111	114	116	119	121	123	126
38.89	102	81	84	86	89	91	93	96	98	101	103	106	108	111	113	116	118	120	123	125	128
39.44	103	82	84	87	89	92	94	97	100	102	105	107	110	112	115	117	120	122	125	127	130
40.00	104	83	85	88	90	93	96	98	101	103	106	108	111	114	116	119	121	124	127	129	132
40.56	105	83	86	89	91	94	97	99	102	105	107	110	113	115	118	121	123	126	129	131	134
41.11	106	84	87	89	92	95	98	100	103	106	109	111	114	117	120	122	125	128	131	133	136
41.67	107	84	87	90	93	96	99	101	104	107	110	113	116	119	121	124	127	130	133	136	138
42.22	108	85	88	91	94	97	100	103	106	108	111	114	117	120	123	126	129	132	135	138	141
42.78	109	86	89	92	95	98	101	104	107	110	113	116	119	122	125	128	131	134	137	140	143
43.33	110	86	90	93	96	99	102	105	108	111	114	117	120	124	127	130	133	136	139	142	145
43.89	111	87	90	93	97	100	103	106	109	113	116	119	122	125	128	132	135	138	141	144	148
44.44	112	88	91	94	98	101	104	107	111	114	117	121	124	127	130	134	137	140	143	147	150
45.00	113	88	92	95	99	102	105	109	112	115	119	122	126	129	132	136	139	142	146	149	153
45.56	114	89	93	96	99	103	106	110	113	117	120	124	127	131	134	138	141	145	148	152	155
46.11	115	90	93	97	100	104	108	111	115	118	122	125	129	133	136	140	143	147	150	154	158
46.67	116	90	94	98	101	105	109	112	116	120	123	127	131	134	138	142	146	149	153	157	160
47.22	117	91	95	99	102	106	110	114	118	121	125	129	133	136	140	144	148	152	155	159	163
47.78	118	92	96	100	103	107	111	115	119	123	127	131	134	138	142	146	150	154	158	162	166
48.33	119	92	96	100	104	108	112	116	120	124	128	132	136		144	148	152	156	160	164	168
48.89	120	93	97	101	105	110	114	118	122	126	130	134	138	142	147	151	155	159	163	167	171
N	lotes:	Calcu	lated	values	assu	me ou	tdoor	work i	n full s	sun, w	ith a li	ght (<	5 mph	) wind							

Notes: Calculated values assume outdoor work in full sun, with a light (<5 mph) win WBGT of green-shaded cells is less than dry-bulb temperature.



Revision Date: 12/19/2018

Document Control Number:

SWP 5-15

Page 12 of 22

### 6.4 Heat Stress Exposure Guidelines

Heat stress exposure guidelines recommended by the American Conference of Governmental Industrial Hygienists (ACGIH) are shown in Table 3: ACGIH Screening Criteria for Heat Stress Exposure. This table is used to determine the allocation of work in a work/rest cycle, which is dependent on the type of work and WBGT values.

Table 3: ACGIH Screening Criteria for Heat Stress Exposure

	PERMISSIBLE HEAT EXPOSURE THRESHOLD LIMIT VALUE														
Clothing Type	Summer Lightweight			Cotton Coveralls				Winter Work Permeable Water (Tyvek)				arrier	Fully-Er	Fully-Encapsulating Suit (Level 4	
Work Load	Light	Moderate	Heavy	Light	Moderate	Heavy	Light	Moderate	Heavy	Light	Moderate	Heavy	Light	Moderate	Heavy
Work/Rest Schedule / WBGT	(°F)	(°F)	(°F)	(°F)	(°F)	(°F)	(°F)	(°F)	(°F)	(°F)	(°F)	(°F)	(°F)	(°F)	(°F)
Continuous Work	86	80	77	82	76	73	79	73	70	75	69	66	68	62	59
75% Work, 25% Rest / Hr	87	82	79	83	79	75	80	75	71	76	72	68	69	64	61
50% Work, 50% Rest / Hr	89	85	82	85	81	79	81	78	75	78	74	71	71	67	64
25% Work, 75% Rest / Hr	90	88	86	86	84	82	83	81	79	79	77	75	72	70	68

**Notes:** Temperature is approximate WBGT from accompanying tables, based on outdoor work, temperature, and relative humidity measurement during work activities. Light Work includes walking, writing notes, handling samples, and similar activities (metabolic rate up to 200 kilocalories [kcal]/hour). Medium Work includes bailing wells, moving light equipment, driving nails, and similar tasks (metabolic rate of 200-350 kcal/hour). Heavy Work is digging, heavy lifting, cutting trees, using heavy hand tools, and similar tasks (metabolic rate above 350 kcal/hour).



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 13 of 22

Table 3 is based on five-day work weeks and eight-hour work days with conventional breaks. Conventional breaks include a 15-minute break in a four-hour period and a half-hour lunch in an eight-hour period. The ACGIH exposure limits are intended to protect most workers from heat-related illnesses. The limits are higher than that if they had been developed to prevent discomfort. A safety factor should be used to protect sensitive individuals or increase comfort. Examples to clarify work load intensity:

- Rest: sitting (quietly or with moderate arm movements).
- Light work: sitting or standing to control machines, performing light hand or arm work (e.g., using a table saw), occasional walking, driving.
- Moderate work: walking about with moderate lifting and pushing or pulling, walking at a moderate pace, scrubbing in a standing position.
- Heavy work: digging, carrying, pushing/pulling heavy loads, walking at a fast pace, pick and shovel work, carpenter sawing by hand.
- Very heavy: very intense activity at a fast to maximum pace (e.g., shoveling wet sand).

For example, in order to minimize heat stress exposure, an employee who is acclimated and is performing heavy work such as shoveling dirt in a temperature of 78°F (25.6°C), would fall into a work/rest regimen of 100% work.

TLVs assume that workers who are exposed to these conditions are adequately hydrated, are not taking medication, are wearing lightweight clothing and are in generally good health.

When the WBGT is at a temperature that exceeds the TLV, 'Stop Work' should be enforced.

### 6.5 Heat Stress and Clothing Guidelines

The exposure limit should be adjusted for workers wearing heavy clothing. ACGIH recommendations for these conditions are listed in Table 4: Correction of TLV for Clothing.



Revision Date: 12/19/2018

Document Control Number:

SWP 5-15

Page 14 of 22

Table 4: Correction of TLV for Clothing

Clothing Type	WBGT Correction (in °F [°C])
Work Clothes (long-sleeved shirts and pants)	0 (0)
Cloth coveralls (woven material)	+3 (0)
Spunbonded Meltdown Spunbonded polypropylene coveralls	+6 (+0.5)
Polyolefin coveralls	+8 (+1)
Double-layer woven clothing	+9 (+3)
Limited-use vapor-barrier coveralls	+18 (+11)

For example, an acclimated worker wearing double-layer woven clothing doing moderate work in  $30^{\circ}$ C would have a corrected exposure level of  $30 + 3 = 33^{\circ}$ C ( $91.4^{\circ}$ F). This would lower the allowable exposure to 0-25% work from 25-50% work.

For Fire Retardant Clothing (FRC), there is no WBGT correction. FRC can be obtained in various weight materials. The lightest weight FRC should be worn during work in warm environments. No second layer of clothing should be worn except for cotton undergarments.

These values are not to be used for completely encapsulating suits. The assumption is that coveralls are worn with only modest clothing underneath, not a second layer of clothing.

### 6.6 Identifying At-risk Employees

A screening program for identifying at risk employees shall include identification of health conditions that are aggravated by extreme environmental temperatures. How a person functions under conditions of heat stress will be unique that person and will depend on:

- Age.
- Weight.
- Metabolism.
- Alcohol or drug use.
- Pre-existing medical conditions.



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 15 of 22

- Level of physical fitness.
- Use of medications.
- Individual sensitivity to heat.
- Possibility of hypertension.

Note: Employees with any 'at-risk' conditions shall have more stringent work/rest regimens or controls

### 6.7 Health and Safety Controls

Controls shall be based on a risk assessment approach. Conditions and available controls will vary from site to site. Therefore, the HASP shall define and document the site-specific control plan. Controls shall be appropriate for the risks that are associated with heat hazards.

### 6.7.1 Acclimation

The human body can adapt to heat exposure to some extent. This physiological adaptation is called acclimation. Acclimation is a response by the body that results in increased heat tolerance.

People differ in their ability to acclimate to heat. Usually, acclimation is obtained in four to five days. However, it is lost in approximately the same amount of time. After a period of acclimation, the same activity will produce fewer cardiovascular demands. The worker will perspire more efficiently, leading to better evaporative cooling, and thus will more easily be able to maintain normal body temperatures.

All site workers who could be exposed to hot weather conditions shall be acclimated or go through an acclimation process, as necessary. Where workers are already acclimated, no acclimation process is necessary. A previously acclimated person is someone who has already been in similar working and heat conditions. Employees newly assigned to a high heat area will be closely observed by the SSC or designees for the first 14 days of the employee's assignment.

All employees shall be closely observed by a supervisor or designee during a heat wave. For acclimization purposes only, a heat wave is defined as any day in which the predicted high temperature for the day will be at least 80 degrees Fahrenheit and at least ten degrees Fahrenheit higher than the average high daily temperature in the preceding five days.

### 6.7.2 Fluid and Nutrient Replacement



Revision Date: 12/19/2018

**Document Control Number:** 

SWP 5-15

Page 16 of 22

Cool (50°-60°F [10°-15°C]) water or other cool liquid, except alcoholic beverages, should be made available to workers.

### Provision of Water (Not Temperature Dependent)

Water is the principal preventive measure to minimize the risk of heat-related illnesses. Tetra Tech employees shall have access to potable drinking water (or electrolytic sports drink). Where the supply of water is not plumbed or otherwise continuously supplied, water shall be provided in sufficient quantity at the beginning of the work shift to provide **1 quart per employee per hour for drinking for the entire shift**. Frequent drinking of water shall be encouraged by the SSC. Water provision requirements include the following:

- At least 2 quarts of water per employee will be available at the start of the shift.
- The SSC will monitor water containers every 30 minutes, and employees are encouraged to report low levels or dirty water to the SSC when observed.
- The SSC will provide reminders to the field team members to drink frequently, and more water breaks will be provided as needed.
- During the daily tailgate safety meeting each morning, the SSC will remind the field team about the importance of frequent water consumption throughout the shift.
- Water containers will be placed as close to the workers as safety conditions allow.
- When drinking water levels within a container drop below 50%, the water shall be replenished immediately.
- If a common water source is used, disposable/single-use drinking cups will be provided to employees each day.
- Communication devices such as radios, cell phones, or air horns may be used to remind field team members to take water breaks.



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 17 of 22

Although some commercial replacement drinks contain salt, this is not necessary for acclimated people, because most people have enough salt in their normal diets. Commercial replacement drinks contain high amounts of sugar and may contribute to an individual's inability to cope with the warm environment. If used, commercial replacement drinks should not be used at full strength and should be diluted with water on at least a one-to-one ratio.

Energy drinks shall not be used while working in warm environments.

Poor nutrition, over eating and under eating are factors contributing to heat stress. During hot conditions, employees should eat small, regular meals.

### 6.7.3 Additional Control Measures

Outdoor workers are exposed to not only potential heat illness, but also UV radiation. Long-term exposure to UV radiation poses additional risks and can lead to a variety of skin disorders, including skin cancer and cataracts of the eyes.

Protection from UV exposure, sunscreen and appropriate eye protection should be considered in addition to the additional controls listed below:

### Access to Shade

Access to rest and shade or other cooling measures are important preventative steps to minimize the risk of heat-related illnesses and exposure to UV radiation. Tetra Tech employees working in temperatures exceeding 80 degrees Fahrenheit for any period shall be provided access to an area with shade that is either open to the air or provided with ventilation or cooling. Such access to shade shall be permitted at all times. The amount of shade present shall be at least enough to accommodate the number of employees on recovery or rest periods, so that they can sit in a normal posture fully in the shade without having to be in physical contact with each other. When the outdoor temperature in the work area does not exceed 80 degrees Fahrenheit, shade shall be made available as addressed in this section or employees may be provided timely access to shade upon request.

Procedures for the provision of shade include the following:



Revision Date: 12/19/2018

**Document Control Number:** 

SWP 5-15

Page 18 of 22

- SSC will set up an adequate number of shaded areas as needed. Examples of shaded areas include vehicles with air conditioning, umbrellas, canopies, or other portable devices. Shading should be placed in close proximity to the work activity (no more than 50-100 yards away, or at the closest location safety conditions allow). Employees will be allowed and encouraged to take preventative cool down rest in the shade when they feel the need to do so to protect themselves from overheating. Employees should have access to an office, construction trailer, or other places with air conditioning.
- Any individual who takes a preventative cool down rest shall be monitored and asked if they are experiencing symptoms of heat illness.
- If an employee exhibits signs or reports symptoms of heat illness while taking a
  preventative cool down rest or during a preventative cool down rest period,
  appropriate first aid or emergency response measures must be provided.
- Any employee experiencing signs and symptoms of heat illness shall not be ordered back to work until signs and symptoms of heat illness have abated but in no event less than 5 minutes in addition to the time needed to access the shade.
- Every morning a short tailgate meeting will occur to remind workers about the importance of rest breaks and the location of shade.
- As safety conditions allow, SSCs shall provide areas for employee breaks that are:
  - Readily accessible
  - In the shade, open to air, and ventilated
  - Near sufficient supplies of drinking water, shade provided during meal periods shall be enough to accommodate the number of employees who remain outside.

### 7.0 Heat Illness Monitoring



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 19 of 22

A medical monitoring program shall be planned with the assistance of a medical or industrial hygiene professional. The monitoring program shall be specify the leading indicators to be used (e.g. heat rate, body temperature, blood pressure, respiration rate, and other) and frequency of measurement.

Heat illness monitoring will be conducted by the SSC or his/her designee when work conditions warrant implementation of a work/rest schedule based on temperature conditions and PPE requirements associated with project activities. Monitoring will be conducted as follows:

- Heart Rate: Count the radial (wrist) pulse during a 30-second period as early as
  possible in the rest period; if heart rate exceeds 110 beats per minute at the
  beginning of the rest period, shorten the next work cycle by one-third without
  changing the rest period.
  - If the heart rate still exceeds 110 beats per minute at the next period, shorten the following work cycle by one-third.
- Body Temperature: If body temperature exceeds 99.6 degrees Fahrenheit (°F) (37.6 degrees Celsius [°C]), shorten the next work cycle by one-third without changing the rest period. If body temperature still exceeds 99.6 °F at the beginning of the next rest period, shorten the following work cycle by one-third. Do not permit a worker to wear impermeable PPE when his or her body temperature exceeds 100.6 °F (38.1 °C). Use any of the following thermometers:
  - Oral Thermometer Use a clinical thermometer (3 minutes under the tongue) to measure the oral temperature at the end of the work period.
  - Tympanic (ear) Thermometer
  - Temporal (swipe) Thermometer

The SSC will document throughout the entire work shift results of heat illness monitoring for each team member participating in work activities. Any employee exhibiting signs and symptoms of heat illness shall not be left alone or sent home without being offered onsite first aid and/or being provided with necessary emergency medical services in accordance with Site HASP emergency response procedures.



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 20 of 22

### 8.0 HIGH HEAT PROCEDURES

### Extra Measures During Heat Waves

Extreme environmental conditions during a heat wave can cause an employee's physical and mental conditions to change rapidly into a serious medical condition. Workers previously fully acclimatized are at risk for heat illness during a heat wave because during a heat wave, the body does not have enough time to adjust to a sudden, abnormally high temperature or other extreme conditions. The onset of heat illness may be confused with other problems and may not always be obvious before it becomes life-threatening. Therefore, the following extra measures may be required to prevent and/or respond to heat illness during heat waves or when temperatures exceed 95 degrees Fahrenheit. These measures will be discussed at the preshift tail gate meeting before commencement of work.

- Communication Make sure voice, observation or electronic means of communication (text messaging or cell phone if service is available) is maintained so that site personnel can contact a supervisor when necessary. Designate one or more employees at the site as authorized to call for emergency services, when designated person(s) are not available any employee can call for emergency services.
- Alertness to the Weather Make sure to monitor the weather and the specific
  locations where work activities are occurring. Continue to stay updated throughout
  the work shift on the changing air temperatures and other environmental factors.
  Use current weather information to make the appropriate adjustments in work
  activities throughout the workday.
- Extra Vigilance and Observation Apply real-time communication methods as stated above as well as a mandatory "Buddy System" to account for the whereabouts of employees at more frequent intervals throughout the work shift and at the end of the work shift. Employee observation methods may also include, supervisor or designee direct observation if less than 20 employees are at the site.
- Additional Water Consumption Remind employees throughout the work shift to drink small quantities of water more frequently and have effective replenishment measures in place for provision of extra drinking water to ensure available supplies.



Revision Date: 12/19/2018

**Document Control Number:** 

**SWP 5-15** 

Page 21 of 22

- Additional Cooling Measures Other alternative cooling measures may be necessary in addition to shade (e.g., allowing employees to spend time in airconditioned places or having them spray themselves with water).
- Additional and/or Longer Rest Breaks Remind employees of their right to take a
  more frequent and cool down rests when necessary.
- Change of Work Scheduling and Assignments One or more of the following additional measures may be necessary:
  - Start the work shift earlier in the day or later in the evening.
  - Cut work shifts short or stop work altogether.
  - Bring in more personnel to accommodate longer, more frequent breaks as necessary to meet production requirements.
  - Reduce the severity of work by scheduling slower paced, less physically demanding work during the hot parts of the day, and the heaviest work activities during the cooler parts of the day (early morning or evening).

### 9.0 Establish Emergency Response

Specific procedures to be followed for heat related first aid and emergency response shall be established relevant to project location and task and documented in the Site-specific HASP. The HASP emergency response procedures must include clear and concise directions to the work site that can be provided to emergency responders. The HASP will also identify local emergency services and if necessary provide a means to transport employees to a place where they can be reached by emergency responders.

### 10.0 Variation to the Heat Illness Prevention and Monitoring Program



Revision Date: 12/19/2018

Document Control Number:

SWP 5-15

Page 22 of 22

Before deviation from the requirements of this document, a designated manager shall authorize the variation. The exception process does not need to be followed for variations that impose more stringent requirements that those outlined in this document.

### 11.0 Disclaimer

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Revision	Docum	ent Authorizer	Revision Details		
Date	Name	Approval Date			
			Revision to align with Cal-OSHA		
12/19/2018	Chris McClain	12/21/2018	Heat Illness Prevention		



### TETRA TECH, INC. PREVENTION of SUN EXPOSURE

Revision Date: 10/1/2008

Document Control Number:

SWP 5-26

Page 1 of 1

By far, the most common cause of skin cancer is overexposure to the sun. Ninety percent of all skin cancers occur on parts of the body that not usually covered by clothing. People who sunburn easily, and those with fair skin and red or blond hair are more prone to develop skin cancer. The amount of time spent in the sun also affects a person's risk of skin cancer. Premature aging of the skin also occurs with prolonged sun exposure. Tetra Tech encourages personnel to avoid prolonged exposure to the sun, and recommends the following:

- Sunburn can occur during any time of the year. To avoid sunburn, wear hats with wide brims.
- Use sunscreen with a Sun Protective Factor (SPF) rating of 15 or higher.
- To prevent skin cancer:
  - o Cover up with a wide brimmed hat and a bandanna for your neck. Wear long-sleeved shirts and pants which the sun cannot penetrate.
  - Use sunscreens to help prevent skin cancer as well as premature aging of your skin. Use a Sun Protective Factor (SPF) rating of 15 or higher.
  - Apply sunscreen at least an hour before going into the sun and again after swimming or perspiring a lot.
  - o Do not use indoor sun lamps, tanning salons/parlors, or tanning pills.
- You can still get burned on a cloudy day. Try to stay out of the direct sun at midday, because sun rays are their strongest between 10 a.m. and 3 p.m. Beware of high altitudes - where there is less atmosphere to filter out the ultraviolet rays. Skiers should remember that snow reflects the sun's rays, too.
- Know your skin. Whatever your skin type, do a monthly self-examination of your skin to
  note any moles, blemishes or birthmarks. Check them once a month and if you notice
  any changes in size, shape or color, or if a sore does not heal, see your physician without
  delay.

**Disclaimer**: This safe work practice (SWP) is the property of Tetra Tech, Inc. (Tetra Tech). Any reuse of the SWP without Tetra Tech's permission is at the sole risk of the user. The user will hold harmless Tetra Tech for any damages that result from unauthorized reuse of this SWP. Authorized users are responsible for obtaining proper training and qualification from their employer before performing operations described in this SWP.

Revision Date	Document Authorizer	Revision Details
10/1/2008	Chris McClain	NEW



May 23, 2024

Ms. Marianne Fuji Rossio Hawaii Department of Health (HDOH) Clean Air Branch Kinau Hale 1250 Punchbowl Street Honolulu, Hawaii 96813

Subject: Community Air Monitoring and Sampling Plan Rev 2 – Specialty

Laboratory and Field Support for HDOH CAB – Ambient Community Air Monitoring in Response to Wildfire Cleanup Actions – Lahaina, Maui

County, Hawaii

Task Order 23206 / Project Number 103S9230

Dear Ms. Fuji Rossio:

Tetra Tech, Inc. (Tetra Tech) is submitting the enclosed Community Air Monitoring and Sampling Plan (CAMSP) for ambient community air monitoring during cleanup actions (debris removal and asbestos abatement operations) in Lahaina, Maui County, Hawaii. This plan summarizes ambient community air monitoring and sampling to be conducted during debris removal and asbestos abatements activities. If you have any questions regarding this plan, please call me at (570) 417-1280.

Sincerely,

Chris Burns Project Manager

Enclosure

### COMMUNITY AIR MONITORING AND SAMPLING PLAN SPECIALTY LABORATORY AND FIELD SUPPORT FOR HDOH – AMBIENT COMMUNITY AIR MONITORING IN RESPONSE TO WILDFIRE CLEANUP ACTIONS – LAHAINA, MAUI COUNTY, HAWAII

### **REVISION 2**

Prepared for

### Hawaii Department of Health Clean Air Branch

Kinau Hale 1250 Punchbowl Street Honolulu, Hawaii 96813

Submitted by

Tetra Tech, Inc.

737 Bishop St., Suite 2000 Honolulu, HI 96813-3201

Contract No. TO 23206 / Project Number 103S9230

May 23, 2024 Prepared By

Chris Burns

Project Manager

### **CONTENTS**

Sect	<u>ion</u>		<u>Page</u>
1.0	INTF	RODUCTION	1
2.0		BACKGROUND	
3.0		JECT DESCRIPTION	
4.0		JECT OBJECTIVES AND DATA USE	
5.0	FIEL	D INVESTIGATION ACTIVITIES	
	5.1	METEOROLOGICAL MONITORING	
	5.2	BACKGROUND AIR MONITORING AND SAMPLING	
	5.3	COMMUNITY AIR LOCATIONS AND IDENTIFIERS	4
	5.4	SITE-SCREENING ACTION LEVELS	5
	5.5	PARTICULATE MONITORING	
	5.6	AIR SAMPLING	6
	5.7	DOCUMENTATION AND REPORTING	9
6.0	QUA	LITY ASSURANCE PROJECT PLAN	11
	6.1	QUALITY ASSURANCE/QUALITY CONTROL DATA OBJECTIVES	11
	6.2	CHAIN OF CUSTODY MAINTENANCE	13
	6.3	CALIBRATION PROCEDURES AND FREQUENCY	14
	6.4	ANALYTICAL METHODS	14
	6.5	DATA REDUCTION, VERIFICATION, AND REPORTING	14
	6.6	FIELD QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) CHECKS	15
	6.7	LABORATORY QA/QC CHECKS	16
	6.8	PERFORMANCE AND SYSTEM AUDITS	16
	6.9	PREVENTIVE MAINTENANCE	16
	6.10	DATA QUALITY ASSESSMENT	17
	6.11	CORRECTIVE ACTION	
7.0	RFFI	FRENCES	20

### **FIGURE**

1 AIR SAMPLING LOCATIONS

### **TABLES**

- 1 COMMUNITY AIR SCREENING LEVELS AND LABORATORY REPORTING LIMITS
- 2 PROJECT-SPECIFIC PARTICULATE MONITORING
- 3 SAMPLING REQUIREMENTS WORKSHEET
- 4 FIELD EQUIPMENT CALIBRATION REQUIREMENTS
- 5 LABORATORY MEASUREMENT PERFORMANCE CRITERIA
- 6 ANALYTICAL INSTRUMENT CALIBRATION
- 7 ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION

### **APPENDICES**

- A TETRA TECH STANDARD OPERATING PROCEDURES
- B METHODOLOGY TO ESTABLISH SITE-SPECIFIC ACTION LEVELS

### **ATTACHMENTS**

- 1 EPA ERT STANDING OPERATING PROCEDURES
- 2 LABORATORY STANDARD OPERATING PROCEDURES, QUALITY ASSURANCE MANUALS, AND CERTIFICATIONS

#### 1.0 INTRODUCTION

The Hawaii Department of Health (HDOH) Clean Air Branch (CAB) contracted Tetra Tech, Inc. (Tetra Tech) to perform ambient community air monitoring and sampling during cleanup actions (debris removal and asbestos abatement operations) in Lahaina, Hawai'i. This work supports the "Specialty Laboratory and Field Support for HDOH – Ambient Community Air Monitoring in Response to Wildfire Cleanup Actions" project.

The primary potential hazards that may be associated with debris removal and asbestos abatement operations related to the 2023 Fires include asbestos-containing materials (ACM), heavy metals, and fugitive dusts. This Community Air Monitoring and Sampling Plan (CAMSP) describes the design, setup, and operation of air monitoring systems and sampling procedures that Tetra Tech will implement during cleanup activities conducted by the U.S. Army Corps of Engineers (Army Corps). Cleanup activities include removing burned debris (including stucco, roofing, floor tile, linoleum, fireplaces, furnaces, vinyl tiles and mastic, sheetrock and joint compound, cement pipe, exterior home siding, thermal system insulation, concrete, white goods, vehicles, vegetation, construction debris, metal debris, electronic waste, and household hazardous chemicals) and abating asbestos.

Tetra Tech will perform air monitoring to document the levels of particulates (dust) in the ambient air at areas in Lahaina where sensitive or active populations are found in the surrounding communities. Community locations have been proposed and confirmed by the HDOH CAB and Maui County. Tetra Tech will also collect air samples from these locations; these will be analyzed for select metals and asbestos. In addition, meteorological data will be monitored and logged at each community location. Tetra Tech will perform ambient air sampling on a daily basis during debris removal and asbestos abatements in Lahaina. If Tetra Tech begins work before debris removal and/or asbestos abatement activities have commenced, background air monitoring and sampling collection for these parameters will be attempted to establish baseline levels for air contaminants.

This CAMSP presents site background information in Section 2.0, contains a project description in Section 3.0, describes project objectives and data use in Section 4.0, presents the proposed field investigation and sampling activities in Section 5.0, and describes the quality assurance project plan (QAPP) in Section 6.0. References cited in this CAMSP are listed in Section 7.0. Tetra Tech's Standard Operating Procedures (SOPs) to be used during the assessment are included as Appendix A. Appendix B provides the methodology used to develop this project's Site Screening Action Levels (SSALs). Where Tetra Tech SOPs are insufficient, U.S. Environmental Protection Agency Emergency Response Team

(EPA ERT) SOPs will be followed and are included in Attachment 1. Laboratory SOPs, quality assurance manuals, and certifications are included in Attachment 2.

# 2.0 SITE BACKGROUND

High winds from Hurricane Dora south of Hawai'i and dry weather caused wildfires to develop in Lahaina, Upper Kula, Pūlehu/Kihei, and Ka'anapali on the island of Maui on August 8, 2023. The wildfires affected approximately 1,550 parcels and 2,200 structures.

The HDOH, Clean Air Branch (CAB) tasked Tetra Tech to create and implement a CAMSP for the Lahaina fire area. Tetra Tech developed this plan in consultation with HDOH CAB and Maui County.

Ash and debris from residential structures burned by fires can contain heavy metals, as discussed in the "Guidance for Conducting Emergency Debris, Waste, and Hazardous Material Removal Actions Pursuant to a State or Local Emergency Proclamation" (California Environmental Protection Agency [CEPA] 2011), "Assessment of Burn Debris – 2007 Wildfires San Bernardino and San Diego Counties, California" (Department of Toxic Substances and Disease Control [DTSC] 2007), and "Assessment of Burned Debris- 2015 Wildfires Lake and Calaveras County, California" (DTSC 2015). The ash and debris may contain concentrations of metals from batteries, treated wood, and melted plumbing. Concentrations of lead specifically may be present for homes built prior 1978 (before lead was banned from household paints in the United States).

Residual materials, such as stucco, roofing, floor tile, linoleum, fireplaces, furnaces, vinyl tiles and mastic, sheetrock and joint compound, cement pipe, exterior home siding, thermal system insulation, and other building materials commonly used in homes built before 1984 may also contain other substances of concern, including asbestos.

#### 3.0 PROJECT DESCRIPTION

This project will implement air monitoring and sampling during debris removal and asbestos abatement activities at four community locations in the Lahaina area. Site activities began January 13, 2024, and are estimated to continue for approximately 12 months, unless otherwise requested by the HDOH CAB.

Air monitoring and sampling are being conducted to demonstrate the effectiveness of best management practices and engineering controls used during debris removal and asbestos abatement activities and ensure that sensitive populations in the community are not impacted by these activities. Data will be compared to project SSALs (identified in Table 1). These data will also be provided to HDOH CAB.

Tetra Tech will conduct air monitoring and sampling in accordance with the "Specialty Laboratory and Field Support for DOH – Ambient Community Air Monitoring in Response to Wildfire Cleanup Actions" project in the area of Lahaina, Maui. Community monitoring locations will monitor PM10 to allow for comparison to the National Ambient Air Quality Standards (NAAQS) and agreed upon regulatory standards for the State of Hawaii. Air monitoring and sampling will be conducted at four community locations identified by the HDOH CAB.

#### 4.0 PROJECT OBJECTIVES AND DATA USE

The objective of air monitoring and sampling is to provide quantitative feedback on the potential impacts to nearby sensitive receptors in the community during debris removal and asbestos abatement operations. As such, data will be reported to the HDOH CAB and any stakeholders identified and approved by the CAB on a weekly basis. Weekly reports will include air monitoring and air sampling results from the previous week (to allow for laboratory sample analysis, and the review of analytical results). Data collected week one will be reported by the lab on week two. This data will then go through Stage 1 data verification and be reported to HDOH CAB two weeks from sample collection. A reporting week will be defined as Wednesday through Tuesday. Results will be compared to the project's SSALs and any exceedances will be clearly highlighted and provided to HDOH CAB for review and for decision-making use. The HDOH may consider additional SSALs based on data review or input from local officials. The community SSALs are listed in in Table 1 are discussed in Section 5.5.

#### 5.0 FIELD INVESTIGATION ACTIVITIES

This section describes the scope of work and associated work activities.

#### 5.1 METEOROLOGICAL MONITORING

Meteorological data will be obtained continuously from a community A10-2 (or equivalent) weather meter. The data obtained will include:

- Temperature (°F)
- Relative humidity
- Wind direction
- Wind speed
- Current weather conditions (such as partly cloudy or raining, recorded when relevant or in relation to exceedances)

#### 5.2 BACKGROUND AIR MONITORING AND SAMPLING

Tetra Tech anticipated conducting background air monitoring and sampling activities, if work began before debris removal and/or asbestos abatement activities commenced. Tetra Tech initiated air monitoring activities on January 13, 2024, and air sample collection activities on January 14, 2024. Debris removal operations occurred on January 13, 2024. Background air monitoring and sampling was not conducted as Tetra Tech could not initiate air monitoring and sampling prior to debris removal and asbestos abatement operations.

# 5.3 COMMUNITY AIR LOCATIONS AND IDENTIFIERS

Four community air monitoring and air sampling locations (community locations) have been selected by the HDOH CAB based on community population density, sensitive receptor groups, prevailing winds, surrounding communities, and power source access. These locations are within the Lahaina area. Community locations are shown on Figure 1 and their air monitoring and sampling ID, name, and GPS coordinates (latitude and longitude) for each location are listed below:

- AM01 Leialii Hawaiian Homelands (20.8998900, -156.6812240)
- AM02 WW Pump #4 (20.8833490, -156.6857760)
- AM03 Lahaina Intermediate School (20.8860230, -156.6631610)
- AM04 Lahaina Boys and Girls Club (20.8689720, -156.6706670)

All air monitoring and sampling location samples will be named according to the following naming conventions: MFL-AMXX-MMDDYY (community locations), where AMXX = a location code assigned for community locations (for example, AM01 = Air Monitoring Location 01), and where MMDDYY = date that sample is collected (for example, 011324 for January 13, 2024). A sample specific identifier will also be added at the end of the ID (-HM for heavy metals and -AB for asbestos).

The locations will be active for 24 hours a day, and the addition and removal of stations will require approval from the HDOH CAB. One station containing co-located air monitoring and air sampling equipment will be placed at each approved location. Equipment siting at approved community locations will be determined with input from community location owners or representatives and will consider location security and safety.

Each community location will include equipment to monitor air for airborne fine particulate matter concentrations (for particulates with an aerodynamic diameter of not more 10  $\mu$ m or less [PM<sub>10</sub>]), and

sample air for select elemental metals (including, at a minimum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, selenium, thallium, vanadium, and zinc) and asbestos. Each community air station will also log meteorological data including temperature (°F), relative humidity, current weather conditions, wind direction, and wind speed. Particulate monitoring and metals and asbestos sampling will be conducted 24 hours a day. All community air sampling will be implemented as directed by HDOH CAB and in accordance with the guidelines described in the January 2020 California Environmental Protection Agency (CalEPA) Department of Toxic Substances Control (DTSC) Community Air Monitoring Plan (CAMP) Guidance. Monitoring and sampling data from community air station locations will be reviewed and any exceedances of the SSALs (Table 1) will be reported to the HDOH CAB. Following review, data from community locations may be further distributed as directed by HDOH CAB.

# 5.4 SITE-SCREENING ACTION LEVELS

Tetra Tech calculated risk-based community Site-Screening Action Levels (SSALs) for this project. These SSALs are presented in Table 1. Appendix B provides the methodology used by Tetra Tech to calculate this project's SSALs. Potential inhalation exposure hazards due to fugitive emissions from debris removal and asbestos abatement activities are expected to be low because removal contractors are required to employ administrative and engineering controls to minimize fugitive emissions (barricades, warning signs, dust suppression measures, and other mitigative actions). It is possible, however, that removal activities could generate low levels of particulates (dust) that could migrate off site. If a project screening level is exceeded at any community location, the HDOH CAB will be notified according to the procedures described in Section 6.2. SSALs will be used as indicators for excessive off-site migration of particulates, metals, or asbestos at debris removal and asbestos abatement locations.

#### 5.5 PARTICULATE MONITORING

Particulate monitoring will be conducted daily throughout debris removal and asbestos abatement efforts at community locations. This section contains general particulate monitoring procedures followed by additional details for particulate monitoring specific to each type of air location.

To conduct particulate monitoring, Tetra Tech will use Met One Instruments, Inc., environmental beta attenuation mass monitors (E-BAM) to allow for comparison to the National Ambient Air Quality Standards (NAAQS). E-BAMs are factory-calibrated annually and do not require daily calibration, except

for a leak check and a flow audit that should be performed before sampling according to the manufacturer's procedures.

Tetra Tech personnel will conduct periodic system checks of air monitoring equipment at a frequency of at least three times per day to ensure equipment is functional. System checks will consist of assessing the physical condition of the equipment, assessing the equipment to ensure it is collecting data, and briefly assessing the current readings to determine whether they are approaching or have exceeded the SSALs contained in Table 1.

Air monitoring data will be recorded on all air monitoring devices and downloaded onto a computer at the end of each day to be reviewed. Any noted data irregularities or equipment problems will be identified, investigated, and addressed. At the end of each working day, data will be reviewed for accuracy before being compiled in a central database. Air monitoring results will be reported to the HDOH CAB according to the procedures described in Section 6.2.

Gas-powered generators will not be used to power the air monitoring devices. Instead, emission-free power sources such as shore power, tandem batteries, or other green power sources will be used. Air monitoring devices will be sited within the breathing zone (4 to 5 feet above ground level), where possible. If these devices must be positioned outside of the breathing zone, justification for this deviance and the actual placement of the equipment will be noted in field logs.

At selected community locations, particulate monitoring will consist of  $PM_{10}$  data collected for 24 hours per day. Daily time weighted averages (TWAs) of community  $PM_{10}$  particulate data will be compared to the project screening levels of 150  $\mu$ g/m<sup>3</sup>, as shown in Table 1. Project-specific particulate monitoring activities are summarized in Table 2.

#### 5.6 AIR SAMPLING

Tetra Tech will collect ambient air samples at community locations throughout debris removal and asbestos abatement operations. Ambient air samples will be collected using high volume air samplers drawing air through filter media at specific (that is, calibrated) flow rates and for defined sample periods. Community air sampling and monitoring activities are anticipated to last for the duration of debris operations, estimated to be 12 months; however, the HDOH CAB may decide to expand, reduce, or stop air monitoring and sampling based on data received and planned operations. Ambient air samples for asbestos and elemental metals will be collected at all community locations. The sampling methods are presented below.

#### Asbestos

Tetra Tech will collect ambient air samples for asbestos at each community locations each day using a QuickTake 30 or similar. Each sample is anticipated to capture 24 hours. Actual sample volumes will vary, depending on the exact duration of the sampling periods and actual sample pump flow rates used. However, Tetra Tech will ensure that an adequate sample volume is collected for each sample sent for analysis, in accordance with the methodology attached to this CAMSP. Sampling flow rates will be determined and documented by pre- and post- calibrating each sampling pump using a primary calibration standard.

The ambient air sampling units at each on-site location will consist of high flow air pumps, such as Casella Vortex 3 or similar, operated at a flow rate of 4-5 liters per minute (L/min). These flow rates are required to collect the additional sample volume necessary to meet the more conservative community screening levels contained in Table 1, which will assess ambient air quality with consideration of potential sensitive receptors. The total daily ambient air sampling time at on-site locations is expected to be approximately 24 hours or 1,440 minutes, during which Tetra Tech will collect approximately 6,480 liters of air through the sample filter media.

At each community sampling location, samples will be collected on a 25-mm open-face 0.45-µm MCE sample cassette with a conductive cowl mounted on a 4- to 5-foot-high cassette tripod stand that is attached to each air pump, with the opening facing at a 45-degree angle downward. The flow rate of the entire assembled ambient air sampling train will be calibrated before sample collection and measured after sample collection using a primary gas flow calibrator, such as a Bios DryCal DC-Lite or similar, capable of calibrating pumps from 1 to 10 L/min. The flow rate during the sample period will be determined by the average of the two readings. Calibration and sampling will be conducted in accordance with Tetra Tech SOPs 064-2, "Calibration of Air Sampling Pump" and 073-3, "Air Quality Monitoring" (Appendix A) and U.S. EPA ERT SOPs No. 2008, "General Air Monitoring and Sampling Guidelines" and 2015 "Asbestos Air Sampling" (Attachment 1).

To avoid the potential for sample filter over-loading (particularly if visibly dusty conditions are observed during sample periods), Tetra Tech staff will perform periodic visual inspections of each filter in the asbestos cowling during each sampling period. If visible loading is observed on a filter, Tetra Tech will replace the sample cassette with a new one, and the analytical results from all cassettes from that overall sampling period will be used to determine the asbestos concentration for that workday.

Community air samples for asbestos will be submitted to a Tetra Tech-procured laboratory for analysis via the International Organization for Standardization (ISO) method 10312, "Ambient Air – Determination of Asbestos Fibres – Direct Transfer Transmission Electron Microscopy Method." Subcontracted laboratories will analyze 10 grid openings. If additional grid openings need to be viewed to meet project detection levels and sensitivity levels, Tetra Tech will authorize up to 10 additional grid openings. If additional grid openings are required, HDOH CAB will be consulted. ISO 10312 results will be reported by transmission electron microscopy (TEM) in structures per cubic centimeter (s/cc) that will be considered equivalent to fibers per cubic centimeters (f/cc) (U.S. EPA 2021). TEM results will be compared to the SSALs referenced in Table 1. If results are above these screening levels, Tetra Tech will authorize up to 10 additional grid openings on the TEM to improve statistical confidence in actual asbestos fibers for the result. If filter particulate loading exceeds 25 percent, upon consultation with the laboratory, Tetra Tech will authorize ISO method ISO-13794 "Ambient air – Determination of Asbestos Fibres - Indirect-Transfer Transmission Electron Microscopy Method." Initial asbestos air sample results will be requested with a turnaround (TAT) time of 72-hours. Additional grid openings and indirect methods, if required, will be requested for the laboratory's standard turnaround time; expedited TAT may be requested if approved by the HDOH CAB. The results of on-site ambient air sampling for asbestos will be evaluated against the SSALs identified in Table 1. Sampling requirements are presented in Table 3.

#### **Elemental Metals**

Tetra Tech will collect ambient air samples for select elemental metals daily at each of the four community locations identified by the HDOH CAB. Ambient air samples for metals will be collected with a Tisch Environmental High Volume Air Sampler, or equivalent, and collocated with the real-time particulate monitors described in Section 5.6 and asbestos samplers described above. The objective of ambient air sampling for metals at community locations is to assess ambient air quality with considerations for potential sensitive receptors.

Ambient air sampling methods require larger sampling devices and media that can draw significantly more air volume than methods intended for occupational exposure assessments. As a result, air sampling for elemental metals at community locations will employ the following air sampling methods:

- EPA Compendium Method IO-2.1, Sampling of Ambient Air for Total Suspended Particulate Matter (SPM) and PM10 Using High Volume (HV) Sampler
- EPA Compendium Method IO-3.5: Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air: Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP/MS). EPA/625/R-96/010a

- EPA 40 Code of Federal Regulations (CFR) Part 50, Method for the Determination of Lead in Total Suspended Particulate Matter.
- EPA 40 CFR Part 58, Appendix E: Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring
- Standard Operating Procedures for Lead Monitoring Using a TSP High Volume Sampler

Results will be reported for elemental metals including, at a minimum, the following: antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, selenium, thallium, vanadium, and zinc. The results of community air sampling for elemental metals will be evaluated against the SSALs identified in Table 1 and by calculating the 95% upper confidence limit of the mean of the data over the project duration intervals. Exceedances over the SSALs will be investigated for potential causes. Sampling requirements are presented in Table 3.

# **Sample Handling**

Sampling locations will be noted on field sheets or digital forms. The collected samples will be labeled, packaged, and shipped under chain-of-custody in accordance with approved sampling methods and with the procedures outlined in Tetra Tech SOP No. 019-9, "Packing and Shipping Samples."

#### 5.7 DOCUMENTATION AND REPORTING

Tetra Tech will document all activities, including sampling locations, equipment set up, field measurements, samples collected, analytical reports, and deviations from this CAMSP in a field logbook, on field sheets, or using digital forms.

Tetra Tech will document work using photographs. Tetra Tech will document field work in accordance with Tetra Tech SOP No. 024-4, "Recording of Notes in Field Logbook" (Tetra Tech 2022, attached in Appendix A).

Tetra Tech will verify and manage data as described in this plan (see also Section 6.5). Tetra Tech will share data through secure means with HDOH and will report any results above the SSALs for each location type to HDOH CAB. A summary report of particulate monitoring and air sample results will be made available to the HDOH CAB on a weekly basis. These reports will comprise of a written summary detailing the project objectives, meteorological conditions, results for the community locations, and quality control measures. Attached to the reports will be a figure depicting the community locations, analytical summary tables for ambient air asbestos and heavy metal sample results, the particulate daily

24-hour TWA calculated results, and meteorological data. The analytical laboratory reports including stage 1 verification documents will be attached at the end of the weekly reports. The reporting week schedule is as follows: samples will be shipped on Mondays and/or Thursdays each week, with results received Fridays and Tuesdays. These results will then go through Stage 1 data verification and be reported to HDOH CAB. Revised reports may need to be submitted if sample results were not received in time, especially in the case of asbestos air samples that needed additional TEM analysis. Following the conclusion of the project, a final summary report will be provided to HDOH.

# 6.0 QUALITY ASSURANCE PROJECT PLAN

This section serves as the quality assurance project plan (QAPP) for air monitoring and sampling during debris removal and asbestos abatements activities in Lahaina for the "Specialty Laboratory and Field Support for HDOH – Ambient Community Air Monitoring in Response to Wildfire Cleanup Actions" project. This QAPP meets the requirements of the Hawai'i Department of Health (HDOH) Hazard Evaluation and Emergency Response Office (HEER) Technical Guidance Manual (TGM), including relevant EPA QAPP guidance as cited in the TGM (HEER 2023). Specifically, this section addresses quality assurance/quality control data objectives; chain of custody maintenance; calibration procedures and frequency; analytical methods; data reduction, verification, and reporting; field quality control checks; laboratory quality control checks; performance and system audits; preventative maintenance; data quality assessment; and corrective action.

# 6.1 QUALITY ASSURANCE/QUALITY CONTROL DATA OBJECTIVES

As part of the project planning process, Tetra Tech used the data quality objective (DQO) process to: (1) clarify study objectives and decisions to be made based on the data collected; (2) define the information to be collected; (3) specify the methods for collecting the information; and (4) specify performance or acceptance criteria that will be used as the basis for establishing the quality of information needed to support the decision. The DQO development process is a recommended approach for ensuring appropriate planning for environmental investigations, as described in EPA's "Guidance on Systematic Planning Using the Data Quality Objectives Process" (EPA 2006). Developing DQOs is a seven-step process:

- 1. State the Problem: As documented in Sections 2, 3, and 4, ash and debris from residential and other structures burned by the Lāhainā wildfires can contain heavy metals or asbestos from residual building materials, such as stucco, roofing, floor tile, linoleum, fireplaces, furnaces, vinyl tiles and mastic, sheetrock and joint compound, cement pipe, exterior siding, thermal system insulation, and other materials. Although removal contractors are required to employ administrative and engineering controls to minimize fugitive emissions (barricades, warning signs, dust suppression measures, and other mitigative actions), it is possible that fire debris removal activities, including asbestos abatement, could generate low levels of particulates (dust), including heavy metals and asbestos, that could migrate off site.
- 2. Identify the Goal of the Study: The objective of air monitoring and sampling is to ensure that administrative and engineering controls employed during debris removal are successful in

- maintaining dust (specifically  $PM_{10}$ ), heavy metals, and asbestos concentrations to below community SSALs (see Table 1).
- 3. Identify Information Inputs: Tetra Tech will deploy three types of equipment at each of four air monitoring stations: (1) Met One Instruments, Inc., E-BAMs to provide real-time PM<sub>10</sub> concentrations, (2) SKC, Inc. QuickTake® 30 (QuickTake 30), or equivalent, air sampling pumps with attached a 25-mm open-face 0.45-µm MCE sample cassette with a conductive cowl to collect high-volume samples for asbestos analysis, and (3) Tisch Environmental High Volume Air Samplers, or equivalent, with 1,600 cubic meter, 8 by 10-inch quartz filters for metals analysis. See Section 5 for additional sample collection information. Tetra Tech will also monitor and log the meteorological data daily at one or more of the community locations using the meteorological data obtained will include temperature (°F), relative humidity, wind direction, wind speed, and current weather conditions (such as partly cloudy or raining). Section 3.1 and Figure 1 present the community air monitoring and sampling station locations.

Particulate concentrations will be continuously logged by the EBAMs. Asbestos samples will be submitted to EMSL Analytical, Inc. in Cinnaminson, New Jersey for asbestos analysis via transmission electron microscopy (TEM) using International Organization for Standardization (ISO) 10312 "Ambient Air – Determination of Asbestos Fibres – Direct Transfer Transmission Electron Microscopy Method." If filter particulate loading exceeds 25 percent, upon consultation with the laboratory, Tetra Tech will authorize ISO method 13794 "Ambient air – Determination of Asbestos Fibres – Indirect-Transfer Transmission Electron Microscopy Method." Samples collected for metals analysis will be submitted to Eastern Research Group, Inc. (ERG) in Morrisville, North Carolina for analysis by EPA Compendium Method IO-3.5: Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air: Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (EPA 1999) and methods equivalent to 40 CFR Part 50, Method for the Determination of Lead in Total Suspended Particulate Matter.

**4. Define Boundaries of the Study:** Particulate monitoring will be conducted daily throughout debris removal and asbestos abatement efforts at four community locations for up to 12 months (see Figure 1). The sampling frequency may be reduced as requested by the CAB upon the CAB's receipt and evaluation of the test results from the first week of debris removal. However, a minimum 2 weeks of sampling will occur. Also, if debris removal and asbestos abatement have

- not yet commenced upon startup of air monitoring and sampling activities, Tetra Tech will attempt to collect up to 5 days of daily background samples.
- 5. Develop the Analytic Approach: Tetra Tech will compare the verified results to the SSALs in Table 1. Tetra Tech will notify HDOH CAB of any particulate monitoring or air sampling results from community locations that exceed SSALs within 24 hours of collection for particulate monitoring results or within 24 hours of receiving results for air samples. Tetra Tech will also investigation potential causes of the SSAL exceedances.
- **6. Specify Performance or Acceptance Criteria:** Laboratory QA/QC and related requirements are provided in Section 6.7, Tables 5 through 7, and relevant laboratory-specific SOPs, quality assurance manuals, and certifications included in Attachment 2. Stage 1 data verification requirements are listed in Step 5.
- 7. **Develop a Plan for Obtaining Data:** The plan for obtaining data is documented in Section 5.

For this project the DQOs established for this project are those in the list above; the QA/QC procedures to achieve these DQOs are discussed throughout this CAMSP and detailed in its tables, appendices, attachments, and references.

#### 6.2 CHAIN OF CUSTODY MAINTENANCE

Samplers must collect samples according to strict sampling procedures and plans. A chain-of-custody form will used to document the transfer of custody of samples from the field to the designated analytical laboratory. The chain-of-custody form is critical to the chain-of-custody process and is used to identify the samples in each shipping container to be shipped or delivered to the laboratory for analysis. Tetra Tech will label, package, and ship the samples under chain-of-custody in accordance with approved sampling methods and with the procedures outlined in Tetra Tech SOP No. 019-9, "Packing and Shipping Samples." The chain-of-custody must not be broken between the sampler and the laboratory sample receiving personnel. The chain-of-custody will include the name of the shipping company, if the samples need to be shipped. A copy of the chain-of-custody form is shipped with the samples and accompanies them from sampler to laboratory. The laboratory will provide a copy of the final chain of custody form with the laboratory report and Tetra Tech will maintain these records in the project database.

# 6.3 CALIBRATION PROCEDURES AND FREQUENCY

Field equipment calibration procedures and frequency are detailed in Sections 5.6 and 5.7 and Table 4. Field equipment calibration and equipment field checks will be logged in a field logbook and/or on a calibration log sheet accompanying the instrument. Laboratory instrument calibration requirements are summarized in Table 6 and are addressed in the laboratory SOPs in Attachment 2; laboratory calibrations will be documented by the laboratory.

#### 6.4 ANALYTICAL METHODS

Analytical methods include ISO methods 10312 "Ambient Air – Determination of Asbestos Fibres – Direct Transfer Transmission Electron Microscopy Method" and 13794 "Ambient air – Determination of Asbestos Fibres – Indirect-Transfer Transmission Electron Microscopy Method." Metals analytical methods include EPA Compendium Method IO-3.5: Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air: Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (EPA 1999) and methods equivalent to 40 CFR Part 50, Method for the Determination of Lead in Total Suspended Particulate Matter. Laboratory analysis based on these methods is detailed in the laboratory SOPs and quality assurance manuals in Attachment 2. Attachment 2 also contains the relevant laboratory certifications for these methods.

# 6.5 DATA REDUCTION, VERIFICATION, AND REPORTING

Tetra Tech will use direct-reading instruments with data logging capability for particulate monitoring. Monitoring data will be downloaded from instruments at the end of each day and peer reviewed and verified for the appropriate QA objectives. Daily TWAs will be calculated from the logged data from each community particulate monitor and any readings above the SSALs will be reported to HDOH CAB the following day.

Tetra Tech will record field measurements collected via monitoring on field data sheets or on digital forms using tablets, as well as direct downloads. Data will be evaluated daily as it is collected (particulate monitoring) and received from the laboratory (air sample analytical results) to identify and report any elevated results or screening level exceedances. All electronic data will be stored in a central database (SQL server) that is managed by Tetra Tech.

Asbestos and heavy metals air sampling analytical results provided by the laboratories in Level I electronic data deliverables (EDD) and portable document format (PDF) file format data packages will undergo a Stage 1 data verification under the supervision of a chemist. Stage 1 data verification consists

of a compliance review for sample receipt conditions, the chain of custody, all samples are accounted for, the requested analytical methods were performed, the analytes dates and results are provided, all qualifications are defined, and the lab report contains the units, method detection limits, and reporting limits. In addition, Tetra Tech will verify that no analytes are detected in field blanks and lot blanks. If data quality issues are identified, Tetra Tech will identify the cause and consult with HDOH CAB. The verified results will be maintained in an electronic database and compared to the SSALs in Table 1 and by calculating the 95% upper confidence limit of the mean of the data over the project duration intervals. Following data verification, data will also be uploaded and available via a shared site folder to review by HDOH. Tetra Tech will notify HDOH CAB of any particulate monitoring or air sampling results from community locations that exceed SSALs within 24 hours of collection for particulate monitoring results or within 24 hours of receiving results for air samples. Tetra Tech will also investigation potential causes of the SSAL exceedances.

A summary report of particulate monitoring and air sample results will be made available to the HDOH CAB on a weekly basis. These reports will show verified data results from samples collected two weeks prior. A reporting week schedule is as follows: samples will be shipped on Mondays and/or Thursdays each week, with results received Fridays and Tuesdays. These results will then go through Stage 1 data verification and be reported to HDOH CAB. Revised reports may need to be submitted if sample results were not received in time, especially in the case of asbestos air samples that needed additional TEM analysis. Following the conclusion of the project, a final summary report will be provided to HDOH.

# 6.6 FIELD QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) CHECKS

Field QA/QC measures will include following Tetra Tech and EPA SOPs. Tetra Tech will implement proper equipment calibration; and adhere to instrument manufacturer user manuals, published and peer-reviewed sampling methods, and Tetra Tech and EPA SOPs for air monitoring, sampling, and documenting activities on field sheets and digital forms. Table 4 describes the equipment QA/QC measures and specifications related to equipment calibration.

The Tetra Tech field team manager will be responsible for ensuring that sample quality and integrity are maintained, and that sample label and documentation procedures are in accordance with each SOP and guidance or manual. Table 3 summarizes field QC sample frequency.

# 6.7 LABORATORY QA/QC CHECKS

Laboratory QA and QC checks are documented in Tables 5 through 7, and relevant laboratory-specific SOPs, quality assurance manuals, and certifications included in Attachment 2.

#### 6.8 PERFORMANCE AND SYSTEM AUDITS

A field audit will be conducted to verify that field sampling and monitoring procedures are performed in accordance with the procedures and requirements established in this CAMSP. Tetra Tech will conduct semi-annual field audits. Tetra Tech or HDOH CAB may conduct other performance and system audits of both field and laboratory activities based on evolution of project needs and to verify that activities conducted are performed in accordance with this CAMSP.

The Tetra Tech project manager will be responsible for directing internal audit activities and selecting the appropriate personnel to conduct each internal audit. Auditors will be independent of the activities being audited. The field audit will include the following checklist:

Item	Description of Field Audit Activities
1.	Review of field-sampling records
2.	Review of equipment operation, maintenance, and calibration records
3.	Examination of the application of sample identifications following the specified protocol
4.	Review of the sample handling and packaging procedures
5.	Review of COC procedures

If deficiencies are observed, they will be noted in writing, and corrective actions may need to be implemented by the Tetra Tech project manager or field team manager. Corrective actions in response to audit findings, if conducted, will be initiated, implemented, and checked according to this plan. Additional information regarding corrective actions is provided in Section 6.11. A report summarizing audit activities including findings, recommendations for improvement, and corrective actions taken, if applicable, will be prepared by Tetra Tech and submitted to HDOH CAB.

# 6.9 PREVENTIVE MAINTENANCE

Analytical instrument and equipment maintenance, testing, and inspection is summarized in Table 7 and included in relevant laboratory SOPs in Attachment 2. Field equipment will be maintained in accordance with manufacturer requirements, Tetra Tech and EPA SOPs, and other guidance documents referred to in this CAMSP.

# 6.10 DATA QUALITY ASSESSMENT

All monitoring and sampling analytical results will be evaluated in accordance with precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters to document data quality and to ensure that the data are of sufficient quality to meet project objectives. The sections below describe how each PARCC parameter will be assessed and how we will determine that DQOs have been met.

# **Precision**

Precision is the degree of mutual agreement between individual measurements under similar conditions. Laboratory analytical precision is evaluated by analyzing laboratory duplicates or MS and MSD, typically utilizing the following formula:

$$RPD = \frac{|A-B|}{(A+B)/2} \times 100\%$$

where:

A = First duplicate concentration

B = Second duplicate concentration

The results of the analysis of each MS/MSD and sample duplicate pairs will be used to calculate an RPD for evaluating precision by the laboratory. Laboratory acceptance criteria are presented in Table 5.

#### Accuracy

Accuracy is a measure of the agreement between a measurement and a known value. Sample spiking will be conducted to evaluate laboratory accuracy. This includes analysis of the MS and MSD samples, laboratory control samples (LCS) and laboratory control sample duplicates (LCSD), or blank spikes and method blanks. MS and MSD samples will be prepared and analyzed at a frequency of 5 percent. LCS or blank spikes are also analyzed at a frequency of 5 percent. Results of the spiked samples are used to calculate the percent recovery for evaluating accuracy using the following equation:

Percent Recovery = 
$$\frac{S-C}{T}$$
 x 100

where:

S = Measured spiked sample concentration

C = Sample concentration

T = True or actual concentration of the spike

Results that fall outside the accuracy goals specified in Table 5 will be qualified by the laboratory.

# Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent the characteristics of a population, variations in a parameter at a sampling point, or an environmental condition that those data are intended to represent. Tetra Tech will ensure representativeness by implementing the monitoring, sampling, analytical and associated quality program as documented in this plan. Potential outside impacts on sample results will be valuated through review of field QC samples (i.e., field blanks and equipment blanks) to ensure no contaminants are detected, which may indicate that non-site related contaminants may have been introduced may indicate that compounds have been introduced field QC samples (i.e., field blanks and equipment blanks) may indicate that compounds have been introduced into the samples. Laboratory quality control, as documented in previous sections, will also ensure sample representativeness.

If analyses of field or lot blank samples result in detected contaminants, the field procedures for sample handling and sample transport will be evaluated for how well procedures were followed, for any potential introduction of contaminants from outside sources, or for potential losses in the course of sample handling or transport.

Data determined non-representative will be used only if accompanied by appropriate qualifiers and specified limits of uncertainty.

#### **Completeness**

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures outlined in this QAPP and laboratory SOPs and QAMs, and when the QC criteria that affect data usability are met. When all data verification is completed, the percent completeness will be calculated by dividing the number of useable sample results by the total number of sample results planned for this investigation. Completeness goals are specified in Table 5.

# **Comparability**

Comparability expresses the confidence with which one data set can be compared with another.

Comparability of data for this investigation will be achieved by following standard field and laboratory procedures, and using standard measurement units in reporting analytical data.

# **Determining if Data Meets DQOs**

As specified by HDOH, Tetra Tech will conduct Stage 1 verification as specified in Section 6.5 to determine if monitoring and analytical results meet project specific DQOs. Laboratories will evaluate results in accordance with relevant laboratory SOPs, quality assurance manuals, and associated analytical methods and qualify the analytical results as appropriate.

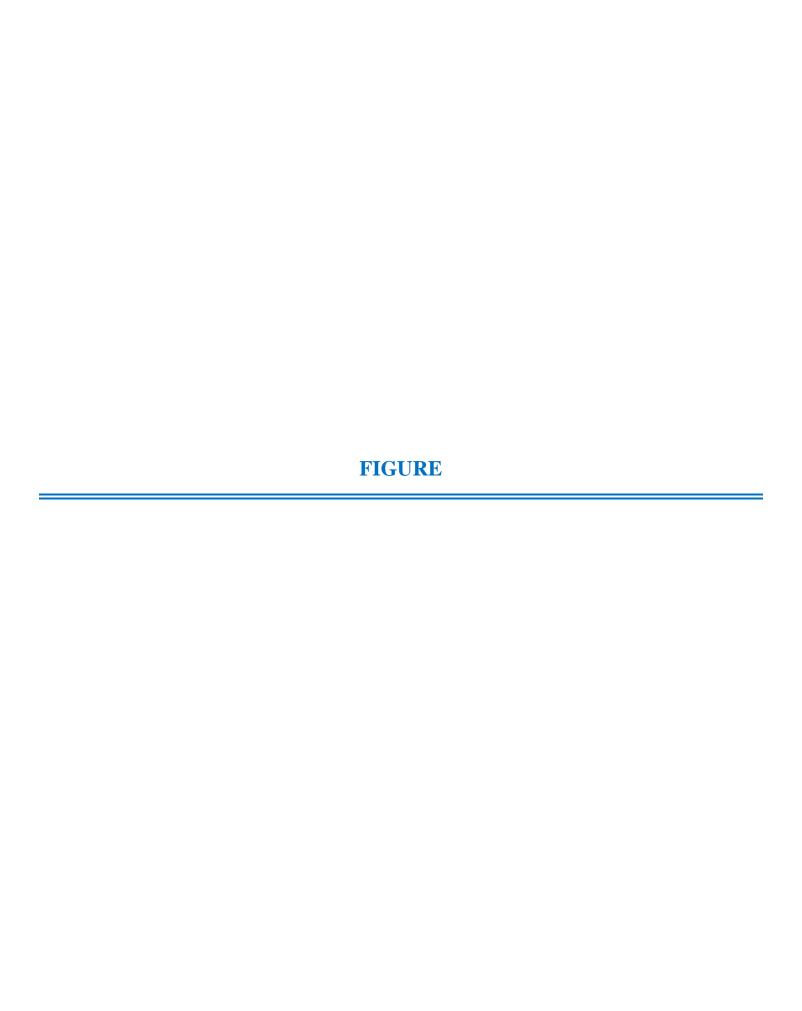
#### 6.11 CORRECTIVE ACTION

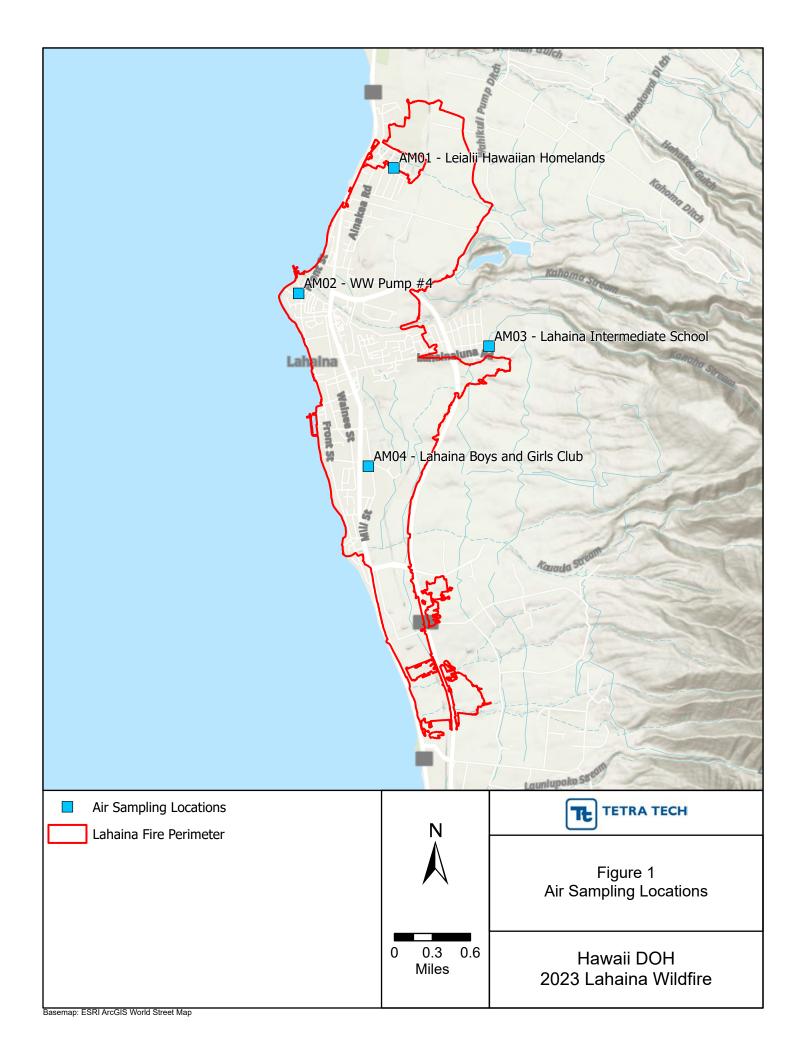
Any DQO non-conformances identified during field checks, field audits, or Stage 1 verification will be investigated by Tetra Tech to identify the potential origin of the issue. If necessary, Tetra Tech initiate any appropriate corrective action. Corrective action will involve evaluating potential impacts that these exceedances have on data quality and therefore usability of the data. Any non-conformances will be reported to HDOH.

#### 7.0 REFERENCES

- California Environmental Protection Agency (CEPA). 2011. "Guidance for Conducting Emergency Debris, Waste, and Hazardous Material Removal Actions Pursuant to a State or Local Emergency Proclamation." October 7.
- California Department of Toxic Substances Control (DTSC). 2007. "Assessment of Burn Debris 2007 Wildfires San Bernardino and San Diego Counties, California" December 27.
- DTSC. 2015. "Assessment of Burned Debris- 2015 Wildfires Lake and Calaveras County, California"

  December 7
- DTSC. 2020. "Community Air Monitoring Plan Guidance" January.
- Hawai'i Department of Health (HDOH) Hazard Evaluation and Emergency Response Office (HEER). 2023. Technical Guidance Manual (TGM). July. Online Address: <a href="https://health.hawaii.gov/heer/tgm/">https://health.hawaii.gov/heer/tgm/</a>.
- United States Environmental Protection Agency (EPA). 1999. Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.5, Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP/MS). Center for Environmental Research Information, Office of Research and Development. EPA/625/R-96/010a. June. Online Address: <a href="https://www.epa.gov/sites/default/files/2015-07/documents/epa-io-3.5.pdf">https://www.epa.gov/sites/default/files/2015-07/documents/epa-io-3.5.pdf</a>.
- EPA. 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA QA/G-4). Office of Environmental Information. Washington, DC. EPA/240/B-06/001. February.
- EPA. 2021. Framework for Investigating Asbestos-Contaminated Comprehensive Environmental Response, Compensation and Liability Act Sites. OLEM Directive #9200.0-90.





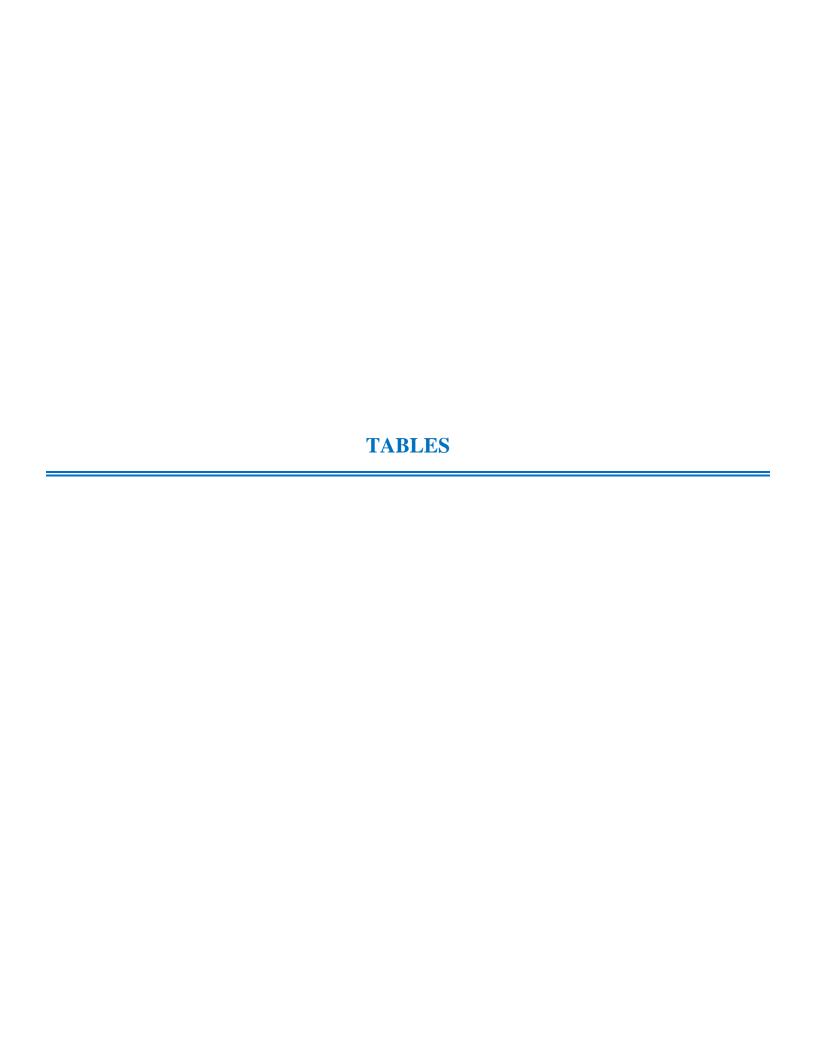


TABLE 1

COMMUNITY SITE-SPECIFIC AIR SCREENING LEVELS AND LABORATORY REPORTING LIMITS

Contaminant or Hazard	Selected SSAL <sup>3</sup>	Regulatory/ Guidance Value Used to Derive SSAL <sup>1, 2</sup>	Laboratory Reporting Limit <sup>4</sup>
Particulate Matter $(PM_{10})$	150 μg/m³	NAAQS	NA
Antimony CAS RN 7440-36-0	0.7 μg/m³	ATSDR MRL (Chronic; non-carc) 2019	0.0001 μg/m³
Arsenic CAS RN 7440-38-2	0.05 μg/m <sup>3</sup>	IRIS (carc)	0.00003 μg/m³
Barium CAS RN 7440-39-3	1.2 μg/m³	HEAST (non-carc; per USEPA RSL Table)	0.0034 μg/m³
Beryllium CAS RN 7440-41-7	0.05 μg/m³	IRIS (non-carc)	0.00001 μg/m <sup>3</sup>
Cadmium CAS RN 7440-43-9	0.02 μg/m <sup>3</sup>	ATSDR MRL (Chronic; non-carc) 2012	0.0003 μg/m³
Chromium CAS RN 7440-47-3	12 μg/m³	ATSDR MRL (Intermediate, non-carc, insoluble particulates) 2012	0.0070 μg/m³
Cobalt CAS RN 7440-48-4	0.01 μg/m³	EPA PPRTV (non-carc), 2008. (More conservative than 2023 MRL chronic, which is 0.1ug/m3. Same study and POD, but PPRTV applied 300 UF and MRL 10 UF)	0.0001 μg/m³
Copper CAS RN 7440-50-8	240 μg/m³	NIOSH REL/ 10x safety factor; Professional judgment	0.008 μg/m³
Lead CAS RN 7439-92-1	1.5 μg/m³	Hawaii Administrative Rules §11-59-4 (during calendar quarter)	0.0007 μg/m <sup>3</sup>
Manganese CAS RN 7439-96-5	0.12 μg/m <sup>3</sup>	IRIS (non-carc)	0.0060 μg/m³
Molybdenum CAS RN 7439-98-7	4.8 μg/m³	ATSDR MRL (Chronic; non-carc) 2020	0.0011 μg/m <sup>3</sup>
Nickel CAS RN 7440-20-0	0.02 μg/m³	ATSDR MRL (Chronic; non-carc) 2023	0.0021 μg/m³
Selenium CAS RN 7782-49-2	48 μg/m³	OEHHA Chronic REL (non-carc)	0.00003 μg/m³
Thallium CAS RN 7440-28-0	24 μg/m³	NIOSH REL/ 10x safety factor; Professional judgment	0.000002 μg/m³
Vanadium CAS RN 114-62-1	0.24 μg/m <sup>3</sup>	ATSDR MRL (Chronic; non-carc) 2012	0.0002 μg/m³
Zinc CAS RN 7440-66-6	1,200 μg/m³	NIOSH REL for zinc oxide/ 10x safety factor; Professional judgment	0.244 μg/m³
Asbestos	0.003 f/cc <sup>5</sup>	NIOSH REL of 0.1 f/cc (OSHA Table Z-3 TWA)/ 30x safety factor	0.0025 s/cc <sup>6</sup>

#### TABLE 1 (CONTINUED)

# COMMUNITY SITE-SPECIFIC AIR SCREENING LEVELS AND LABORATORY REPORTING LIMITS

#### Notes:

- 1: Guidance values were used to calculate SSALs based on risk calculations outlined in Appendix B
- <sup>2</sup>: Exposure Assumptions for risk screening values:

Exposure frequency = 313 days per year i.e., 12 months, six days per week project duration.)

Exposure time = 10 hours/day

Lifetime (for carcinogenic risk screening calculations): 70 years

- <sup>3</sup>: Analytical method recommendation for metals: High volume, ICP/MS. Sampling Method EPA IO-2.1 and analytical method EPA IO-3.5
- <sup>4</sup> Metals reporting limits are from Eastern Research Group, Inc. and based on a sample volume of 1,627 cubic meters. Asbestos reporting limit is based on the limit of detection provided by EMSL Analytical, Inc. for a sample volume of 6,480 liters of air.
- <sup>5</sup> The NIOSH REL (up to 10-hour TWA) of 0.1 f/cc with an applied safety factor of 30 is selected as the asbestos SSAL to be both protective of human health and meet the laboratory detection limit.
- <sup>6</sup> Asbestos sample analytical results in structures per cubic centimeter (s/cc) will be considered equivalent to fibers per cubic centimeters (f/cc) (U.S. EPA 2021).

ATSDR - Agency for Toxic Substances and Disease Registry

Carc - risk screening level based on carcinogenic effects

CAS RN - Chemical Abstracts Service Registry Number

EPA PPRTV - U.S. Environmental Protection Agency Provisional Peer-Reviewed Toxicity Values

FAA methods from Hawaii Analytical Laboratory, LLC

f/cc – fibers per cubic centimeter

IRIS - Integrated Risk Information System

ISO 10312 - Determination of asbestos fibers- Direct transfer transmission electron microscopy method

MRL - Minimal Risk Level

NA – Not applicable

NAAQS - National Ambient Air Quality Standards

NIOSH - National Institute for Occupational Safety and Health

Non-carc - Risk screening level based on noncarcinogenic effects

OEHHA - Office of Environmental Health Hazard Assessment

REL - Reference Exposure Level

s/cc – structures per cubic centimeter

SSAL - Site Screening Action Level

μg/m<sup>3</sup> - micrograms per cubic meter

TABLE 2
PROJECT-SPECIFIC PARTICULATE MONITORING

<b>Location Type</b>	Monitoring Fraction / Frequency	Monitoring Device	Data Review	Screening Level - Reference	Alarm & Excursion Reporting	Alarm or Screening Level Excursion – Potential Outcomes
Community	PM <sub>10</sub> / 24 hours	Particulate Monitor (E- BAM)	Daily, between operational periods	150 μg/m <sup>3</sup> - NAAQS 24- hour average	HDOH CAB within 24 hours	Data will be reported to HDOH CAB and used at the discretion of HDOH CAB

# Notes:

The National Ambient Air Quality Standard (NAAQS) for PM<sub>10</sub> is based on a 24-hour average.

E-BAM: Environmental Beta Attenuation Mass Monitor

HDOH CAB: Hawaii Department of Health (Clean Air Branch)

μg/m<sup>3:</sup> micrograms per cubic meter

PM: particulate matter

TABLE 3
SAMPLING REQUIREMENTS WORKSHEET

	Parameter and Method			Number of Quality Control (QC) Samples					Total	Total
Parameter and Method		and Investigat	Number of Investigative Samples	Matrix Spike / Matrix Spike Duplicate (MS/MSD)	Field Duplicate or Split	Equipme nt Blank	Field Blank	Trip Blank	Number of Investigativ e and QC Samples	Number of Sample Containers
Community Elemental Metals <sup>1</sup> (minus mercury and silver)	Elemental Metals <sup>1</sup> (Minus mercury and silver) / Metals analysis in accordance with EPA Compendium Method IO-3.5, or equivalent.	1,627 cubic meters 8x10 quartz filters	TBD	1 every 20	NA	1 / lot	1 / day	NA	TBD	TBD
Community Asbestos High-Flow	Asbestos / ISO 10312 (direct) or ISO 13794 (indirect)	6,480 liters of air / 25-mm 0.45-µm MCE cassette with cowl	TBD	NA	NA	1 / lot (minimum of 2 per 100 filter)	1 / day	NA	TBD	TBD

<sup>1</sup>Elemental metals include: antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, selenium, thallium, vanadium, zinc

QC: quality control mm: millimeter µm: micrometer TBD: to be determined

MS/MSD: matrix spike/matrix spike duplicate

NA: not applicable

TABLE 4
FIELD EQUIPMENT CALIBRATION REQUIREMENTS

Instrument	Parameters to Measure	Frequency of Calibration	Calibration gas/concentration	
E-BAM	Particulates	Leak test and flow audit before start of sampling and monthly; factory calibration annually	Not applicable (NA) (annual factory calibration)	
Casella Vortex 3	Asbestos	Twice Daily (Pre- and Post-Sampling)	NA	
Tisch	Elemental Metals	Upon installation, after routine maintenance, once every 3 months, after 360 sampling hours	NA	

TABLE 5

LABORATORY MEASUREMENT PERFORMANCE CRITERIA

Matrix	Air			
Analytical Group	Asbestos TEM by ISO 10312 <sup>a</sup>			
Sampling Procedure <sup>b</sup>	Analytical Method SOP <sup>c</sup>	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance
Various including but not limited to Tetra Tech SOPs 064-1,	EMSL Analytical SOP "ISO 10312 Ambient air –	Accuracy/Bias-Contamination	Asbestos structures < QL	Laboratory blank Lot blank Field blank
"Calibration of Air Sampling Pump," and 073-2 Air Quality Monitoring, and U.S. EPA Emergency	Determination of asbestos fibres – Direct-transfer transmission electron microscopy method	Accuracy/Bias	Re-analysis by same analyst and re-analysis by different analyst each at frequencies of 4% and 2%, respectively. Pass/fail criteria for repeat results should not differ at the 5% significance level.	Re-analysis
Response Team (ERT) SOPs No. 2008, "General Air Monitoring and Sampling Guidance" and 2015, "Asbestos Air Sampling"	[ASB-SOP-417, Revision 10]"	Precision	The analytical precision is dependent upon the number of structures counted and the uniformity of the particulate deposit. Assuming a uniform distribution and structure loading of at least 3.5 structures per grid opening, the coefficient of variance of the counting procedure can be estimated at 10 %.	Analysis
		Sensitivity	0.00046 s/cc	Analysis
		Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after verification

# TABLE 5 (CONTINUED)

# LABORATORY MEASUREMENT PERFORMANCE CRITERIA

Matrix	Air			
Analytical Group	Asbestos Indirect- Transfer TEM by ISO 13794-Backup method if filter is overloaded <sup>a</sup>			
Sampling Procedure <sup>b</sup>	Analytical Method SOP <sup>c</sup>	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance
Various including but not limited to Tetra Tech SOPs 064-1, "Calibration of Air Sampling Pump," and 073-2 Air Quality Monitoring, and U.S. EPA Emergency Response Team	EMSL Analytical SOP "Ambient air – Determination of asbestos fibres – Indirect-transfer transmission electron microscopy method" ISO 13794 SOP, Revision 1.2	Accuracy/Bias-Contamination  Accuracy/Bias	Asbestos structures < QL  Re-analysis by same analyst and re- analysis by different analyst each at a frequency of at 4% of the same grid opening; re-analysis results should not differ greater than a 5% significance level	Laboratory blank Process blanks (asher and filtration) Lot blank Field blank Re-analysis
(ERT) SOPs No. 2008, "General Air Monitoring and Sampling Guidance" and 2015, "Asbestos Air Sampling"		Precision  Completeness	Results should be $\leq 80$ % of true positives, $\leq 20$ % false negatives, and $\leq 10$ % false positives of the Total Structure count $\geq 90$ %	Analysis  Data completeness defined as data not qualified as rejected after verification

# **TABLE 5 (CONTINUED)**

# LABORATORY MEASUREMENT PERFORMANCE CRITERIA

Matrix	Ambient Air	1	SOREMENT LEAF ORMAINCE CRITERI	
<b>Analytical Group</b>	Metals <sup>d</sup>			
Concentration Level / Filter Size and Type	Low / 8x10" Quartz Fiber Filters			
Sampling Procedure <sup>b</sup>	Analytical Method SOP <sup>c</sup>	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance
		Accuracy	Recovery must be between 60 and 125% of the measured intensity of the calibration blank.	Internal Standard Response
		Accuracy/Bias- Contamination	Absolute value must be <mdl.< td=""><td>Laboratory Reagent Blank (LRB/BLK1)</td></mdl.<>	Laboratory Reagent Blank (LRB/BLK1)
		Accuracy/Bias- Contamination	Absolute value must be <mdl.< td=""><td>Method Blank (MB/BLK2)</td></mdl.<>	Method Blank (MB/BLK2)
	Eastern Research Group SOP ERG- MOR-095	Accuracy/Precision	Recovery 79.9-120.1% and <20.1% RPD.	Laboratory Control Sample (LCS/BS/BSD)
		Accuracy/Bias	Recovery 79.9-120.1%.	Reagent Blank Spike (RBS)
		Precision	<20.1% RPD when concentration of either sample is $\geq$ 5x the MDL (see Sections 16.4.4 and 16.4.3 or lab SOP for details)	Collated Samples (C1/C2)
Compendium Method IO-2.1		Precision	<10.1% RPD when concentration of the parent sample is $\geq$ 5x the MDL (see Section 16.4.5 of lab SOP for details)	Replicate Analyses (Analytical Duplicate)
		Accuracy/Bias	Recovery within 79.9% and 120.1% of the true value.	Matrix Spike / Matrix Spike Duplicate
		Precision	RPD < 20.1%	Matrix Spike / Matrix Spike Duplicate
		Precision	$\pm 10\%$ RPD of the undiluted sample if the parent sample concentration is $> 2.5x$ the MDL.	Serial Dilution (SRD)
		Accuracy/Bias	Recovery 74.9%-125.1%	Post Digestion Spike (PDS)
		Accuracy/Bias	Recovery within 20% of the expected value.	Interference Check Sample (ICS) using two different standards (ICSA and ICSAB)
		Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after verification

Notes:

Asbestos samples will be analyzed by EMSL Analytical, Inc. in Cinnaminson, New Jersey.

b Sampling SOPs are included in Appendix A, Attachment 1, and <a href="https://www.epa.gov/sites/default/files/2019-11/documents/mthd-2-1.pdf">https://www.epa.gov/sites/default/files/2019-11/documents/mthd-2-1.pdf</a>.

# **TABLE 5 (CONTINUED)**

# LABORATORY MEASUREMENT PERFORMANCE CRITERIA

- c Analytical SOPs are included in Attachment 2.
- d Metals samples will be analyzed by Eastern Research Group, Inc. in Morrisville, North Carolina.

DOI	Data o	auality	indicator

QC Quality control

QL Quantitation limit

RPD Relative percent difference

SOP Standard operating procedure

TEM Transmission electron microscopy

mm<sup>2</sup> square millimeters

μm micrometers

MDL method detection limit

LRB lab reagent blank

MB method blank

BLK blank

LCS laboratory control sample

BS blank spike

BSD blank spike duplicate

RBS reagent blank spike

ICS Interference check sample

ICSA Interference check standard which contains known concentrations of the interference analytes of interest with no addition of target elements.

ICSAB Interference check standard which contains the same concentration of interference analytes in the ICSA plus a known concentration of reported analytes.

TABLE 6

ANALYTICAL INSTRUMENT CALIBRATION

Instrumenta	Calibration Procedure or Parameter	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA
Transmission electron	Alignment	Daily	Uniform and centered alignment	Adjustments	Laboratory Analyst
microscope (EMSL SOP ISO	Camera constant	Weekly	± 5%	Adjustments	Laboratory Analyst
10312 Ambient air – Determination	Magnification	Monthly	± 5%	Adjustments	Laboratory Analyst
of asbestos fibres – Direct-transfer	Beam dose	Quarterly	Fibril remains visible for 15 seconds	Adjustments	Laboratory Analyst
transmission electron	Detector resolution	Quarterly	< 175 eV	Adjustments	Laboratory Analyst
microscopy method [ASB-	K-factors	Semi-annually	Various sensitivity factors	Adjustments	Laboratory Analyst
SOP-417, Revision 10])	Spot size	Quarterly	< 250 nm	Adjustments	Laboratory Analyst
	Selected Area Electron Diffraction	As needed	80% accuracy	Adjustments or replacements	Laboratory Analyst
	Al, Cu	Daily	± 0.02 keV	Adjustments	Laboratory Analyst
Inductively coupled plasma / mass spectrometry (ICP/MS) (SOP ERG-MOR-095, For Quartz Fiber Filters)	Calibration curve with at least blank and five standard solutions. A minimum of three replicate integrations are required.	Daily	Curve must have correlation coefficient of at least 0.995; and deviation from the initial calibration verification: metals 90-110%. Must also be ±30% or ±10% of the nominal concentrations of the standards. RSDs<10.1% for all non-zero calibration standards for Tier I elements. All other elements may be reported with RSDs > 10% with qualification.	Inspect the system for problems, clean the system, verify operating conditions, and take corrective actions to achieve the technical acceptance criteria.	Laboratory Analyst

# **TABLE 6 (CONTINUED)**

# ANALYTICAL INSTRUMENT CALIBRATION

Instrument <sup>a</sup>	Calibration Procedure or Parameter	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA
	Initial Calibration Verification (ICV)	Daily	Recovery 89.9 – 110.1% non-Tier I elements	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	Laboratory Analyst
Inductively coupled plasma / mass spectrometry (ICP/MS)	Initial Calibration Blank (ICB)	Daily	Absolute value must be <s*k< td=""><td>Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.</td><td>Laboratory Analyst</td></s*k<>	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	Laboratory Analyst
(SOP ERG-MOR- 095, For Quartz Fiber Filters) (continued)	High Calibration Verification (HCV)	Daily	Recovery 94.9-105.1%, non-Tier 1	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	Laboratory Analyst
	Calibration Range Check	Daily	Reporting elements for unknown / field samples within range of highest calibration standard (or LDR if one is in use).	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	Laboratory Analyst

#### Notes:

Analytical SOPs are included in Attachment 2.

nm Nanometers

RRF Relative response factor
RSD Relative standard deviation
S/N Signal-to-noise ratio

SOP Standard operating procedure

LDR Linear dynamic range

<s\*K less than the standard deviation times a multiplier for a tolerance limit based on the 99th percentile for n-1 degrees of freedom

STD Standard

KED Kinetic energy discrimination

eV Electron volts keV kiloelectron volt

TABLE 7

ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION

Instrument/	Maintenance			Acceptance		Responsible
Equipment	Activity	Inspection Activity	Frequency	Criteria	<b>Corrective Action (CA)</b>	Person
Transmission electron microscope (EMSL SOP ISO 10312 Ambient air – Determination of asbestos fibres – Direct-transfer transmission electron microscopy method [ASB-	Daily check, calibration verification	Check screen magnification at 20,000x Check film magnification at 20,000x Verify spot size <250 nm	As required by SOP	As required by SOP	As required by SOP	Laboratory Analyst
SOP-417, Revision 10]) Inductively coupled plasma / mass spectrometry (ICP/MS) (SOP ERG-MOR-095, For Quartz Fiber Filters)	Daily check, calibration verification	Nebulizer, injection tube, plasma optimization, gas supply, and detector	Daily before use	As required by SOP	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	Laboratory Analyst

# Notes:

<sup>a</sup> Analytical SOPs are included in Attachment 2.

SOP Standard operating procedure

nm nanometers

# **APPENDIX A**

**TETRA TECH SOPS** 

#### **SOP APPROVAL FORM**

## TETRA TECH, INC.

#### EMI OPERATING UNIT

## ENVIRONMENTAL STANDARD OPERATING PROCEDURE

## PACKAGING AND SHIPPING SAMPLES

**SOP NO. 019** 

**REVISION NO. 9** 

Last Reviewed: November 2023

Carlo Jamilo	
The second second	November 2023
Quality Assurance Approved	Date

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 1 of 20

Title: Packaging and Shipping Samples Last Reviewed: November 2023

#### 1.0 BACKGROUND

In any sampling program, the integrity of a sample must be ensured from its point of collection to its final disposition. This standard operating procedure (SOP) describes procedures for packaging and shipping samples. Steps in the procedures should be followed to ensure sample integrity and to protect the welfare of persons involved in shipping and receiving samples.

#### 1.1 PURPOSE

This SOP establishes the requirements and procedures for packaging and shipping nonhazardous environmental samples. It has been prepared in accordance with the U.S. Environmental Protection Agency (EPA) "Contract Laboratory Program Guidance for Field Samplers." Procedures described in this SOP should be followed for all routine sample packaging and shipping of nonhazardous samples. If procedures are to be modified for particular contract- or laboratory-specific requirements, modified procedures should be clearly described in site-specific plans such as work plans, field sampling plans (FSP), or quality assurance project plans (QAPP). Deviations from the procedures in this SOP must be documented in a field logbook. This SOP assumes that samples are already in the appropriate sample jars and that the sample jars are labeled.

#### This SOP does not cover the packaging and shipment of Dangerous Goods or Hazardous Materials.

The shipment of Dangerous Goods (by air) and Hazardous Materials (by ground) requires specialized training. If you have NOT received this training in the last 2 years, you are NOT qualified to package or ship these materials and may be personally liable for any damages or fines. Contact one of Tetra Tech's shipping experts for assistance. Instructions to access the training course, shipping experts, and health and safety (H&S) contacts, and general information on packaging and shipping hazardous substances and dangerous goods can be obtained by checking the links provided in Section 1.4 (References) and communicating with appropriate Tetra Tech H&S contacts listed on the EMI Operating unit internal H&S web site.

#### 1.2 SCOPE

This SOP applies to packaging and shipping of environmental and nonhazardous samples. This SOP does not address shipping dangerous goods or hazardous materials.

#### 1.3 **DEFINITIONS**

**Airbill:** An airbill is a shipping form (such as a FedEx shipping form) acquired from the commercial shipper and is used to document shipment of the samples from the sampler to the designated analytical laboratory (see Figure 1).

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 2 of 20

Title: **Packaging and Shipping Samples** Last Reviewed: November 2023

**Blank:** A blank is any sample that is used to assess cross-contamination from sampling and sample management procedures. A typical blank sample will consist of distilled or deionized (DI) water (water sampling) or an air filter cartridge (air sampling) that is then analyzed by the laboratory to evaluate whether cross-contamination has been introduced. Each blank is assigned its own unique sample number. Blanks collected in the field include trip blanks, field blanks, and equipment blanks, all intended to assess potential cross-contamination. For example, a trip blank checks for contamination during sample handling, storage, and shipment from the field to the laboratory. Field blanks assess the contamination of water or soil from ambient air. Equipment blanks (also known as rinse blanks) assess contamination from incomplete decontamination procedures.

**Chain-of-Custody form:** A chain-of-custody form is used to document the transfer of custody of samples from the field to the designated analytical laboratory (see <u>Figure 2</u>). The chain-of-custody form is critical to the chain-of-custody process and is used to identify the samples in each shipping container to be shipped or delivered to the laboratory for chemical or physical (geotechnical) analysis. A copy of the chain-of-custody form is shipped with the samples and accompanies them from sampler to laboratory (see Figure 3).

**Custody seal:** A custody seal is a tape-like seal and is used to indicate that samples are intact and have not been disturbed during shipping or transport after the samples have been released from the sampler to the shipper (see <u>Figure 4</u>). The custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they have been packaged for shipping (see <u>Figure 5</u>).

**Environmental samples:** Environmental samples include drinking water, groundwater, surface water, soil, sediment, treated municipal and industrial wastewater effluent, indoor and ambient air, nonhazardous bulk materials, soil gas, dust, asbestos, and biological specimens. Environmental samples typically contain low concentrations of contaminants and, when handled, require only limited precautionary procedures.

**Nonhazardous samples:** Nonhazardous samples are those samples that do not meet the definition of a hazardous sample AND do not need to be packaged and shipped in accordance with the International Air Travel Association's (IATA) "Dangerous Goods Regulations" (DGR) or U.S. Department of Transportation's "Hazardous Materials Regulations" defined in Title 49 *Code of Federal Regulations* (CFR).

The following definitions are provided to further distinguish environmental and nonhazardous samples from dangerous goods and hazardous samples:

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 3 of 20 Last Reviewed: November 2023

**Packaging and Shipping Samples** 

**Dangerous goods:** Dangerous goods are articles or substances that can pose a significant risk to health, safety, or property when transported by air; they are classified as defined in Section 3 of the DGR (IATA 2020).

Hazardous samples: Hazardous samples include dangerous goods and hazardous substances. Hazardous samples shipped by air should be packaged and labeled in accordance with procedures specified by the DGR; ground shipments should be packaged and labeled in accordance with the Hazardous Material Regulations.

Hazardous substance: A hazardous substance is any material, including its mixtures and solutions, that is listed in 49 CFR 172.101 and its quantity, in one package, equals or exceeds the reportable quantity listed in Table 1 to Appendix A of 49 CFR 172.101.

#### 1.4 **REFERENCES**

- General Awareness, H&S Contacts, and Course Training Information (Tetra Tech, Inc., EMI Operating Unit. Intranet) On-line address: Dangerous Goods Shipping (sharepoint.com)
- International Air Transport Association (IATA). 2024. "Dangerous Goods Regulations. 2024." For sale at: https://www.iata.org/en/publications/dgr/. Updated annually, with new edition available late in year. 2024 Dangerous Goods Regulations (DGR), 65th Edition.
- U.S. Environmental Protection Agency (EPA). 40 CFR, 763 Subpart F, Asbestos Hazards Emergency Response Act (AHERA). On-line address: <a href="https://www.ecfr.gov/current/title-40/chapter-">https://www.ecfr.gov/current/title-40/chapter-</a> I/subchapter-R/part-763?toc=1
- EPA. 2014. "Contract Laboratory Program Guidance for Field Samplers." EPA 540-R-014-013. October. On-line address: https://www.epa.gov/sites/production/files/2015-03/documents/samplers guide.pdf.
- EPA. 2023. "Packing, Marking, Labeling and Shipping of Environmental and Waste Samples." EPA Region 4, LSASDPROC-209-R5. April . On-line address: https://www.epa.gov/sites/production/files/2015-06/documents/Shipping-Environmental-and-Waste-Samples.pdf

#### 1.5 REQUIREMENTS AND RESOURCES

The procedures for packaging and shipping samples require the following:

- Coolers (insulated ice chest) or other shipping containers appropriate to sample type
- Bubble wrap or similar cushioning material
- Chain-of-custody forms and seals
- Airbills

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019

Page 4 of 20

Title: **Packaging and Shipping Samples**Last Reviewed: November 2023

- Resealable plastic bags for sample jars and ice
- Tape (strapping and clear)
- Large plastic garbage bags for lining the cooler
- Temperature blank sample bottle filled with distilled water can be included in the cooler if appropriate to sample type

Trip blank samples used to check for volatile contamination during sample handling in the field should accompany sample containers during shipment from laboratory to field (empty containers) and from field to laboratory (filled containers). It should remain in the cooler with sample containers during the sampling event. Trip blanks should be requested from the laboratory when containers are initially ordered.

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 5 of 20

#### 2.0 PROCEDURES

The following procedures apply to packaging and shipping nonhazardous and environmental samples.

#### 2.1 PACKAGING SAMPLES

After they have been appropriately containerized and labeled, environmental samples should be packaged as described in this section. This section covers procedures for packing samples for delivery by commercial carrier (air or ground) and hand delivery of environmental samples (by employee or courier), as well as shipping asbestos and air quality samples. Note that these instructions are general; samplers also should be aware of client-specific requirements concerning the placement of custody seals or other packaging provisions.

#### 2.1.1 Packaging Samples for Delivery by Commercial Carrier (Air or Ground)

Samples shipped by commercial carriers should be packed for shipment using the following procedures and in compliance with all carrier requirements:

#### Preparing the sample:

- 1. Allow a small amount of headspace in all bottles, or as instructed by the laboratory (except volatile organic compound [VOC] containers with a septum seal) to compensate for any changes in pressure and temperature during transfer.
- 2. Be sure the lids on all bottles are tight (will not leak). Lids maybe taped or sealed with custody seals as added protection or as required. For any sample containers that are not marked with a tare weight by the laboratory, cover the completed sample label on the container with clear tape to protect the label.
- 3. Place sample containers in resealable plastic bags.

#### Preparing the cooler:

- 1. Secure and tape the drain plug of the cooler with fiber or duct tape.
- 2. Line the cooler with a large plastic garbage bag before samples, ice, and absorbent packing material are placed in the cooler.
- 3. Wrap the sample containers in bubble wrap or line the cooler (bottom and sides) with a cushioning material to prevent breakage of bottles or jars during shipment.
- 4. If required by the laboratory for the analytical method, add a sufficient quantity of ice to the cooler to cool samples to 4 °C (± 2 °C). Ice should be double bagged in resealable plastic bags to prevent the melted ice from leaking out. If required, include one temperature blank (a sample bottle filled with distilled water) per cooler.

- 5. For VOC samples only, include one trip blank for VOC analysis per shipment matrix in each cooler.
- 6. Fill all remaining space between the bottles or jars with bubble wrap.
- 7. As each container is placed in the cooler, verify the sample information on the chain-of-custody form. The samples listed on the chain-of-custody form must match exactly with the contents of the cooler.
- 8. Securely fasten the top of the large garbage bag with tape (preferably plastic electrical tape).
- 9. If more than one cooler is being shipped, mark each cooler as "1 of 2," "2 of 2," and so forth.
- 10. Place the chain-of-custody forms (see Figure 2) into a resealable plastic bag, and tape the bag to the inner side of the cooler lid (see Figure 3). If you are shipping more than one cooler, copy the chain-of-custody form so that there is one copy of all forms in each cooler. The samples listed on the chain-of-custody form must match exactly with the contents of the cooler. Tape any instructions for returning the cooler to the inside of the lid.
- 11. Close the lid of the cooler and tape it shut by wrapping strapping tape around both ends and hinges of the cooler at least once.
- 12. Place two signed custody seals (see Figure 4) on opposite sides of the cooler, ensuring that each one covers the cooler lid and side of the cooler (see Figure 5; note that in contrast to the figure, the seals should be placed on the opposite sides of the cooler and offset from each other, rather than directly across from each other as shown in Figure 5). Place clear plastic tape over the custody seals so that the cooler cannot be opened without breaking the seal.
- 13. Shipping containers should be marked "THIS END UP." Arrow labels, which indicate the proper upward position of the container, may also be affixed to the container. As appropriate, the containers should also be labeled for Saturday delivery or other special requirements.
- 14. Ship samples overnight using a commercial carrier such as FedEx. As a best practice, electronic sample shipping labels should be prepared by the shipping agency's employees, at the direction of Tetra Tech employees or sampling personnel. This allows the sampling personnel to confirm special shipping requirements, such as Saturday delivery, and verify that samples will be shipped that day (that is, the last shipment of the day has not already occurred). If this is not possible, the airbill can be prepared by hand (see Figure 1), but samples should still be handed over directly to shipping agency employees and shipping details should be verified. The shipping label should be placed on the outside of the container.
- 15. A copy of the receipt with sample tracking number should be retained by the sampling personnel and delivery should be verified the next day.

#### 2.1.2 Hand Delivery of Environmental Samples (by Employee or Courier)

Samples hand-delivered to the laboratory should be packed for shipment using the following procedures:

Preparing the sample:

1. Bottles can be filled completely with sample (required for VOC containers with a septum seal).

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019

Page 7 of 20

Title: **Packaging and Shipping Samples**Last Reviewed: November 2023

2. Be sure the lids on all bottles are tight (will not leak).

#### Preparing the cooler:

1. Secure and tape the drain plug of the cooler with fiber or duct tape.

- 2. Wrap the sample containers in bubble wrap or line the cooler (bottom and sides) with a cushioning material to prevent breakage of bottles or jars during shipment.
- 3. As each container is placed in the cooler, verify the sample information on the chain-of-custody form. The samples listed on the chain-of-custody form must match exactly with the contents of the cooler.
- 4. If required for by the laboratory for the analytical method, add a sufficient quantity of ice to the cooler to cool samples to 4 °C. Ice should be double bagged in resealable plastic bags to prevent the melted ice from leaking out. If required, include one temperature blank (a sample bottle filled with distilled water) per cooler.
- 5. For VOC samples only, include one trip blank for VOC analysis per shipment matrix in each cooler.
- 6. If more than one cooler is being shipped, mark each cooler as "1 of 2," "2 of 2," and so forth.
- 7. Place the chain-of-custody form (see Figure 2) in a resealable plastic bag and tape to the inside of the cooler lid (see Figure 3), close the lid, and seal with custody seals (see Figure 5; note that in contrast to the figure, the seals should be placed on the opposite sides of the cooler and offset from each other, rather than directly across from each other as shown in Figure 5). Place clear plastic tape over the custody seals so that the cooler cannot be opened without breaking the seal. Transfer the cooler to the courier. When samples will be delivered directly to the laboratory, it is sufficient to close the cooler and hand-deliver it with the chain-of-custody form.
- 8. Include any instructions for returning the cooler to the inside of the lid.
- 9. If the cooler is being transferred to a courier, the shipping containers should be marked "THIS END UP," and arrow labels, which indicate the proper upward position of the container should be affixed to the container.

#### 2.1.3 Shipping Asbestos Samples

Asbestos samples shipped by commercial carriers should be packed for shipment using the following procedures and in compliance with all carrier requirements:

- 1. Place each asbestos sample in a small resealable plastic bag or Whirl-pak sealable bag. Seal the bags carefully and place the sample bags in a larger resealable plastic bag.
- 2. Select a rigid shipping container and pack the samples upright in a noncontaminating, nonfibrous medium such as a bubble pack to minimize excessive movement during shipping.
- 3. Avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials because of possible contamination.

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 8 of 20 Last Reviewed: November 2023

## Title: Packaging and Shipping Samples

4. Affix custody seals to the samples or outer sample bag so that the bags cannot be opened without breaking the seal.

- 5. Insert the chain-of-custody form in the box. Include a shipping bill and a detailed listing of samples shipped, their descriptions and all identifying numbers or marks, sampling data, shipper's name, and contact information.
- 6. Ship bulk samples in a separate container from air samples. Bulk samples and air samples delivered to the analytical laboratory in the same container will be rejected.
- 7. For each sample set, designate which are the ambient samples, which are the abatement area samples, which are the field blanks, and which is the sealed blank if sequential analysis is to be performed.
- 8. Hand-carry samples to the laboratory in an upright position if possible; otherwise, choose that mode of transportation least likely to shake the samples in transit.
- 9. Address the package to the laboratory sample coordinator by name when known and alert him or her of the package description, shipment mode, and anticipated arrival as part of the chain-ofcustody and sample tracking procedures. This information will also help the laboratory schedule timely analysis for the samples when they are received.

#### 2.1.4 **Shipping Air Samples**

Packaging and shipping requirements for air samples vary depending on the media used to collect the samples and the analyses required. Sampling media typically include Summa canisters and Tedlar bags for whole air samples, filters for metals and particulate matter, and sorbent tubes for organic contaminants. This section of the SOP provides general guidelines for packaging and shipping air samples collected using these media. The project FSP or QAPP should also be reviewed for any additional project-specific requirements or instructions.

#### **Summa Canister Samples**

- 1. Close the canister valve by tightening the knob clockwise or flipping the toggle switch. Replace the brass cap on the canister inlet.
- 2. If a flow controller was used to collect the air sample over a specified time interval, the flow controller should be removed before replacing the brass cap.
- 3. Fill out the sample tag on the canister with the sample number and the date and time of collection. Include the identification number of the flow controller on the sample tag if one was used. Make sure the information on the sample tag matches the chain-of-custody form.
- 4. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final Summa canister vacuum readings; Summa canister identification number; and flow controller identification number.

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 9 of 20 Last Reviewed: November 2023

#### Title: Packaging and Shipping Samples

5. Package the Summa canister (and flow controller) in its original shipping box with the original packaging material. Tape the box shut and apply custody seals if required. Note: Summa canisters should never be packaged with ice.

- 6. Summa canister shipments typically include several canisters, and may include more than one shipping box. The chain-of-custody form for the shipment should be sealed within one of the shipping boxes. If more than one box is being shipped, mark each box as "1 of 2," "2 of 2," and so forth.
- 7. Ship the samples by a method that will meet the holding time. Summa canister samples should be analyzed within 30 days of sample collection.

#### **Tedlar Bag Samples**

- 1. Before removing it from the sample port, close the Tedlar bag by tightening the valve clockwise. The bag should only be approximately half-full to allow for pressure changes during shipping and handling of the sample. Keep the Tedlar bag out of direct sunlight to preserve the sample.
- 2. Fill out the label on the bag with the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.
- 3. Complete the chain-of-custody form.
- 4. Package the Tedlar bag in a shipping box with appropriate packing material to prevent the bag from being punctured or damaged. Multiple bags can be packaged in the same box. Tape the box shut and apply custody seals if required. Note: Tedlar bag samples should not be cooled or packaged with ice, although they can be shipped in an ice chest to protect the samples.
- 5. Tedlar bag shipments may include more than one shipping box. The chain-of-custody form for the shipment should be sealed within one of the shipping boxes. If more than one box is being shipped, mark each box as "1 of 2," "2 of 2," and so forth.
- 6. Ship the samples using priority overnight delivery. Tedlar bag samples should be analyzed within 3 days of sample collection.

#### **Filter Cassette Samples**

- 1. Disconnect the filter cassette from the air sampling pump and replace the plastic caps on the inlet and outlet openings.
- 2. Attach a label to the sample that includes the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.
- 3. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final air flow rates (or average flow rate); volume of air sampled; and sampling pump identification number.
- 4. Package the filter cassettes in a shipping box (such as a FedEx box). Use an appropriate packing material (such as bubble wrap) to separate the samples and prevent damage.
- 5. Place the chain-of-custody form within the box, seal the box, and apply custody seals if required. Filter cassette samples typically do not need to be cooled, but check the FSP or QAPP for projectspecific requirements.

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019

Page 10 of 20

Title: **Packaging and Shipping Samples**Last Reviewed: November 2023

6. Ship the samples by a method that will meet the holding time.

#### **Sorbent Tube Samples**

1. Disconnect the sample tube from the air sampling pump and seal both ends of the tube with plastic caps.

- 2. Complete a sample label that includes the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.
- 3. If the tube is small and the label cannot be attached to the tube, the tube can be placed in a small resealable plastic bag and the label can be attached to the bag or placed inside the bag with the tube.
- 4. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final air flow rates (or average flow rate); volume of air sampled; and sampling pump identification number.
- 5. Packaging requirements for the sample tubes will depend on the analysis required, and the sampler should check the FSP or QAPP for project-specific requirements (for example, tubes may need to be wrapped in aluminum foil to prevent exposure to light). Packaging containers and methods include (1) shipping boxes (as described under filter cassette samples), (2) small sample coolers filled with double-bagged ice, and (3) small sample coolers filled with blue (reusable) ice.
- 6. Place the chain-of-custody form within the box or container, seal the box or container, and apply a custody seal if required.
- 7. If coolers are used for shipping, tape instructions for returning the cooler to the inside of the lid.
- 8. Ship the samples by a method that will meet the holding time.

#### Polyurethane Foam (PUF) Tube Samples

- 1. Disconnect the PUF tube from the air sampling pump and wrap the tube in aluminum foil.
- 2. Attach a label to the wrapped sample tube that includes the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.
- 3. Wrap the PUF tube in bubble wrap and place the tube in a glass shipping jar.
- 4. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final air flow rates (or average flow rate); volume of air sampled; and sampling pump identification number.
- 5. Package the PUF tube jars in a cooler that is filled with double-bagged ice. Use bubble wrap or other cushioning material to separate the samples and prevent breakage.
- 6. Place the chain-of-custody form within the cooler, seal the cooler, and apply a custody seal if required.
- 7. If coolers are used for shipping, tape instructions for returning the cooler to the inside of the lid.
- 8. Ship the samples by a method that will meet the holding time. Samples collected in PUF tubes typically must be extracted within 7 days of collection.

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 11 of 20

Title: Packaging and Shipping Samples Last Reviewed: November 2023

#### 2.2 SHIPPING DOCUMENTATION FOR SAMPLES

Airbills, chain-of-custody forms, and custody seals must be completed for each shipment of nonhazardous environmental samples.

Field staff collecting samples should also review their field work plans to confirm what documentation must be completed during each sampling event, including client-specific requirements. For example, some EPA programs have a specific requirement to use Scribe software, an environmental data management system, to create sample documentation, electronically input information into Traffic Report or chain-of-custody forms, and enter other data.

- The Scribe software can be accessed from the EPA Environmental Response Team (ERT) at the following address: <a href="https://www.epa.gov/ert/ert-information-management">https://www.epa.gov/ert/ert-information-management</a>
- The ERT User Manual for Scribe, reference, and training materials can be accessed from the Scribe Support Web site at the following address: <a href="https://epaosc.org/site/site\_profile.aspx?site\_id=ScribeGIS">https://epaosc.org/site/site\_profile.aspx?site\_id=ScribeGIS</a>

Note that some laboratories must routinely return sample shipping coolers within 14 calendar days after the shipment has been received. Therefore, the sampler should also include instructions for returning the cooler with each shipment, when possible. The sampler (not the laboratory) is responsible for paying for return of the cooler and should include shipping airbills bearing the sampler's shipping account number, as well as a return address to allow for return of the cooler. Samplers should use the least expensive option possible for returning coolers.

#### 2.3 SHIPMENT DELIVERY AND NOTIFICATION

A member of the field sampling team must contact the laboratory to confirm it accepts deliveries on any given day, especially Saturdays. In addition, samplers should ensure the laboratory has been notified in advance of the pending shipment and notify any additional parties as required. The sampler needs to know the laboratory's contact name, address, and telephone number and be aware of the laboratory's requirements for receiving samples.

In addition, samplers should be aware of the sample holding times, shipping company's hours of operation, shipping schedule, and pick-up and drop-off requirements to avoid delays in analytical testing.

Priority Overnight Delivery

Priority overnight delivery is typically the best method for shipment. Delays caused by longer shipment times may cause the sample temperature to rise above the acceptable range of  $4^{\circ}$  C ( $\pm$  2  $^{\circ}$  C) and technical holding time may expire, which in turn may compromise sample integrity and require recollection of

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 12 of 20

requirements, the procedures should be clearly described in site-specific plans such as work plans, FSPs, or QAPPs.

#### Saturday Delivery

If planning to ship samples for Saturday delivery, the laboratory must be contacted in advance to confirm it will accept deliveries on Saturdays or arrange for them to be accepted. In addition, samplers should ensure the laboratory has been notified in advance of the pending shipment and notify any additional parties as required.

#### 2.4 **HEALTH AND SAFETY CONSIDERATIONS**

In addition to the procedures outlined in this SOP, all field staff must be aware of and follow the health and safety practices that result from the Activity Hazard Analyses (AHA) for the project. The AHAs include critical safety procedures, required controls, and minimum personal protective equipment necessary to address potential hazards. The hazards specific to project tasks must be identified and controlled to the extent practicable and communicated to all project personnel via the approved, projectspecific health and safety plan (HASP).

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 13 of 20

Title: Packaging and Shipping Samples Last Reviewed: November 2023

#### 3.0 POTENTIAL PROBLEMS

The following potential problems may occur during sample shipment:

• Leaking package. If a package leaks (either from broken sample containers or melting ice), the carrier may open the package and return the package. Special care should be taken during sample packaging to minimize potential leaks.

- Improper labeling and marking of package. If mistakes are made in labeling and marking the package, the carrier will most likely notice the mistakes and return the package to the shipper, thus delaying sample shipment. A good practice is to have labels, forms, and container markings double checked by a member of the field team.
- Bulk samples and air samples delivered to the analytical laboratory in the same container. If samples are combined in this way, they will be rejected. Always ship bulk samples in separate containers from air samples.
- Issues in packing asbestos samples. When asbestos samples are shipped, avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials with asbestos samples because of possible contamination.
- Improper, misspelled, or missing information on the shipper's declaration. The carrier will most likely notice these errors as well and return the package to the shipper. A good practice is to have another field team member double check this information.
- Missed drop off time or wrong location. Missing the drop off time or having the wrong location
  identified for drop off will delay delivery to the laboratory and may cause technical holding times
  to expire. Establish the time requirements in advance of completing the field effort and be sure
  and provide some contingency time for potential delays such as traffic or checking and redoing
  paperwork.
- Incorrectly packaging samples for analysis at multiple laboratories. For example, inorganic samples may be shipped to one laboratory for analysis, while organic samples may need to be shipped to another laboratory. All field staff should be aware which samples are to be shipped to which laboratory when they package samples for multiple types of analysis.
- Holidays or weather-related delays. Be aware of holidays and weather forecasts that could cause delays in delivery. Delays caused by longer shipping times may cause technical holding times to expire, which in turn may compromise sample integrity or require recollection of samples.
- Not noting field variances in field logbook. Field variances should be noted in the field logbook and the project manager notified. Common field variances include:
  - Less sample volume collected than planned. Notify appropriate staff and the laboratory to ensure there is an adequate amount for analysis.
  - Sample collected into incorrect jar because of broken or missing bottle-ware. Notify appropriate laboratory staff to ensure there is no confusion regarding the analysis of the sample.

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 14 of 20 Last Reviewed: November 2023

**Packaging and Shipping Samples** Title:

#### FIGURE 1

#### EXAMPLE OF A FEDEX US AIRBILL FOR LOW-LEVEL ENVIRONMENTAL SAMPLES



#### Filling Out the FedEx US Airbill

- The sender *must complete* the following fields on the pre-printed airbill:
  - Section 1: Date
  - Section 1: Sender's FedEx Account Number (available from your office administrator)
  - Section 1: Sender's Name, Company, Address, and Phone Number
  - Section 2: Internal Billing Reference (Project Number) (this field may not be present on newer airbills)
  - Section 3: Recipient's Name, Company, Address, and Phone Number
  - Section 4: Express Package or Freight Services (Priority Overnight)
  - Section 5: Packaging (usually "Other," your own packaging)
  - Section 6: Special Handling (Saturday delivery if prearranged with receiving laboratory; "No" dangerous goods contained in shipment)
  - Section 7: Payment ("Bill to Sender")
  - Section 7: Total Number of Packages
  - Section 7: Total Weight (completed by FedEx employee)
  - Section 8: Delivery Signature Options ("No Signature Required")

#### Completing a Sample Chain-of-Custody Form (See Also Section 2.2 on SCRIBE for Forms)

After samples have been collected, they will be maintained under chain-of-custody procedures. These procedures are used to document the transfer of custody of the samples from the field to the designated analytical laboratory. The same chain-of-custody procedures will be used for the transfer of samples from one laboratory to another, if required.

Page 15 of 20

The field sampling personnel will complete a Chain-of-Custody and Request for Analysis (CC/RA) form for each separate container of samples to be shipped or delivered to the laboratory for chemical or physical (geotechnical) analysis. These forms are often triplicate, carbonless forms. Care should be taken when completing the form that all copies are legible—PRESS FIRMLY WHEN WRITING. Information on the form will include:

- 1. Project identification (ID) (for example, contract and task order number);
- 2. Project Contract Task Order (CTO) number;
- 3. Laboratory Project Order (PO) number;
- 4. Tetra Tech Technical Contact;
- 5. Tetra Tech Project Manager;
- 6. Laboratory name;
- 7. Field sampler names;
- 8. Field sampler signature;
- 9. Sample ID;
- 10. Date and time of sampling;
- 11. Sample matrix type;
- 12. Sample preservation method; note "NONE" if no preservatives;
- 13. Number and types of containers per sample;
- 14. Sample hazards (if any);
- 15. Requested analysis;
- 16. Requested sample turnaround time or any special remarks (for example, possible presence of free product or high screening concentrations);
- 17. Page \_\_ of \_\_;
- 18. Method of shipment;
- 19. Carrier/waybill number (if any);
- 20. Signature, name, and company of the person relinquishing the samples and the person receiving the samples when custody is transferred;

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 16 of 20 Last Reviewed: November 2023

**Packaging and Shipping Samples** 

21. Date and time of sample custody transfer;

22. Condition of samples when they are received by the laboratory.

The sample collector will cross out any blank space on the CC/RA form below the last sample number listed on the part of the form where samples are listed. If more than one CC/RA form is used for the samples within one shipping container, all of the forms must have completed header information and original signatures on each page.

The sampling personnel whose signature appears on the CC/RA form is responsible for the custody of a sample from time the sample is collected until the custody of the sample is transferred to a designated laboratory, a courier, or to another Tetra Tech employee for transporting a sample to the designated laboratory. A sample is considered to be in custody when the custodian: (1) has direct possession of it; (2) has plain view of it; or (3) has securely locked it in a restricted access area.

Custody is transferred when both parties to the transfer complete the portion of the CC/RA form under "Relinquished by" and "Received by" or a sample is left at a FedEx facility pending shipment. Signatures, printed names, company names, and date and time of custody transfer are required. When custody is transferred, the Tetra Tech sampling personnel who relinquished the samples will retain the third sheet (pink copy) of the CC/RA form. When the samples are shipped by a common carrier, a Bill of Lading supplied by the carrier will be used to document the sample custody, and its identification number will be entered on the CC/RA form. Receipts of Bills of Lading will be retained as part of the permanent documentation in the Tetra Tech project file.

Last Reviewed: October 2023

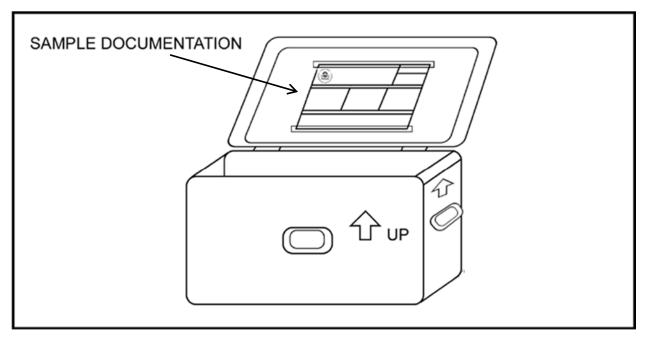
FIGURE 2 **EXAMPLE OF A CHAIN-OF-CUSTODY FORM (WHITE COPY)** 

	Tetra Tech EM Inc. Oakland Office  1999 Harrison Street, Suite 500	Ch	ain of	Custo	dy Reco	ord	ΙN	10.	98	14		13	G	Į 7	vative	Page —	l of	
	Oakland, CA 94612	Lab PO#:	Lab:								Page of Preservative Added							
	510.302.6300 Phone 510.433.0830 Fax	130AK27	XAMS				No./Container Types											
	Project name: DA PN	Tremi technical contact: Sava Woolley	Field samples Sand Rebe	٩														
	Project (CTO) number: 1036 11 590 29	Steve Del Honene	Field samplers' signatures:				0 0					ırgeables						
	Sample ID	Point ID/Depth	Date	Time	Matrix 2	40 ml VOA	1 liter	Sleeve	Glass Jar 250 ml Pol	Encore	VOA	Pest	Metals TPH Pur	TPH E				
123456	0295RE 55ØI 0293RE 55ØZ 029PGC 3D55ØI 029C3D55ØZ 029C3D55Ø3 029C3D55Ø3		7/22/13	1245	55.1				2 1 1 1 1		,	κ κ χ	× × × × ×	× × × × × × × × × ×				
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	Received by: Pelitie C	la s	Ceci	(a)	haver	Ī	3	MI	DO				7	13	1/30	13	09	30
	Relinquished by: Received by:					+									,			
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Turnaround time/remarks: Standard TAT  Temp-2  Riovitize: SVO(5, TPH-E on 029C3D5501 >> BY thenmetals										2.0°C								
	Fed Ex#: 8612 4667 77	215																
		WHITE-LE	aboratory Copy Y	ELLOW-Samp	e Tracker PINK-Fi	le Co	ру											

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 18 of 20

Title: Packaging and Shipping Samples Last Reviewed: October 2023

## FIGURE 3 EXAMPLE OF A SAMPLE COOLER WITH ATTACHED DOCUMENTATION



Source: U.S. Environmental Protection Agency. 2014.

Place the necessary paperwork (chain-of-custody form, cooler return instructions, and associated paperwork) in the shipping cooler or acceptable container. All paperwork must be placed in a plastic bag or pouch and then secured to the underside of the shipping container lid.

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019

Title: **Packaging and Shipping Samples**Page 19 of 20

Last Reviewed: October 2023

## FIGURE 4

## **EXAMPLE OF A CUSTODY SEAL**

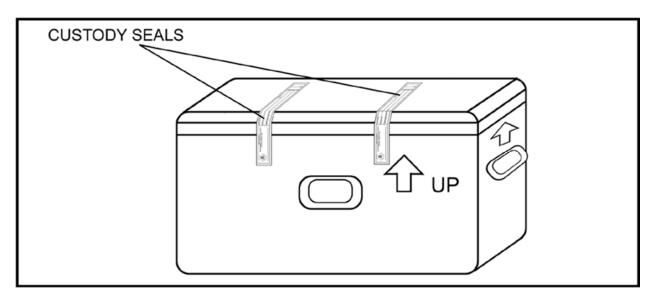
## **Custody Seal**

SIGNATURE

Tetra Tech, Inc. EMI Operating Unit - SOP No. 019 Page 20 of 20

Title: Packaging and Shipping Samples Last Reviewed: October 2023

## FIGURE 5 EXAMPLE OF SHIPPING COOLER WITH CUSTODY SEALS



Source: U.S. Environmental Protection Agency. 2014.

Please note that the two seals typically are affixed to opposite sides of the cooler and offset from each other, although the offset is not depicted on the EPA figure above.

## **SOP APPROVAL FORM**

## TETRA TECH, INC.

#### EMI OPERATING UNIT

#### ENVIRONMENTAL STANDARD OPERATING PROCEDURE

## RECORDING NOTES IN FIELD LOGBOOKS

**SOP NO. 024** 

**REVISION NO. 4** 

Last Reviewed: December 2022

(ada Jamik	December 2022
Quality Assurance Approved	Date

Tetra Tech, Inc. EMI Operating Unit - SOP No. 024 Page 1 of 8

Title: **Recording Notes in Field Logbooks**Last Reviewed: December 2022

#### 1.0 BACKGROUND

Complete and accurate field documentation is critical to a successful project and the field logbook is an important tool to support field documentation needs. The field logbook should include detailed records of all field activities, document interviews with people, and record observations of conditions at a site. Entries should be described in a level of detail to allow personnel to reconstruct, after the fact, activities and events that occurred during their field assignments. Furthermore, entries should be limited to facts. Avoid speculation related to field events and do not record hearsay or unfounded information that may be presented by other parties during field activities. For example, do not record theories regarding the presence or absence of contamination when you are collecting field screening data or speculation regarding the reasons for a property owner's refusal to grant access for sampling.

Field logbooks are considered accountable documents in enforcement proceedings and may be subject to review. Therefore, the entries in the logbook must be accurate and detailed but should not contain speculative information that could conflict with information presented in subsequent project deliverables and correspondence. Also be aware that the field logbooks for a site may be a primary source of information for depositions and other legal proceedings that may occur months or years after field work is complete and long after our memories have faded. The accuracy, neatness, and completeness of field logbooks are essential for recreating a meaningful account of events.

Field notes may also be recorded digitally, using a variety of software programs. The requirements and use of digital recording programs is not addressed in this standard operating procedure (SOP) because many items are unique to the selected software system. However, many of the principles discussed in this SOP will apply to the digital recording of field notes.

#### 1.1 PURPOSE

The purpose of this SOP is to provide guidance to ensure that field logbook documentation collected during field activities meets all requirements for its later use. Among other things, field logbooks may be used for:

- Identifying, locating, labeling, and tracking samples
- Recording site activities and the whereabouts of field personnel throughout the day
- Documenting any deviations from the project approach, work plans, quality assurance project plans, health and safety plans, sampling plans, and any changes in project personnel
- Recording arrival and departure times for field personnel each morning and evening and weather conditions each day

Tetra Tech, Inc. EMI Operating Unit - SOP No. 024

**Recording Notes in Field Logbooks** Title:

Page 2 of 8

Last Reviewed: December 2022

Describing photographs taken during the project.

In addition, the data recorded in the field logbook may later assist in the interpretation of analytical results. A complete and accurate logbook also aids in maintaining quality control, because it can verify adherence to project scope and requirements.

#### 1.2 **SCOPE**

This SOP establishes the general requirements and procedures for documenting site activities in the field logbook.

#### 1.3 **DEFINITIONS**

None.

#### 1.4 REFERENCES

Compton, R.R. 1985. Geology in the Field. John Wiley and Sons. New York, NY.

#### 1.5 REQUIREMENTS AND RESOURCES

The following items are required for field notation:

- Bound (sewn) notebooks
- Ballpoint pens or Sharpies with permanent waterproof ink
- 6-inch ruler (optional)

Field logbooks should be bound (sewn) with water-resistant and acid-proof covers, and each page should have preprinted lines or grids and numbered pages. They should be approximately  $7^{1}/_{2}$  by  $4^{1}/_{2}$  inches or  $8^{1}/_{2}$ by 11 inches in size. Loose-leaf sheets are not acceptable for use as a field logbook, although logs and field forms used to record field measurements and data are acceptable as loose-leaf sheets maintained in a threering binder with numbered pages, as a supplement to the logbook. If notes are written on loose paper, they must be transcribed as soon as possible into a bound field logbook by the same person who recorded the notes originally.

Ideally, distribution of logbooks should be controlled by a designated person in each office. This person assigns a document control number to each logbook and records the assignment of each logbook distributed (name of person, date distributed, and project number). The purpose of this procedure is to ensure the integrity of the logbook before its use in the field, and to document each logbook assigned to a project. In the event that more than one logbook is assigned to a project, this process will ensure that all logbooks are accounted for at project closeout.

#### 2.0 PROCEDURES

The following subsections provide general guidelines and formatting requirements for field logbooks, and detailed procedures for completing field logbooks.

#### 2.1 GENERAL GUIDELINES

- A separate field logbook must be maintained for each project. If a site consists of multiple subsites (or operable units), designate a separate field logbook for each subsite. Similarly, if multiple activities are occurring simultaneously requiring more than one task leader (for example, well installation, private well sampling, or geophysical survey), each task leader should maintain a separate field logbook to ensure that each activity is documented in sufficient detail.
- At larger sites, a general field log may be kept at the site trailer or designated field office to track site visitors, document daily safety meetings, and record overall site issues or occurrences.
- Data from multiple subsites may be entered into one logbook that contains only one type of information for special tasks, such as periodic well water-level measurements.
- All logbooks must be bound and contain consecutively numbered pages. If the pages are not prenumbered, the sequential page number should be written at the top of each page.
- No pages can be removed from the logbook for any purpose.
- All information must be entered using permanent, waterproof ink, either a traditional ballpoint
  pen or a permanent marker. Do not use pens with water-based ink (typically identified as
  rollerball or gel ink pens) because the ink may wash out if the paper gets wet. Pencils are not
  permissible for field notes because information can be erased. The entries should be written dark
  enough so that the logbook can be easily photocopied.
- Be sure that all entries are legible. Use print rather than cursive writing and keep the logbook pages free of dirt and moisture to the extent possible.
- Set apart critical information such as sample numbers by circling or drawing a box around the critical data.
- Do not enter information in the logbook that is not related to the project. The language used in the logbook should be factual and objective. Avoid speculation that could conflict with information presented in subsequent project deliverables and correspondence (see Section 1.0 above).
- Use military time, unless otherwise specified by the client. If a logbook entry is not related to a specific event, set it aside with the identification as a "NOTE."
- Include site sketches, as appropriate.
- Begin a new page for each day's notes.

- Include the date, project number, and location (if the project has multiple locations) at the top of each page.
- At the end of a day, draw a single diagonal line through any unused lines on the page, and sign at the bottom of the page. Note and implement any client-specific requirements (for example, some clients require each logbook page to be signed).
- Write notes on every line of the logbook. Do not skip any pages or parts of pages unless a day's activity ends in the middle of a page.
- If a line is left blank for some reason, cross it out (with a single line) and initial to prevent unauthorized entries.
- Cross out (with a single line) and initial any edits to the logbook entries. Note and implement any client-specific requirements (for example, some clients also require that edits be dated). Edits should only be made if the initial entry is illegible or erroneous. Do not make corrections for grammar or style.

#### 2.2 LOGBOOK FORMAT

The layout and organization of each field logbook should be consistent and generally follow the format guidelines presented below. Some clients or contracts may have specific formatting guidelines that differ somewhat from this SOP; review client requirements at the start of the project to help ensure any client-specific guidelines are integrated.

#### 2.2.1 Logbook Cover

Spaces are usually provided on the inside front cover (or the opening page in some logbooks) for the company name, address, contact names, and telephone numbers. If preprinted spaces for this information are not provided in the logbook, write the information on the first available page. Information to be included on the inside front cover or first page includes:

- Logbook document control number (assigned by issuer)
- "Book # of #" (determined by the project manager if there is more than one logbook for the project)
- Contract and task order numbers
- Name of the site and site location (city and state)
- Name of subsite (or operable unit), if applicable
- Type of activity, if the logbook is for a specific activity, such as well installation or indoor air sampling
- Beginning and ending dates of activities entered into the logbook (that is, dates of the activities performed or overseen on site)

Tetra Tech, Inc. EMI Operating Unit - SOP No. 024 Page 5 of 8

#### 2.2.2 Inside Cover or First Page

Spaces are usually provided on the inside front cover (or the opening page in some logbooks) for the company name, address, contact names, and telephone numbers. If preprinted spaces for this information are not provided in the logbook, write the information on the first available page. Information to be included on the inside front cover or first page includes:

- Tetra Tech project manager and site manager names and telephone numbers
- Tetra Tech office address
- Client contact and telephone number
- Site safety officer and telephone number
- Emergency contact telephone number (911, if applicable, or nearest hospital)
- Subcontractor contacts and telephone numbers
- Site property owner or property manager contact information

Note—some clients prohibit the inclusion of personally identifiable information such as personal mobile telephone numbers on official project records.

#### 2.3 ENTERING INFORMATION IN THE LOGBOOK

The following lists provide guidance on the types of information to be included in a typical field logbook. This guidance is general and is not intended to be all-inclusive. Certain projects or clients may specify logbook requirements that are beyond the elements presented in this SOP; as appropriate, any client- or program-specific guidance regarding logbook notation requirements can be referenced in the logbook.

#### 2.3.1 General Daily Entries

- Document what time field personnel depart the Tetra Tech office and arrive at the hotel or site. If permitted by the client to charge travel time for site work, document what time personnel leave and arrive at the hotel each day. (This information may be needed at remote sites where hotel accommodations are not near the site.)
- Indicate when all subcontractors arrive and depart the site.
- Note weather conditions at the time of arrival on site and any changes to the weather that might affect completion of project tasks during the day.
- Include the date and project number at the top of each page.
- Document that a site safety meeting was held and include the basic contents of the meeting.

Tetra Tech, Inc. EMI Operating Unit - SOP No. 024 Page 6 of 8

Last Reviewed: December 2022

## Title: Recording Notes in Field Logbooks

• List the level of personal protection to be used for health and safety.

- Summarize the day's planned activities.
- Summarize which activities each field team member will be doing.

#### 2.3.2 Field Activity Entries

- Refer to field data collection forms for details about field data collection activities (for example time, date, depth of samples, and field measurements). If separate field sampling sheets are not used, see Section 2.3.3 regarding logbook entries for sampling activities.
- Refer to well purge forms, well construction logs, and other activity-specific forms as applicable
  rather than including this type of information in the field logbook. These other forms allow the
  information to be more accessible at a later date.
- List any air monitoring instrumentation used, with readings and locations.
- Refer to instrument field logs for equipment calibration information.
- Summarize pertinent conversations with site visitors (agency representatives, property owners, client contacts, and local citizens).
- Summarize any problems or deviations from the quality assurance project plan (QAPP) or field sampling plan.
- Document the activities and whereabouts of each team member. (As indicated in Section 2.1, multiple logbooks may be required to ensure sufficient detail for contemporaneous activities).
- Indicate when utility clearances are completed, including which companies participated and specific ticket numbers issued by the utility locate vendor.
- Indicate when verbal access to a property is obtained.
- Include names, addresses, and telephone numbers of any pertinent site contacts, property owners, and any other relevant personnel.
- Document when lunch breaks or other work stoppages occur.
- Include approximate scale for all diagrams. If a scale is not available, write "not to scale" on the diagram. Indicate the north direction on all maps and cross-sections, and label features on each diagram.

#### 2.3.3 Sampling Activity Entries

The following information should typically be on a sample collection log and referenced in the logbook. If the project does not use sample sheets as a result of project-specific requirements, this information should be included in the logbook.

Location description

Tetra Tech, Inc. EMI Operating Unit - SOP No. 024 Page 7 of 8

Last Reviewed: December 2022

#### Title: Recording Notes in Field Logbooks

- Names of samplers
- Collection time
- Designation of sample as a grab or composite sample
- Identification of blind duplicates or split samples
- Type of sample (water, sediment, soil gas, or other medium)
- Sample number
- On-site measurement data (such as pH, temperature, and specific conductivity)
- Field observations (odors, colors, weather)
- Preliminary sample description
- Type of preservative used
- Instrument readings, if applicable

#### 2.3.4 Closing Daily Entries

- Describe decontamination procedures (personnel and equipment).
- Describe handling and disposition of any investigation-derived wastes.
- Summarize which planned activities were completed and which ones were not.
- Note the times that personnel depart the site for the day.
- Summarize any activities conducted after departing the site (paperwork, sample packaging, etc.). This may be required to document billable time incurred after field activities were completed for the day.

#### 2.3.5 Photographic Log Entries

- Before using a digital camera, ensure that the system date and time are correct. Verify whether the timestamp is being recorded on the image, if required.
- Indicate in the text that photographs were taken and the location where the photographs can be found (for example, in the project file) and identify the photographer.
- Begin a new photolog page for each new field day.
- Record the time of photograph so that the image can be generally identified when reviewing the digital files.
- Note the direction in which the photograph was taken, along with any relevant details that might not be understood when looking at the photograph.
- In the event that a film camera is used, the sequential number of the image should also be recorded, and the time from the logbook will be the recorded time for the photograph.

Tetra Tech, Inc. EMI Operating Unit - SOP No. 024 Page 8 of 8

Title: **Recording Notes in Field Logbooks**Last Reviewed: December 2022

#### 2.4 LOGBOOK STORAGE

Custody of logbooks must be maintained at all times. During field activities, field personnel must keep the logbooks in a secure place (locked car, trailer, or field office) when the logbook is not in personal possession. When the field work is over, the logbook should be included in the project file, which should be in a secured file cabinet; in addition, if directed by the project manager, scan logbook pages for electronic file management upon returning to the office. The logbook may be referenced in preparing subsequent reports and scanned logbook pages may be included as an appendix to a report. However, it is advisable to obtain direction directly from the client before including the logbook as a report appendix, because its inclusion may not be appropriate in all cases.

#### 2.5 HEALTH AND SAFETY CONSIDERATIONS

In addition to the procedures outlined in this SOP, all field staff must be aware of, and follow, the health and safety practices that result from the Activity Hazard Analyses (AHA) for a project. The AHAs include critical safety procedures, required controls, and minimum personal protective equipment necessary to address potential hazards. The hazards specific to project tasks must be identified and controlled to the extent practicable and communicated to all project personnel via the approved, project-specific health and safety plan. If health and safety conditions warrant a change to sampling approaches or locations, this should be documented in the field logbook.

## **SOP APPROVAL FORM**

## TETRA TECH, INC.

## EMI OPERATING UNIT

## ENVIRONMENTAL STANDARD OPERATING PROCEDURE

## CALIBRATION OF AIR SAMPLING PUMP

**SOP NO. 064** 

**REVISION NO. 2** 

Last Reviewed: November 2023

Carlo Jamik	November 2023
Quality Assurance Approved	Date

Tetra Tech, Inc. EMI Operating Unit - SOP No. 064

Title: Calibration of Air Sampling Pump

Last Reviewed: November 2023

Page 1 of 6

#### 1.0 BACKGROUND

Several instruments are available to calibrate low air flow rate. This standard operating procedure (SOP) will assume the use of a MesaLabs Bios DryCal Defender 510 or 520 for calibration of high volume (flow rate greater than 5 liters per minute (L/min)) and low volume (flow rate of 5 L/min or lower). This SOP also assumes the use of a low volume air sampling pump similar to the Sensidyne GilAir Plus, a digital air sampling pump, and the use of a high-volume air sampling pump similar to the SKC QuickTake® 30. The air calibration procedure discussed in this SOP will work for analog air sampling pumps as well, such as the Sensidyne GilAir 5 or Sensidyne Aircon 2. A DryCal calibrator is used to calibrate sample collecting devices, which includes an air sampling pump with attached media, such as filters, impingers, sampling tubes, and color detector tubes.

#### 1.1 PURPOSE

This SOP establishes the requirements and procedures for calibrating a digital air sampling pump using a MesaLabs Bios DryCal Defender.

#### 1.2 SCOPE

This SOP provides instruction on the calibration of a digital air sampling pump and sample media using a BIOS DryCal Defender Calibrator.

#### 1.3 REFERENCES

Bios International Corporation. 2007. "Defender 500 Series User Manual" Form #MK01-25, Rev B. Bios, 2007.

Sensidyne, LP. 2011. "GilAir Plus Operation Manual." Form #360-0132-01, Rev D. Sensidyne.

SKC Inc. 2010. "QuickTake 30 Sample Pump Operating Instructions." Form #40079, Rev 1910. SKC Inc.

U.S. Environmental Protection Agency (EPA). 2020. "Operation of DryCal Defender Series Primary Flow Calibrator." Environmental Response Team. EPA-PROC-2130-20. January. <u>ERT-PROC-2130-20-R1 SOP Manual.pdf</u> (epa.gov)

Tetra Tech, Inc. EMI Operating Unit - SOP No. 064 Page 2 of 6 Last Reviewed: November 2023

Title: Calibration of Air Sampling Pump

#### 1.4 REQUIREMENTS AND RESOURCES

To calibrate an air sampling pump, the following equipment is needed:

- Air sampling pump (GilAir Plus or QuickTake 30, or similar digital pump)
- DryCal calibrator (Bios Defender 500 series or similar)
- Sample media (mixed cellulose ester [MCE] membrane filter, polyurethane foam [PUF] tube, phase contrast microscopy [PCM] filter, etc.)
- Logbook or field data forms (can be electronic)
- Sample labels

The DryCal defender must be calibrated annually by an accredited vender to ensure accuracy and function.

#### 2.0 **PROCEDURE**

The following procedure is used to calibrate a sampling pump with a Bios DryCal Defender 520 or 530. If only using a low volume air sampling pump, use a "medium" (50-5,000 milliliters per minute [mL/min]) model of the 520 or 530. If using only a high volume, or a high volume and a low volume air sampling pump, use a "high" (300-30,000 mL/min) model.

Before calibration can occur, establish a flow rate for the air sampling pump. This will require a review of the method, a discussion with the analytical laboratory, and a review of the established reporting limits. Check the calibration at the beginning and end of the sampling event to establish an average flow rate for the sampling event. Calibrate equipment in the field as close to the start of the sample time as possible in order to obtain an accurate representation of field conditions during the time of sampling.

The procedure for calibrating an air sampling pump is as follows:

- Attach the tubing to the suction fitting (Figure 1). 1.
- Attach the sample media, additional tubing, and then the sample pump (Figure 2). If sampling with multiple pumps and media, calibrate each pump with the media that will be attached to that pump. Use flexible tubing to attach the sample media and pump to the DryCal calibrator. Ensure no kinks are present and use the shortest length of tubing as reasonable.
- Turn on the DryCal calibrator. To power on, press the On/Off button in the lower right corner of the control panel for one second.

Tetra Tech, Inc. EMI Operating Unit - SOP No. 064

Page 3 of 6

Title: Calibration of Air Sampling Pump

Last Reviewed: November 2023

4. Navigate through the Defender menu screen using the four arrows on the control panel. Select your desired menu option using enter in the center of the four arrows. Ensure in "SETUP" that the units are set to your preference and continuous flow is set up to take 10 readings with one second between each reading.

- 5. Navigate to the "Measure" option on the menu screen. Select continuous and press enter.
- 6. Turn the sampling pump on. The continuous measurement on the DryCal calibrator should start automatically. You will see a reading count at the bottom of the display. To receive the average flow rate, pause at the 10<sup>th</sup> reading by disconnecting the pump or hitting the enter button. This reading will be the flow rate you record for that pump.
- 7. Record the calibration information in a logbook or on electronic form where minute and total flow volume calculations will be calculated electronically. An example of an electronic form is shown in Figure 3. Record the following information in the form:
  - Flow rate (start and end)
  - Serial number of the pump
  - Pump number
  - Date
  - Sample start and end times
  - Location of sample
  - Analysis
- 8. At the end of the sampling event, collect the end flow rate and end sample time. Put sample name, date, and end time on the sample label. Enter all information into an air calculation form and calculate total air volume for your samples. This can be done by hand, but it is recommended to use an electronic air calculation form. In order to calculate total air volume, average the start and end flow rates, determine the total minutes the pump was ran for using the start and flow times<sup>1</sup>, multiply the average flow rate and total minutes.

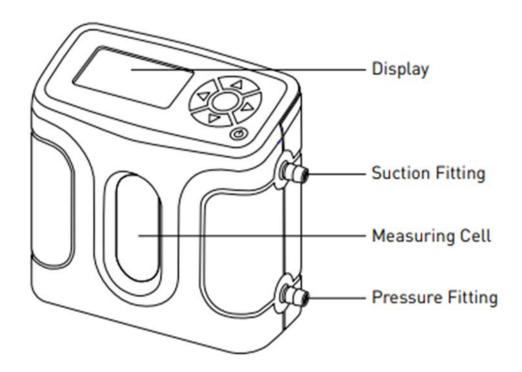
#### **Example:**

Name	Pump Serial #	Location	Analysis	Start Time	End Time	Total Minutes	Start Flow (L/min)	End Flow (L/min)	Average Flow (L/min)	Total volume (Liters)
Sample 1	0001	East Perimeter	Metals	0702	1503	481	4.12	3.98	4.05	1948.05

**Note**: <sup>1</sup>Air sample pumps can fault. If the pumps are digital you can get the total run time from the pump. If the pumps are analog, the end time will be the time when the pump fault was observed. It is recommended to check air sampling pumps every hour to minimize time discrepancies.

9. Fill out the chain of custody with the information shown in the above example. Double check ALL calculations, be especially cautious that total minutes were calculated correctly. Double check the chain matches your total volume calculations.

FIGURE 1
BIOS DRYCAL DEFENDER LAYOUT



# FIGURE 2 CALIBRATION SET UP



Tetra Tech, Inc. EMI Operating Unit - SOP No. 064

Title: Calibration of Air Sampling Pump

Page 6 of 6

Last Reviewed: November 2023

### FIGURE 3

# ELECTRONIC FORM EXAMPLE

	Air Sampling Form									
				Cra	awford Dem	olition ER				
TOLIN:										
Site: Crawford										
	NIOSH 7400 and NIOSI									
	pling Technician: C.Re	enner								
Date: 6/										
Primary Calibration: Bios DryCal D.C. L			ite							
Pump ID	Sample I.D.	Start Time	Stop Time	Total Min.	Start Flow Rate (L/min)	Stop Flow Rate (L/min)	Corrected Flow Rate	Total Volume (liters)	Pump Fault	Location
QT080	CPP-SP-200601	7:15	12:00	284.00	9.98	9.98	9.980	2834.32		South Asbestos
GA208	CPP-SP-200601	7:15	12:00	285.00	4.02	3.82	3.920	1117.20		South Metals
GA116	CPP-SP-200601	7:15	12:00	285.00	5.01	5.06	5.035	1434.98		South PCB

### **SOP APPROVAL FORM**

### TETRA TECH, INC.

# EMI OPERATING UNIT ENVIRONMENTAL STANDARD OPERATING PROCEDURE

### AIR QUALITY MONITORING

**SOP NO. 073** 

**REVISION NO. 3** 

Last Reviewed: November 2023

and famile	November 2023
Quality Assurance Approved	Date

Tetra Tech EM Inc. - Environmental SOP No. 073 Page 1 of 12

Title: **Air Quality Monitoring**Last Reviewed: November 2023

#### 1.0 BACKGROUND

Air quality monitoring is performed to evaluate concentrations of airborne chemicals or other materials. Particulate matter, volatile organic compounds (VOC), semivolatile organic compounds (SVOC), and other gases and vapors in the air can present potential health risks to workers and other receptors. Reliable measurements of airborne contaminants are necessary for selecting or upgrading personal protective equipment (PPE), delineating areas where protection is needed, assessing the potential health effects of exposure, evaluating site conditions, and evaluating the need for specific medical monitoring. Air monitoring is frequently used in emergency responses, where immediate information is needed about a release or threatened release of hazardous substances that present an imminent danger to public health, welfare, or the environment. Air monitoring is also commonly used as a screening tool to identify relatively impacted environmental media and to provide a real-time basis for selecting samples for laboratory analysis.

Various types of air monitoring instruments are available to monitor each type of airborne contaminant. Common air monitoring instruments used by Tetra Tech include photoionization detectors (PIDs), flame ionization detectors (FIDs), oxygen meters, combustible gas indicators, multigas meters that combine organic and toxic sensors for inorganics, single point monitors, colorimetric instruments, compound-specific instruments, and particulate matter monitors. It should be noted that this standard operating procedure (SOP) discusses only some of the air monitoring instruments available to field personnel. The particular type of meter or monitoring system to be used should be identified in the project work plan, field sampling plan, or health and safety plan, and should be selected on a site-specific basis depending on the data collection needs, the airborne contaminants to be monitored, and the sampling procedures to be used.

#### 1.1 PURPOSE

This SOP establishes the requirements and procedures for using various instruments to conduct air monitoring in the field. It also discusses general factors to consider when conducting air monitoring.

### 1.2 SCOPE

This SOP provides only a broad overview of recommendations for monitoring air quality and applies to general procedures for calibrating and operating air monitoring equipment in the field. The project work plan or field sampling plan should identify types of instruments to be used and the actual project-specific field parameters to be measured. The project-specific healthy and safety plan should identify chemical-

specific action levels for health and safety purposes. For each type of air monitoring instrument, the manufacturer's manual should be consulted for specific operating instructions.

This SOP is to be used in conjunction with Tetra Tech EMI SOP No. 003, "Organic Vapor Air Monitoring," U.S. Environmental Protection Agency (EPA) Environmental Response Team (ERT) SOPs, and instrument manuals and quick start guides developed by manufacturers or by qualified users of the monitoring equipment.

#### 1.3 **DEFINITIONS**

**Breathing Zone:** The area where field workers would be inhaling potentially impacted air, generally from about 3 to 5 feet above ground surface. The breathing zone will vary depending on the types of work activities being performed. Air monitoring is conducted in this zone to ensure that it is representative of the air being breathed by field team members.

**Direct Reading Instrument:** Instrumentation operating on various detection principles such as photoionization, photometry, or spectroscopy to provide real time readings of ambient contaminants in air.

#### 1.4 REFERENCES

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Tetra Tech EM Inc. - Environmental SOP No. 073

Page 3 of 12

Title: Air Quality Monitoring

Last Reviewed: November 2023

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- EPA. 2020c. "Operation of the DustTrak Aerosol Monitors; Desktop DRX Model 8533EP and Handheld DRX Model 8534." Environmental Response Team. EPA-PROC-2078-20. June.
- EPA. 2020d. "Operation of the SPM Flex Gas Detector." Environmental Response Team. EPA-PROC-2071-20. May.
- EPA. 2020e. "Operation of the Jerome J505 Mercury Analyzer." Environmental Response Team. EPA-PROC-2080-20. June.

#### 1.5 REQUIREMENTS AND RESOURCES

Depending on the type of air quality monitoring to be conducted, some or all of the following equipment will be required:

- Particulate monitoring meter, such as the TSI, Inc. DustTrak II Aerosol Monitor Model 8530 or DustTrak DRX Model 8533/8534 or similar
- Organic Vapor Air Monitoring Meter, such as the RAE Systems AreaRAE Pro, MultiRAE Pro, or UltraRAE 3000
- Compound-specific or compound-class-specific air monitoring meters, such as the Honeywell Single Point Monitor or SPM Flex, Lumex RA-915M mercury vapor analyzer, Jerome mercury vapor analyzers, or RAE Systems MultiRAE Benzene
- Manufacturer-supplied calibration kits, including tubing, regulators, and zeroing equipment
- Manufacturer-supplied calibration gas
- Telemetry hardware, such as RAE Systems RAELink 3 modems, Safe Environment Engineering VIPER Lincs and Gateways, or Field Data Solutions radio modems
- Logbook or field data sheets (may be in electronic form)
- Laptop equipped with telemetry software and wireless internet

Tetra Tech EM Inc. - Environmental SOP No. 073 Page 4 of 12

#### 2.0 APPLICATIONS, DETECTION METHODS, AND LIMITATIONS

All direct-reading instruments have inherent constraints in their ability to detect gaseous organic or inorganic compounds and particulates. They usually detect or measure only specific classes of chemicals or particulates. Generally, many direct-reading instruments that have been designed to detect one particular substance also respond to other substances, causing interferences and possibly resulting in false readings. Applications, detection methods, and limitations of organic air monitoring instruments can be found in Tetra Tech SOP No. 003. The following subsections discuss general applications, detection methods, and limitations when using air monitoring equipment.

### 2.1 APPLICATIONS

Air monitoring is used to help establish criteria for worker safety, document potential exposures, evaluate site conditions, and determine protective measures for site personnel and surrounding public. Air monitoring instruments can be used alone or as a network integrated using wireless telemetry to monitor for multiple contaminants at numerous locations.

Multigas meters such as the RAE Systems MultiRAE Pro and AreaRAE Pro have slots for electrochemical, carbon dioxide, oxygen, or combustible sensors in addition to the PID. Electrochemical sensors are available for a wide range of toxic gases. A list of available MultiRAE Pro and AreaRAE Pro sensors is available on the manufacturer's website and in the instrument user guides. When operated in datalog mode, the instrument records sensor readings to internal memory for download after the monitoring event. Refer to EPA ERT SOP No. ERT-PROC-2139-20, "Operation of the MultiRAE Pro Wireless Portable Detector" and EPA ERT SOP No. ERT-PROC-2066-20, "Operation of the AreaRAE Wireless Multi-Gas Monitor" for comprehensive standard operating procedures for the MultiRAE Pro and AreaRAE Pro (EPA 2020a; EPA 2020b).

TSI DustTrak DRX monitors or similar particulate monitors can be used to obtain real-time readings of size-segregated mass fractions of particulates with aerodynamic diameters of 1 micron (PM<sub>1</sub>), 2.5 micron (PM<sub>2.5</sub>), 4 micron or respirable particles (PM<sub>4</sub>), 10 micron or thoracic particles (PM<sub>10</sub>), and total suspended particulates (TSP). DustTrak II monitors can provide real-time readings of PM<sub>2.5</sub> or any other mass fraction if the impactor for the specified particle size is installed in the monitor. Both the DustTrak DRX and DustTrak II electronically record particulate data as specified in the "RunMode" menu on the instrument screen. Refer to EPA ERT SOP No. EPA-PROC-2078-20, "Operation of the DustTrak Aerosol Monitors; Desktop DRX Model 8533EP and Handheld DRX Model 8534" for comprehensive standard operating procedures for the DustTrak DRX (EPA 2020c).

Tetra Tech EM Inc. - Environmental SOP No. 073 Page 5 of 12

Title: Air Quality Monitoring Last Reviewed: November 2023

Single Point Monitors (SPM) such as the SPM Flex can detect specific chemicals or classes of chemicals, depending on the Chemcassette cartridge installed. A wide range of Chemcassettes are available for toxic gases; a full list is available on the manufacturer's website. The SPM Flex can provide real-time readings of concentrations of the specified gas or gas family and electronically logs the data for download after the monitoring event. Refer to EPA ERT SOP No. EPA-PROC-2071-20, "Operation of the SPM Flex Gas Detector" for comprehensive standard operating procedures for the SPM Flex (EPA 2020d).

Mercury Vapor Analyzers (MVAs) such as the Lumex RA-915+ Mercury Analyzer, RA-915M Mercury Analyzer, and the Jerome J505 Mercury Vapor Analyzer are capable of detecting mercury vapor in ambient air at concentrations from 2 to 200,000 nanograms per cubic meter (ng/m³) (Lumex RA-915M) and 0.05 to 500 micrograms per cubic meter (μg/m³) (Jerome J505 MVA). Both the Lumex RA915-M and Jerome J505 MVA provide real-time readings of mercury vapor concentrations and have the ability to electronically log the data for download after the monitoring event. When operated in "Monitoring" mode, the Lumex RA915-M is capable of running for a designated amount of time with periodical zero checks and datalogging. Refer to EPA Scientific, Engineering, Response and Analytical Services (SERAS) SOP No. 1729, "Operation of the Lumex RA-915+ Analyzer for Measuring Mercury Vapor Concentrations in Air" and EPA ERT SOP No. EPA-PROC-2080-20, "Operations of the Jerome J505 Mercury Analyzer" for comprehensive standard operating procedures for the Lumex RA-915+, Lumex RA-915M, and Jerome J505 MVA (EPA 2016; EPA 2020e)

### 2.2 DETECTION METHODS

The MultiRAE Pro and AreaRAE Pro electrochemical sensors measure the concentration of the toxic gas within an external circuit using oxidation and reduction reactions. Electrochemical sensors for oxygen (O<sub>2</sub>) are available as well. The toxic sensor must be calibrated to the specific toxic gas the sensor is designed to measure, or a member of the group of compounds the sensor is designed to measure. While some sensors are toxic compound specific, all sensors may respond negatively or positively to other compounds in the environment. Some electrochemical sensors such as nitric oxide (NO) and ammonia (NH<sub>3</sub>) require a bias voltage to detect the gas and require an equilibrium (or warm-up) time of at least 6 hours after installation. Unbiased sensors require at least 10 minutes to stabilize.

There are two types of combustible sensors available for detecting LEL and methane, protected catalytic bead sensors and non-dispersive infrared (NDIR) sensors. Protected catalytic bead sensors contain a catalyzer that allows for oxidation of the combustion gas at temperatures well below their normal flash point. NDIR sensors utilize light and measure IR absorption at specified wavelengths to estimate

Tetra Tech EM Inc. - Environmental SOP No. 073 Page 6 of 12

Title: Air Quality Monitoring

Last Reviewed: November 2023

concentrations of a gas. While catalytic bead sensors are not sensitive to only one chemical, NDIR sensors rely on chemical-specific IR absorbance to detect a single combustible gas.

TSI DustTrak monitors utilize light-scattering laser photometers to determine mass-based measurements of particulates. Both the DustTrak II and the DustTrak DRX draw aerosols into a sensing chamber using a diaphragm pump, and a sample flow is illuminated by a laser light. While the DustTrak II utilizes the voltage across the photodetector to determine particulate concentration, the DustTrak DRX utilizes the individual pulses from the photometer to make single particle measurements, allowing the monitor to simultaneously measure both the aerodynamic diameter of an individual particle, as well as its mass.

The Honeywell SPM Flex draws a gas sample to the Chemcassette tape-based optical gas detection system. The SPM Flex uses an optical scanning system to detect the presence of a gas based on a color change on the installed Chemcassette. Chemcassettes are available for a wide range of both specific toxic gases and classes of toxic gases; a list of available cassettes can be found in the SPM Flex User's Manual. It is important to note that when using a Chemcassette designed to monitor for a class of toxic gases, such as a mineral acid Chemcassette, the monitor is configured to report the concentration of one specific member of the target compound class. However, a color-change will be induced in the Chemcassette by any member of the target compound class. The specific configuration of the SPM Flex monitor, limit of detection, and instrument response time can vary widely by both Chemcassette and the specific target compound.

The Lumex RA-915+ and RA-915M utilize differential Zeeman atomic absorption spectrometry using high frequency modulation of light polarization (ZAAS-HFM). A mercury lamp, which is a radiation source, is placed into a permanent magnetic field. Two beams are propagated from the lamp to a photodetector, one passes through an analytical cell and one does not. When mercury vapor from a sample is introduced into the analytical cell, the difference between the intensities of the beams increases proportionately to mercury vapor concentration. The Jerome J505 MVA uses atomic fluorescence spectroscopy (AFS), which uses a light source with a wavelength of 254nm. Any mercury present in the sample will absorb and re-emit the light, which is then measured by a photo multiplier tube (PMT).

Tetra Tech EM Inc. - Environmental SOP No. 073

Page 7 of 12

Title: Air Quality Monitoring

Last Reviewed: November 2023

### 2.3 LIMITATIONS

Toxic sensors in the MultiRAE Pro and AreaRAE Pro have the following limitations:

- Cannot be exposed to precipitation or operate in temperatures outside of the 14 to 113-degree Fahrenheit (°F) range.
- Cannot be used when liquid may be inadvertently drawn into the probe.
- Electrochemical sensors can respond to chemicals other than those they are designed to detect. Some sensors, such as formaldehyde (HCHO) and HCN, have extremely high cross-sensitivities. Sensor-specific RAE Systems Technical Notes should be consulted prior to use. RAE Systems Technical notes are available on the manufacturer's website.
- When used in the field, it is recommended that the instrument's accuracy be checked on a daily basis. Silicones, phosphates and phosphorus containing compounds, acidic or basic atmospheres, and overexposure (especially for oxygen [O<sub>2</sub>] and VOC sensors) can damage sensors or the PID lamp.
- Most RAE System toxic sensors have a life span of two years.

Combustible sensors in the MultiRAE Pro and AreaRAE Pro have the following limitations:

- Protected catalytic bead sensors must be used in an environment with sufficient oxygen (greater than 10%) in order to operate.
- Protected catalytic bead sensors do not distinguish between combustible gases. If concentrations of a specific combustible gas are desired, use an NDIR sensor for the desired gas.
- Combustible sensors have a shorter life span (about 1 year) than electrochemical sensors. Ensure that the sensor is not expired prior to use.
- Combustible sensors are susceptible to sensor poisoning, in which the presence of certain compounds (typically silicones and phosphorous-containing compounds) can degrade the performance of the sensor over time, resulting in under-reporting of combustible atmospheres.

Photometric particulate monitors such as the DustTrak DRX have the following limitations:

- High humidity or fog may cause elevated particulate readings. The use of the Heated Inlet Sample Conditioning module may mitigate these issues by maintaining a stable relative humidity level.
- Measurement accuracy is dependent on a wide particle size distribution and by the optical characteristics of the particles, such as density, size, shape, and composition.

Single Point Monitors such as the SPM Flex have the following limitations:

- Chemcassette tapes are highly sensitive to rain, fog, humidity, and condensation.
- Cannot be used in temperatures outside of the 32 to 104 °F range.
- Operation is calibrated for use at altitudes below 3,000 feet. Work at elevations above 3,000 ft may require manufacturer calibration.
- Not all Chemcassette reactions are chemical specific. A colorimetric indicator may react with several similar chemicals, but the concentration determined by the monitor will be for a specific compound, based on a compound-specific conversion factor.
- Chemcassettes have a short life span and often need to be refrigerated or stored in freezers. Additionally, there is a relatively long lead-time to obtain new Chemcassettes.

Tetra Tech EM Inc. - Environmental SOP No. 073

Page 8 of 12

Title: Air Quality Monitoring

Last Reviewed: November 2023

The Lumex RA-915+, Lumex RA-915M, and Jerome J505 MVA have the following limitations:

- MVAs should never be exposed to metallic mercury.
- The Lumex RA-915+ and Lumex RA-915M should not be used for long durations in environments with high concentrations of mercury vapor (greater than 10,000 ng/m³). If mercury vapors are present at high concentrations, operate the Lumex instruments in "High Concentrations" measuring mode or use a Jerome J505 MVA.
- Some interference with high levels of VOCs has been observed and noted. If operating any MVA in an environment with VOCs present, consider collecting air samples for laboratory analysis via National Institute for Occupational Safety and Health (NIOSH) Method 6009, *Mercury*.
- MVAs are sensitive to environmental conditions. If operating an MVA in an environment with
  variable environmental conditions, re-zero the instrument frequently. The MVA should never be
  exposed to water or dust.

#### 3.0 PROCEDURES

#### 3.1 TESTING AND CALIBRATION PROCEDURES

Each air monitoring instrument should be calibrated according to the manufacturer's specifications. General procedures applicable to most equipment are as follows:

- Equipment should be thoroughly cleaned, and then calibrated and tested before the startup of monitoring at each site.
- Equipment should be calibrated and tested using manufacturer-provided calibration gas and calibration connector kits.
- Batteries should be charged before the startup of field work, and the battery charge level should be checked at the start of each day. The battery charge life will vary depending on the monitoring instrument used, the application, and environmental conditions such as the temperature. Some instruments are equipped with an adapter that will allow the unit to be plugged into a car charger or charge from a car battery. It is recommended that extra batteries be kept on hand when conducting field work.
- Calibration and testing of field equipment should be documented every time it is performed.
   Calibration and testing information should be recorded in field logbooks, field data sheets, or electronic data capture forms.
- If testing and calibration measurements are out of tolerance, the instrument must be serviced or repaired.
- The manufacturer-recommended factory calibration schedule should be followed. If an instrument is due for factory calibration it should not be used in the field.

Tetra Tech EM Inc. - Environmental SOP No. 073 Page 9 of 12

Title: Air Quality Monitoring

Last Reviewed: November 2023

### 3.2 FIELD MEASUREMENT PROCEDURES

Each air monitoring instrument should be operated according to manufacturer's specifications. The actual field procedures will vary depending on the type of monitoring to be conducted. Many instruments are affected by moisture, humidity, and dust. Additionally, many instruments include a datalogging option. Even if wireless telemetry is utilized, the datalogging option should be selected to prevent losing air monitoring data. General procedural summaries and processes for air monitoring associated with initial surveys and offsite or perimeter monitoring are presented below.

### 3.2.1 Initial Surveys

Initial surveys are conducted upon first entry to an area with a potentially hazardous atmosphere. Initial surveys are performed relatively rapidly and are a screening process for collecting preliminary data to use for determining levels of PPE, establishing work zones, and establishing contaminated areas for further monitoring or sampling.

In most cases, project teams will have prior knowledge of the site and site contaminants, allowing air monitoring instrumentation to be selected based on the site-specific contaminants of concerns. When entering unknown environments, utilize the EPA Emergency Response Technical Group "Hazard Evaluation Flow Chart for Unknown" (EPA 2005). Intrinsically safe instrumentation should be used when there is a threat of atmospheres that may be explosive or conducive to ignition.

General procedures for conducting initial surveys are as follows:

- Following the instrument manual, calibrate and test air monitoring equipment. Multiple instruments may be necessary depending on the nature of the site.
- Record calibration information, site name, date and time, and pertinent weather information in a field logbook or on field data sheets.
- Approach the entrance to the site from the upwind direction, if possible. At the entry of the site, record the readings from each instrument in the breathing zone. Negative or non-detect results should also be recorded to demonstrate that the measurement was taken.
- Enter the site and record a measurement in each area of interest (ex: spill areas, frequently utilized spaces, current or historical chemical storage areas).
- Determine if measurements indicate sustained concentrations of contaminants in the breathing zone which are above action levels specified in the site-specific health and safety plan, or which otherwise do not meet healthy and safety criteria. Upgrade the level of PPE, implement engineering controls, and/or stop work if necessary.
- Once the initial survey is complete, download all instrument data and save in the project folder for site records or for future analysis.

Information obtained during initial surveys may lead to further monitoring. A more comprehensive survey and analysis for specific compounds may be warranted.

Tetra Tech EM Inc. - Environmental SOP No. 073 Page 10 of 12

Title: Air Quality Monitoring Last Reviewed: November 2023

### 3.2.2 Off-Site and Perimeter Monitoring

Off-site and perimeter monitoring is typically conducted with the same instrumentation that is used for onsite monitoring. Since air is dynamic by nature, physical boundaries do not necessarily delineate the extent of contamination or the boundary of an area affected by a release. Monitoring at the perimeter of the site, or at locations off site provides information about pollutant migration. General procedures for conducting off-site and perimeter monitoring are as follows:

- Following the instrument manual, calibrate and test air monitoring equipment. Multiple instruments may be necessary depending on the nature of the site.
- Record calibration information, site name, date and time, and pertinent weather information in a field logbook or on field data sheets.
- Identify perimeter or offsite locations for monitoring. Generally, cardinal directions are acceptable for perimeter monitoring. Monitors may be adjusted or added based on wind direction and wind speed, or the presence of priority populations for monitoring, such as sensitive receptors.
- Once monitoring locations have been determined, place instruments in the breathing zone and record monitoring start times and readings. Ensure instruments are not in the way of traffic, work, or other activities that could damage or disturb them.
- If the monitoring period is prolonged, check on the instrument periodically to ensure proper function and accuracy. If sensor drift is suspected or observed, recalibrate the instrument and restart monitoring.
- At the end of the monitoring period, record the monitoring stop time and readings.
- Download all instrument data and save in the project folder for site records or for future analysis.

Lack of detections or negative results should not be interpreted as complete absence of airborne contaminants. It is possible that the compound class the instrument detects is not present or that the concentrations of the compounds are below the instrument's detection limits. The instrument detection limits should be presented with all site data.

#### 3.3 AIR MONITORING TELEMETRY

Many air monitoring instruments are capable of generating a real-time data stream that can be incorporated into telemetry systems for remote monitoring. Telemetry systems make use of wireless data transmission technologies to convey monitoring results to a central location for viewing and storage. Common telemetry systems used for transmitting air monitoring data include EPA ERT's VIPER system and Honeywell's ProRAE Guardian system. The VIPER system can be operated using either wireless local area networks (WLAN)/cellular system or 900 MHz radio modems. Numerous air monitoring instruments can be incorporated in the VIPER system, including DustTrak DRX monitors, SPM Flex monitors, and RAE Systems equipment (for a full list of supported monitoring equipment, see www.response.epa.gov/VIPER). The ProRAE Guardian system utilizes 900 MHz radio modems or

WLAN to transmit data. The ProRAE Guardian system supports most RAE Systems instruments, including AreaRAE Pro and MultiRAE Pro monitors.

When monitoring instruments are configured as part of a telemetry network, monitoring data are compiled and visualized in a telemetry software. Most telemetry software applications provide tools for visualizing trends in monitoring results, configuring alarms when results exceed action levels, and store monitoring data in a database application. Data transmitted to the telemetry software can be viewed in near real-time from a remote location, including simultaneous viewing of multiple types of instruments, positioned at multiple locations.

#### 3.4 ACCURATE RECORDING AND INTERPRETATION

Air monitoring instruments must be operated, and the data interpreted by individuals who understand the operating principles and limitations of the instruments. At hazardous waste sites, where unknown and multiple contaminants are frequently encountered, instrument readings should be interpreted conservatively.

The following guidelines promote accurate recording and interpretation:

- Calibrate instruments in accordance with the manufacturer's instructions before and after every
- Conduct additional monitoring at any location where a positive response occurs.
- Report any readings of zero as nondetectable (ND) rather than as "clean." Quantities of chemicals may be present but at concentrations that are not detected by the instrument.

#### 3.5 VARIABLES AFFECTING AIR MONITORING

Complex environments containing many substances, such as those associated with hazardous waste sites, pose significant challenges to assess airborne contaminants accurately and safely. Several independent and uncontrollable variables can affect airborne concentrations and instrument performance. These factors must be considered when conducting air monitoring and interpreting data. The following variables must be considered:

- **Temperature:** An increase in temperature increases the vapor pressure of most chemicals. Generally, worst-case emission of VOCs and SVOCs occur on the hottest day of site activities.
- Wind speed and direction: An increase in wind speed can affect airborne contaminant concentrations near free-standing contamination or a release. Wind speed and direction impacts the extent and paths of plumes of airborne contamination.

Tetra Tech EM Inc. - Environmental SOP No. 073

Page 12 of 12

Title: **Air Quality Monitoring**Last Reviewed: November 2023

• Rainfall: Water from rainfall can cap vapors, gases, or dust from a site, reducing concentrations of airborne contaminants.

- Moisture: Particulates, including finely divided hazardous solids, are highly moisture-sensitive.
   Moisture can vary significantly with respect to location and time and can impact the accuracy of monitoring results across all instruments.
- **Background emissions:** Background emissions from other activities in the proximity of a site can affect instrument readings. Operations such as vehicle maintenance, fueling facilities, and road or landscape work can affect readings associated with perimeter monitoring. Any potential outside sources of emissions should be recorded in the logbook or on field data sheets.
- Work activities: Work activities often require the mechanical disturbance of contaminated materials, which may change the concentration and composition of airborne contaminants at a site. Monitoring should be conducted at all phases of a project to ensure no work activities create hazardous work environments that require upgraded PPE, engineering controls, or stop work.

Consult the manufacturer's manual to determine the instrument-specific impacts of environmental conditions.

### **APPENDIX B**

**COMMUNITY SSAL METHODOLOGY** 



To: Matt Soltis

Tetra Tech

From: Karren Wood

Tetra Tech

Date: March 12, 2024

Subject: DRAFT Community Site Screening Action Level (SSAL) Calculations for Metals

and Asbestos, Lahaina, Maui County, Hawaii

The following paragraphs present the methodology used by Tetra Tech to calculate risk-based community Site Screening Action Levels (SSALs) for ambient community air monitoring, planned in response to debris operations as a result of the Maui wildfires. This methodology is based on the Community Air Monitoring Plan (CAMP) guidance (CalEPA DTSC, 2020). Community SSALs were calculated for the airborne chemicals of concern (COCs) listed in Table 1 using the most recent toxicity criteria available and project-specific exposure assumptions. The SSALs represent a conservative estimate of the analyte concentrations in air that off-site populations, including sensitive receptors such as children and the elderly, can be safely exposed to over the duration of on-site work activities.

Ambient air community SSALs for COCs listed in Table 1 were calculated using standard United States Environmental Protection Agency (USEPA) risk-assessment methodology, exposure assumptions, and toxicological data preferentially from USEPA sources.

### **Equations for Calculating SSALs**

Cancer and noncancer health effects are considered separately for carcinogens and noncarcinogens, respectively. As presented in the 2020 CAMP Guidance, the SSAL for each COC is "back-calculated" from a target cancer risk or noncancer hazard in accordance with USEPA inhalation risk assessment methodology, as described below.

#### Cancer-Based SSAL

A cancer-based SSAL (SSALc) was calculated for each carcinogenic COCs using the following equation:

$$SSAL_C = TR \times \frac{1}{IUR} \times \frac{AT_C}{ET \times EF \times ED}$$

#### Where.

- SSAL<sub>C</sub> = cancer-based screening action limit for COC in air (micrograms per cubic meter [µg/m³] for metals or fibers per cubic centimeter [f/cc] for asbestos)
- TR = target inhalation cancer risk (unitless)

- IUR = inhalation unit risk (per μg/m³ for metals; per f/cc for asbestos)
- ATc = averaging time for carcinogenic effects (hours)
- ET = exposure time (hours/day)
- EF = exposure frequency (days/year)
- ED = exposure duration (year)

A target excess risk level of one in a million  $(1\times10^{-6})$  was used as the point of departure for developing the SSALc, as recommended by USEPA. Note that cancer risks that are less than 1 x  $10^{-6}$  are considered *de minimis* by any regulatory agency. The IUR values and exposure assumptions are discussed in separate sections below.

#### Noncancer-Based SSAL

A noncancer-based SSAL (SSAL<sub>NC</sub>) was calculated for each noncarcinogenic COCs in using the following equation:

$$SSAL_{NC} = THQ \times REL \times \frac{AT_{NC}}{ET \times EF \times ED}$$

### Where,

- SSAL<sub>NC</sub> = noncancer-based screening action limit of COC in air (μg/m³ for metals; f/cc for asbestos)
- THQ = target inhalation noncancer hazard quotient (unitless)
- REL = inhalation reference exposure level or reference concentration (μg/m³ for metals; f/cc for asbestos)
- AT<sub>NC</sub> = averaging time for non-carcinogenic effects (hours)
- ET = exposure time (hours/day)
- EF = exposure frequency (days/year)
- ED = exposure duration (year)

A target noncancer hazard quotient (HQ) of 1 was used to develop the SSAL<sub>NC</sub>, as recommended by USEPA. An HQ is a numerical indicator of exceedance of an acceptable threshold for noncarcinogenic effects. An HQ equal to or less than 1 indicates that no adverse noncarcinogenic health effects are expected to occur for a select COC. The REL values and exposure assumptions are discussed in separate sections below.

### Toxicity Values used in the SSAL Calculations

There are different toxicity databases that regulatory agencies rely on for the purposes of quantifying the toxicity of chemicals in the environment. The sources of the carcinogenic toxicity values (IURs) and noncarcinogenic toxicity values (RELs or reference concentrations [RfC]) used in the SSAL calculations in priority order are as follows:

- USEPA Integrated Risk Information System (IRIS) (USEPA, 2023a)
- USEPA Provisional Peer-Reviewed Toxicity Values (PPRTVs) (USEPA, 2023b)
- Agency for Toxic Substances and Disease Registry (ATSDR) (ATSDR, 2023)

- California Office of Environmental Health Hazard Assessment (OEHHA) or California Environmental Protection Agency (CalEPA) Department of Toxic Substances Control (DTSC) (OEHHA, 2023)
- USEPA Health Effects Assessment Summary Tables (HEAST) (USEPA, 2023c)

### Carcinogenic Effects

Potential carcinogenic effects resulting from human inhalation exposure to constituents are estimated quantitatively using IURs, which represent the theoretical increased risk per exposure concentration. Of the COCs for which SSALs were calculated, only six (asbestos, arsenic, beryllium, cadmium, cobalt, lead, and nickel) have been assessed as potentially having carcinogenic effects via the inhalation pathway. The sources for the chemical specific IURs used in the SSALc calculations are presented in Table 1.

### Noncarcinogenic Effects

Potential noncarcinogenic effects resulting from human inhalation exposure to constituents are estimated quantitatively using RELs (or RfCs), which represent estimates of the daily maximum level of exposure to human populations (including sensitive subpopulations) that are likely to be without an appreciable risk of deleterious effects (USEPA, 1989). Except for particulate matter, all COCs listed in Table 1 have been assessed as potentially having noncarcinogenic effects via the inhalation pathway.

Most noncancer toxicity values are developed for long-term or chronic exposures. Chronic RELs are designed to address continuous exposures for one year or longer up to a lifetime (ATSDR, 2023). However, noncancer toxicity values for short-term or acute exposures have been developed for some chemicals. For example, for shorter exposure periods, ATSDR has acute (1 to 14 day duration) and intermediate (15 to 364 day duration – akin to subchronic) Minimal Risk Levels (MRLs). When available, noncancer toxicity values for acute exposure are generally an order of magnitude or higher than chronic toxicity values. That is, a higher level of exposure for a short duration may not lead to adverse health effects. While the duration of the site activities for this project may lead to short term, or acute, exposure circumstances, chronic RELs were conservatively applied over acute or subchronic toxicity values in order to be protective of sensitive subpopulations. The sources for the chemical specific RELs/RfCs used in the SSAL<sub>NC</sub> calculations are presented in Table 1.

There were uncertainties associated with the RELs for select COCs. The following substitutions were conservatively applied:

- For lead, there is an OEHHA IUR, but there is no established noncancer toxicity REL.
   There is a Hawaii-specific Ambient Air Quality Standard (AAQS) for lead (per Hawaii Administrative Rule §11-59-4), which is 1.5 μg/m³ averaged over a calendar quarter (that is, 3 months) (Hawaii Administrative Rules, 2001). The AAQS is lower than the SSALc calculated with the IUR. Therefore, the AAQS was used as the SSAL for lead.
- Copper, thallium, and zinc RELs were not available from IRIS, ATSDR, or OEHHA,.
   Therefore, National Institute for Occupational Safety and Health (NIOSH)

   Recommended Exposure Limits with applied safety factors of 10 were conservatively applied (NIOSH, 2023).

### **Exposure Assumptions**

Exposure assumptions were applied in the SSAL calculations based on project specific activities. The project is assumed to be comprised of 10-hour workdays for 6 days per week over a time period of 1 year (assumed to be a conservative estimate). The AT is the amount of time over which the exposure is averaged and is equal to the project duration for noncancer effects and 70 years, or assumed lifetime, for cancer effects.

The exposure parameters are summarized in the table that follows:

Exposure Parameter	SSALc	SSALnc
ET (hours)	10	10
EF (days per year) <sup>(1)</sup>	313	313
ED (years)	1	1
AT (hours) <sup>(2)</sup>	613,200	7,509

- 1 EF for days worked in 1 year is calculated as follows:
  - EF = 6/7 (6 days/week work schedule) \* 365 days (total number of days in 1 year)
- 2 AT is calculated as follows:
  - AT (cancer) = 70 years (lifetime) \* 365 days per year \* 24 hours per day
  - AT (noncancer) = 313 days (project duration) \* 24 hours per day

### **Summary and Recommendations**

The selected SSALs were calculated as described above and are presented in Table 1. SSALs were calculated for both cancer and noncancer endpoints, as applicable. The lower (more conservative) of the two values was selected as the recommended SSAL for each COC. Note that in some cases, the SSALc was greater than the SSAL<sub>NC</sub>. In these cases, due to the short term duration of the project (1 year), the predicted cancer risk from short-term exposure was less significant than potential noncancer health effects.

As noted in Table 1, lower of the asbestos SSAL $_{\rm C}$  and SSAL $_{\rm NC}$  is the SSAL $_{\rm C}$  of 0.0009 f/cc, The SSAL $_{\rm C}$ , which is based on a target cancer risk of 1E-06, falls below the laboratory method detection limit (MDL) (0.0025 f/cc) for ISO 10312 (Determination of Asbestos Fibers). Therefore, in order to be protective of human health, while being able to meet the MDL, the SSAL for asbestos is set at the NIOSH REL (up to 10-hour TWA) of 0.1 f/cc with a safety factor of 30 applied (professional judgment to account for some uncertainty associated with using an occupational exposure limit when also considering sensitive subpopulations). The resulting SSAL $_{\rm C}$  is 0.003 f/cc, which is above the MDL. The SSAL of 0.003 f/cc is considered protective of human health for the Lahaina cleanup efforts based on the following factors.

- First, the ISO 10312 method measures all types of asbestos fibers and the inhalation unit risk is based on only one type of asbestos fiber (chrysotile). Therefore, the analytical method is likely to overestimate exposure levels to chrysotile and therefore may also overestimate the potential cancer risk level based on the total fiber count. Even with this conservatism, the cumulative risk at the detection limit of the analytical method would result in a cumulative risk of 2.5×10-6 from possible asbestos exposure assumed to occur 10 hours per day each day of the debris removal project.
- Given the heterogeneity of the debris in the fire zone, it is unlikely that asbestos will be
  present across the entire project area and therefore nearby receptors would not be
  expected to be exposed to asbestos in ambient air throughout the project period.

- Additionally, the SSAL of 0.003 f/cc is below the USEPA's asbestos clearance level (found within the Asbestos Hazard Emergency Response Act [AHERA]) of 0.01 f/cc, which is the benchmark used in air testing to assure that asbestos fibers released during an abatement action have been reduced to an acceptable level. AHERA is USEPA's regulation pertaining specifically to schools and has become the general standard used for all asbestos clearance levels.
- Background asbestos levels can be higher (e.g., ambient air levels from traffic and wear
  of vehicle brake pads). Studies to determine asbestos concentrations vary based on
  the sources of exposure in the region or city studied, and as such there is no specific
  level for ambient asbestos in air. However, the intensity of <u>vehicular traffic</u> is one of the
  most relevant sources found to be related to concentrations of <u>asbestos fibers</u> in the air.

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**ATTACHMENT 1** 

**EPA ERT SOPs** 



SOP: ERT-PROC-2015-20

PAGE: 1 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

### **ASBESTOS AIR SAMPLING**

#### **CONTENTS**

1	וח	rc	C	[ A	TN	ΛE	P	C
			١.	, <b>,</b> ,		/	. 11	. 7

1.0	SCOPE.	AND	A PPI	<b>ICA</b>	TION
1.0	DCOLL.	$\alpha$	$\alpha$	$\mu \cup D$	

- 2.0 METHOD SUMMARY
- 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE
  - 3.1 Sample Preservation
  - 3.2 Sample Handling, Container and Storage Procedures
- 4.0 INTERFERENCES AND POTENTIAL PROBLEMS
  - 4.1 Filter Preparation Methods
    - 4.1.1 ISO 10312 Direct-Transfer TEM Specimen Preparation Method
    - 4.1.2 ISO 13794 Indirect TEM Specimen Preparation Method
  - 4.2 NIOSH Method for TEM
  - 4.3 NIOSH Method for PCM
- 5.0 EQUIPMENT/APPARATUS
  - 5.1 Sampling Pump
  - 5.2 Filter Cassette
    - 5.2.1 TEM Cassette Requirements
    - 5.2.2 PCM Cassette Requirements
  - 5.3 Other Equipment
- 6.0 REAGENTS
- 7.0 PROCEDURES
  - 7.1 Flow Rates, Air Volumes and Analytical Sensitivity
    - 7.1.1 Occupational Methods
    - 7.1.2 Risk-based Methods
  - 7.2 Calibration Procedures
    - 7.2.1 Calibrating a Personal Sampling Pump with a Rotameter
    - 7.2.2 Calibrating a Personal Sampling Pump with an Electronic Calibrator
  - 7.3 Meteorology



8.0

9.0

10.0

11.0

12.0

13.0

APPENDICES A - Figures SOP: ERT-PROC-2015-20

PAGE: 2 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

## **ASBESTOS AIR SAMPLING**

### CONTENTS (Cont'd)

7.4	Ambie	Ambient Sampling Procedures				
	7.4.1 7.4.2 7.4.3	1 0				
7.5	Indoor	Indoor Sampling Procedures				
	7.5.1	Aggressive Sampling Procedures				
CALC	CALCULATIONS					
QUAL	QUALITY ASSURANCE/QUALITY CONTROL					
9.1 9.2						
DATA VALIDATION						
HEALTH AND SAFETY						
REFE	REFERENCES					



SOP: ERT-PROC-2015-20

PAGE: 3 of 25 REV: 1.1

EFFECTIVE DATE: 06/18/20

### **ASBESTOS AIR SAMPLING**

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SOP: ERT-PROC-2015-20 PAGE: 4 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

### ASBESTOS AIR SAMPLING

#### 1.0 SCOPE AND APPLICATION

Asbestos are naturally occurring fibrous minerals including: chrysotile, crocidolite, amosite, tremolite, actinolite, anthophylite, and any of these minerals that have been chemically treated and/or altered. The precise chemical formulation of each species will vary with the location from which it was mined. Asbestos has been used in many commercial products, including building materials such as flooring tiles and sheet goods, paints and coatings, insulation, and roofing asphalts. These products and others may be found at hazardous waste sites, hanging on overhead pipes, contained in drums, abandoned in piles, or as part of a structure are also known as asbestos containing material (ACM). Naturally occurring asbestos (NOA) in multiple forms can also be found. NOA associated with vermiculite mining and operations, and asbestos tailing piles from vermiculite mining operations can be a source of ambient asbestos fibers.

Asbestos is a known carcinogen, and air sampling is necessary to assess the potential for airborne exposure as part of a human health risk assessment. This standard operating procedure (SOP) provides procedures for asbestos air sampling based on drawing a known volume of air through a mixed cellulose ester (MCE) filter, which is then sent to a laboratory for analysis. One of the following four analytical methods is typically used for determining asbestos in air by ERT. These methods consist of: (1) National Institute for Occupational Safety and Health (NIOSH) Method 7400 for Phase Contrast Microscopy (PCM); (2) NIOSH Method 7402, Asbestos by TEM; (3) ISO 10312, Ambient Air - Determination of Asbestos Fibers - Direct Transfer Transmission Electron Microscopy (TEM) Method; and (4) ISO 13794 Ambient Air - Determination of Asbestos Fibres - Indirect-Transfer Transmission Electron Microscopy Method. Each method has specific sampling and analytical requirements (i.e. sample volume and flow rate) for determining asbestos in air.

The United States Environmental Protection Agency (U.S. EPA)/Environmental Response Team (ERT) typically follows procedures outlined in the TEM methods for determining mineralogical types of asbestos in air and for distinguishing asbestos from non-asbestos minerals. The PCM method is primarily used as a screening tool since it is less costly than TEM. TEM is used to distinguish asbestos fibers from non-asbestos fibers and characterize asbestos mineral species. The PCM method can be used to estimate asbestos concentrations, but the PCM method cannot distinguish asbestos from non-asbestos fibers. The TEM method, therefore, may be necessary to confirm analytical results. For example, if an action level for the presence of fibers has been set and PCM analysis indicates that the action level has been exceeded, TEM analysis can be used to quantify and identify asbestos structures through examination of their morphology, crystalline structures (through electron diffraction), and elemental composition (through energy dispersive X-ray analysis). In this instance, samples can be collected for both analyses on the same filter or in side by side sampling trains. The direct preparation method is designed specifically to provide results suitable for supporting human health risk assessments at Superfund sites, and the direct and indirect preparation methods are applicable to a wide range of ambient air situations at hazardous waste sites. The PCM and TEM NIOSH analytical methods require lower sample volumes than the ISO methods; however, both NIOSH methods can be adapted for ambient air sampling if the sampling volume is increased appropriately.

Regulations pertaining to asbestos have been promulgated by the U.S. EPA and Occupational Safety and Health Administration (OSHA). The U.S. EPA National Emission Standards for Hazardous Air Pollutants (NESHAP) regulates asbestos-containing waste materials. NESHAP establishes management practices and standards for the handling of asbestos and emissions from waste disposal operations under 40 Code of Federal Regulations (CFR) Part 61, Subparts A and M. Comprehensive rules for the asbestos abatement industry were promulgated under 40 CFR 763. State and local regulations regarding asbestos management and assessment practices vary and may be more stringent than federal requirements. The OSHA regulations in



SOP: ERT-PROC-2015-20 PAGE: 5 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

### **ASBESTOS AIR SAMPLING**

29 CFR 1910.1001 (general industry; formerly designated 29 CFR 1926.58), 29 CFR 1926.1101 (construction industry), and 29 CFR 1915.1001 (shipbuilding industry) specify work practices and safety equipment including respiratory protection and protective clothing when handling asbestos or ACM. The OSHA 8-hour, time-weighted average (TWA) is 0.1 fibers per cubic centimeter (f/cc) of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length greater than 5 microns ( $\mu$ m). Assessment and monitoring of airborne asbestos is required by OSHA to determine if asbestos is present in the workplace and if the work will generate airborne fibers. In addition, employers may be required to establish an employee training program, medical surveillance program, and install engineering or institutional controls in conjunction with an asbestos assessment or monitoring program.

A Quality Assurance Project Plan (QAPP) in Uniform Federal Policy (UFP) format describing the project objectives must be prepared prior to deploying for a sampling event. The sampler needs to ensure that the methods used are adequate to satisfy the data quality objectives listed in the QAPP for a particular site.

The procedures in this SOP may be varied or changed as required, dependent on site conditions, equipment limitations, or other procedural limitations. In all instances, the procedures employed must be documented on a Field Change Form that is attached to the QAPP. These changes must also be documented in the final deliverable.

#### 2.0 METHOD SUMMARY

If possible and prior to sampling, the site should be characterized by identifying on-site and off-site sources of airborne asbestos. The array of proposed sampling locations and the schedule for sample collection are critical to the success of an investigation. In general, sampling strategies to characterize a single point source are fairly straightforward, while multiple point sources and/or area sources increase the complexity of the sampling strategy. Experience, objectives, and site characteristics will dictate the sampling strategy.

During a site investigation, sampling stations should be arranged to support the evaluation of spatial trends in airborne asbestos concentrations, and sampling schedules should be fashioned to establish temporal trends. The sampling strategy typically requires that the concentration of asbestos at the source (worst case), area of concern (downwind), crosswind areas, and background areas (upwind) contributions be quantified. Indoor air asbestos sampling events require a different type of sampling strategy. Consult the site-specific QAPP for all sampling activities. It is critical to establish background levels of contaminants in order to develop a reference point from which to evaluate the source data. Field blanks and lot blanks can be utilized to determine other sources.

Prior to sampling, the site sampling objectives should be identified in the site-specific QAPP to support the selection of the most appropriate analytical method. Additionally, the specific sampling requirements, required equipment and sample preparation, and quality control data quality objectives (DQOs) should be identified in the QAPP prior to sampling. Each analytical method has specific sampling requirements and produce results which may or may not be applicable to a specific sampling effort. Consult with the end line stakeholders, toxicologists and risk assessors in the early stages of project planning in order to determine the best course of action.

The following sampling and analytical protocols may be used for asbestos determination.

• NIOSH 7400 – Determination of Asbestos and Other fibers by Phase Contrast Microscopy.



SOP: ERT-PROC-2015-20 PAGE: 6 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

### ASBESTOS AIR SAMPLING

- ASTM International (ASTM) D7200-12 Sampling and Counting Airborne Fibers, Including Asbestos Fibers, in Mines and Quarries, by Phase Contrast Microscopy and Transmission Electron Microscopy.
- ASTM D7201-06 (Reapproved 2011) Sampling and Counting Airborne Fibers, Including Asbestos Fibers in the Workplace, by Phase Contrast Microscopy (with an Option of Transmission Electron Microscopy.
- International Organization for Standardization (ISO) 8672:2014 Determination of the Number Concentration of Airborne Inorganic Fibres by Phase Contrast Optical Microscopy – Membrane Filter Method.
- NIOSH 7402 Asbestos by Transmission Electron Microscopy.
- Asbestos Hazard Emergency Response Act (AHERA) 40 CFR Part 763 Appendix A to Subpart E Interim Transmission Electron Microscopy Analytical Method.
- ISO Method 10312: Ambient Air Determination of Asbestos Fibres Direct Transfer Transmission Electron Microscopy Method.
- ISO Method 13794: Ambient Air Determination of Asbestos Fibres Indirect Transfer Transmission Electron Microscopy Method.

### 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Preservation

No preservation is required for asbestos samples.

- 3.2 Sample Handling, Container and Storage Procedures
  - 1. Place a sample label on the cassette that indicates a unique sampling number. Do not put sampling cassettes in shirt or coat pockets as the filter can pick up interfering fibers. The original cassette box can be used to hold the samples.
  - 2. Upon completing the sampling, store the cassettes individually in a manila envelope. Each envelope should be labeled with the sample identification number, location, total sampling time, total volume and sampling date.
  - 3. The wrapped sampling cassettes should be placed upright in a rigid container to ensure that the cassettes are oriented vertically (i.e., the caps for the top and bottom align with the top and bottom of the container). Use enough packing material to prevent jostling or damage. Do not use vermiculite as packing material for samples. If possible, hand deliver the samples to the laboratory.



SOP: ERT-PROC-2015-20 PAGE: 7 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

### **ASBESTOS AIR SAMPLING**

4. Provide appropriate documentation with samples (i.e., chain of custody, requested analytical methodology, and other information specific to the laboratory analyzing samples).

#### 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates exceeding 16 liters/minute (L/min) that could result in filter destruction due to (a) failure of its physical support under force from the increased pressure drop; (b) leakage of air around the filter mount so that the filter is bypassed, or (c) damage to the asbestos structures due to increased impact velocities.

The filter and it associated back pressure will limit flow rates that are able to be achieved. When utilizing a 0.8-µm filter cassette higher flow rates are achievable than when using a 0.45-µm filter cassette. To the best extent possible do not operate pumps at their maximum capacity. When calibrating a pump, there is a point at which the maximum flow through a filter is achieved before the pump flow adjustment has been maximized. Even though the pump may sound indicates it is still accelerating, the flow will not increase. In this case turn the adjustment back to the point below maximum draw. Higher than acceptable flow rates can damage the filter and pump. Not all pumps are created equally, even those of identical make and manufacturer. Total sampling times will vary based on battery draw.

For high-volume samples, the filter overload should be no higher 25 percent (%). If a high-volume sample is determined to be overloaded and a lower volume collocated sample was collected, the corresponding low-volume sample can be analyzed. For low-volume samples that are greater than 25% overloaded, the laboratory must have the capability of analyzing these samples using ISO Method 13794 (indirect method).

#### 4.1 Filter Preparation Methods

4.1.1 ISO 10312 Direct-Transfer TEM Specimen Preparation Method

Direct-transfer TEM specimen preparation methods have the following interferences:

- This method cannot discriminate between individual fibers of asbestos and elongate fragments (cleavage fragments and acicular particles) from non-asbestos analogues of the same amphibole mineral.
- Complete identification of every chrysotile and/or amphibole fiber is not possible due
  to instrumental limitations and the nature of some of the fibers. Additionally, complete
  identification of every amphibole fiber is not practical due to the limitations of both
  time and cost.
- On rare occasions, some amphibole fibers can visually be misidentified as chrysotile.
   Particles of other minerals having visual compositions similar to those of some amphiboles could be erroneously classified as amphibole. Use of energy dispersive X-ray analysis (from the method) can eliminate the misidentification.
- The achievable detection limit (DL) is restricted by the particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled.



SOP: ERT-PROC-2015-20 PAGE: 8 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

### **ASBESTOS AIR SAMPLING**

- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.
- Air samples should be collected to ensure that the particulate and fiber loadings are within a specified narrow range. If particulate loading on the filter is too high, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. In this case, even if satisfactory TEM specimens can be prepared, accurate fiber counting may not be possible.

#### 4.1.2 ISO 13794 Indirect TEM Specimen Preparation Method

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified.
- There is increased opportunity for fiber loss or introduction of extraneous contamination.
- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.
- There is a possibility of misidentification of fibers for which both the morphologies and the electron diffraction (ED) patterns are reported on the basis of visual observation only. This can be rectified by the use of energy-dispersive x-ray analysis (EDXA) and the observation of the 0.73 nanometer (nm) reflection of chrysotile in the ED pattern.
- Particles of a number of other minerals may have compositions similar to amphiboles.
   These could be classified as amphibole when the classification criteria does not include zone-axis ED techniques

It can be argued that direct methods yield an under-estimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, indirect methods can be considered to yield an over-estimate of the asbestos structure concentration because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

### 4.2 NIOSH Method for TEM

Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere with the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification.



SOP: ERT-PROC-2015-20 PAGE: 9 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

### ASBESTOS AIR SAMPLING

#### 4.3 NIOSH Method for PCM

PCM cannot distinguish asbestos from non-asbestos fibers; therefore, all particles meeting the counting criteria are counted as total asbestos fibers. Fibers less than  $0.25~\mu m$  in length will not be detected by this method. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the DL.

#### 5.0 EQUIPMENT/MATERIALS

#### 5.1 Sampling Pump

The constant flow or critical orifice controlled sampling pump should be capable of a flow rate and pumping time sufficient to collect the desired air sample volume

The low flow personal sampling pumps generally provide a flow rate of 20 cubic centimeters per minute (cc/min) to 4 L/min, and these pumps are usually battery powered

High-flow pumps are utilized when flow rates between 2 L/min to 16 L/min are required. High-flow pumps are generally used for short sampling periods. A stand should be used to hold the filter cassette at the desired height for sampling and the filter cassette shall be isolated from the vibrations of the pump. Given that the proposed sampling locations may be located in remote areas or a significant distance from a stationary alternating current (AC) power source, consideration of how to provide electrical power for the pumps must be considered prior to sampling. High-flow pumps operate preferentially on AC power, but a generator or an external battery supply can be utilized as an alternative source of power. If a generator is used, the generator should be positioned downwind from the sampling stands to avoid cross contamination or interference with the samples being collected. Voltage and amperage should be closely monitored when running more than one high flow pump using the same power source.

#### 5.2 Filter Cassette

The cassettes are purchased with the required filters in position. A shrink cellulose band or adhesive tape is usually applied to the cassette joints to prevent air leakage.

#### 5.2.1 TEM Cassette Requirements

For NIOSH Method 7402, TEM, commercially available 25-millimeter (mm) diameter two-piece cassette with a conductive extension cowl will be used for sample collection. The cassette must be new and not previously used. The cassette will be loaded with a MCE filter with a pore size between 0.45 to 1.2  $\mu$ m that was supplied from a lot number that was qualified as low background for asbestos determination. The cowls should be constructed of electrically-conducting material to minimize electrostatic effects. The filter will be backed by a 5- $\mu$ m pore size MCE diffuser and support pad (Figures 1 and 2, Appendix A).



SOP: ERT-PROC-2015-20

PAGE: 10 of 25 REV: 1.1

EFFECTIVE DATE: 06/18/20

### **ASBESTOS AIR SAMPLING**

#### 5.2.2 PCM Cassette Requirements

For NIOSH Method 7400, PCM, a 25-mm, 0.45 or 0.8-µm MCE filter cassette (typical pore sizes) with conductive extension cowl will be used for sample collection. The MCE filter will be backed by a support pad (Figures 1 and 2, Appendix A). Some labs are able to perform both the PCM and TEM analyses on the same filter; however, this should be discussed with the laboratory prior to sampling.

ERT typically uses the 0.8-µm MCE filter cassette. Consult with the end line stakeholders, toxicologists and risk assessors in order to determine the best course of action.

### 5.3 Other Equipment

- Sampling trains
- Manila envelopes (#6 coin size preferred) for cassettes
- Tools small screwdrivers
- Container (to keep samples upright)
- Generator or electrical outlet (may not be required)
- Extension cords (may not be required)
- Multiple plug outlet (may not be required)
- Sample labels
- Air sampling data sheets
- Chain of custody records
- Scribe

#### 6.0 REAGENTS

Reagents are not required for the collection or preservation of asbestos samples.

### 7.0 PROCEDURES

#### 7.1 Flow Rates, Air Volumes and Analytical Sensitivity

Occupational Safety and Health Administration (OSHA) method ID-161, *Asbestos in Air*, shows an acceptable flow rate from 0.5 to 5 liters per minute (LPM). National Institute of Occupational Safety and Health (NIOSH) methods 7400, *Asbestos and Other Fibers by PCM* and 7402, *Asbestos by TEM* refer to flow rates ranging from 0.5 to 16 LPM.

These methods were developed for occupational settings where asbestos was expected to be present and would demonstrate the compliance or non-compliance of the OSHA permissible exposure level (PEL) of 0.1 fiber per cubic centimeter.

#### 7.1.1 Occupational Methods

The listed minimum recommended volume for TEM and PCM is 400 L at 0.1 f/cc. The sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for eight hours (480 to 1920 L) is appropriate in non-dusty atmospheres



SOP: ERT-PROC-2015-20 PAGE: 11 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

# ASBESTOS AIR SAMPLING

containing 0.1 f/cc. Dusty atmospheres (i.e., areas with the potential for high levels of airborne asbestos) require smaller sample volumes (sometimes significantly less than 400 L) to obtain countable samples.

If a time-integrated sample is required, when collecting samples in dusty environments, collect short, consecutive samples and average the results over the total collection time.

For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres where targeted fiber concentrations are significantly less than 0.1 f/cc, collect a larger sample volume (3,000 to 10,000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If loading is greater than 20-25% of the filter surface covered with particles, the measured fiber concentration may be biased, or the filter may be too overloaded to count.

#### 7.1.2 Risk-based Methods

OSWER Directive 9345.4-05 recommended the development of risk-based, site-specific air action levels to determine if response actions for asbestos in soil/debris should be undertaken. Because inhalation is the exposure pathway of concern for asbestos, an action (or screening) level for asbestos in air is an appropriate metric for site managers in making the determination of whether a response action, no action, or further, more detailed investigation at a given site is warranted. This led to the development of OSWER's Framework for Investigating Asbestos-Contaminated Superfund Sites in 2008, which recommends the use of ISO 10312.

Sampling volumes will be determined, based on the analytical sensitivity required for a particular task. The sensitivity is defined as the structure concentration corresponding to the detection of one structure by ISO 10312. Sensitivities can vary from project to project and within tasks on a project. The desired sensitivity can be met through a combination of volume collected and number of filter grids counted by the microscopist for the TEM by the direct transfer method. Limit of detection is usually defined as 3 times the analytical sensitivity. Before collecting any samples, consult with the end line stakeholders, toxicologists and risk assessors in the early stages of project planning in order to determine the required analytical sensitivity.

The following example, of number of grids to be counted, is a condensed excerpt from the Code of Federal Regulations Chapter 40 Part 763, Subpart E, Appendix A. In this example the number of grids to be counted will reflect an analytical sensitivity of 0.005 structures per cubic centimeter from a 25-millimeter (mm) filter with an effective area of 385 mm<sup>2</sup>.



SOP: ERT-PROC-2015-20

PAGE: 12 of 25 REV: 1.1

EFFECTIVE DATE: 06/18/20

### ASBESTOS AIR SAMPLING

Volume in	Grid
Liters	Openings
600	23
1200	11
1800	8
2400	6
3000	5
3600	4

Typically, battery-operated (personal) pumps are utilized for sampling at flow rates less than 5 LPM. Battery operated pumps are also capable of collecting samples at flow rates up to 16 LPM. Generally, the higher the flow rate required the heavier the sampling pump. AC operated pumps are also capable of sampling at flow rates up to 16 LPM.

#### 7.2 Calibration Procedures

To determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps are calibrated before and after each use. Preliminary calibration is conducted using a primary or secondary calibration device such as an electronic calibrator or rotameter with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette can be reused for calibrating other pumps that will be used for asbestos sampling. A cassette from the same lot used for sampling should also be used for the calibration. A sticker can be affixed to the outside of the extension cowl marked "Calibration Cassette." The calibration cassette cannot be used as a sampling cassette.

Constant flow calibration readings are obtained before and after sampling. If the flow rate changes by more than 5% during the sampling period, the average of the pre- and post-calibration rates will be used to calculate the total sample volume. The sampling pump used will provide a non-fluctuating air-flow through the filter, and the flow rate should be maintained within 10% of the initial volume flow rate throughout the sampling period. The value of these flow-rate measurements will be used to calculate the total air volume sampled. A constant flow or critical orifice controlled pump meets these requirements. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling may be terminated. It will be determined by the end user of the data if the sample should be analyzed. Flexible tubing is used to connect the filter cassette to the sampling pump. Sampling pumps can be calibrated prior to coming on-site to reduce the amount of time spent performing on-site calibration activities.

### 7.2.1 Calibrating a Personal Sampling Pump with a Rotameter

- 1. For ERT rotameters, perform calibrations following directions established in ERT SOP, *Rotameter Calibration*.
- 2. Set up the calibration train as shown in (Figure 3, Appendix A) using a rotameter, sampling pump, and a representative sampling cassette.
- 3. To set up the calibration train, attach one end of the Tygon tubing (approximately 2 feet) to the cassette cap air inlet or cover open inlet-end of cowl with rubber



SOP: ERT-PROC-2015-20 PAGE: 13 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

### **ASBESTOS AIR SAMPLING**

cassette calibration adapter (shown in figure); attach the other end of the tubing to the air outlet (top fitting) on the rotameter. Another piece of tubing is attached from the cassette base air outlet to the inlet of the sampling pump.

- 4. Ensure that the rotameter is as level as possible when recording sampling flow rates.
- 5. Turn the sampling pump on.
- 6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the pre-calibrated flow rate value. A sticker on the rotameter should indicate this value. Confirm the flow rate after approximately 10 seconds. Adjust flow rate accordingly.
- 7. A verification of calibration is generally performed on-site in the clean zone immediately prior to sampling.

### 7.2.2 Calibrating a Personal Sampling Pump with an Electronic Calibrator

- 1. Refer to the manufacturer's manual for operational instructions. Ensure that the unit has been calibrated within the past year.
- 2. Set up the calibration train as shown in (Figure 4, Appendix A) using a sampling pump, electronic calibrator, a representative filter cassette and Tygon tubing. A cassette from the same lot used for sampling should also be used for the calibration.
- 3. To set up the calibration train remove the air outlet plug of the sampling cassette and attach one end of the Tygon tubing (approximately 2 feet) to the cassette outlet; attach the other end of the tubing to the inlet of the sampling pump. Another piece of tubing is attached from the cassette inlet cap to the inlet of the electronic calibrator or cover open inlet-end of cowl with rubber cassette calibration adapter (shown in figure).
- 4. Turn the electronic calibrator and sampling pump on.
- 5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained. Confirm the flow rate after approximately 10 seconds. Adjust flow rate accordingly.

### 7.3. Meteorology

It is recommended that an onsite, portable, 3-meter meteorological station be established. If possible, sample after two to three days of dry weather when wind conditions are representative for the climatology of the location based on month and time of day. Historical hourly wind speed and wind direction data should be analyzed before mobilization. Wind speed, wind direction, temperature and



SOP: ERT-PROC-2015-20 PAGE: 14 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

### **ASBESTOS AIR SAMPLING**

station pressure should be recorded and real-time data should be available for review. A nearby representative meteorological station, may be used to acquire the necessary data.

#### 7.4 Ambient Sampling Procedures

### 7.4.1 Pre-Site Sampling Preparation

- 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.
- 2. Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).
- 3. Perform a general site survey prior to site entry in accordance with the site-specific Health and Safety Plan.
- 4. Once on-site, perform calibration of the sampling equipment in the clean zone. The calibration procedures are summarized in Section 7.2.
- 5. After calibrating the sampling pump, mobilize to the sampling location.

#### 7.4.2 Site Sampling

- 1. Prior to sampling, assemble the sampling train as illustrated in Figures 5 (Personal Sampling Train for Asbestos) and Figure 6 (High Flow Stationary Sampling Train for Asbestos), Appendix A, by connecting one end of the Tygon tubing to the cassette cap (air inlet); connect the other end of the tubing to the inlet of the sampling pump. The cassette should be positioned downward or at a 45 degree angle.
- 2. If AC or direct current (DC) electricity is required, turn the power on. If used, a generator should be placed at least 10 feet downwind of the sampling pump.
- 3. Turn the pump on. Record the following parameters or air sampling worksheets or in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.
- 5. Check the pump at the sampling mid-point if the sampling duration is longer than 4 hours. If generators are used, they may need to be refueled depending on tank size. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.
- 6. At the end of the sampling period, orient the cassette up, and turn the pump off.
- 7. Check the flow rate of each sampling cassette as described in Section 7.2 before removing from the pump. Note that increased dust/fiber loading may have altered



SOP: ERT-PROC-2015-20 PAGE: 15 of 25

REV: 1.1
EFFECTIVE DATE: 06/18/20

### ASBESTOS AIR SAMPLING

the flow rate.

- 8. Record the post flow rate, cumulative time or pump run sample end time and date.
- 9. Remove the tubing from the sampling cassette. Still holding the cassette upright, replace the cassette cap and the inlet and outlet plugs.

#### 7.4.3. Post Site Sampling

Follow handling procedures described in Section 3.2.

#### 7.5 Indoor Sampling Procedures

The inlet of the sampling pump should be placed at a (breathing) height four to five feet above floor level and away from obstructions that may affect air flow. For example, the pump can be placed on a table or counter. Sensitive receptor areas should be given priority. Refer to the site-specific QAPP for a summary of indoor sampling locations and rationale for selection. The optimum positions for collection of air samples should be determined after a complete survey of the unit is conducted. A sufficient number of samples should be collected in areas where known asbestos materials are present to characterize the unit. Control samples should be collected in adjacent areas where no airborne asbestos would be expected (i.e, near intakes for the air conditioning system). Indoor sampling volumes should consider the analytical sensitivity, in order to meet project goals.

Depending on project needs, indoor sampling may consist of static/normal operation conditions or utilizing disturbed/aggressive procedures.

#### 7.5.1 Aggressive Sampling Procedures

Sampling equipment at fixed locations may fail to detect the presence of asbestos fibers. Due to limited air movement, many fibers may settle out of the air onto the floor and other surfaces and may not be captured on the filter. A quick and effective screening to capture asbestos fibers is to circulate the air artificially so that the fibers remain airborne during sampling. The result from this sampling option typifies a worst-case condition, and this is referred to as aggressive air sampling for asbestos. Refer to the site-specific QAPP and HASP prior to initiation of site activities.

- 1. Before starting the sampling pumps, direct forced air (such as a leaf blower or large fan) against walls, ceilings, floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces. This should take at least 5 minutes per 1,000 square feet of floor area.
- 2. Place a 20-inch fan (or similar piece of equipment) in the center of the room. Place the fan on slow speed and point it toward the ceiling. Use one fan per 10,000 cubic feet of room volume.
- 3. Follow the procedures described in Sections 7.4.1 and 7.4.2. When sampling is completed, turn off the pump first, and turn off the fans second.



SOP: ERT-PROC-2015-20 PAGE: 16 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

#### **ASBESTOS AIR SAMPLING**

4. Follow the handling procedures described in Section 3.2.

#### 8.0 CALCULATIONS

The sample volume is calculated by multiplying the average flow rate of the pump by the number of minutes the pump was running (sample volume = flow rate X time in minutes). The sample volume should be submitted to the laboratory and identified on the chain of custody for each sample. Note that a sample volume of zero will be indicated for lot and field blanks.

The concentration result is calculated using the sample volume and the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

#### 9.0 QUALITY ASSURANCE/QUALITY CONTROL

Specific QA/QC activities that apply to the implementation of these procedures will be listed in the QAPP prepared for the applicable sampling event. The following general quality assurance (QA) procedures also apply:

- 1. All sample collection data, including sample number, sample location, start and end times, start and end flow rates, pump number, media used and analysis/method must be documented on site logbooks or Field Sampling Worksheets.
- 2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer or instrument-specific SOPs, unless otherwise specified in the QAPP. Equipment check-out and calibration is necessary prior to sampling and must be done according to the instruction manuals supplied by the manufacturer.
- 3. Records must be maintained, documenting the training of the operators that use instrumentation and equipment for the collection of environmental information.

The following quality control (QC) requirements are applicable:

#### 9.1 TEM Requirements

- 1. Examine lot blanks to determine the background asbestos structure concentration.
- 2. Examine field blanks to determine if there is the presence of contamination associated with extraneous asbestos structures introduced during specimen preparation.
- 3. Examine laboratory blanks to determine if contamination was introduced during critical phases of the laboratory program.
- 4. To determine if the laboratory can satisfactorily analyze samples of known asbestos structure concentrations, reference filters can be examined. Reference filters can be maintained as part of the laboratory's QA program.



SOP: ERT-PROC-2015-20 PAGE: 17 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

#### **ASBESTOS AIR SAMPLING**

- 5. To minimize subjective effects, some specimens should be recounted by a different microscopist.
- 6. It is preferred that asbestos laboratories be accredited by the National Voluntary Laboratory Accreditation Program (NVLAP).
- 7. At this time, performance evaluation samples for asbestos in air are not available for Removal Program Activities.

#### 9.2 PCM Requirements

- 1. Examine reference slides of known concentration to determine the analyst's ability to satisfactorily count fibers. Reference slides should be maintained as part of the laboratory's quality assurance program.
- 2. Examine field blanks to determine if there is the presence of contamination associated with extraneous structures introduced during sample handling.
- 3. Some samples should be relabeled then submitted for counting by the same analyst to determine possible bias by the analyst.
- 4. Participation in a proficiency testing program such as the American Industrial Hygiene Association (AIHA)-NIOSH proficiency analytical testing (PAT) program is recommended.

#### 10.0 DATA VALIDATION

Data verification (completeness checks) must be conducted to ensure that all data inputs are present for ensuring the availability of sufficient information. This may include but is not limited to: location information, start and end times, sampling method and total volume sampled. These data are essential to providing an accurate and complete final deliverable. The contractor's Task Leader (TL) is responsible for completing the UFP-QAPP verification checklist for each project.

Results of the QA/QC samples will be evaluated for contamination during the data validation process. This information will be utilized to qualify the environmental sample results accordingly with the data quality objectives of the project.

#### 11.0 HEALTH AND SAFETY

Based on Occupational Safety and Health Administration (OSHA) requirements, a site-specific health and safety plan (HASP) must be prepared for response operations under the Hazardous Waste Operations and Emergency Response (HAZWOPER) standard, 29 CFR 1910.120. Field personnel working for EPA's ERT should consult the Emergency Responder Health and Safety Manual currently located at <a href="https://response.epa.gov/">https://response.epa.gov/</a> HealthSafetyManual/manual-index.htm for the development of the HASP, required PPE and respiratory protection.



SOP: ERT-PROC-2015-20 PAGE: 18 of 25

EFFECTIVE DATE:

REV: 1.1

06/18/20

#### **ASBESTOS AIR SAMPLING**

#### 12.0 REFERENCES

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#### 13.0 APPENDICES

A - Figures



SOP: ERT-PROC-2015-20

PAGE: 19 of 25 REV: 1.1

EFFECTIVE DATE: 06/18/20

#### **ASBESTOS AIR SAMPLING**

APPENDIX A
Figures
SOP: ERT-PROC-2015-20
June 2020



SOP: ERT-PROC-2015-20

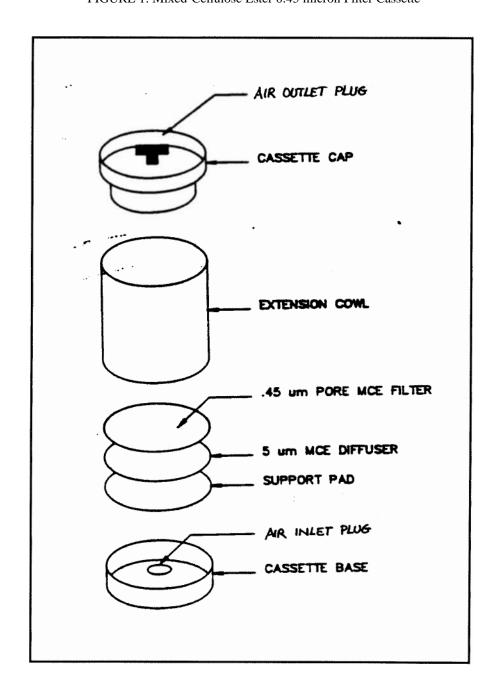
PAGE: 20 of 25

REV:

1.1 06/18/20

EFFECTIVE DATE:

FIGURE 1. Mixed-Cellulose Ester 0.45 micron Filter Cassette





SOP: ERT-PROC-2015-20

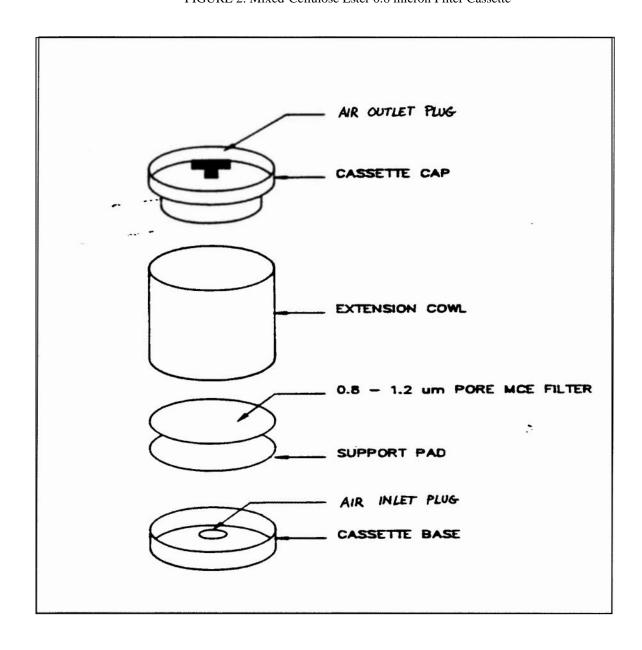
PAGE: 21 of 25

REV:

1.1 06/18/20

EFFECTIVE DATE:

FIGURE 2. Mixed-Cellulose Ester 0.8 micron Filter Cassette

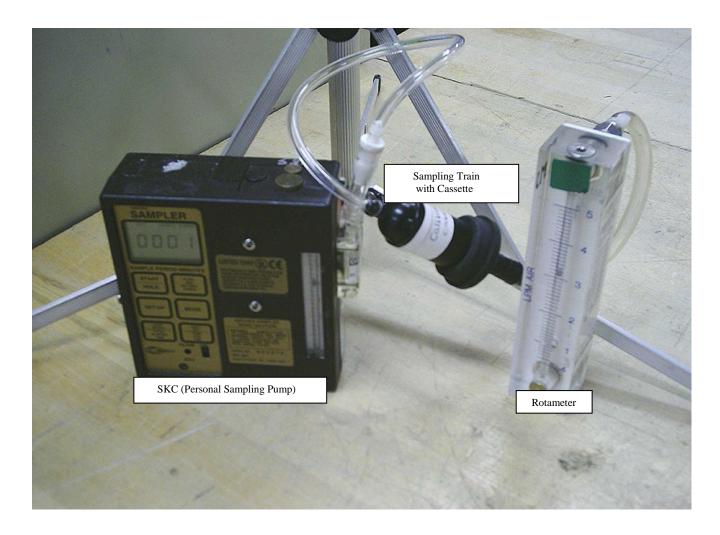




SOP: ERT-PROC-2015-20 PAGE: 22 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

FIGURE 3. Calibration of Personal Sampling Pump Using a Rotameter



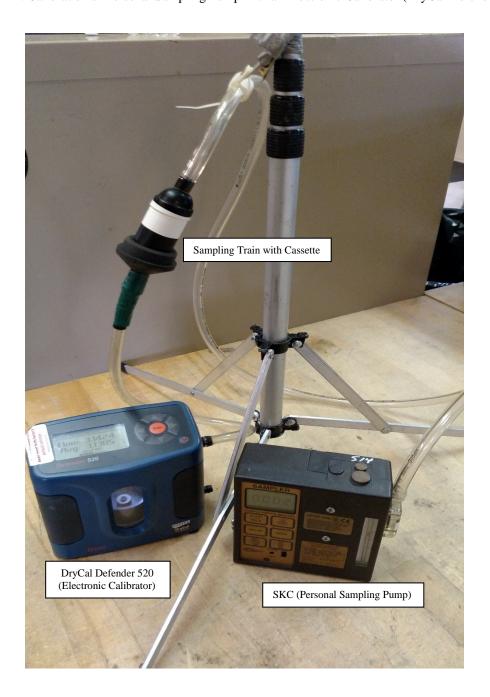




SOP: ERT-PROC-2015-20 PAGE: 23 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

FIGURE 4. Calibration of Personal Sampling Pump with an Electronic Calibrator (DryCal Defender 520)





SOP: ERT-PROC-2015-20

PAGE: 24 of 25

REV: 1.1 EFFECTIVE DATE: 06/18/20

FIGURE 5. Personal Sampling Train for Asbestos



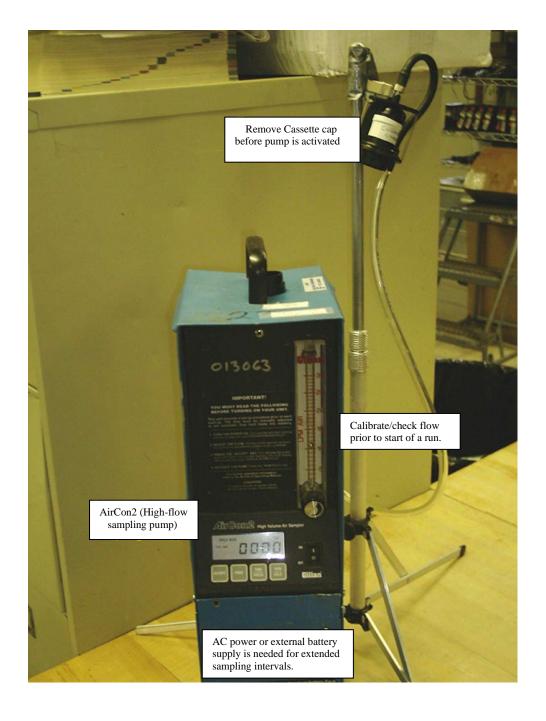


SOP: ERT-PROC-2015-20

PAGE: 25 of 25 REV: 1.1

EFFECTIVE DATE: 06/18/20

FIGURE 6. High Flow Stationary Sampling Train for Asbestos



#### **ATTACHMENT 2**

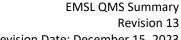
LABORATORY STANDARD OPERATING PROCEDURES, QUALITY ASSURANCE MANUALS, AND CERTIFICATIONS



# EMSL Analytical, Inc.

**Quality Management Program Summary** 

Based on Rev. 26 of the Quality Management System (QMS) Manual





Revision Date: December 15, 2023

The quality program at EMSL is built on a commitment to quality and continued improvement. This program is a primary part of our everyday work; developed, utilized, and maintained by all the dedicated staff at EMSL.

#### Introduction

This program outline presents an overview of the quality assurance program. It provides the reader with a summary of EMSL laboratory policies and procedures as they relate to the technical elements of the corporate quality objectives.

This program follows quality guidelines as documented by ISO/IEC 17025:2005/2017, the American Industrial Hygiene Association Laboratory Accreditation Program (AIHA LAP, LLC), the EPA's National Voluntary Laboratory Approval Program (NVLAP), The NELAP Institute (TNI), A2LA, Canadian Association for Laboratory Accreditation (CALA), Analytical Laboratory Accreditation Program (PALA), and other applicable state and federal regulatory agencies.

This QA program is designed to ensure the highest level of quality professional services and technical excellence is provided to our customers. This is accomplished by the implementation of program policies including:

- Development of company standard quality control programs
- Standardization of reporting formats
- The monitoring of laboratory QC performance
- Providing technical training for all staff levels
- Achieving traceability of data
- Performance of internal quality audits
- Participation in applicable accreditation programs
- Participation in third party proficiency testing programs

The objectives of these program policies ensure the quality, accuracy and integrity of our analytical data.

The quality assurance objectives, policies and procedures are formally documented in the EMSL Quality Management System Manual and program-specific Modules. A summary of this manual is presented on the following pages.

#### Topics covered are:

1. Or	ganization	Structure

2. Document Control

3. Purchasing

4. Complaint Handling

5. Corrective and Preventive Action

6. Control of Records

7. Internal and External Audits

8. Management Reviews

9. Personnel and Training

10. Lab Conditions

11. Equipment Calibration

12. Measurement Traceability

13. Sample Handling

14. Data Quality Programs

15. Ethics

16. Customer Communication

17. Notice of Performance

18. Estimate of Analytical Uncertainty

19. Addressing Risk and Opportunities



#### **EMSL Quality Policy Statement**

EMSL is committed to providing a high standard of service, and to producing dependable, accurate and technically defensible test results, in order to best serve our customers. EMSL will avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgment, or operational integrity. Our experienced and qualified technical personnel are committed to providing data of the highest quality achievable.

The senior management of EMSL Analytical, Inc. is committed to adopting the quality standards utilized by the various accrediting authorities (e.g., NVLAP, AIHA LAP LLC, A2LA, CALA, PALA and other State and Federal authorities), and those requirements documented in the ISO/IEC 17025 and TNI standards. The major goal of the laboratory and its personnel will be toward constant improvement in the quality management system, which has been designed with the purpose of ensuring consistent operations leading to quality data.

The senior management staff of EMSL acknowledges and accepts the responsibility for the overall quality of the data produced by the laboratory, and makes a commitment toward continual improvement of the final product and the management system. In doing so, management provides the laboratory manager and the Quality Assurance Department with full authority to accomplish this end. Management is committed to providing all of the resources necessary to provide high quality analytical data.

All personnel concerned with testing within the laboratory must familiarize themselves with the quality documentation, and must implement the policies and procedures addressed in the EMSL Quality Management System Manual.

Commitment to ISO Standard

 Starting with corporate management, and extending to regional and local laboratory management, EMSL is committed to ensuring the standards documented in ISO/IEC 17025:2005/2017 are upheld in all aspects of company affairs.

By way of authority, it is corporate management who implements, maintains and monitors compliance.

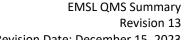
This statement is issued under the authority of company President, Peter Frasca, Ph.D.

#### 1. Organization Structure

The corporate headquarters of EMSL Analytical, Inc. operates out of the Cinnaminson, NJ location. The corporate management oversees the laboratory operations located there, as well as the branch laboratory locations. Corporate headquarters are responsible for the management of all company activities.

EMSL's approximately 46 branch laboratories (as well as the laboratory located in Cinnaminson, NJ) perform the company's analytical services. They report to the corporate headquarters on quality control, productivity, staffing and market issues. Each branch laboratory holds specific accreditations relevant to market requirements and the scope of their analytical work. A copy of each branch lab's qualifications is available on the EMSL Analytical website.

The Quality Program for EMSL Analytical, Inc. is established and maintained by the corporate Quality Assurance Department, and implemented at the lab level by local branch lab managers and quality control personnel. All changes to corporately issued procedures, processes and policies must be approved by the QA Department and/or executive management of EMSL Analytical, Inc. Changes are controlled to ensure affected personnel are notified and the changes do not impact EMSL's compliance with quality standards, regulations and accreditation requirements.





Revision Date: December 15, 2023

Due to this multi-laboratory structure, the EMSL Quality Program is in a constant state of review by outside assessors from multiple agencies. With outside assessments of the quality program occurring on average at least once per month, the program has been thoroughly vetted, and is always in a state of continual improvement.

EMSL Analytical goes by three additional DBA names: LA Testing, operating in Southern California; Advanced MicroAnalytical, a materials testing lab in Salem, New Hampshire; and MPL Laboratories, a GMP-microbial lab in Sparta, New Jersey. The policies and procedures documented in this manual apply to all facilities, including those doing business as LA Testing and Advanced MicroAnalytical. MPL Laboratories operates under its own QMS Manual, and is not covered by this manual.

#### 2. Document Control

In order to prepare and distribute documents in an organized fashion, and ensure the most up-to-date documents are available to the end user, procedures for initiation, preparation, review, approval and distribution of controlled copies have been established. EMSL's document control program is a coordinated effort involving both technical review and custodial control. Laboratories are to use only approved, controlled and current documents for calibrations, analyses, final reports, and other activities performed in our laboratories. Most documents are controlled at the corporate level on EMSL's E-Link SharePoint site, and may not be altered by branch laboratories without permission of corporate management. This ensures consistent operations across all EMSL branch laboratories and minimizes the risk for local inconsistencies. The document control system that has been established has proven to be effective, efficient and sustainable.

#### 3. Purchasing

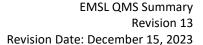
Quality results begin with supplies and services that meet necessary quality specifications. Vendors utilized by EMSL are selected based on the establishment of their own quality programs, their reputation in the industry, as well as historic performance as an EMSL service/supply provider. Supplies and services are ordered from approved vendors, and any non-conforming product received from these vendors is isolated and reported to both laboratory management and the purchasing department. Vendors are evaluated on a regular basis to ensure products and services provided continue to meet EMSL expectations, and vendors are contacted for corrective action if products are found to be unacceptable.

#### 4. Use of Customer Feedback

Customer feedback, both positive and negative, provides consistent input to the evaluation of the effectiveness of EMSL's quality program. Customer feedback is solicited through the use of customer surveys, as well as during routine communications with our customers. This data feeds directly into our continual improvement processes, such as corrective and preventive actions and management reviews. Feedback is considered a crucial aspect of improving our quality program, which the QA Department oversees. Laboratory personnel contacts customers regarding final resolution of any complaints.

#### 5. Corrective and Preventive Actions

The heart of EMSL's continual improvement process is the use of corrective and preventive actions to identify areas for improvement. Inputs to the corrective and preventive action programs come from all other quality systems implemented in the lab including, but not limited to, audit findings (internal and external), complaints, QC results, and customer and employee suggestions. The corrective action process includes an evaluation of a problem, a root cause analysis, and then the selection of corrective actions necessary to prevent recurrence. This investigation includes evaluation of risk created from the situation, and an opportunity for improvement, to determine the appropriate plan to avoid recurrence. Preventive actions are improvements based on prospective problems that may arise in order to ensure they never occur. In both cases, follow-up actions ensure the actions taken have proven effective. EMSL also reviews corrective actions for trends which may indicate the need for





further root cause analysis to improve systems across the entire EMSL enterprise. Corrective and preventive action records are reviewed by management during annual management reviews.

#### 6. Control of Records

EMSL recognizes the importance of maintaining accurate records in a manner that prevents degradation, allows for timely retrieval, and protects customer confidentiality. Policies and procedures for controlling records have been established for implementation at both the lab and corporate level. These policies include general requirements for how changes to records must be handled, confidentiality of records, and how records are to be stored. Each branch laboratory is responsible for how these policies are implemented in their laboratory based on the size, scope and volume of records generated. Electronic records are backed up according to established procedures to ensure they are retrievable in case of a computer error, and where offsite storage of archived records is necessary, storage is contracted with established record management services. Tape backups of many crucial electronic records are also stored offsite to prevent loss in case of an unexpected disaster at the corporate headquarters. EMSL has also established a standard 5-year record retention schedule; although some types of analyses require longer retention times as a function of accreditation requirements, or through customer agreement.

It is understood confidentiality and proprietary rights must be respected throughout the performance of services for any customer, or for those that may include national security concerns. Information will not be given to those for whom it is not intended, and the proprietary rights of our customer will be protected. Data reports and/or other related information will not be given out to any person or agency other than the customer, unless we have received prior approval from the customer.

#### 7. Internal and External Audits

EMSL's quality processes and procedures are continually being audited. One of the benefits of having multiple labs is each is accredited independently, and therefore, each lab audit contains a review of the quality system. Between all branch labs, the EMSL Quality System is being assessed by a 3<sup>rd</sup> party approximately once per month. Non-conformities noted in these audits are evaluated, and where necessary, quality system improvements are implemented for the company as a whole. As a result, the program is continually being improved, and over time has become more efficient and effective. External audit findings are directed to the corporate Quality Assurance Department and, therefore, systematic defects are more easily identified and corrected.

In addition to external assessments of the quality program, EMSL also has established policies and procedures for conducting internal audits of each laboratory. Internal audits use checklists similar to those used by outside agencies, but with additional information on how to audit EMSL specific systems. Findings from internal audits go through the same corrective action process as external findings. Internal audit findings and responses are forwarded to the corporate QA department, and are reviewed by corporate management as part of the annual management review process.

#### 8. Management Reviews

The management team conducts annual management reviews for each branch laboratory for the previous year. Input to the management reviews include information from monthly and quarterly quality reports, results from internal and external audits, corrective and preventive actions, results from lab participation in proficiency testing programs and inter-lab round robin exchanges, summaries of customer feedback, and input on resources and staff training. From this input, a management review report is generated which documents management's findings on the suitability of policies and procedures currently in place, and makes recommendations on actions that need to be completed by the branch laboratory or corporate management. Follow-up is conducted on these recommendations for improvement to ensure they have been carried out.



#### 9. Personnel and Training

The EMSL Quality Program has established minimum requirements for the technical personnel hired by the laboratory. In addition, analysts must complete an EMSL training program in order to perform analysis independently. EMSL provides in-house training pertinent to areas of analysis. Laboratory managers are responsible for ensuring appropriate training is provided to every analyst, and they are completely qualified to perform analysis, including demonstrating competency with the methods used.

In addition to initial training for lab personnel, ongoing evaluations of competency are conducted at least annually (some areas require 6-month evaluations) for each analyst. Where it is determined additional training may be beneficial, this will be conducted immediately. These evaluations will include review of routine QC, trends in analyst performance, performance in proficiency testing and/or blind sample rounds, and other data. This ongoing evaluation process ensures the performance of EMSL analysts meet expectations at all times.

#### 10. Lab Conditions/Environmental Monitoring

EMSL has established processes and procedures for ensuring lab spaces are free from contamination which could jeopardize the health of its employees, customers, or affect the quality of results. In addition to general good housekeeping routines, quarterly monitoring is performed in the lab areas to ensure there is no contamination above acceptable levels. In addition, blank samples are run alongside most analyses which may be affected by cross-contamination. Whenever contamination is detected or suspected, corrective actions will be implemented and documented before re-evaluating to ensure the corrections were effective.

In addition to contamination, lab areas will be monitored for temperature, humidity, etc., where such conditions may have an affect on method performance or stability of samples. Refrigerators, freezers and incubators are monitored according to defined criteria to ensure they are performing as expected. These checks are maintained in laboratory logs and referenced in lab records, as appropriate.

In some more specialized areas, additional precautionary design features may be in place, such as UV lighting, additional access controls, and continual monitoring systems.

#### 11. Equipment Calibration

EMSL has established calibration programs for instrumentation which may affect the quality of results. These calibration programs define the frequency, parameters and acceptance criteria for equipment calibrations and calibration verifications. Standards used in in-house calibrations and routine calibration verifications are all traceable to NIST, and come from approved providers. Where external calibrations are performed, these are performed by approved calibration services accredited to ISO 17025:2005/2017 when available, and calibration reports must comply with the requirements of that standard. Equipment found to fail performance criteria must be either repaired and re-calibrated, or taken out of service.

#### 12. Measurement Traceability

EMSL processes and procedures are established to ensure measurement traceability to recognized standards whenever such standards are available. NIST traceable standards are mandatory for EMSL laboratories whenever available. Where no NIST-traceable standard exists, alternative standards may be considered, but shall come from sources reviewed and approved by EMSL management (e.g., non-NIST sources such as ATCC, or where no external source is available, consensus standards such as graded proficiency testing samples). Calibrations shall be performed using traceable standards, so equipment performance is traceable to NIST or other approved source. Instruments shall be identified in lab records for the analysis, along with standards used in calibration to ensure final measurement results are traceable to the original standards.



#### 13. Sample Handling

#### **Chain of Custody**

In order to ensure the integrity of any sample, records of its custody must be maintained throughout the sample collection in the field, acknowledgement of receipt, acceptance by the laboratory, and through analysis.

In general, EMSL does not perform sampling for customers, nor is it present at the time of sampling, and as a result, cannot be responsible for issuing a chain of custody at the time of sampling. However, the laboratory can advise customers regarding sampling requirements (sampling materials, recommended sampling volumes, packaging, instructions for shipping, etc.) and chain-of-custody, and recommends customers use available EMSL chain-of-custody forms whenever possible.

Once the sample is accepted for analysis by the laboratory, the EMSL "Internal Chain of Custody" is used to document the handling of the samples throughout the analytical process.

#### Sample Acceptance Criteria

In addition to acknowledgement of the receipt of samples, samples must also be accepted for analysis. Prior to accepting samples, the person preparing the samples for analysis inspects them to determine if they conform to laboratory acceptance criteria. If they do not, or if this person has any question as to the validity of the sample, the laboratory manager or an analyst trained to analyze such samples will determine whether the questionable circumstance is sufficient to cause rejection. Rejections of samples are to be followed up by immediate notification to the customer with an explanation. The sample will be returned upon request.

#### Login

Information is entered for samples received into the Laboratory Information Management system (LIMS). LIMS is a computer laboratory information management system which serves to track samples from receipt through the analysis, reporting, and billing processes. Samples are tagged with a project number label at this time to ensure they cannot be separated from others in the project.

#### **Archival and Disposal of Samples**

Once the analysis is complete and the analysis worksheet is signed, the analyst stores the sample in the appropriate storage area as defined in relevant SOPs. All storage containers are to be stored in a safe manner for the period indicated for that category of waste, and in accordance with regulatory requirements for sample retention.

Samples which are not completely consumed in analysis are retained as detailed in the EMSL QMS Manual.

#### 14. Data Control Programs

The ultimate purpose of the EMSL Quality Program is to ensure "Final Test Reports," which are EMSL's final product, are reliable and defensible within the confines of the scope of work. Data is checked through a number of interrelated programs throughout the process, from receipt to final approval.

Checks on lab and analyst proficiency are conducted using the following tools:

- External proficiency testing programs
- Round Robin programs
- Blind sample re-analysis
- Routine QC samples for evaluating accuracy and precision against established control and acceptance limits
- Analysis of data over time using control charts



Any out-of-specification result in a graded proficiency test or round robin will be evaluated, and corrective action performed, as appropriate.

Specific quality control requirements are established in the QMS Manual and SOPs. These requirements are established by corporate management and the QA department and are implemented by lab managers. In addition to quality samples such as blanks and verifications analyzed to ensure accuracy of a particular run, interand intra-analyst reanalysis is performed, where appropriate, to verify accuracy and precision of each analyst. QC data is charted over time against established warning and acceptance limits in order to identify potentially significant trends which may lead to non-conformities. Whenever QC results are out-of-specification, this shall be evaluated by the analyst and lab management. Corrective action is taken where necessary.

In addition, for each project there is a continuous data review process culminating in a review of the final report, usually by a second independent individual. At each stage, the information on the chain of custody and internal chain of custody is reviewed for errors, and any errors are corrected. The final report review includes a thorough review of both completeness and accuracy. A check on any QC analysis performed in association with the samples may be completed to ensure QC passed, or the final report is adequately notated. If any errors or suspected errors are identified, this will be discussed with the analyst prior to release of the final report.

#### 15. Ethics

One of the objectives of the quality assurance program is to ensure the staff of EMSL is provided information regarding ethics as they pertain to corporate policy. The goals of the EMSL ethics policy and ongoing training program are:

- For each staff member to understand the responsibility to provide true and accurate information
- To understand the consequences of unethical conduct
- Provide direction to employees regarding prevention of unethical conduct
- Define right and wrong (as it is job related)
- To ensure all employees are free of undue pressures

EMSL's laboratory activities have been evaluated and designed to avoid bias. Impartiality is maintained through peer review and training. Supervisory staff, in additional to the QA Department, emphasize the importance of reporting results without bias on a routine basis.

EMSL's Conflict of Interest statement is included in Appendix B of the QMS Manual.

#### 16. Customer Communication

Clear, continuous and open communication between the laboratory and the customer is one of the keys to maintaining a successful, quality operation. Communication is established prior to the start of any work. Information must be clearly understood between laboratory management and the customer. EMSL provides quality assurance information and technical support to the customer to ensure continued quality service. The support and information provided in relation to the work performed includes:

- Field sampling guides
- Availability of pertinent QC records
- Access to the Quality Assurance Department for technical assistance
- Security of data (confidentiality), including information relayed to a third party
- Reasonable access to the relevant areas of the laboratory for the witnessing of analysis



If a major deficiency in policy or procedure is identified which directly affects customer results, the customer will

#### 17. Notice of Performance

be notified immediately of the problem.

The laboratory manager shall provide the customer with information as it relates to the performance of the analysis and turnaround time, where necessary. The laboratory shall notify the customer if:

- Analysis cannot be performed on time
- Integrity of the sample has been jeopardized (either by the laboratory or the customer)
- A discrepancy in the analysis has been found during QC analysis

#### 18. Estimate of Analytical Uncertainty

When requested by the customer or required by regulatory agency, an estimate of analytical uncertainty can be provided with customer results. Generally, the uncertainty will be reported at a confidence interval of 95%. It should be noted this is only analytical uncertainty, and does not cover those contributors to uncertainty that may arise from sample collection activities.

#### 19. Addressing Risk and Opportunities

EMSL has implemented procedures and policies to mitigate risk. The quality system is designed to provide the necessary resources for employees to perform their job and limit risk. Should undesired outcomes arise, EMSL's non-conformance work policy will be followed. Any questionable judgements are evaluated by an Ethics Committee.



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EMSL Analytical, Inc. Management



EMSL

Revision Date: September 30, 2021 Effective Date: October 25, 2021

# **EMSL Analytical S.O.P.**

#### ISO 10312

Ambient air - Determination of asbestos fibres - Direct-transfer transmission electron microscopy method

#### [ASB-SOP-417]

#### 1.0 Method Description

**1.1** Applicable Matrix: This method is appropriate for air samples only.

#### 1.2 Scope and Application

This method is for the determination of asbestos concentrations in air samples by transmission electron microscopy (TEM); it is applicable to the determination of airborne asbestos in a wide range of ambient air situations, both exterior and interior. The range of concentrations which can be determined is 50 structures/mm² to 7000 structures/mm² on the filter.

#### 1.3 Summary of Method

Samples are collected on a membrane filter, prepped via a direct prep method, and analyzed with an electron microscope at ≈20,000 X magnification. Asbestos structures are identified by a combination of morphology, elemental chemistry via Energy Dispersive X-Ray Analysis (EDXA), and Selected Area Electron Diffraction (SAED). All structures ≥0.5 micron in length, with at least a 5:1 aspect ratio, are counted and recorded. Structures are differentiated as primary or total structures; a primary structure may be composed of numerous total structures. The report includes a breakdown of fibers and bundles > 5 microns, as well as true PCM equivalent fibers (>5microns in length, diameter between 0.2 and 3 microns, and a 3:1 aspect ratio). Unless the client specifies otherwise, the lab will attempt to reach an analytical sensitivity of 0.005 structures/cc.

#### 1.4 Detection Limit

The analytical sensitivity is defined as the calculated airborne asbestos structure concentration in asbestos structures per Liter, equivalent to the counting of 1 asbestos structure in the analysis. The analytical sensitivity can be lowered by increasing the volume of air collected, and also by increasing the area of the filter analyzed. The Limit of Detection is defined by this method as the upper, one-sided 95% confidence limit of the Poisson distribution for a count of 0 structures. In the absence of background contamination, this is equivalent to 2.99 asbestos structures. The limit of detection can be lowered by collecting a larger initial volume of air, or by analyzing additional surface area of the filter (grid openings).

#### 2.0 Interferences

Interferences for this method include, but are not limited to:

- 2.1 Non-regulated asbestos minerals, such as the two polymorphs of Chrysotile, Lizardite and Antigorite
- 2.2 Non-regulated amphiboles, such as winchite and richterite, and pyroxenes
- **2.3** Cleavage fragments of the regulated asbestos types, which may at times have aspect ratios similar to the true asbestiform varieties
- 2.4 Clay minerals that can have similar morphology to asbestos, such as sepiolite and pallygorskite
- 2.5 All non-asbestos particulate, fibrous or not, which can partially or wholly obscure asbestos fibers

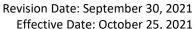


Revision Date: September 30, 2021 Effective Date: October 25, 2021

#### 3.0 Definitions

- 3.1 Analytical Sensitivity: The airborne concentration represented by one asbestos structure counted under the electron microscope. The air volume collected and the proportion of the filter examined determine the analytical sensitivity. There is no set target A.S. with this method. If not directed otherwise by the client, EMSL will attempt to reach an A.S. of 0.005 s/cc up to 10 G.O. unless the method stopping rule of 100 asbestos structures is reached. A minimum of 4 grid openings must be analyzed.
- **3.2 Asbestos:** Generic term for a group of hydrated mineral silicates
- **3.3 Aspect Ratio:** The ratio of the length to the width of a particle. Minimum aspect ratio for this method is 5:1 (3:1 for optional PCMe phase of analysis).
- **3.4 Bundle:** A grouping of apparently attached parallel fibers. A structure composed of parallel, smaller diameter fibers attached along their lengths. A bundle may exhibit diverging fibers at one or both ends.
- **3.5 Cluster:** A structure in which 2 or more fibers or fiber bundles are randomly oriented in a connected grouping. There are two types of clusters; disperse and compact.
- **3.6 Compact Cluster:** Complex and tightly bound network in which one or both ends of each individual fiber or bundle is (are) obscured, such that the dimensions of individual fibers and bundles cannot be unambiguously determined. As such, no subcomponents or residuals will be listed.
- **3.7 Compact Matrix:** A structure consisting of one or more particles, greater than 0.5 microns, in which fibers or bundles can be seen either within the structure or projecting from it, such that the dimensions of individual fibers and bundles cannot be unambiguously determined. As such, no subcomponents or residuals will be listed.
- **3.8 Disperse Cluster:** A disperse and open network in which at least one of the individual fibers or bundles can be separately identified, and its dimensions measured
- 3.9 Disperse Matrix: A structure consisting of one or more particle greater than 0.5 microns, with overlapping or attached fibers or bundles, in which at least one of the individual fibers or bundles can be separately identified and measured
- **3.10 EDXA:** Energy Dispersive X-ray Analysis
- **3.11 Fiber:** An elongated particle which has parallel or stepped sides. For this method, it must have a minimum length of 0.5 µm and an aspect ratio of 5:1 or greater.
- **3.12** Fiber Bundle: See 'bundle'
- **3.13 Grid:** A thin metal (usually Cu) foil with openings that the sample is mounted on to aid in its examination in the TEM
- **3.14 Grid Opening (GO):** One opening of the grid. For this method, the area of the grid opening must be known.
- **3.15 Matrix:** One or more fibers or fiber bundles attached to or partially concealed by a single particle or group of overlapping non fibrous particles. There are two types of matrices; disperse and compact.
- **3.16** MCE Filters: Mixed Cellulose Ester filter
- **3.17 PC Filters:** Polycarbonate filters
- **3.18 PCM Equivalent Fiber:** A fiber of aspect ratio 3:1 or greater, longer than 5 microns, with a diameter between 0.2 and 3 microns
- **3.19 PCM Equivalent Structure:** A fibrous structure of aspect ratio 3:1 or greater, longer than 5 microns, with a diameter between 0.2 and 3 microns
- **3.20 Primary Structure:** A fibrous structure that is a separate entity in the TEM image, and shall be recorded as one of the fundamental structures; fiber, bundle, cluster, or matrix







- **3.21** Residual: A localized group of fibers that remain after the 5 largest prominent features of a primary structure has been documented. Residuals are named cluster residual (CR) or matrix residual (MR), depending on the identity of the primary structure. Record up to 5 residuals for each primary structure.
- 3.22 SAED: Selected Area Electron Diffraction
- 3.23 Structure: A single fiber, fiber bundle, cluster, or matrix which may contain asbestos
- **3.24 TEM:** Transmission Electron Microscopy
- 3.25 Total Structure: Structure or structures associated with the primary structure. Total structures are enumerated separately from primary structures, and are used to classify and measure the important features of each primary structure.

#### Safety 4.0

All personnel performing preparation and/or analysis of samples must be familiar with the EMSL Chemical Hygiene and Safety Plan. Specific hazards and precautions associated with this analysis include:

#### **Asbestos**

- 4.1.1 Prudent measures must be taken to prevent any possible airborne asbestos fiber release from occurring during sample handling.
- Any filter handling performed prior to the filter collapse step should be performed under the 4.1.2 safety hood.

#### 4.2 Acetone

- 4.2.1 Keep away from heat, sparks, and flame.
- **4.2.2** Avoid breathing vapors; use with adequate ventilation.
- 4.2.3 Avoid contact with eyes.
- 4.2.4 Prevent prolonged or repeated contact with skin.

#### 4.3 Carbon Spark

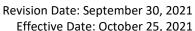
Shield eyes from the glow of the spark during the carbon coating process with welder's type goggles or similar protection.

#### 4.4 Oxygen

- 4.4.1 The oxygen used with the plasma asher is an explosion hazard. Use only vacuum pumps filled with non-hydrocarbon (Fomblin) oil.
- Keep oxygen tank strapped to wall at all times. 4.4.2

#### **Equipment and Supplies**

- ≤0.45 micron MCE or ≤0.4 micron PC filters 5.1
- 5.2 Glass Petri dishes
- 5.3 Glass microscope slides
- Low temperature plasma asher
- 5.5 Vacuum evaporator (carbon coater)
- 5.6 Graphite or carbon rods
- 5.7 **HEPA** laminar flow hood
- 5.8 Grids; copper, gold
- 5.9 Fine forceps
- **5.10** Grid clips, grid storage boxes
- **5.11** Jaffe wick or sponge
- **5.12** Kimwipes or alternative paper





**5.13** Transmission Electron Microscope with the following capabilities:

- **5.13.1** 100 Kev
- **5.13.2** Fine probe size <250 nm
- 5.13.3 Elemental chemistry via X-Ray detector

#### 6.0 Reagents and Standards

All reagents should be of recognized <u>analytical grade</u> or better:

- **6.1** Acetone
- **6.2** Di-Methyl Formamide (DMF)
- **6.3** Glacial Acetic Acid
- 6.4 NIST SRM 1876b
- **6.5** NIST SRM 2063
- **6.6** Albite standard
- **6.7** Aluminum-coated copper grid standard
- **6.8** Gold-coated grid standard
- 6.9 Magnification (cross grating replica) calibration standard
- **6.10** NIST-traceable asbestos on grids
- **6.11** Non-asbestos standards on grids
- **6.12** Ethylenediamine
- 6.13 1-methyl 2-pyrrolidone

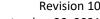
#### 7.0 Sample Collection, Preservation, Shipment and Storage

- 7.1 Samples are collected on  $\leq$  0.45 micron Mixed Cellulose Ester (MCE) or  $\leq$  0.4 micron Polycarbonate (PC) filter cassettes (EMSL recommends MCE filters).
- 7.2 Air flow should be between 2.4 LPM and 15 LPM (not to exceed 10 LPM recommended).
- **7.3** Typically, 1800 liters or more are collected. The total volume that can be collected will be partially determined by the air being sampled.
- **7.4** No sample preservation is needed, and samples can be stored indefinitely prior to analysis.
- **7.5** Samples are best transported to the lab by hand. When mailing, try to package samples carefully to minimize disturbance and possible dislocation of particulate from the filter surface. Use packing materials that will minimize static charge.
- **7.6** All air cassettes must be retained in an easily retrievable manner for a minimum of 60 days.
- **7.7** All samples prepped to grid (both on the grid clip and in the grid box), will be retained for a minimum of 3 years.

#### 8.0 Calibration and Standardization

Each major component of the method is calibrated and/or standardized, including the analyst. Examples follow:

- **8.1** Sample collection vacuum pumps are calibrated at the beginning and end of a sampling event (with the sample cassette in line) using a rotameter.
- **8.2** The rotameter needs to be calibrated to a primary standard periodically. (Rotameter use and calibration is the responsibility of the sample collection entity.)
- **8.3** The Electron Microscope is calibrated with various standards for
  - **8.3.1** Exact screen magnification at 20,000 X
  - **8.3.2** Exact film magnification at 20,000 X





Revision Date: September 30, 2021 Effective Date: October 25, 2021

- 8.3.3 Spot Size (must be less than 250 nm)
- 8.4 The X-ray system attached to the Electron Microscope is calibrated with various standards for:
  - 8.4.1 Proper location of Al and Cu peaks
  - 8.4.2 Resolution <175 eV at the Mn peak
  - 8.4.3 Relative Sensitivity (K Factors) determined for various elements
  - 8.4.4 A background-subtracted NaKα integrated peak count rate of more than 1 count per second from a fiber of UICC Crocidolite, 50nm diameter or smaller at an accelerating voltage of 80 kV. The peak to background ratio should exceed 1.0.
    - **8.4.4.1** The mineral used for calibration of the EDXA system for sodium shall be prepared using a TEM gold grid.

#### 9.0 Procedure

#### 9.1 Sample Receipt

- Upon receipt of samples, check the sample information on the Chain of Custody (COC) matches the information on the samples and other paperwork. Any discrepancies must be resolved before proceeding.
- If the samples do not have a COC, one is completed at time of login. Have the client fill out 9.1.2 the necessary information completely.
- Information required on the Chain of Custody includes: 9.1.3
  - 9.1.3.1 Client name, address, telephone number, contact person, fax number
  - **9.1.3.2** Project number/ name, state where samples were taken
  - 9.1.3.3 Number of samples sent and sample IDs
  - **9.1.3.4** Type of analysis requested
  - **9.1.3.5** Sample volumes or areas if applicable
  - **9.1.3.6** Turnaround time; "RUSH" is not acceptable
  - **9.1.3.7** A date and signature of the person relinquishing the samples
  - **9.1.3.8** All samples MUST be accounted for with the proper sample IDs.
  - **9.1.3.9** All samples MUST be sealed, properly bagged and undamaged.
- All samples must be clocked in at the time of receipt, and signed and dated by an EMSL 9.1.4 employee. If the lab does not have a clock for sample receipt, the receiving employee should record the time of receipt also.
- 9.1.5 Check to ensure the samples match the COC, and if the cassettes are open, damaged, or contaminated. If samples are delivered in the same container or bag as bulk asbestos material samples, if the samples are damaged, or if the COC does not match the samples, notify the client.

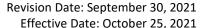
#### **Sample Login** 9.2

If all of the above criteria for sample receiving are met, the samples can be logged into Sample Master (LIMS) as per GEN-SOP-702 Sample Receiving and Chain of Custody.

- 9.2.1 This process will assign a unique EMSL order number for the project, as well as unique lab sample IDs.
- 9.2.2 Sample Master generates an Internal Chain of Custody; however, at this time, it does not create bench sheets or produce reports. For samples analyzed by ISO 10312, bench sheets and the final report are generated using an EMSL prepared Excel spreadsheet.

#### 9.3 **Sample Preparation**

ISO 10312 samples may be collected on mixed cellulose ester (MCE) filters (≤0.45 μm) or Polycarbonate (PC) filters (≤0.4 μm).





- **9.3.2** A direct-transfer preparation is required for this method.
- **9.3.3** The method specifies MCE filters are collapsed using a mixture 35 ml DMF / 15 ml Glacial Acetic Acid / 50 ml fiber free DI water. The dissolution of the filters after collapsing may be performed with acetone.
- **9.3.4** A minimum of 3 grids per sample are prepped and stored.
- **9.3.5** The specific procedures for direct transfer preparation of the filters are outlined in ASB-SOP-102 TEM Direct Prep.

#### 9.4 Grid Storage

- **9.4.1** Grids are stored and location recorded following Section 9.5 of ASB-SOP-102 TEM Direct Prep.
- **9.4.2** All ISO 10312 grid clips and grid boxes (analyzed preps and backup grids not mounted on clips), are stored for 3 years, or as specified by client/project requirements.

#### 9.5 Sample Preparation Acceptance

- **9.5.1** Remove the first sample grid clip from the box and insert it into the TEM.
- **9.5.2** Bring the TEM to a magnification of 300 to 500x, and inspect the grids to determine if all of the following conditions are being met:
  - **9.5.2.1** More than 75% of the grid must be covered with replica
  - **9.5.2.2** Grid must have at least 75% intact grid openings
  - **9.5.2.3** Grids must not have more than 10% opaque area due to incomplete filter dissolution
  - **9.5.2.4** Total grid area must have <25% overlapping or folded replica
  - 9.5.2.5 Sample must have <10% overall particulate loading
  - **9.5.2.6** Particulate loading is uniform from grid opening to grid opening
  - **9.5.2.7** The fibrous structure loading should be  $\leq$  7000 str/mm<sup>2</sup>
  - **9.5.2.8** Grid openings selected must not have rips or overlapping folds
- **9.5.3** If the samples are judged to be unacceptable due to poor prep quality, re-prep the samples as necessary.
- 9.5.4 If the samples are judged to be overloaded (>10% particulate loading) or if the loading is non-uniform, it may be possible to achieve satisfactory preps and the samples analyzed using ISO 13794. This procedure allows for an indirect transfer preparation and can eliminate the problems of overloading and non-uniform distribution. The client needs to authorize this option if it is deemed appropriate.

#### 9.6 Sample Analysis

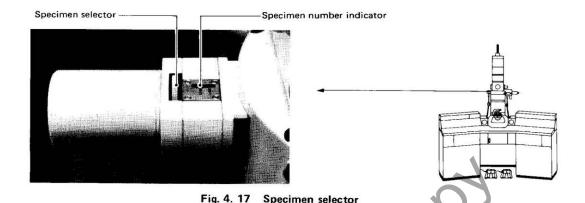
**9.6.1** At a magnification of 100X, first locate and center the grid to be analyzed, using both the specimen selector (located on the left side of the TEM column on top of the left translator where it enters the column) and if present, the grid selector knob, located on the specimen arm (see diagram below).





EMSL

Revision Date: September 30, 2021 Effective Date: October 25, 2021



- **9.6.2** The first grid should be located on screen as the grid to the extreme left.
- **9.6.3** The specimen locator on the scope should be used to find the first grid, but only the grid selector knob (if present) located on the specimen arm needs to be employed to move from grid to grid. (Detailed instruction on the use of both long and short clips and their associated sample arms can be found in EMSL's Grid Clip Loading SOP).
- **9.6.4** Initially, no matter which grid is to be analyzed, locate grid number one, and then proceed to the grid of interest (if not grid one). As analysis proceeds from grid to grid, simply proceed in the same direction (at 100X) to the next grid.
- **9.6.5** Next, orient an intact grid opening on the middle of the screen with the left and right stage controls.
- **9.6.6** Choose an acceptable grid opening to analyze.
- **9.6.7** Increase magnification to approximately 20,000X, taking care to remain in the chosen grid opening.
- **9.6.8** Log the grid opening identification on the sample worksheet.
- **9.6.9** Move to the upper left corner of the grid square and begin traversing the grid, using only one directional control.
- 9.6.10 Once the opposite grid bar has been reached, move over/down one field of view using the other directional control (approximately one large circle width  $\approx 5 \mu m$ ) and proceed scanning with the original directional controller in the opposite direction.
- **9.6.11** Repeat this procedure until the entire grid opening has been scanned. Take care not to count any structure twice or to miss any area of the grid opening.
- **9.6.12** If no fibrous structures have been located, record "ND" for the grid opening and repeat steps 9.6.5 to 9.6.11 until one of the stopping procedures in step 9.6.21 have been reached.
- **9.6.13** Do not analyze adjacent grid openings. Grid openings selected should be split evenly and randomly between a minimum of 2 grid preps.
- 9.6.14 Do not count structures that intersect the top and left grid bars. Structures that intersect the bottom and right grid bar are to be counted as twice the length observed extending from the grid bar.
- **9.6.15** If a fibrous structure has been located, (minimum fiber length = 0.5μm with 5:1 aspect ratio) follow the analysis protocol for each structure encountered as outlined in the flowchart on

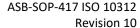


Revision Date: September 30, 2021 Effective Date: October 25, 2021



page 38 of the method for fibers with tubular morphology, and the flowchart on page 41 of the method for fibers without tubular morphology. Both flowcharts are in Section 16 of this procedure. Detailed procedures for SAED and EDXA analysis are found in their respective EMSL SOPs (ASB-SOP-414 SAED; ASB-SOP-411 TEM EDXA PGT Spirit or ASB-SOP-415 TEM EDXA IXRF Ultra).

- **9.6.16** Record only fibrous Non-asbestos Minerals (NAMs) that required close inspection to identify. Enumerate NAM structures with a "0" in the total column and proceed to record all pertinent classifications and distinctions. Both the mineral type and level of ID will be a NAM.
- 9.6.17 Unless the client specifies otherwise, EMSL will attempt to classify Chrysotile to at least a level of "CD" (Table D.1, page 37 of the method) and Amphiboles to at least "ADX" (Table D.2, page 37 of the method); both tables are located in Section 16 of this procedure.
- **9.6.18** Record at least one SAED pattern on film for each type of asbestos, per sample. Record diffraction information on the sample worksheet. See ASB-SOP-414 SAED for detailed information on obtaining and recording SAED patterns.
- **9.6.19** At least one EDXA spectra per asbestos and non-asbestos type, per sample, should be printed out or alternately stored to disk as a .pgt file, if possible. Record EDXA information on the sample worksheet.
- **9.6.20** Record all structure information on the bench sheet as follows:
  - **9.6.20.1** Primary Structure # <u>Tabulate Primary Structures SEQUENTIALLY</u>
  - **9.6.20.2** Total Structure # <u>Tabulate Total Structures SEQUENTIALLY</u>
  - **9.6.20.3** Length of all structures, both primary and total
  - **9.6.20.4** Width of all structures, both primary and total
  - **9.6.20.5** Level of ID (for primary structures, this will be the highest level of identification achieved on the associated total structures)
  - **9.6.20.6** Mineral Identification
  - 9.6.20.7 Sketches and/or comments
  - **9.6.20.8** Any photo ID information for negatives taken
  - 9.6.20.9 Whether the EDXA was printed/saved and which structure it corresponds to
- **9.6.21** Definitions for the basic structure types are found in the definitions section of this SOP. Example sketches of various structure types are found in the ISO 10312 method and in Section 16 of this procedure. Record the structures using the following notation.
  - **9.6.21.1** Structure types and abbreviations are:
    - **9.6.21.1.1** F Fiber
    - **9.6.21.1.2** B Bundle
    - **9.6.21.1.3** CD Cluster (disperse)
    - **9.6.21.1.4** CF Cluster Fiber
    - **9.6.21.1.5** CB Cluster Bundle
    - **9.6.21.1.6** CR Cluster residual
    - **9.6.21.1.7** CC Cluster (compact)
    - **9.6.21.1.8** MD Matrix (disperse)
    - **9.6.21.1.9** MF Matrix Fiber
    - 9.6.21.1.10 MB Matrix Bundle
    - 9.6.21.1.11 MR Matrix Residual
    - **9.6.21.1.12** MC Matrix (compact)
  - **9.6.21.2** Following all primary structures, except for fibers (F) and bundles (B), are two characters:





Revision Date: September 30, 2021

Effective Date: October 25, 2021

- **9.6.21.2.1** The first character is an estimate of fibers and bundles comprising the structure. A "+" is recorded when there are more than 9 component fibers or bundles within a structure.
- 9.6.21.2.2 The second character corresponds to the number of component structures described in the first character that are also > 5µm. A "+" is recorded when there are more than 9 subcomponents that are > 5 μm.
- 9.6.21.3 Record the 5 largest prominent subcomponents, (fibers or bundles), that make up the structure. Fill in the same information on the bench sheet for these compositional structures as were completed for the primary structures. Do not record more than 5 prominent subcomponents for each primary structure.
- **9.6.21.4** If, after accounting for prominent component structures, a group of fibers remains, record this as a residual. Do not record more than 5 residuals for each primary structure. A residual is measured and assigned a two digit number derived in the same manner as specified in the primary structure coding above.
- 9.6.21.5 Compact structures (cluster and matrix), by definition, have no sub-components that cannot be isolated and labeled; therefore, sub-components or residuals should **not be** recorded.
- 9.6.21.6 Cluster and matrix structures can occur in which the characteristics of both types, disperse and compact, happen in the same structure. In these instances, the structure should be assigned as a disperse structure, and a logical procedure should be followed for recording the subcomponents.
- 9.6. 21.7 When in a matrix, the proportion of the length of a fiber or bundle obscured by other particles shall be used as the basis for determining whether it is to be recorded as a separate component (see flowchart in Section 16.7).
  - 9.6.21.7.1 If obscured length could not be more than one third of the total length, record that fiber or bundle accordingly, as either a Matrix Fiber (MF) or Matrix Bundle (MB).
  - 9.6.21.7.2 If obscured length could be more than one third of the total length, the fiber or bundle shall be recorded as Compact Matrix (MC) or a Matrix Residual (MR).
    - 9.6.21.7.2.1 Record as MC if no other subcomponent can be separately identified in the structure.
    - **9.6.21.7.2.2** Record as MR if other subcomponents can be separately identified in the structure.
  - **9.6.21.7.3** If a fiber or bundle crosses a matrix particle (greater than or equal to 0.5 microns) and both ends can be located, the structure should be recorded as a Matrix Fiber (MF) or Matrix Bundle (MB).
- **9.6.22** Examples of proper syntax follows:
  - **9.6.22.1** MD31 is a disperse matrix comprised of 3 distinct fibers or structures, one of which is greater than 5 microns.
  - 9.6.22.2 MC30 is a compact matrix estimated to have no more than 3 subcomponents, none of which is greater than 5 microns.
  - 9.6.22.3 CD+1 is a disperse cluster with more than 9 fibers or structures ("+" means more than 9), one greater than 5 microns.



Effective Date: October 25, 2021



**9.6.22.4 MD+0** is a disperse matrix with more than 9 fibers or structures, none greater than 5 microns.

- 9.6.23 Stopping Criteria
  - **9.6.23.1** Analysis may be terminated after the required analytical sensitivity is achieved, or
  - **9.6.23.2** Analysis may be terminated after the completion of analysis of the grid opening in which the 100<sup>th</sup> structure was encountered.
  - **9.6.23.3** Regardless of which step above terminates the analytical process, a minimum of 4 grid openings must be analyzed.
- **9.6.24** Blanks
  - **9.6.24.1** One laboratory blank must be processed with each slide of samples. (Only one of the laboratory blanks needs to be analyzed per Order ID.)
  - **9.6.24.2** At least one field blank should be processed with each batch of samples.
  - 9.6.24.3 Filter lot blanks are required at a minimum of two for every 100 filters.
- **9.6.25** There is no method required analytical sensitivity for this procedure; the target analytical sensitivity is set with mutual consent of the client. Unless otherwise specified, EMSL will analyze sample to an analytical sensitivity of 0.005 s/cc.
- **9.6.26** A Pass / Fail criteria is not given for this method.

#### 10.0 Calculations

Examples of the calculations are displayed below. Variables include, but are not limited to the volume and GOA (grid opening area) which is calculated with every batch of grids.

- EFA Effective filter area of a 25mm cassette = 385mm<sup>2</sup>
- GOA Grid opening area (0.00635 mm<sup>2</sup>)
- N Number of fibers (If N=0 then default to 1 structure)
- V Volume (1200 liters)
- AA Area analyzed (0.06985 mm<sup>2</sup>)
- NO Number of grid openings analyzed (11)
- n Number of Samples
- AS Analytical Sensitivity
- 10.1 Area analyzed (AA)

$$AA = GOAxNO$$
  $AA = 0.00635mm^2x11$   $AA = 0.06985mm^2$ 

10.2 Structures per square millimeter (str/mm<sup>2</sup>)

$$str/mm^2 = (N/AA)$$
  $str/mm^2 = (1/0.06985mm^2)$   $str/mm^2 = 14.3$ 

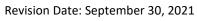
10.3 Structures per cubic centimeter of air (str/cc)

$$str/cc = \frac{EFAxN}{AAx1000xV}$$
  $str/cc = \frac{385mm^2x5}{0.06985mm^2x1000x1200L} = 0.023$ 

#### 10.4 Analytical Sensitivity (AS)

To calculate the analytical sensitivity, calculate the str/cc using 1 for the number of structures (N) in the formula above.





EMSL

Effective Date: October 25, 2021

$$AS = \frac{EFAxN}{AAx1000xV}$$

$$AS = \frac{385mm^2x1}{0.06985mm^2x1000x1200L} = 0.0046$$

#### 10.5 Grid Openings (NO) required to be analyzed

The number of grid openings (NO) required to be analyzed is dependent on grid opening area (GOA), the volume (V) of sample collected, and the requested analytical sensitivity (AS) of the analysis.

$$NO = \frac{EFA}{ASxGOAx1000xV} \qquad NO = \frac{385}{0.005x0.00635x1000x1200} = 10.1 = 11$$

#### Important!!

For this calculation, round the result to an integer, and ALWAYS round the result <u>UP</u> regardless of rounding rules. In the above example, even though the first decimal place is below 5 (10.1), the result is rounded up to produce 11 required openings.

#### 10.6 Chi Square to test for uniform fiber distribution

This test is performed to quantify the randomness of the fibers distributed on the filter surface. It is calculated with the following equation:

$$\sum_{i=1}^{j=k} \frac{(n_i - np_i)^2}{np_i}$$

Where  $(p_i)$ ; is the grid opening area divided by the total area of the TEM grid examined (see 10.1, above)

Where  $n_i$  is the observed number of primary structures on that grid opening

Where n is the total number of primary structures observed in the sample

The value from the above calculation is compared with the significance points of the Chi Square table, having (k-1) degrees of freedom at the 0.001 significance level, where k is the total number of grid openings analyzed.

#### 11.0 Reporting

The following items are included in the final report (on EMSL letterhead) to the client.

- **11.1** Asbestos concentration in structures/liter (str/L) categorized as follows:
  - **11.1.1** Total asbestos concentration for all asbestos types meeting minimum Identification required, typically CD and ADX
    - **11.1.1.1** For sample where three or less structures are counted, report the concentration as less than the corresponding one-sided upper 95% confidence limit for the Poisson distribution.
    - **11.1.1.2** For samples where four or greater structures are counted, report the mean asbestos structure concentration.
  - **11.1.2** Chrysotile asbestos concentrations are reported as follows:
    - **11.1.2.1** Determine which identification and structure categories will be used to calculate the concentration. This is specified by the client. If it is not specified by the client, use all Total Structures identified as CD or better.





Revision Date: September 30, 2021 Effective Date: October 25, 2021

- 11.1.2.2 For sample where three or less chrysotile structures are counted, report the concentration as less than the corresponding one-sided upper 95% confidence limit for the Poisson distribution.
- 11.1.2.3 For samples where four or greater structures are counted, report both
  - **11.1.2.3.1** The mean chrysotile concentration
  - **11.1.2.3.2** The upper and lower chrysotile concentrations based on the corresponding two-sided Poisson 95% confidence intervals
- **11.1.3** Total amphibole asbestos concentrations are reported as follows:
  - 11.1.3.1 Determine which identification and structure categories will be used to calculate the concentration. This is specified by the client. If it is not specified by the client, use all Total Structures identified as ADX or better.
  - 11.1.3.2 For samples where three or less amphibole structures are counted, report the concentration as less than the corresponding one-sided upper 95% confidence limit for the Poisson distribution.
  - 11.1.3.3 For samples where four or greater structures are counted, report both
    - **11.1.3.3.1** The mean amphibole concentration
    - **11.1.3.3.2** The upper and lower amphibole concentrations based on the corresponding two-sided Poisson 95% confidence intervals
- 11.2 The compositional data for the principle varieties of amphiboles present (if any)
- **11.3** The analytical sensitivity in str/L
- **11.4** The detection limit in str/L
- 11.5 Total number of primary structures counted
- 11.6 Number of total asbestos structures counted
- 11.7 Number of asbestos structures > 5μm
- 11.8 Number of asbestos fibers and bundles (combined) > 5µm
- 11.9 Number of PCM equivalent asbestos structures
- 11.10 Number of PCM equivalent asbestos fibers
- 11.11 The level of analysis used when identifying chrysotile and amphibole structures
- 11.12 Aspect ratio used to define fibers for the analysis
- **11.13** Reference to the Method
- 11.14 Client Identification and contact information
- 11.15 EMSL's order ID
- **11.16** Date of receipt of the samples
- **11.17** Date of sample collection (if provided)
- **11.18** Date of the report
- **11.19** EMSL and client sample ID numbers
- **11.20** Volume of air collected in the sample
- 11.21 Effective filter area of the sample media
- **11.22** Area of the grid openings used during analysis
- 11.23 Microscope magnification used during analysis
- **11.24** Initials of the analyst
- 11.25 Count sheets are supplied with final results.

#### 12.0 Method Performance

Method performance data can be found in the method Ambient air - Determination of asbestos fibres -Direct-transfer transmission electron microscopy method ISO 10312.



Revision Date: September 30, 2021 Effective Date: October 25, 2021

- **12.1 MDL:** When no structures are counted, the detection limit is considered to be the upper limit of the one-sided Poisson confidence interval, or 2.99 structures
- **12.2 DOCs:** Demonstrations of Capability are required for each analytical method.
- **12.3 PTs:** Proficiency tests do not exist for this procedure at the current time.
- **12.4 Accuracy:** Since it is not possible to create a standard filter with a known structure concentration, the exact accuracy of the method cannot be determined.
- **12.5 Precision:** The analytical precision is dependent upon the number of structures counted and the uniformity of the particulate deposit. Assuming a uniform distribution and structure loading of at least 3.5 structures per grid opening, the coefficient of variance of the counting procedure can be estimated at 10%. It should be noted that in practice, particulate deposits obtained by filtration of ambient air samples are rarely ideally distributed.

#### 13.0 Quality Control

- **13.1** All QC data must be maintained and available for easy reference and inspection.
- **13.2** Blanks
  - **13.2.1** Laboratory Blanks: A lab blank is included with each slide of samples prepared. The last slide's lab blank will be prepared for analysis, At least one lab blank for every Order ID should be analyzed.
  - **13.2.2** Field Blanks: At least one field blank should be processed with each sample set.
  - **13.2.3** Lot Blanks: A minimum of two unused filters from each filter lot of 100 will be analyzed.
- **13.3** Inter-analyst QC is at least 4% reanalysis of the same grid openings.
- **13.4** Intra-analyst QC is at least 2% reanalysis of the same grid openings.
- **13.5** Verified analysis is used for training, and will be performed as need for discordant sample QC reconciliation.
- **13.6** R-preparation and analysis of samples filter QC = 0.5%
- **13.7** Inter-laboratory analysis is performed in order to monitor systematic errors among microscopists as needed or as determined by client.

#### 14.0 Data Assessment

- 14.1 Acceptance Criteria for QC Measures: These are addressed in EMSL's QMS Manual, Module A
  - **14.1.1** If a sample falls outside the acceptable limits, it needs to be reconciled with participating analysts and/or a third analyst, when necessary.
  - **14.1.2** The Pass/Fail criteria for repeat results (inter- and intra-analyst) should not differ at the 5% significance level.
  - **14.1.3** For verified analysis, the results should be  $\leq 80\%$  of true positives,  $\leq 20\%$  false negatives, and  $\leq 10\%$  false positives of the Total Structure count.
  - **14.1.4** Repreparations from different sectors of the filter is a test for reproducibility of the whole method. Since this type of QC is impacted by circumstances outside the control of the analysis, no statistical evaluation is currently performed on this data. The data is simply compiled for informational purposes only.
  - 14.1.5 Criteria for the maximum allowable contamination levels for laboratory blanks: on all filter types (MCE and PC) filters, cannot exceed the methods limit for lot blanks, which is 10 str/mm², or if the mean fiber count for asbestos fibers and bundles longer than 5 microns is more than 0.1 fiber/mm².
  - **14.1.6** The Chi Square uniformity test shall be conducted using the number of primary structures found on individual grid openings (see Section 10.6 of this SOP). This value will be compared



Revision Date: September 30, 2021

Effective Date: October 25, 2021

with significance points of the Chi Square distribution. If the structure count fails this test, the precision of the result may be in question, and if new air samples cannot be collected, additional grid openings may need to be examined, or alternatively, the sample should be prepared indirectly by ISO 13794.

- 14.2 Corrective Actions: These policies are addressed fully in EMSL's QMS Manual.
  - **14.2.1** All corrective actions should look for the root cause of the error.
  - 14.2.2 All out-of-control or unacceptable data must be brought to the attention of the laboratory manager.
  - **14.2.3** The laboratory manager is responsible for generating a corrective action, including an investigation of calibration procedures, a review of analytical technique, and investigation of training policies and compliance.
  - **14.2.4** Corrective actions will be reported to the QA Department by means of the Quarterly Management Report, or sooner when appropriate.
- 14.3 Contingencies for Handling Out-of-control or Unacceptable Data: Any quality control requirements not met must have an explanation to their non-conformance.

#### 15.0 Pollution Prevention / Waste Management

#### **15.1** Pollution Prevention

EMSL Analytical makes all efforts to reduce the volume and toxicity of the waste generated by the laboratory. An effort to manage procurement of hazardous materials has been implemented in order to avoid over ordering. Hazardous waste is classified for proper disposal.

#### 15.2 Waste Management

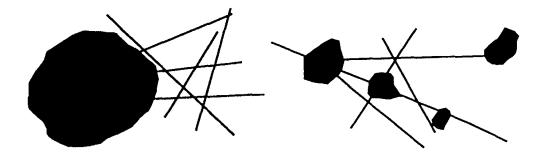
The waste generated during prep and analysis will be disposed of following safety procedures outlined in the chemical hygiene and safety plan.

#### 16.0 Tables, Diagrams, Flowcharts, and Validation Data

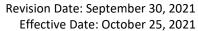
16.1 Example of disperse clusters:



**16.2** Examples of disperse matrices:

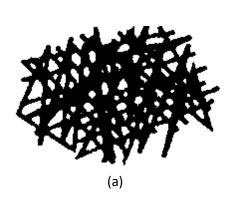


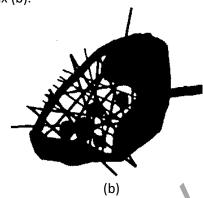






#### **16.3** Examples of compact Cluster (a) and Matrix (b):





#### 16.4 Structure Classification Tables

Table D.1 — Classification of fibres with tubular morphology

Category	Description
TM	Tubular Morphology, not sufficiently characteristic for classification as chrysotile
CM	Characteristic Chrysotile Morphology
CD	Chrysotile SAED pattern
CO	Chrysotile composition by Quantitative EDXA
СМО	Chrysotile Morphology and composition by Quantitative EDXA
CDQ	Chrysotile SAED pattern and composition by Quantitative EDXA
NAM	Non-Asbestos Mineral

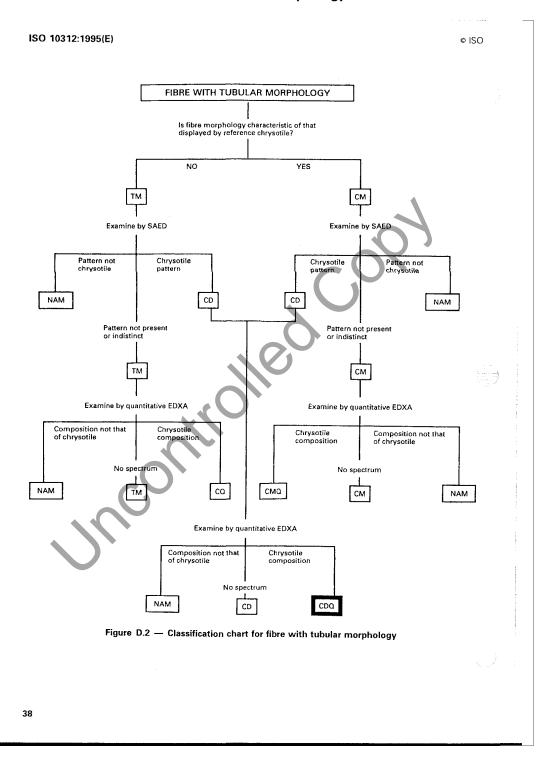
Table D.2 — Classification of fibres without tubular morphology

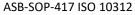
Category	Description
UF	Unidentified Fibre
AÐ	Amphibole by random orientation SAED (shows layer pattern of 0,53 nm spacing)
AX	Amphibole by qualitative EDXA. Spectrum has elemental components consistent with amphibole
ADX	Amphibole by random orientation SAED and qualitative EDXA
AQ	Amphibole by Quantitative EDXA
AZ	Amphibole by one Zone-axis SAED pattern
ADQ	Amphibole by random orientation SAED and Quantitative EDXA
AZQ	Amphibole by one Zone-axis SAED pattern and Quantitative EDXA
AZZ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle
AZZQ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle, and Quantitative EDXA
MAM	Non-Asbestos Mineral





#### 16.5 Identification Flowchart for Fibers with Tubular Morphology









Effective Date: October 25, 2021

#### 16.6 Identification Flowchart for Fibers without Tubular Morphology

ISO 10312:1995(E) o ISO

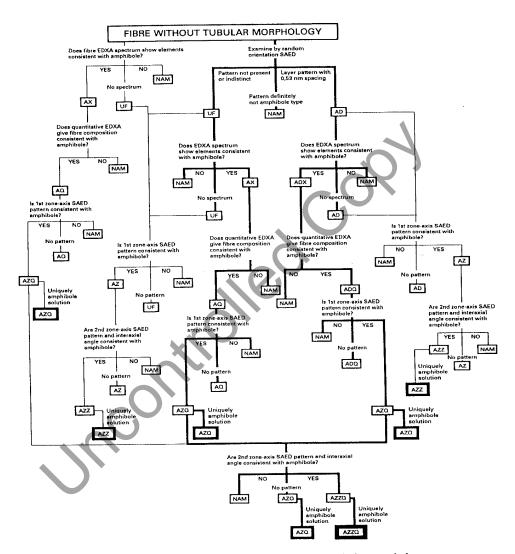


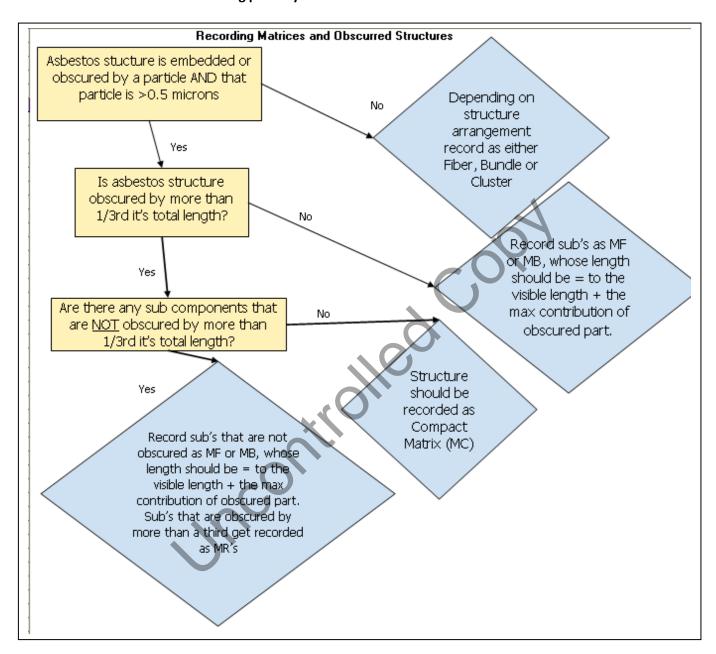
Figure D.4 — Classification chart for fibre without tubular morphology

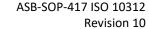
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#### 16.7 Flow chart for recording partially obscured fiber and bundles







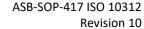
#### 17.0 References

- 17.1 Ambient air Determination of asbestos fibres Direct-transfer transmission electron microscopy method ISO 10312.
- **17.2** Less is Better- Guide to Minimizing Waste in Laboratories prepared by the Task Force on Laboratory Environment, Health and Safety- American Chemical Society 2002.
- 17.3 EMSL QMS Manual
- 17.4 SAF-SOP-2 Chemical Hygiene and Safety Plan

#### **18.0** Revision History

Rev. #	Rev. Date	Changes in this Revision	Initials
10	9/30/2021	12.0, 17.1: Updated method reference (removed 1 <sup>st</sup> Edition 1995-05-01 after ISO 10312) 17.3, 17.4: Updated QMS Manual, SAF-SOP-2 references Updated SOP reference names throughout Renamed per protocol	KN
9.1	2/16/2012	1.3: Revised definition of True PCMe fiber	RD
9	2/06/2012	8.4: Added Na sensitivity requirements 10.6: Added chi square test 11.0: Added provide statement re which structure category, ID category to include in calculation of asbestos concentration 13.0, 14.0: Revised to conform to QA measurements defined in method 16.0: Added flow chart detailing recording of partially obscured fibers or bundles Revised blank QC determination	RD

Ken Najuch	Authorizing Signatures	9/30/2021
Author/Revised by (print)	Author Signature	Date
Michelle McGowan QA Reviewer (print)	Michille McKonan Reviewer Signature	10/08/2021 Date
Michelle McGowan	Michele Mc Assoan	10/08/2021
Corporate Approval (print)	Corporate Approval Signature	Date





# Document Review and Acknowledgement Form

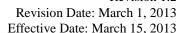
Instructions: Ensure all personnel in laboratory affected by this document read and acknowledge receipt of document. Once completed, page shall be retained on file along with personnel training.

	d, understood, and have had the opportunity to ask questio comply with, implement, and enforce all requirements, proc ng reference materials or methodologies.	
Print Name	Signature	Date
Acknowledged by:		
Lab Manager/QA Officer (Print Name	e) Signature	Date



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EMSL Analytical, Inc. Management





# **EMSL Analytical S.O.P.**

# Ambient air - Determination of asbestos fibres - Indirect-transfer transmission electron microscopy method

ISO 13794:1999 First edition 1999-07-15

#### 1.0 Method Description

#### 1.1 Applicable Matrix

This method is appropriate for air samples only.

#### 1.2 Scope and application

This method is for the determination of asbestos concentrations in air samples by transmission electron microscopy (TEM); it is applicable to the determination of airborne asbestos in a wide range of ambient air situations both exterior and interior. The range of concentrations which can be determined is 50 structures/mm² to 7000 structures/mm² on the filter.

#### 1.3 Summary of Method

Samples are collected on a membrane filter, prepped first via an indirect transfer method and then by direct prep of the subsequent filters. The samples are then analyzed with an electron microscope at ≈20,000 X magnification. Asbestos structures are identified by a combination of morphology, elemental chemistry via Energy Dispersive X-Ray Analysis (EDXA), and Selected Area Electron Diffraction (SAED). All structures ≥0.5 micron in length with at least a 5:1 aspect ratio are counted, and recorded. Structures are differentiated as primary or total structures, a primary structure may be composed of numerous total structures. The report includes a breakdown of fibers and bundles > 5 microns as well as true PCM equivalent fibers (>5microns in length, >0.25 microns in width and a 3:1 aspect ratio). Unless the client specifies otherwise, the lab will attempt to reach an analytical sensitivity of 5 structures/L.

#### 1.4 Detection Limit

The analytical sensitivity is defined as the calculated airborne asbestos structure concentration in asbestos structures per Liter, equivalent to the counting of 1 asbestos structure in the analysis. The analytical sensitivity can be lowered by increasing the volume of air collected, increasing the proportion of aliquot of sample suspension filtered and also by increasing the area of the filter analyzed. The Limit of Detection is defined by this method as the upper, one-sided 95% confidence limit of the Poisson distribution for a count of 0 structures. In the absence of background contamination this is equivalent to 2.99 asbestos structures. The limit of detection can be lowered by collecting a larger initial volume of air or by analyzing additional surface area of the filter (grid openings).

#### 2.0 Interferences

Interferences for this method include but are not limited to:

- **2.1** Non-regulated asbestos minerals such as the two polymorphs of Chrysotile, Lizardite and Antigorite.
- **2.2** Non regulated amphiboles such as winchite and richterite, and pyroxenes.
- **2.3** Cleavage fragments of the regulated asbestos types which may at times have aspect ratios similar to the true asbestiform varieties.

Revision Date: March 1, 2013 Effective Date: March 15, 2013



- Clay minerals that can have similar morphology to asbestos such as sepiolite and pallygorskite.
- **2.5** All non-asbestos particulate, fibrous or not which can partially or wholly obscure asbestos fibers.

#### 3.0 Definitions

- Analytical Sensitivity The airborne concentration represented by one asbestos structure counted under the electron microscope. The air volume collected and the proportion of the filter examined determine the analytical sensitivity. There is no set target A.S. with this method. If not directed otherwise by the client, EMSL will attempt to reach an A.S. of 5 s/L up to 10 G.O. unless the method stopping rule of 100 asbestos structures is reached. A minimum of 4 grid openings must be analyzed.
- 3.2 Asbestos Generic term for a group of hydrated mineral silicates
- 3.3 Aspect Ratio The ratio of the length to the width of a particle. Minimum aspect ratio for this method is 5:1 (3:1 for optional PCMe phase of analysis).
- 3.4 Bundle A grouping of apparently attached parallel fibers. A structure composed of parallel, smaller diameter fibers attached along their lengths. A bundle may exhibit diverging fibers at one or both ends.
- 3.5 Cluster A structure in which 2 or more fibers or fiber bundles are randomly oriented in a connected grouping. There are two types of clusters, disperse and compact.
- 3.6 Compact Cluster Complex and tightly bound network in which one or both ends of each individual fiber or bundle is (are) obscured, such that the dimensions of individual fibers and bundles cannot be unambiguously determined. As such, no subcomponents or residuals will be listed.
- 3.7 Compact Matrix A structure consisting of a particle or linked groups of particles in which fibers or bundles can be seen either within the structure or projecting from it, such that the dimensions of individual fibers and bundles cannot be unambiguously determined. As such, no subcomponents or residuals will be listed.
- 3.8 Disperse Cluster A disperse and open network in which at least one of the individual fibers or bundles can be separately identified and its dimensions measured.
- 3.9 Disperse Matrix A structure consisting of a particle or linked group of particles with overlapping or attached fibers or bundles in which at least one of the individual fibers or bundles can be separately identified and measured.
- 3.10 EDXA -Energy dispersive X-ray analysis.
- **3.11** Fiber An elongated particle which has parallel or stepped sides. For this method it must have a minimum length of 0.5 µm and an aspect ratio of 5:1 or greater.
- **3.12** Fiber Bundle See bundle.
- **3.13** Grid A thin metal (usually Cu) foil with openings that the sample is mounted on to aid in its examination in the TEM.
- **3.14** Grid Opening (GO) One opening of the grid. For this method the area of the grid opening must be known.
- 3.15 Matrix One or more fibers or fiber bundles that are attached to or partially concealed by a single particle or group of overlapping non fibrous particles. There are two types of matrices, disperse and compact.
- **3.16** MCE Filters Mixed cellulose ester filter.
- **3.17** PC Filters Polycarbonate filters.
- **3.18** PCM Equivalent Fiber A fiber of aspect ratio 3:1 or greater, longer than 5 microns and a diameter between 0.2 and 3 microns.

Revision Date: March 1, 2013

Effective Date: March 15, 2013



- 3.19 PCM Equivalent Structure A fibrous structure of aspect ratio 3:1 or greater, longer than 5 microns and a diameter between 0.2 and 3 microns.
- 3.20 Primary Structure A fibrous structure that is a separate entity in the TEM image and shall be recorded as one of the fundamental structures: fiber, bundle, cluster or matrix.
- 3.21 Residual A localized group of fibers that remain after the 5 largest prominent features of a primary structure has been documented. Residuals are named cluster residual (CR) or matrix residual (MR) depending on the identity of the primary structure. Record up to 5 residuals for each primary structure.
- **3.22** SAED Selected area electron diffraction.
- **3.23** Structure A single fiber, fiber bundle, cluster, or matrix which may contain asbestos.
- **3.24** TEM Transmission Electron Microscopy.
- 3.25 Total Structure Structure or structures associated with the primary structure. Total structures are enumerated separately from primary structures and are used to classify and measure the important features of each primary structure.

#### 4.0 Safety

All personnel performing preparation and/or analysis of samples must be familiar with the EMSL Chemical Hygiene Plan (EMSLChemHygiene 200.0). Specific hazards and precautions associated with this analysis include:

#### 4.1 Asbestos

- **4.1.1** Prudent measures must be taken to prevent any possible airborne asbestos fiber release from occurring during sample handling.
- **4.1.2** Any filter handling performed prior to the filter collapse step should be performed under the safety hood.

#### 4.2 Acetone

- **4.2.1** Keep away from heat, sparks, and flame.
- **4.2.2** Avoid breathing vapors use with adequate ventilation.
- 4.2.3 Avoid contact with eyes.
- **4.2.4** Prevent prolonged or repeated contact with skin.

#### 4.3 Carbon Spark

**4.3.1** Shield eyes from the glow of the spark during the carbon coating process with welder's type goggles or similar protection.

#### 4.4 Oxygen

- **4.4.1** The oxygen used with the plasma asher is an explosion hazard. Use only vacuum pumps filled with non hydrocarbon (Fomblin) oil.
- **4.4.2** Keep Oxygen tank strapped to wall at all times.

#### 5.0 Equipment and Supplies

- 5.1 ≤0.22 micron MCE or ≤0.2 micron PC filters with a 5.0 µm backing filter
- 5.2 Glass Petri Dishes
- **5.3** Glass microscope slides
- **5.4** Low Temperature Plasma Asher
- **5.5** Vacuum Evaporator (Carbon Coater)
- **5.6** Graphite or Carbon rods
- **5.7** HEPA Laminar Flow Hood
- **5.8** Grids Copper and Gold
- **5.9** Fine Forceps

Revision Date: March 1, 2013 Effective Date: March 15, 2013



**5.10** Grid Clips and Grid Storage Boxes

- **5.11** Jaffe Wick or Sponge
- **5.12** Kimwipes or alternative paper
- **5.13** Transmission Electron Microscope with the following capabilities:
  - -100 kV
  - -fine probe size <250 nm
  - -Elemental Chemistry via X-Ray Detector

#### 6.0 Reagents and Standards

All reagents should be of recognized analytical grade or better:

- **6.1** Acetone
- **6.2** Di-Methyl Formamide (DMF)
- **6.3** Glacial Acetic Acid
- **6.4** NIST SRM 1876b
- **6.5** NIST SRM 2063
- **6.6** Albite Standard
- 6.7 Aluminum Coated Copper Grid Standard
- **6.8** Gold Coated Grid Standard
- 6.9 Magnification (cross grating replica) Calibration Standard
- 6.10 NIST Traceable Asbestos on Grids
- 6.11 Non Asbestos Standards on Grids
- **6.12** Ethylenediamine
- **6.13** 1-methyl 2-pyrrolidone
- 6.14 Glacial Acetic Acid

#### 7.0 Sample Collection, Preservation, Shipment and Storage

- 7.1 Samples are collected on ≤ 0.45 micron Mixed Cellulose Ester (MCE) or ≤0.4 micron Polycarbonate (PC) filter cassettes (EMSL recommends MCE filters).
- **7.2** Air flow should be between 2.4 LPM and 15 LPM (not to exceed 10 LPM is recommended)
- **7.3** Typically 1800 liters of air or more are collected. The total volume that can be collected will be partially determined by the air being sampled.
- **7.4** No sample preservation is needed and samples can be stored indefinitely prior to analysis.
- **7.5** Samples are best transported to the lab by hand. When mailing, try to package samples carefully to minimize disturbance and possible dislocation of particulate from the filter surface. Use packing materials that will minimize static charge.
- **7.6** All air cassettes must be retained in an easily retrievable manner for a minimum of 60 days.
- 7.7 All samples prepped to grid (both on the grid clip and in the grid box), will be retained for a minimum of 3 years.

#### 8.0 Calibration and Standardization

Each major component of the method is calibrated and/or standardized including the analyst. Examples follow:

8.1 Sample collection vacuum pumps are calibrated at the beginning and end of a sampling event (with the sample cassette in line) using a rotameter.

ISO 13794 SOP

Revision 1.2

EMSL

Revision Date: March 1, 2013 Effective Date: March 15, 2013

- 8.2 The rotameter in turn needs to be calibrated to a primary standard periodically. (Rotameters use and calibration is the responsibility of the sample collection entity).
- **8.3** The Electron Microscope is calibrated with various standards for
  - **8.3.1** Exact screen magnification at 20,000 X.
  - **8.3.2** Exact film magnification at 20,000 X.
  - **8.3.3** Spot Size (must be less than 250 nm).
- **8.4** The X-Ray system attached to the Electron Microscope is calibrated with various standards for:
  - **8.4.1** Proper location of aluminum (Al) and copper (Cu) peaks.
  - **8.4.2** Resolution <175 eV at the manganese (Mn) peak.
  - **8.4.3** Relative Sensitivity (K Factors) determined for various elements.
  - **8.4.4** A background-subtracted NaKα integrated peak count rate of more than 1 count per second from a fiber of UICC Crocidolite 50nm diameter or smaller at an accelerating voltage of 80 kV. The peak to background ratio should exceed 1.0. 8.4.4.1 The mineral used for calibration of the EDXA system for sodium shall be prepared using a TEM gold grid.

#### 9.0 Procedure

#### 9.1 Sample Receipt

- **9.1.1** Upon receipt of samples, check that the sample information on the Chain of Custody (COC) matches the information on the samples and other paperwork. Any discrepancies must be resolved before proceeding.
- **9.1.2** If the samples do not have a COC then one is completed at time of log in. Have the client fill out the necessary information completely.
- **9.1.3** Information required on the Chain of Custody includes:
  - **9.1.3.1** Client name, address, telephone number, contact person, fax number
  - **9.1.3.2** Project number/ name, state where samples where taken
  - **9.1.3.3** Number of samples sent and sample ID's
  - 9.1.3.4 Type of analysis requested
  - **9.1.3.5** Sample volumes or areas if applicable
  - 9.1.3.6 Turn around time. "RUSH" is not acceptable
  - **9.1.3.7** A date and signature of the person relinquishing the samples
  - 9.1.3.8 All samples MUST be accounted for with the proper sample ID's
  - **9.1.3.9** All samples MUST be sealed, properly bagged and undamaged.
- **9.1.4** All samples must be clocked in at the time of receipt and signed and dated by an EMSL employee. If the lab does not have clock for sample receipt the receiving employee should record the time of receipt also.
- 9.1.5 Check to see if the samples match the COC and if the cassettes are open, damaged, or contaminated. If samples are delivered in the same container or bag as bulk asbestos material samples, if the samples are damaged or if the COC does not match, notify the client.

#### 9.2 Sample Log In

If all of the above criteria for sample receiving are met then the sample can be logged in to Sample Master (LIMS) as per the Sample Master SOP.

**9.2.1** This process will assign a unique EMSL order number for the project as well as unique lab sample ID's.





Revision Date: March 1, 2013 Effective Date: March 15, 2013

9.2.2 Sample Master generates an Internal Chain of Custody; however at this time it does not create bench sheets or produce reports. For samples analyzed by ISO 10312, bench sheets and the final report are generated using an EMSL prepared Excel spreadsheet.

#### 9.3 **Sample Preparation**

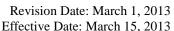
- ISO 13794 samples may be collected on mixed cellulose ester (MCE) filters (≤0.45 μm) or Polycarbonate (PC) filters (≤0.4 μm).
- **9.3.2** An indirect-transfer preparation is required for this method.
- 9.3.4 Initial preparation and filtration of samples should follow the EMSL Indirect Prep SOP. Filters to be used are either 0.22 µm MCE or 0.2 µm PC.
- Section 2.0 of the Indirect Prep SOP (Sonicate and Re-suspend) is omitted and 9.3.5 Section 3.0 (Ash and Re-suspend) is utilized for this method.
- At least one empty beaker should be processed with each batch of samples 9.3.6 processed through the plasma asher. This empty beaker should be processed through both the filtration section (4.0) of the Indirect Prep SOP and the following Direct Prep SOP when the final filtration filters are prepared onto grids.
- For this procedure the final volume of sample suspension is not brought up to 100ml after plasma ashing (section 3.16 in the Indirect Prep SOP).
- In addition to the Plasma Asher process blank, a filtration process blank is also run during sample suspension filtration (Section 4.0 of the Indirect Prep SOP).
- 9.3.9 The method specifies that MCE filters are collapsed using a mixture 35% DMF / 15% Glacial Acetic Acid / 50% fiber free DI water during the direct preparation of the filtered sample suspensions. The dissolution of the filters after collapsing may be performed with acetone.
- **9.3.10** A minimum of 3 grids per sample are prepped and stored.
- **9.3.11** After all samples suspension have been filtered, prepare the filters following a direct transfer preparation technique as outlined in the EMSL Direct Prep of Sample Filters SOP.

#### 9.4 Grid Storage

- Grids are stored and location recorded following section 9.5 of EMSL's Direct Transfer Prep SOP.
- 9.4.2 All ISO 13794 grid clips and grid boxes (analyzed preps and backup grids not mounted on clips, are stored for 3 years.

#### 9.5 **Sample Preparation Acceptance**

- **9.5.1** Remove the first sample grid clip from the box and insert it into the TEM.
- **9.5.2** Bring the TEM to a magnification of 300 to 500x and inspect the grids to determine if all of the following conditions are being met.
  - **9.5.2.1** More than 75% of the grid must be covered with replica
  - **9.5.2.2** Grid must have at least 75% intact grid openings.
  - 9.5.2.3 Grids must not have more than 10% opaque area due to incomplete filter dissolution.
  - **9.5.2.4** Total grid area must have <25% overlapping or folded replica.
  - **9.5.2.5** Sample must have <10% overall particulate loading.
  - **9.5.2.6** Particulate loading is uniform from grid opening to grid opening.
  - **9.5.2.7** The fibrous structure loading should be  $\leq$  7000 str/mm<sup>2</sup>.
  - **9.5.2.8** Grid openings selected must not have rips or overlapping folds.
- 9.5.3 If the samples are judged to be unacceptable due to poor prep quality, reprep the samples as necessary.



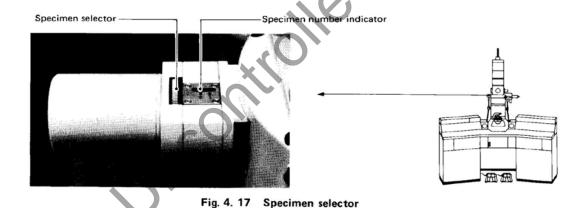


9.5.4 If the samples are judged to be overloaded (>10% particulate loading) or if the loading is non-uniform, it may be possible to achieve satisfactory preps and the samples analyzed using ISO 13794. This procedure allows for an indirect transfer preparation and can eliminate the problems of overloading and non-uniform distribution. The client needs to authorize this option if it is deemed appropriate.

#### 9.6 Sample Analysis

9.6.1 At a magnification of 100X, first locate and center the grid to be analyzed using both the specimen selector (located on the left side of the TEM column on top of the left translator where it enters the column) and if present, the grid selector knob located on the specimen arm (see diagram below).





- **9.6.2** The first grid should be located on screen as the grid to the extreme left.
- 9.6.3 The specimen locator on the scope should be used to find the first grid but only the grid selector knob (if present) located on the specimen arm needs to be employed to move from grid to grid. (Detailed instruction on the use of both long and short clips and their associated sample arms can be found in EMSL's Grid Clip SOP).
- 9.6.4 Initially, no matter which grid is to be analyzed, you will need to locate grid number 1 and then proceed to the grid of interest (if not grid 1). As analysis proceeds from grid to grid you can simply proceed in the same direction (at 100X) to the next grid.
- **9.6.5** Next, orient an intact grid opening on the middle of the screen with the left and right stage controls.
- **9.6.6** Choose an acceptable grid opening to analyze.

Revision 1.2
Revision Date: March 1, 2013
Effective Date: March 15, 2013

- **9.6.7** Increase magnification to approximately 20,000X taking care to remain in the chosen grid opening.
- **9.6.8** Log the grid opening identification on the sample worksheet.
- **9.6.9** Move to the upper left corner of the grid square and begin traversing the grid using only one directional control.
- 9.6.10 Once the opposite grid bar has been reached, move over/down one field of view using the other directional control (approximately one large circle width  $\approx 5 \mu m$ ) and proceed scanning with the original directional controller in the opposite direction.
- **9.6.11** Repeat this procedure until the entire grid opening has been scanned. Take care not to count any structure twice or to miss any area of the grid opening.
- **9.6.12** If no fibrous structures have been located, record "ND" for the grid opening and repeat steps 9.6.5 to 9.6.11 until one of the stopping procedures in step 9.6.21 have been reached.
- **9.6.13** Do not analyze adjacent grid openings. Grid openings selected should be split evenly and randomly between a minimum of 2 grid preps.
- **9.6.14** Do not count structures that intersect the <u>top</u> and <u>left</u> grid bars. Structures that intersect the <u>bottom</u> and <u>right</u> grid bar are to be counted as twice the length observed extending from the grid bar.
- 9.6.15 If a fibrous structure has been located, (remember minimum fiber length = 0.5µm with 5:1 aspect ratio) then follow the analysis protocol for each structure encountered as outlined in the flowchart on page 38 of the method for fibers with tubular morphology, and the flowchart on page 41 of the method for fibers without tubular morphology. Both flowcharts are in section 16 of this procedure. Detailed procedures for SAED and EDXA analysis are in their respective EMSL SOPs (SAED SOP and Energy Dispersive X-Ray Analysis SOP).
- 9.6.16 Record only fibrous Non Asbestos Minerals (NAM's) that required close inspection to identify. Enumerate NAM structures with a "0" in the total column and proceed to record all pertinent classifications and distinctions. Both the mineral type and level of ID will be a NAM.
- 9.6.17 Unless the client specifies otherwise EMSL will attempt to classify Chrysotile to at least a level of "CD" (table D.1 page 37 of the method) and Amphiboles to at least "ADX" (table D.2 page 37 of the method), both tables are located section 16 of this procedure.
- **9.6.18** Record at least one SAED pattern on film for each type of asbestos, per sample. Record diffraction information on the sample worksheet. See SAED SOP for detailed information on obtaining and recording SAED patterns.
- 9.6.19 At least 1 EDXA spectra per asbestos and non-asbestos type, per sample should be printed out or alternately stored to disk as a .pgt file if possible. Record EDXA information on the sample worksheet.
- **9.6.20** Record all structure information on the bench sheet as follows:
  - **9.6.20.1** Primary Structure # <u>Tabulate Primary Structures SEQUENTIALLY</u>
    - **9.6.20.2** Total Structure # Tabulate Total Structures SEQUENTIALLY
    - **9.6.20.3** Length of all structures, both primary and total.
    - **9.6.20.4** Width of all structures, both primary and total.
    - **9.6.20.5** Level of ID (for primary structures, this will be the highest level of identification achieved on the associated total structures).
    - **9.6.20.6** Mineral Identification





Revision Date: March 1, 2013 Effective Date: March 15, 2013

9.6.20.7	Sketches and/or comments
9.6.20.8	Any photo ID information for negatives taken.
9.6.20.9	Whether the EDXA was printed/saved and which structure it corresponds to.

**9.6.21** Record the structure using the following notation.

9.6.21.1	Structure types and abbreviations a	re:
----------	-------------------------------------	-----

F - Fiber
B - Bundle
CD - Cluster (disperse)
CF - Cluster Fiber
CB - Cluster Bundle
CR - Cluster residual
CC - Cluster (compact)
MD - Matrix (disperse)
MF - Matrix Fiber
MB - Matrix Bundle
MR - Matrix Residual
MC - Matrix (compact)

**9.6.21.2** Following all primary structures, except for fibers (F) and bundles (B), are two characters:

9.6.21.2.1 The first character is an estimate of fibers and bundles comprising the structure. A "+" is recorded when there are more than 9 component fibers or bundles within a structure.

The second character corresponds to the number of component structures described in the first character that are also > 5µm. A "+" is recorded when there are more than 9 subcomponents that are > 5 µm.

9.6.21.3 Record the 5 largest prominent subcomponents, (fibers or bundles), that makeup the structure. Fill in the same information on the bench sheet for these compositional structures as were completed for the primary structures. Do not record more than 5 prominent subcomponents for each primary structure.

- 9.6.21.4 If, after accounting for prominent component structures, a group of clustered fibers remains, record this as a residual. Do not record more than 5 residuals for each primary structure. A residual is measured and assigned a two digit number derived in the same manner as specified in the primary structure coding above.
- 9.6.21.5 Definitions for the various structure types are found in the definitions section of this SOP. Example sketches of various structure types are found in the ISO 10312 method and in section 16 of this procedure.
- **9.6.22** Examples of proper syntax follows:
  - **9.6.22.1 MD31** is a disperse matrix comprised of 3 distinct fibers or structures one of which is greater than 5 microns.
  - **9.6.22.2 MC30** is a compact matrix estimated to have no more than 3 subcomponents none of which is greater than 5 microns.



Revision Date: March 1, 2013 Effective Date: March 15, 2013



**9.6.22.3 CD+1** is a disperse cluster with more than 9 fibers or structures ("+" means more than 9), one greater than 5 microns.

**9.6.22.4 MD+0** is a disperse matrix with more than 9 fibers or structures none greater than 5 microns.

#### 9.6.23 Stopping Criteria

**9.6.23.1** Analysis may be terminated after the required analytical sensitivity is achieved, or

**9.6.23.2** Analysis may be terminated after the completion of analysis of the grid opening in which the 100<sup>th</sup> structure was encountered.

**9.6.23.3** Regardless of which step above terminates the analytical process, a minimum of 4 grid openings must be analyzed.

#### **9.6.24** Blanks

9.6.24.1 One laboratory blank must be processed with each slide of samples. (Only 1 of the laboratory blanks needs to be analyzed per Order ID.)

**9.6.24.2** At least one field blank should be processed with each batch of samples.

**9.6.24.3** Filter lot blanks are required at a minimum of 2 for every 100 filters.

9.6.25 There is no method required analytical sensitivity for this procedure; the target analytical sensitivity is set with mutual consent of the client. Unless otherwise specified EMSL will analyzed sample to an analytical sensitivity of 0.005 s/cc.

9.6.26 A Pass / Fail Criteria is not given for this method.

#### 10.0 Calculations

Examples of the calculations are displayed below. Variables include but are not limited to the volume and GOA (grid opening area) which is calculated with every batch of grids.

EFA - Effective filter area of a 25mm cassette = 385mm<sup>2</sup>

GOA - Grid opening area (0.00635 mm<sup>2</sup>)

N - Number of fibers (If N=0 then default to 1 structure)

V - Volume (1200 liters)

AA - Area analyzed (0.06985 mm<sup>2</sup>)

NO - Number of grid openings analyzed (11)

n - Number of Samples

AS - Analytical Sensitivity (s/L)

F - Fraction of the original sample filter sampled for resuspension

V<sub>d</sub> - Final volume of the sample suspension (ml)

V<sub>f</sub> - Volume of the final sample suspension filtered for analysis (ml)

#### **10.1** Area analyzed (AA)

$$AA = GOAxNO$$
  $AA = 0.00635mm^2x11$   $AA = 0.06985mm^2$ 

#### **10.2** Structures per square millimeter (str/mm²)

$$str/mm^2 = \frac{NxV_d}{AAxV_f xF}$$
  $str/mm^2 = \frac{1x40}{0.06985mm^2 x34x0.5}$   $str/mm^2 = 33.7$ 



Revision Date: March 1, 2013

Effective Date: March 15, 2013

Structures per cubic centimeter of air (str/L)

$$L = \frac{EFAxNxV_d}{AAxVxV_f xF} \qquad str/cc = \frac{385mm^2 x5x40}{0.06985mm^2 x1800Lx34x0.5} = 36$$

10.4 Analytical Sensitivity (AS)

> To calculate the analytical sensitivity, calculate the str/L using 1 for the number of structures (N) in the formula above.

$$str/L = \frac{EFAxNxV_d}{AAxVxV_fxF}$$
  $AS = \frac{385mm^2x1x40}{0.06985mm^2x1800Lx34x0.5} = 7.2$ 

10.5 Grid Openings (NO) required to be analyzed.

Grid Openings (NO) required to be analyzed. The number of grid openings (NO) that are required to be analyzed is dependent on grid opening area (GOA), the volume (V) of sample collected and the requested analytical sensitivity (AS) of the analysis.

$$NO = \frac{EFAxV_d}{ASxGOAx1000xVxFxV_f}$$

$$NO = \frac{385x40}{5x0.00635x1800x0.5x34} = 15.9 = 16$$

#### Important!!

For this calculation round the result to an integer and ALWAYS round the result UP regardless of rounding rules. As an example, even though the first decimal place may be below 5 (10.1), the result is rounded up to produce 11 required openings.

#### 11.0 Reporting

The following items are included in the final report (on EMSL letterhead) to the client:

- Asbestos concentration in structures/liter (str/L) categorized as follows
  - **11.1.1** Total aspestos concentration for all aspestos types meeting minimum Identification required, CM and ADX
    - 11.1.1.1 For sample where 3 or less structures were counted, report the concentration as less than the corresponding one-sided upper 95% confidence limit for the Poisson distribution.
    - 11.1.1.2 For samples where 4 or greater structures are counted report the mean asbestos structure concentration.
  - **11.1.2** Chrysotile asbestos concentrations are reported as follows:
    - 11.1.2.1 Determine which identification and structure categories will be used to calculate the concentration. This is specified by the client. If it is not specified by the client, use all Total structures identified as CD or
    - **11.1.2.2** For sample where 3 or less chrysotile structures were counted, report the concentration as less than the corresponding one-sided upper 95% confidence limit for the Poisson distribution.
    - **11.1.2.3** For samples where 4 or greater structures are counted report both 11.1.2.3.1 The mean chrysotile concentration

Revision 1.2

Revision Date: March 1, 2013 Effective Date: March 15, 2013



- 11.1.2.3.2 The upper and lower chrysotile concentrations based on the corresponding two-sided Poisson 95% confidence intervals.
- **11.1.3** Total amphibole asbestos concentrations are reported as follows:
  - **11.1.3.1** Determine which identification and structure categories will be used to calculate the concentration. This is specified by the client. If it is not specified by the client, use all Total structures identified as ADX or
  - **11.1.3.2** For samples where 3 or less amphibole structures were counted, report the concentration as less than the corresponding one-sided upper 95% confidence limit for the Poisson distribution.
  - **11.1.3.3** For samples where 4 or greater structures are counted report both
    - The mean amphibole concentration 11.1.3.3.1
    - The upper and lower amphibole concentrations based on 11.1.3.3.2 the corresponding two-sided Poisson 95% confidence intervals.
- The compositional data for the principle varieties of amphiboles present (if any). 11.2
- 11.3 The analytical sensitivity in str/L.
- The detection limit in str/L. 11.4
- Total number of primary structures counted. 11.5
- 11.6 Number of total asbestos structures counted.
- 11.7 Number of asbestos structures > 5µm.
- Number of asbestos fibers and bundles (combined) > 5µm. 11.8
- Number of PCM equivalent asbestos structures. 11.9
- 11.10 Number of PCM equivalent asbestos fibers.
- **11.11** The level of analysis used when identifying chrysotile and amphibole structures.
- **11.12** Aspect ratio used to define fibers for the analysis.
- **11.13** Reference to the Method.
- 11.14 Client Identification and contact information
- 11.15 EMSL's order ID.
- **11.16** Date of receipt of the samples.
- 11.17 Date of sample collection (if provided).
- **11.18** Date of the report.
- **11.19** EMSL and client sample ID numbers.
- **11.20** Volume of air collected in the sample.
- **11.21** Effective filter area of the sample media.
- **11.22** Area of the grid openings used during analysis.
- **11.23** Microscope magnification used during analysis.
- **11.24** Initials of the analyst.
- **11.25** Count sheets are supplied with final results.

#### 12.0 **Method Performance**

Method performance data can be found in the method Ambient air - Determination of asbestos fibres - Indirect-transfer transmission electron microscopy method ISO 13794 First edition 1999-07- 15.

#### 12.1 MDL

When no structures are counted the detection limit is considered to be the upper limit of the one-sided Poison confidence interval, or 2.99 structures

Revision Date: March 1, 2013 Effective Date: March 15, 2013



12.2 DOC's

Demonstrations of Capability are required for each analytical method.

12.3 PT's

Proficiency tests do not exist for this procedure at the current time.

12.4 Accuracy

Since it is not possible to create a standard filter with a known structure concentration the exact accuracy of the method cannot be determined.

#### 12.5 Precision

The analytical precision is dependent on the number of structures counted and the uniformity of the particulate deposit. Assuming a uniform distribution and structure loading of at least 3.5 structures per grid opening, the coefficient of variance of the counting procedure can be estimated at 10%. It should be noted that in practice, particulate deposits obtained by filtration of ambient air samples are rarely ideally distributed.

#### 13.0 Quality Control

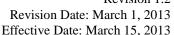
- **13.1** All QC data must be maintained and available for easy reference and inspection.
- 13.2 Blanks
  - **13.2.1** Laboratory Blanks: A lab blank is included with each slide of samples prepared. The last slide's lab blank will be prepared for analysis. At least one lab blank for every Order ID should be analyzed.
  - **13.2.2** Field Blanks: at least 1 field blank should be processed with each sample set.
  - **13.2.3** Lot Blanks: A minimum of 2 unused filters from each filter lot of 100 will be analyzed.
  - **13.2.4** Process Blank Asher: A clean blank beaker that is included with and processed through each step of the procedure including plasma ashing, filtration, preparation and analysis.
  - **13.2.5** Process Blank Filtration: A quantity of water (at least 40ml), filtered, prepared and analyzed along with the samples.
- **13.3** Inter-Analyst QC is at least 4% reanalysis of the same grid openings.
- **13.4** Intra-Analyst QC is at least 2% reanalysis of the same grid openings.
- **13.5** Verified analysis is used for training and will be performed as need for discordant sample QC reconciliation. .
- **13.6** 0.5% repreparation and analysis of sample filter QC.
- **13.7** Inter-laboratory analysis are performed in order to monitor systematic errors among microscopists as needed or as determined by client.

#### 14.0 Data Assessment

#### 14.1 Acceptance criteria for QC measures

These are addressed in the EMSL's QA Manual Module A section A.12.6.2

- **14.1.1** If a sample falls outside the acceptable limits it needs to be reconciled with participating analysts and/or a third analyst when necessary.
- **14.1.2** The Pass/Fail criteria for repeat results (inter and intra analyst) should not differ at the 5% significance level.
- **14.1.3 For** Verified Analysis, the results should be  $\leq 80$  % of true positives,  $\leq 20$ % false negatives, and  $\leq 10$  % false positives of the Total Structure count.
- **14.1.4** Repreparations from different sectors of the filter is a test for the reproducibility of the whole method. Since this type of QC is impacted by circumstances outside





the control of the analysis, no statistical evaluation is currently performed on this

data. The data is simply compiled for informational purposes only. **14.1.5** Criteria for the maximum allowable contamination levels for laboratory blanks: on MCE Filters, a cumulative average of < 5 str/mm<sup>2</sup>, or any single preparation

individual less than 15. For PC filters, a cumulative average of <18 structures/mm<sup>2</sup>, or any single preparation < 53 structures/mm<sup>2</sup>.

**14.1.6** The Chi Square uniformity test shall be conducted using the number of primary structures found on individual grid openings. This value will be compared with significance points of the chi square distribution. If the structure count fails this test, the precision of the result may be in question, and if new air samples cannot be collected, additional grid openings may need to be examined or alternatively the sample should be prepared indirectly by ISO 13794.

#### 14.2 Corrective actions

These policies are addressed fully in the EMSL's QA manual section 18.

- **14.2.1** All corrective actions should look for the root cause of the error.
- 14.2.2 All out of control or unacceptable data must be brought to the attention of the Laboratory Manager.
- 14.2.3 The Laboratory manager is responsible for generating a corrective action including an investigation of calibration procedures, a review of analytical technique and investigation of training policies and compliance.
- 14.2.4 Corrective actions will be reported to the QA Department by means of the Quarterly Management Report or sooner when appropriate.
- 14.3 Contingencies for handling out-of control or unacceptable data.

Any quality control requirements not met must have an explanation to their Non-conformance.

#### 15.0 Pollution Prevention / Waste Management

#### 15.1 Pollution Prevention

EMSL Analytical makes all efforts to reduce the volume and toxicity of the waste generated by the laboratory. An effort to manage procurement of hazardous materials has been implemented in order to avoid over ordering. Hazardous waste is classified for proper disposal.

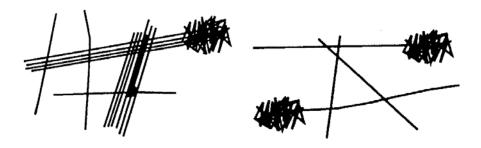
#### 15.2 Waste Management

The waste generated during prep and analysis will be disposed of following safety procedures outlined in the chemical hygiene plan (EMSLChemHygiene 200.0).

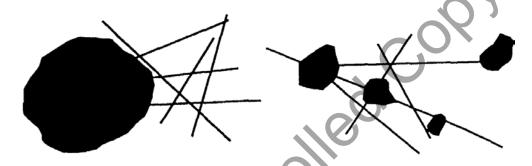


### 16.0 Tables, Diagrams, Flowcharts, and Validation Data

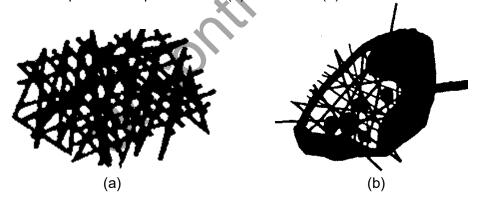
Example of disperse clusters: 16.1



16.2 Examples of disperse matrices:



Examples of compact Cluster (a) and Matrix (b): 16.3





Revision Date: March 1, 2013 Effective Date: March 15, 2013

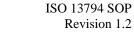
#### 16.4 Structure Classification Tables

Table D.1 — Classification of fibres with tubular morphology

Category	Description	
TM	Tubular Morphology, not sufficiently characteristic for classification as chrysotile	
CM	Characteristic Chrysotile Morphology	
ÇD	Chrysotile SAED pattern	
CO	Chrysotile composition by Quantitative EDXA	
СМО	Chrysotile Morphology and composition by Quantitative EDXA	
CDQ	Chrysotile SAED pattern and composition by Quantitative EDXA	
NAM	Non-Asbestos Mineral	

Table D.2 — Classification of fibres without tubular morphology

Category	Description
UF	Unidentified Fibre
AÐ	Amphibole by random orientation SAED (shows layer pattern of 0,53 nm spacing)
AX	Amphibole by qualitative EDXA. Spectrum has elemental components consistent with amphibole
ADX	Amphibole by random orientation SAED and qualitative EDXA
AQ	Amphibole by Quantitative EDXA
AZ	Amphibole by one Zone-axis SAED pattern
ADQ	Amphibole by random orientation SAED and Quantitative EDXA
AZQ	Amphibole by one Zone-axis SAED pattern and Quantitative EDXA
AZZ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle
AZZQ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle, and Quantitative EDXA
MAM	Non-Asbestos Mineral



Revision Date: March 1, 2013 Effective Date: March 15, 2013



#### 16.5 Identification Flowchart for fibers with Tubular Morphology

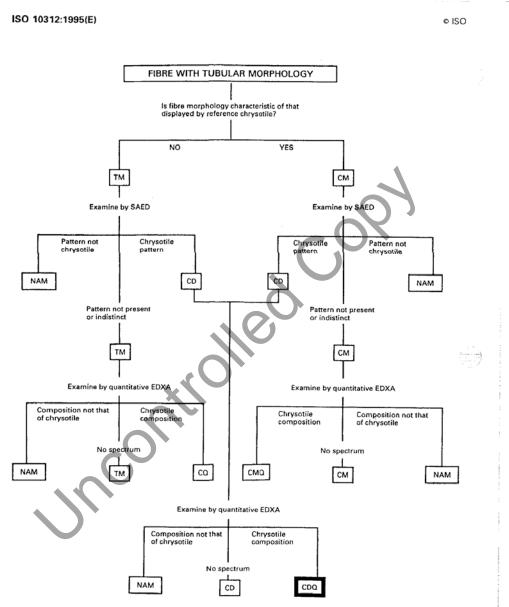
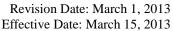


Figure D.2 — Classification chart for fibre with tubular morphology

38







#### 16.6 Identification Flowchart for fibers without Tubular Morphology

o ISO ISO ISO 10312:1995(E)

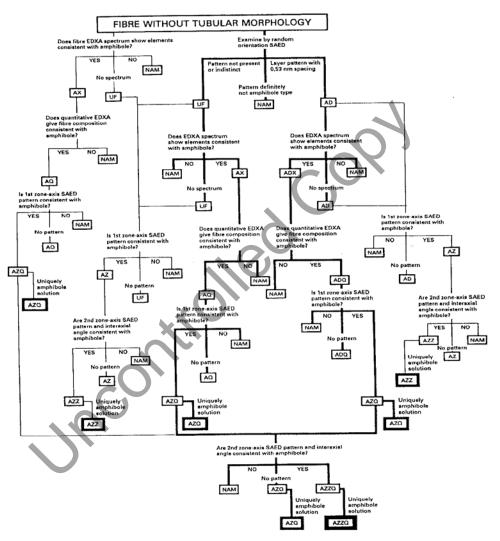


Figure D.4 — Classification chart for fibre without tubular morphology

41

Revision 1.2
Revision Date: March 1, 2013
Effective Date: March 15, 2013



#### 17.0 References

- **17.1** Ambient air Determination of asbestos fibres Indirect-transfer transmission electron microscopy method ISO 13974 First edition 1997- 07- 15.
- **17.2** Ambient air Determination of asbestos fibres Direct-transfer transmission electron microscopy method ISO 10312 First edition 1995- 05- 01.
- 17.3 Less is Better- Guide to Minimizing Waste in Laboratories prepared by the Task Force on
- 17.4 Laboratory Environment, Health and Safety-American Chemical Society 2002.
- **17.5** EMSL Quality Assurance Manual
- 17.7 EMSL Chemical Hygiene Plan

#### 18.0 Revision History

Revision #	Date	Revision	Initials
0.0	3/08/09	First initiation of this SOP	RD
1.0	5/13/09	QC pass fail criteria requirements (14.1,2) were changed to more closely mirror AHERA	KN
1.1	2/15/13	Corrected stated ISO method number. Clarification on which filters to use during filtration. Changes for grammatical errors.	LB
1.2	3/1/13	Minor formatting, grammatical changes and correct method revision date, changed calculations to s/L instead of s/cc.	KN

Authorizing Signatures		
Ken Najuch Author (Print)	Author Signature	03/01/2013 Date
Luis H. Bustillos  Reviewer (Print)	Reviewer Signature	03/01/2013 ————————————————————————————————————
Ed Cahill  Corporate Approval (Print)	Corporate Approval Signature	03/01/2013 ————————————————————————————————————



Revision Date: March 1, 2013 Effective Date: March 15, 2013

SOP Review and Acknowledgement Form Instructions: Ensure all personnel in laboratory affected by this procedure read and acknowledge. Once completed, the page shall be retained in an Acknowledgments binder along with personnel training.

Laboratory Manager Acknowledgement My signature below signifies that I have read and understand the entire conter represents that I agree to fully comply with, implement, and enforce all require specified in these procedures set forth in this document and any supporting re	ments, procedures, and protocols
Laboratory Manager Signature	 Date
, , ,	.01
Laboratory Staff Acknowledgement My signature below signifies that I have read and understand the entire conter represents that I agree to fully comply with, implement, and enforce all require specified in these procedures set forth in this document and any supporting re	ments, procedures, and protocols
Signature	Date



# **Accredited Laboratory**

A2LA has accredited

# EMSL ANALYTICAL, INC.

Cinnaminson, NJ

for technical competence in the field of

## **Environmental Testing**

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2016 TNI Environmental Testing Laboratory Standard, and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.4 of the DoD Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

SEAL 1978 VIOLENTIA AZLA

Presented this 5th day of August 2022.

Vice President, Accreditation Services For the Accreditation Council

Certificate Number 2845.01

Valid to July 31, 2024



#### SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

#### EMSL ANALYTICAL, INC. 200 Route 130 North Cinnaminson, NJ 08077

Nicholas Straccione Phone: 856-303-2550

#### **ENVIRONMENTAL**

Valid To: July 31, 2024 Certificate Number: 2845.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with the 2016 TNI Environmental Testing Laboratory Standard, the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.4 of the DoD/DOE Quality Systems Manual for Environmental Laboratories), and for the test methods applicable to the National Lead Laboratory Accreditation Program (NLLAP), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

ENVIRONMENTAL LEAD		
Test	Test Method(s)	
Total Lead (Pb) in Dust Wipes	EMSL Analytical, Inc. LM-007C (Modified EPA 7000B - (FLAA), 3050 Hotblock Digestion)	
Total Lead (Pb) in Paint Chips	EMSL Analytical, Inc. LM-007B (Modified EPA 7000B - (FLAA), 3050 Hotblock Digestion)	
Total Lead (Pb) in Soil	EMSL Analytical, Inc. LM-007A (Modified EPA 7000B - (FLAA), 3050 Hotblock Digestion)	

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform the following tests in the analyte categories listed below:

AIR MATRIX*		
Test	Test Method(s)	Parameter/Analyte
Acetic Acid	NIOSH 1603 mod.	Acetic Acid
Acid Mist	OSHA 165SG	Nitric Acid
		Hydrochloric Acid
		Sulfuric Acid
		Hydrofluoric Acid
		Hydrobromic Acid
Alcohols	NIOSH 1400 mod.	Isopropyl Alcohol
		Ethanol
Aldrin and Lindane	NIOSH 5502 mod.	Aldrin and Lindane

Test	Test Method(s)	Parameter/Analyte
Aromatic Hydrocarbons	NIOSH 1501 mod.	Benzene Ethylbenzene Toluene o-Xylene p-Xylene m-Xylene
Chlordane	NIOSH 5510 mod.	Chlordane
Chlorine	NIOSH 6011	Chlorine
Combustion-by-Products	ASTM D6602	Black Carbon/Soot
(Black Carbon/Soot, Char, and Ash)	ASTWI DOUGZ	Char
(Black Carbon/Soot, Char, and Ash)		Ash
Diesel Particulate Matter (As Elemental	NIOSH 5040	Elemental Carbon
Carbon)	1410511 3040	Elementar Carbon
Elements by ICP	NIOSH 7300	Aluminum (Al)
Lichions by ici	NIOSH 7300 mod.	Antimony (Sb)
	NIOSH 7303	Arsenic (As)
	NIOSH 7303 mod.	Barium (Ba)
	1410511 7505 mod.	Beryllium (Be)
		Bismuth (Bi)
		Boron (B)
		Cadmium (Cd)
		Cerium (Ce)
		Chromium (Cr)
		Cobalt (Co)
		Copper (Cu)
		Iron (Fe)
		Lead (Pb)
		Lithium (Li)
		Magnesium (Mg)
		Manganese (Mn)
		Molybdenum (Mo)
		Nickel (Ni)
		Phosphorous (P)
		Potassium (K)
		Selenium (Se)
		Silver (Ag)
		Sodium (Na)
		Strontium (Sr)
		Sulfide (S)
		Thalium (Tl)
		Tin (Sn)
		Titanium (Ti)
		Vanadium (V)
		Zinc (Zn)
		Zircon (Zr)
Formaldehyde	NIOSH 2016 mod.	Formaldehyde

Test	Test Method(s)	Parameter/Analyte
Halogenated Hydrocarbons	NIOSH 1003 mod.	1,2-dichlorobenzene 1,4-dichlorobenzene Chlorobenzene 1,1-dichloroethane 1,1-dichloroethene 1,2-dichloroethane 1,2-dichloroethene Carbon Tetrachloride Chloroform Tetrachloroethene
Hexavalent Chromium	OSHA 215	Haxavalent Chromium
Hydrogen Cyanide	NIOSH 6010 mod.	Hydrogen Cyanide
Inorganic Acids	NIOSH 7903	Flourine (F) Bromie (Br) Chlorine (Cl) Nitrate (NO <sub>3</sub> ) Nitrite (NO <sub>2</sub> ) Sulfate (SO <sub>4</sub> ) Phosphate (PO <sub>4</sub> )
Inorganic Fibrous Particles by SEM method	German VDI 3492	Fibrous Glass Mineral Wool Refractory Ceramic Fibers Asbestos
Inorganic Fibrous Particles by SEM method	ISO 14966	Fibrous Glass Mineral Wool Refractory Ceramic Fibers Asbestos
Mercury	NIOSH 6009 mod., OSHA 140 mod.	Mercury
Metalworking Fluids (MWF) All Categories	NIOSH 5524	Metal Working Fluids N.O.S.
Methamphetamine on Wipes	NIOSH 9111	Methamphetamine
Methanol	NIOSH 2000 mod.	Methanol
Methylene Chloride	NIOSH 1005	Methylene Chloride
Naphthas	NIOSH 1550 mod.	WMP Naphtha
Ozone	OSHA 214	Ozone
Polychlorinated Biphenyls	NIOSH 5503 mod.	Aroclor-1016 Aroclor-1221 Aroclor-1232 Aroclor-1242 Aroclor-1248 Aroclor-1254 Aroclor-1260 Aroclor-1262 Aroclor-1268

Test	Test Method(s)	Parameter/Analyte
Polynuclear Aromatic Hydrocarbons by HPLC	NIOSH 5506 mod.	Naphthalene Acenaphthylene Acenaphthene Fluorene Phenanthrene Anthracene Fluoranthene Pyrene Benzo(a)anthracene Chrysene Benzo(e)pyrene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
Silica, Crystalline	NIOSH 7500 mod., OSHA 142	Indeno(1,2,3-c,d)pyrene a-quartze Cristobalite Trydimite
Sulfur Dioxide	NIOSH 6004 mod.	Sulfur Dioxide
Total Lead (Pb)	NIOSH 7082 - (FLAA)	Lead
Total Metals	EMSL Analytical, Inc. LM-003 (Modified NIOSH 7300 for ICP/ICP-MS)	Beryllium Oxide (BeO) Beryllium (Be)
Volatile Organic Compounds	TO-15	Acetaldehyde Acetone Acetonitrile Acetophenone Acrolein Acrylamide Acrylic acid Acrylonitrile Allyl chloride Aniline Benzene Benzyl chloride Bis (2-chloroethyl) ether Bis (chloromethyl) ether Bromodichloromethane Bromoform Bromomethane Butadiene (1,3-) Butadiene (2-chloro-1,3-) Carbon disulfide Carbon oxysulfide (Carbonyl sulfide)

Test	Test Method(s)	Parameter/Analyte
Volatile Organic Compounds in Air	TO-15 (cont)	Carbon tetrachloride
(cont)		Catechol
		Chloroacetic acid
		Chlorobenzene
		Chloroethane
		Chloroform
		Chloromethane
		Chloromethyl methyl ether
		Chlorotoluene (2-)
		Cresols/Cresylic acid
		Cyclohexane
		Diazomethane
		Dibromo-3-chloropropane (1,2-)
		Dibromochloromethane
		Dibromoethane (1,2-) (EDB)
		Dichlorobenzene (1,2-)
		Dichlorobenzene (1,3-)
		Dichlorobenzene (1,4-)
		Dichlorodifluoromethane
		Dichloroethane (1,1-)
		Dichloroethane (1,2-)
		Dichloroethene (1,1-)
		Dichloroethene (cis-1,2-)
		Dichloroethene (trans-1,2-)
		Dichlorofluoromethane
		Dichloropropane (1,2-)
		Dichloropropene (cis-1,3-)
		Dichloropropene (trans-1,3-)
		Dichlorotetrafluoroethane (1,2-)
		Diethyl sulfate
		Dimethyl formamide (N, N-)
		Dimethyl hydrazine (1,1-)
		Dimethyl sulfate
		Dimethylaniline (N, N-)
		Dimethylcarbamoyl chloride
		Dioxane (1,4-)
		Epichlorohydrin
		Epoxybutane (1,2-)
		Ethanol
		Ethyl acetate
		Ethyl acrylate
		Ethyl carbamate (Urethane)
		Ethylbenzene
		Ethylene Oxide
		Ethyleneimine
		Ethyltoluene (4-)
		Formaldehyde
		Heptane (n-)
		Hexachlorobutadiene (1,3-)
	L	Tieracinorouadiene (1,5-)

Test	Test Method(s)	Parameter/Analyte
Volatile Organic Compounds in Air	TO-15 (cont)	Hexachloroethane
(cont)		Hexane (n-)
		Hexanone (2-)
		Isophorone
		Isopropanol
		Isopropylbenzene
		Methyl alcohol (Methanol)
		Methyl ethyl ketone (MEK)
		Methyl iodide
		Methyl isobutyl ketone (MIBK)
		Methyl isocyanate
		Methyl methacrylate
		Methyl tert-butyl ether
		Methylene chloride (Dichloromethane)
		Methylhydrazine
		Methylphenol (2-)
		Naphthalene
		Nitrobenzene
		Nitropropane (2-)
		N-Nitrosodimethylamine
		N-Nitrosomorpholine
		N-Nitroso-N-methylurea
		Phenol
		Phosgene
		Propane sultone (1,3-)
		Propiolactone (beta-)
		Propionaldehyde
		Propylene
		Propylene oxide
		Propyleneimine (1,2-)
		Styrene
		Styrene oxide
		Tert-butyl alcohol
		Tetrachloroethane (1,1,2,2-)
		Tetrachloroethene
		Tetrahydrofuran
		Toluene
		Trichloro (1,1,2-) trifluoroethane (1,2,2-)
		Trichlorobenzene (1,2,4-)
		Trichloroethane (1,1,1-)
		Trichloroethane (1,1,2-)
		Trichloroethene
		Trichlorofluoromethane
		Triethylamine
		Trifluoromethane
		Trimethylbenzene (1,2,4-)
		Trimethylbenzene (1,3,5-)

Test	Test Method(s)	Parameter/Analyte
Volatile Organic Compounds in Air (cont)	TO-15 (cont)	Trimethylpentane (2,2,4-) Vinyl acetate Vinyl bromide Vinyl chloride Xylene (m-) Xylene (o-) Xylenes (total) Xylene (p-)

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform the following tests in the analyte categories listed below:

SOIL/SOLIDS/BULK MATRIX*		
Test	Test Method(s)	Parameter/Analyte
Combustion-by-Products (black carbon/soot, char and ash)	ASTM D6602	Black Carbon/Soot Char Ash
Determination of Asbestos in Technical Products by SEM method	German VDI 3866 Part 5	Asbestos
Separatory Funnel Liquid/Liquid Extractions	EPA 3510C	
Microwave Sample Preparation	EPA 3546	
Polychlorinated Biphenyls (PCBs)	EPA 8082A	Aroclor-1016 Aroclor-1221 Aroclor-1232 Aroclor-1242 Aroclor-1248 Aroclor-1254 Aroclor-1260 Aroclor-1262 Aroclor-1268
Silica Gel Cleanup	EPA 3630C	
Soxhlet Sample Preparation	EPA 3540C	
Sulfur Extract Cleanup	EPA 3660B	
Sulfuric Acid Cleanup	EPA 3665A	
Waste Dilution Sample Preparation	EPA 3580A	

<sup>\*</sup>Not NLLAP program

Page 7 of 1

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform the following tests in the analyte categories listed below:

ASBESTOS ANALYSIS		
Test	Test Method(s)	Parameter/Analyte
Phase Contrast Microscopy	NIOSH 7400	Asbestos: Chrysotile Asbestos: Amosite Asbestos: Crocidolite Asbestos: Anthophyllite Asbestos: Tremolite Asbestos: Actinolite
Polarized Light Microscopy	SAE J2975, EPA 600/R-93/116 NIOSH 9002 ASTM D7521-16	Asbestos: Other non-regulated amphibole fibers  Asbestos: Chrysotile Asbestos: Amosite Asbestos: Crocidolite Asbestos: Anthophyllite Asbestos: Tremolite Asbestos: Actinolite Asbestos: Other non-regulated amphibole fibers
Sample Preparation by Drilling	SAE J2975	Asbestos: Chrysotile Asbestos: Amosite Asbestos: Crocidolite Asbestos: Anthophyllite Asbestos: Tremolite Asbestos: Actinolite Asbestos: Other non-regulated amphibole fibers
Transmission Electron Microscopy – Air	ISO 10312 (direct method)	Asbestos: Chrysotile Asbestos: Amosite Asbestos: Crocidolite Asbestos: Anthophyllite Asbestos: Tremolite Asbestos: Actinolite Asbestos: Other non-regulated amphibole fibers
Transmission Electron Microscopy – Bulk	ISO 13794 (indirect method)	Asbestos: Chrysotile Asbestos: Amosite Asbestos: Crocidolite Asbestos: Anthophyllite Asbestos: Tremolite Asbestos: Actinolite Asbestos: Other non-regulated amphibole fibers
Transmission Electron Microscopy – Surfaces	ASTM D6480-99 ASTM D5755-95	Asbestos: Chrysotile Asbestos: Amosite Asbestos: Crocidolite Asbestos: Anthophyllite Asbestos: Tremolite Asbestos: Actinolite Asbestos: Other non-regulated amphibole fibers

Test	Test Method(s)	Parameter/Analyte
Transmission Electron Microscopy –	ASTM D7521-16	Asbestos: Chrysotile
Soil		Asbestos: Amosite
		Asbestos: Crocidolite
		Asbestos: Anthophyllite
		Asbestos: Tremolite
		Asbestos: Actinolite
		Asbestos: Other non-regulated amphibole fibers
Transmission Electron Microscopy	EPA 100.2	Asbestos: Chrysotile
		Asbestos: Amosite
		Asbestos: Crocidolite
		Asbestos: Anthophyllite
		Asbestos: Tremolite
		Asbestos: Actinolite
		Asbestos: Other non-regulated amphibole fibers

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform the following tests in the analyte categories listed below:

RADIOCHEMISTRY and CHEMISTRY DRINKING WATER, NON_POTABLE, SOLID/CHEMICAL MATRIX		
Parameter/Analyte	Test Method(s)	
Alpha Spectroscopy (Pu-238, Pu-239/240, U-235, U-234/238, Am-241, Th-230/232)	EMSL RC-SOP-007, EPA 907.0 mod.	
Alpha/Beta Scan	EMSL RC-SOP-003, EPA 900 mod.	
Gamma Scan	EMSL RC-SOP-002, EPA 901.1 mod.	
Gross Alpha/Beta	EPA 900, EPA 900 mod.	
Nickel (Ni-63)	EMSL RC-SOP-201	
Radium (Ra-226)	EPA 903, EPA 903 mod.	
Radium (Ra-228)	EPA 904, EPA 904 mod.	
Strontium (Sr-89/-90)	EPA 905, EPA 905 mod.	
Tritium	EPA 906, EPA 906 mod.	
Total Organic Carbon in Water and Wastewater; Persulfate Oxidation Method SM 5310C	MS-SOP-R2	

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform the following tests on  $\underline{\text{Children's Products:}}^{(1)}$ 

CHEMICAL		
Test Method(s)		
Lead in Paint and Surface Coatings	16 CFR 1303 (using ASTM E1613 and E1645); CPSC-CH-E1003-09.1	
Phthalates	CPSC-CH-C1001-09.4 (using EPA SW-846 8270)	
Soluble Heavy Metals Content	ASTM F 963-17 Section 4.3.5.1 & Section 4.3.5.2	
(As, Ba, Cd, Cr, Pb, Hg, Sb, Se)	(EMSL Analytical, Inc. LM-032)	

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Test	Test Method(s)
Total Cadmium in Children's Metal Products	EMSL Analytical, Inc. LM-016, (Modified CPSC-CH-
Including Children's Metal Jewelry	E1001-08.1)
Total Cadmium in Children's Non-Metal Products	EMSL Analytical, Inc. LM-016, (Modified CPSC-CH-
	E1002-08)
Total Lead in Children's Metal Jewelry	CPSC-CH-E1001-08.1
Total Lead in Children's Metal Products	CPSC-CH-E1001-08.1
Total Lead in Children's Non-Metal Products	CPSC-CH-E1002-08

<sup>&</sup>lt;sup>1</sup> The Consumer Product Safety Improvement Act (CPSIA) requires that every children's product subject to a federal consumer product safety requirement be tested by a Consumer Product Safety Commission (CPSC) accepted laboratory for compliance with the applicable federal children's product safety requirements. Accreditation by A2LA does not infer acceptance by the CPSC. Please verify this organization's acceptance status by using the CPSC's searchable database, located at <a href="http://www.cpsc.gov/cgi-bin/labsearch/">http://www.cpsc.gov/cgi-bin/labsearch/</a>.

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO/IEC 17025:2017 and the 2016 TNI Environmental Testing Laboratory Standard), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

POTABLE, NON-POTABLE, SOLIDS/SOILS MATRIX		
Test	Test Method(s)	Parameter/Analyte
PFAS	EPA 537.1	11Cl-PF3OUdS
	EPA 537 Modified	9CL-PF3ONS
	EPA 533	ADONA
		HFPO-DA
		PFNS
		PFBA
		PFDS
		N-MeFOSAA
		N-EtFOSAA
		PFBS
		8:2 FTS
		PFDA
		PFDoA
		PFOSA
		PFHpA
		4:2 FTS
		PFHxS
		PFHxA
		PFTrDA
		PFTeDA
		PFNA
		6:2 FTS
		PFOS
		PFOA

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Test	Test Method(s)	Parameter/Analyte
PFAS	EPA 537.1, EPA 533	PFPeA
	EPA 537	PFPeS
		PFUnA
Lead	EPA 7420/7000B	Lead
Metals	SW 846 6010D	Aluminum
Wettis	SW 846 6020B	Antimony
	5 W 040 0020B	Arsenic
		Barium
		Beryllium
		Boron
		Cadmium
		Calcium
		Chromium
		Cobalt
		Copper
		Iron
		Lead
		Lithium
		Magnesium
		Manganese
		Molybdenum
		Nickel
		Phosphorus
		Potassium
		Selenium
		Silver
		Sodium
		Strontium
		Thallium
		Tin
		Titanium
		Vanadium
		Zinc
		Zirconium
Mercury	SW 846 7470B (NPW)	Mercury
Wicicary	SW 846 7471A (SCN)	Wiciedly
PCB's	SW 846 8082A	PCB 1016
TCD 3	5 17 040 000211	PCB 1221
		PCB 1232
		PCB 1232 PCB 1242
		PCB 1242 PCB 1248
		PCB 1254
		PCB 1260
		PCB 1262
		PCB 1268

Test	Test Method(s)	Parameter/Analyte
Pesticides	SW 846 8081B	Aldrin
Testicides		Alpha BHC
		Beta BHC
		Chlordane (alpha) (cis-)
		Chlordane (gamma) (trans-)
		DDD (4,4'-)
		DDE (4,4'-)
		DDT (4,4'-)
		Delta BHC
		Dieldrin
		Endosulfan I
		Endosulfan II
		Endosulfan sulfate
		Endosurran surrate Endrin
		Endrin Endrin aldehyde
		Endrin aldenyde Endrin ketone
		Heptachlor
		Heptachlor epoxide
		Lindane (gamme BHC)
		Methoxychlor
		Mirex
	GWI 046 02505	Toxaphene
Semi-Volatiles	SW 846 8270E	Acenaphthene
		Acenaphthylene
		Acetophenone
		Acetylaminofluorene (2-)
		Aminobiphenyl (4-)
		Aniline
		Anthracene
		Aramite
		Atrazine
		Benzaldehyde
		Benzidine
		Benzo(a)anthracene
		Benzo(a)pyrene
		Benzo(b)fluoranthene
		Benzo(ghi)perylene
		Benzo(k)fluoranthene
		Benzoic acid
		Benzyl alcohol
		Biphenyl (1,1'-)
		Bis (2-chloroethoxy) methane
		Bis (2-chloroethyl) ether
		Bis(2-chloroisopropyl)etherl2,2'-
		oxybis(1- chloropropane)
		Bis (2-ethylhexyl) phthalate
		Bromophenyl-phenyl ether (4-)
		Butylbenzylphthalate
		Caprolactam

Test	Test Method(s)	Parameter/Analyte
Semi-Volatiles (cont)	SW 846 8270E	Carbazole
		Chloroaniline (4-)
		Chlorobenzilate
		Chloronaphthalene (1-)
		Chloronaphthalene (2-)
		Chlorophenol (2-)
		Chlorophenyl-phenyl ether (4-)
		Chrysene
		Decane (n-)
		Diallate (cis)
		Diallate (trans)
		Dibenzo(a,h)anthracene
		Dibenzofuran
		Dichlorobenzene (1,2-)
		Dichlorobenzene (1,3-)
		Dichlorobenzene (1,4-)
		Dichlorobenzidine (3,3'-)
		Dichlorophenol (2,4-)
		Dichlorophenol (2,6-)
		Diethyl phthalate
		Dimethoate
		Dimethyl benzidine (3,3-)
		Dimethyl phthalate
		Dimethylamin oazobenzene
		Dimethylbenz(a)anthracene
		(7,12-)
		Dimethylphenol (2,4-)
		Di-n-butyl phthalate
		Dinitrobenzene (1,3-)
		Dinitrophenol (2,4-)
		Dinitrophenol (2-methyl-4,6-)
		Dinitrototuene (2,4-)
		Dinitrotoluene (2,6-)
		Di-n-octyl phthalate
		Dinoseb
		Dioxane (1,4-)
		Diphenylhydrazine / Azobenzene
		Disulfoton
		Famphur
		Fluoranthene
		Fluorene
		Hexachlorobenzene
		Hexachlorobutadiene (1,3-)
		Hexachlorocyclopentadiene
		Hexachloroethane
		Hexachlorophene
		Hexachloropropene
		Indeno(1,2,3-cd)pyrene

Test	Test Method(s)	Parameter/Analyte
Semi-Volatiles (cont)	SW 846 8270E	lsodrin
		lsophorone
		lsosafrole (cis-)
		lsosafrole (trans-)
		Kepone
		Methanesulfonate (Ethyl-)
		Methanesulfonate (Methyl-)
		Methapyrilene
		Methyl phenol (4-chloro-3-)
		Methylcholanthrene (3-)
		Methylnaphthalene(1-)
		Methylnaphthalene (2-)
		Methylphenol (2-)
		Methylphenol (3-)
		Methylphenol (4-)
		Naphthalene
		Napthoquinone (1,4-)
		Napththylamine(1-)
		Napththylamine (2-)
		Nitroaniline (2-)
		Nitroaniline (3-)
		Nitroaniline (4-)
		Nitrobenzene
		Nitrophenol (2-)
		Nitrophenol (4-)
		N-Nitrosodiethylamine
		N-Nitrosodimethylamine
		N-Nitroso-di-n-butylamine
		N-Nitroso-di-n-propylamine
		N-Nitrosodiphenylamine /
		Diphenylamine
		N-Nitrosomethylethylamine
		N-Nitrosomorpholine
		N-Nitros opiperidine
		N-Nitrosopyrrolidine
		Octadecane (n-)
		Parathian mathyl
		Parathion methyl
		Pentachlorobenzene
		Pentachlo roethane
		Pentachlo ronitrobenzene
		Phenylethylamine (alpha,alpha-
		Dimethyl)
		Phorate
		Phosphorothioate (O,O,O-
		triethyl)
		Phosphorothioate (diethyl-O-2-
		pyrazinyl) [Thionazin]
		Picoline (2-)

Semi-Volatiles (cont)  SW 846 8270E  Pronamide Pyrene Pyridine Quinoline 1-Oxide (4-Nitro) Safrole Sulfotepp Tetrachlorobenzene (1,2,4,5-) Tetrachlorobenol (2,3,4,6-) Toluidine (2-) (2-Methylamiline) Toluidine (2-) (2-Methylamiline) Toluidine (2-) (2-Methylamiline) Toluidine (3-nitro-2-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,6-) Trinitrobenzene (1,3,5-) Acenaphthene Anthracene Benzo(a)amthiacene Benzo(a)phyrene Benzo(y)phyrene Benzo(y)phyrene Benzo(y)phyrene Benzo(y)phyrene Benzo(y)phyrene Benzo(y)phyrene Benzo(y)phyrene Benzo(y)phyrene Benzo(x)hjundratene Chrysene Dibenzo(a,h)amthracene Dimethylbenz(a)amthracene Fluorene Hexachlorobtenzene Hexachlorobtenzene Hexachlorobtenzene Hexachlorobtenzene Hexachlorobtenzene Hexachlorobtenzene Hexachlorobtenzene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethyl alcohol Ethylene glycol Ethylene glycol Triethylene glycol	Test	Test Method(s)	Parameter/Analyte
Pyridine Quinoline -1-Oxide (4-Nitro) Safrole Sulfotepp Tetrachlorophenol (2,3,4,6-) Toluidine (2-) (2-Methylaniline) Toluidine (2-) (1-Methylaniline) Toluidine (2-) (1-Methylaniline) Toluidine (3-intro-2-) Trichlorophenol (2,4,6-) Trichlorophenol (2,4,6-) Trichlorophenol (2,4,6-) Trichlorophenol (2,4,6-) Trinitrobenzene (1,3,5-) Acenaphthene Acenaphthylene Anthracene Benzo(a)anthiacene Benzo(a)mthiacene Benzo(b)fluoranthene Benzo(b)fluoranthene Chrysene Dibenzo(a,h)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene (7,1,2-) Dinitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobenzene Hexachlorobenzene Hexachlorobenzene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (1-) Nethylnaphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethylene glycol Ethylene Gxide Gasoline Range Organic Methyl alcohol (Methanol) Propylene glycol	Semi-Volatiles (cont)	SW 846 8270E	Pronamide
Pyridine Quinoline -1-Oxide (4-Nitro) Safrole Sulfotepp Tetrachlorophenol (2,3,4,6-) Toluidine (2-) (2-Methylaniline) Toluidine (2-) (1-Methylaniline) Toluidine (2-) (1-Methylaniline) Toluidine (3-intro-2-) Trichlorophenol (2,4,6-) Trichlorophenol (2,4,6-) Trichlorophenol (2,4,6-) Trichlorophenol (2,4,6-) Trinitrobenzene (1,3,5-) Acenaphthene Acenaphthylene Anthracene Benzo(a)anthiacene Benzo(a)mthiacene Benzo(b)fluoranthene Benzo(b)fluoranthene Chrysene Dibenzo(a,h)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene (7,1,2-) Dinitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobenzene Hexachlorobenzene Hexachlorobenzene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (1-) Nethylnaphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethylene glycol Ethylene Gxide Gasoline Range Organic Methyl alcohol (Methanol) Propylene glycol			Pyrene
Quinoline -1-Oxide (4-Nitro) Safrole Sulfotepp Tetrachlorobenzene (1,2,4,5-) Tetrachlorophenol (2,3,4-6-) Toluidine (2-) (2-Methylaniline) Toluidine (3-) (2-Methylaniline) Toluidine (3-) (2-4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trinitrobenzene (1,3,5-) Acenaphthene Acenaphthylene Anthracene Benzo(a)pyrene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(b)fluoranthene Chrysene Dibenzo(a,h)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene Hexachlorobenzene Hexachlorobenzene Hexachlorobenzene Hexachlorobenzene Hexachlorobenzene Nethylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethylene glycol Ethylene glycol Ethylene glycol Ethylene glycol Ethylene glycol Ethylene Gvide Gasoline Range Organic Methyl alcohol (Methanol) Propylene glycol			
Sulfotepp Tetrachlorobenzene (1,2,4,5-) Tetrachlorophenol (2,3,4,6-) Toluidine (2-) (2-Methylamiline) Toluidine (5-nitro-2-) Trichlorobenzene (1,2,4-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,6-) Trinitrobenzene (1,3,5-) Acenaphthene Acenaphthylene Anthracene Benzo(a)nthiacene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(b)fluoranthene Chrysene Dibenzo(a,b)anthracene Dimethylbenz(a)anthracene Oinethylbenz(a)anthracene (7,12-) Dinitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobenzene Hexachlorobutadiene (1,3-) Indeno(1,2,3-cd)pyrene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethylene glycol Ethylene glycol Ethylene Oxide Gasoline Range Organic Methyl alcohol (Methanol) Propylene glycol			Quinoline -1-Oxide (4-Nitro)
Tetrachlorobenzene (1,2,4,5-) Tetrachlorophenol (2,3,4,6-) Toluidine (2-) (2-Methylamiline) Toluidine (5-nitro-2-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,6-) Trinitrobenzene (1,3,5-) Acenaphthene Acenaphthylene Anthracene Benzo(a)amthiacene Benzo(a)pyrene Benzo(b)fluoranthene Chrysene Dibenzo(a,b)anthracene Chrysene Dibenzo(a,b)anthracene Dimetrylbenz(a)anthracene (7,12-) Dimitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobenzene Hexachlorobutadinene (1,3-) Indeno(1,3-2-d)pyrene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethylene glycol Ethylene glycol Ethylene Range Organic Methyl alcohol Ethylene Range Organic Methyl alcohol Propylene Blycol			Safrole
Tetrachlorobenzene (1,2,4,5-) Tetrachlorophenol (2,3,4,6-) Toluidine (2-) (2-Methylamiline) Toluidine (5-nitro-2-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,6-) Trinitrobenzene (1,3,5-) Acenaphthene Acenaphthylene Anthracene Benzo(a)amthiacene Benzo(a)pyrene Benzo(b)fluoranthene Chrysene Dibenzo(a,b)anthracene Chrysene Dibenzo(a,b)anthracene Dimetrylbenz(a)anthracene (7,12-) Dimitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobenzene Hexachlorobutadinene (1,3-) Indeno(1,3-2-d)pyrene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethylene glycol Ethylene glycol Ethylene Range Organic Methyl alcohol Ethylene Range Organic Methyl alcohol Propylene Blycol			Sulfotepp
Tetrachlorophenol (2,3,4,6) Toluidine (2-) (2-Methylaniline) Toluidine (5-nitro-2-) Trichlorobenzene (1,2,4-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trinitrobenzene (1,3,5-) Acenaphthlene Acenaphthlene Acenaphthlene Andrracene Benzo(a)myrene Benzo(a)myrene Benzo(b)fluoranthene Benzo(ghi)perylene Benzo(ghi)perylene Benzo(k)fluoranthene Chrysene Dibenzo(a,h)anthracene Dimethylbenz(a)anthracene (7,12-) Dinitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobenzene Hexachlorobutadiene (1,3-) Indeno(1,2,3-cd)pyrene Methylacholanthrene (3-) Methylmaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Dieses Range Organics (DRO) Ethylene glycol Ethylene glycol Ethylene glycol Ethylene glycol Ethylene Range Organics (DRO) Ethyl alcohol Ethylene Range Organics (DRO)			
Toluidine (2-) (2-Methylaniline) Toluidine (5-nitro-2-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,5-) Trichlorophenol (2,4,6-) Trinitrobenzene (1,3,5-) Acenaphthene Acenaphthylene Anthracene Benzo(a)anthiacene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(gh)fluoranthene Chrysene Dibenzo(a,h)anthracene Dimethylben/(a)anthracene Dimethylben/(a)anthracene (7,12-) Dinitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobenzene Hexachlorobutadiene (1,3-) Indeno(1,2,3-cd)pyrene Methylchalathrene (3-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenamthrene Pyrene Diethylene glycol Ethylene Oxide Gasoline Range Orgaines (DRO) Ethylachol (Methanol) Propylene glycol			Tetrachlorophenol (2,3,4,6-)
Trichloropenzene (1,2,4-) Trichlorophenol (2,4,6-) Trichlorophenol (2,4,6-) Trinitrobenzene (1,3,5-) Acenaphthene Acenaphthene Acenaphthylene Anthracene Benzo(a)nthiacene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(b)fluoranthene Chrysene Dibenzo(a,h)anthracene Dibenzo(a,h)anthracene Dibenzo(a,h)anthracene Oinethylbenz(a)anthracene (7,12-) Dinitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobenzene Hexachlorobutadiene (1,3-) Indeno(1,2,3-cd)pyrene Methylcolanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethyl alcohol Ethylene Oxide Gasoline Range Organic Methyl alcohol (Methanol) Propylene glycol			Toluidine (2-) (2-Methylaniline)
Trichlorophenol (2,4,5-) Trichlorophenol (2,4,6-) Trinitrobenzene (1,3,5-) Acenaphthene Acenaphthylene Anthracene Benzo(a)anthiacene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(b)fluoranthene Chrysene Dibenzo(a,h)anthracene Dimethylbenz(a)anthracene (7,12-) Dinitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobutadiene (1,3-) Indenol (1,2,3-cd)pyrene Methylhaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethyl alcohol Ethylene Oxide Gasoline Range Organic Methyl alcohol (Methanol) Propylene glycol			Toluidine (5-nitro-2-)
Trichlorophenol (2,4,6-) Trinitrobenzene (1,3,5-) Acenaphthene Acenaphthylene Anthracene Benzo(a)mthiacene Benzo(a)mthiacene Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(b)fluoranthene Chrysene Dibenzo(a,h)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene (7,12-) Dinitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobutadiene (1,3-) Indeno(1,2,3-cd)pyrene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethylene glycol Ethylene glycol Ethylene glycol Ethylene oxide Gasoline Range Organic Methyl alcohol (Methanol) Propylene glycol (Methanol)			Trichlorobenzene (1,2,4-)
Trichlorophenol (2,4,6-) Trinitrobenzene (1,3,5-) Acenaphthene Acenaphthylene Anthracene Benzo(a)mthiacene Benzo(a)mthiacene Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(b)fluoranthene Chrysene Dibenzo(a,h)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene (7,12-) Dinitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobutadiene (1,3-) Indeno(1,2,3-cd)pyrene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethylene glycol Ethylene glycol Ethylene glycol Ethylene oxide Gasoline Range Organic Methyl alcohol (Methanol) Propylene glycol (Methanol)			Trichlorophenol (2,4,5-)
Trinitrobenzene (1,3,5-) Acenaphthhene Acenaphthylene Anthracene Benzo(a)aptrene Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(k)fluoranthene Chrysene Dibenzo(a,h)anthracene Dimethylbenz(a)anthracene Dimethylbenz(a)anthracene (7,12-) Dinitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobutadiene (1,3-) Indeno(1,2,3-cd)pyrene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethyl alcohol Ethylene Oxide Gasoline Range Organic Methyl alcohol (Methanol) Propylene glycol (Methanol)			
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Dinitrophenol (2-methyl-4,6-) Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobutadiene (1,3-) Indeno(1,2,3-cd)pyrene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethyl alcohol Ethylene glycol Ethylene Oxide Gasoline Range Orgainc Methyl alcohol (Methanol) Propylene glycol			
Dioxane (1,4-) Fluoran thene Fluorene Hexachlorobenzene Hexachlorobutadiene (1,3-) Indeno(1,2,3-cd)pyrene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethyl alcohol Ethylene Oxide Gasoline Range Orgainc Methyl alcohol (Methanol) Propylene glycol			
Fluorene Hexachlorobenzene Hexachlorobutadiene (1,3-) Indeno(1,2,3-cd)pyrene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethyl alcohol Ethylene glycol Ethylene Oxide Gasoline Range Orgainc Methyl alcohol (Methanol) Propylene glycol			
Hexachlorobutadiene (1,3-) Indeno(1,2,3-cd)pyrene Methylcholanthrene (3-) Methylnaphthalene (1-) Methylnaphthalene (2-) Naphthalene N-Nitrosodimethylamine Pentachlorophenol Phenanthrene Pyrene Diethylene glycol Diesel Range Organics (DRO) Ethyl alcohol Ethylene glycol Ethylene Oxide Gasoline Range Orgainc Methyl alcohol (Methanol) Propylene glycol			
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Gasoline Range Orgainc Methyl alcohol (Methanol) Propylene glycol			
Methyl alcohol (Methanol) Propylene glycol			
Propylene glycol			
Themylene grycol			Triethylene glycol

Volatiles	SW 846 8260D	Acetone Acetonitrile Acrolein Acrylonitrile Allyl chloride Benzene Bromobenzene Bromochloromethane
		Acrolein Acrylonitrile Allyl chloride Benzene Bromobenzene
		Acrylonitrile Allyl chloride Benzene Bromobenzene
		Allyl chloride Benzene Bromobenzene
		Allyl chloride Benzene Bromobenzene
		Benzene Bromobenzene
		Bromobenzene
		Bromochloromethane
		Bromodichloromethane
		Bromoform
		Bromomethane
		Butadiene (2-chloro-1,3-)
		Butanone (2-) (Methyl ethyl
		ketone)
		Butylbenzene (n-)
		Carbon disulfide
		Carbon tetrachloride
		Chlorobenzene
		Chloroethane
		Chloroethyl vinyl ether (2-)
		Chloroform
		Chloromethane
		Chlorotoluene (2-)
		Chlorotoluene (4-)
		Cyclohexane
		Cyclohexanone
		Dibromo-3-chloropropane (1,2-)
		Dibromochloromethane
		Dibromoethane (1,2-) (EDB)
		Dibromomethane
		Dichloro-2-butene (trans-1,4-)
		Dichlorobenzene (1,2-)
		Dichlorobenzene (1,3-)
		Dichlorobenzene (1,4-)
		Dichlorodinuoromethane
		Dichloroethane (1,1-)
		Dichloroethane (1,2-)
		Dichloroethene (1,1-)
		Dichloroethene(cis-1,2-)
		Dichloroethene (trans-1,2-)
		Dichloropropane (1,2-)
		Dichloropropane (1,3-)
		Dichloropropane (2,2-)
		Dichloropropene (1,1-)
		Dichloropropene (cis-1,3-)
		Dichloropropene (trans-1,3-)
		Diethyl ether (Ethyl ether)
		Dioxane (1,4-)
		Ethyl acetate

Test	Test Method(s)	Parameter/Analyte
Volatiles (cont)	Test Method(s)  SW 846 8260D	Ethyl methacrylate Ethylbenzene Ethyl-tert-butyl Ether (ETBE) Heptane (n-) Hexachlorobutadiene (1,3-) Hexachloroethane Hexane (n-) Hexanone (2-) Isa-butyl alcohol Isopropanol Isopropylbenzene Isopropyltoluene (4-) Methylcrolonitrile Methyl acetate Methyl aichae Methyl iodide Methyl methacrylate Methyl tert-butyl ether Methylcyclohexane Methylene chloride (Dichloromethane) Naphthalene Nitrobenzene Nitropropane (2-) Pentachloroethane Pentanone (4-methyl-2-) (MIBK) Propionitrile Propylbenzene Styrene tert-Amylmethyl ether (TAME) Tert-butyl alcohol Tert-butylbenzene
		Tetrachloroethane (1,1,1,2-) Tetrachloroethane (1,1,2,2-) Tetrachloroethene Tetrahydrofuran Toluene Trichloro (1,1,2-) trinuoroethane (1,2,2-)
		Trichlorobenzene (1,2,3-) Trichlorobenzene (1,2,4-) Trichloroethane (1,1,1-) Trichloroethane (1,1,2-) Trichloroethene Trichlorofluoromethane Trichloropropane (1,2,3-) Trimethylbenzene (1,2,4-)

Test	Test Method(s)	Parameter/Analyte
Volatiles (cont)	SW 846 8260D	Trimethylbenzene (1,3,5-)
		Vinyl acetate
		Vinyl chloride
		Xylene (m-)
		Xylene (o-)
		Xylene (p-)
		Xylenes (total)
Wet Extraction Test (WET) – Soluble	Colifornia Codo of Doculations Title	
Threshold Limit Concentration/Total	California Code of Regulations, Title	
Threshold Control Limit (STLC-TTLC)	22, Chapter 11, Article 5 Appendix II	

# SUPPORT FOR THE EPA NATIONAL MONITORING PROGRAMS

# NATIONAL HAZARDOUS AIR POLLUTANT SUPPORT

Contract No. 68HERH22D0002

2023-2024

**Quality Assurance Project Plan** 

Category 1

Eastern Research Group, Inc. 601 Keystone Park Drive, Suite 700 Morrisville, NC 27560

Project No. 0475.00
Element No. A2
Revision No. 2
Date August 2023
Page ii of xiv

2023-2024 Quality Assurance Project Plan, Category 1
National Hazardous Air Pollutant Support (Contract No. 68HERH22D0002)

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#### **DISCLAIMER**

This Category 1 Quality Assurance Project Plan has been prepared specifically to address the operation and management of the U.S. EPA National Hazardous Air Pollutant Support Programs (UATMP, NATTS, CSATAM, PAMS and NMOC). The contents have been prepared in accordance with Level I Specifications of the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5 and the EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5.

0475.00
A2
2
August 2023
iii of xiv

PROJEC	CT MANAGEMENT	1
SECTIO	N 1 PROJECT/TASK ORGANIZATION	1
1.1	Assignment of Program Personnel	1
1.	1.1 Program Manager	2
1.	1.3 Program Technical Adviser	3
1.	1.4 Program QA Coordinator	3
1.	1.5 Deputy Program QA Coordinator	3
1.	1.6 Task Leaders	4
SECTIO	N 2 PROBLEM DEFINITION/BACKGROUND	9
SECTIO	ON 3 PROJECT/TASK DESCRIPTION	12
3.1	PAMS, NMOC and SNMOC	
3.2	UATMP, NATTS and CSATAM	13
SECTIO	N 4 DATA QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA	16
SECTIO	N 5 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION	22
5.1	Field Activities Training Personnel	22
5.2	Analytical Laboratory Personnel	23
SECTIO	N 6 DOCUMENTATION AND RECORDS	24
6.1	Data Management	24
6.2	Preliminary Monthly Data Reports	24
6.3	Quarterly QA Report	24
6.4	Annual Summary Reports Submitted to EPA	25
6.5	Records and Supporting Data	26
	5.1 Notebooks	
6.	5.2 Electronic Data Collection	28
6.6	Data Reporting Package Archiving and Retrieval	28
6.7	Quality System Document Control	28
MEASU	JREMENT DATA ACQUISITION	29
SECTIO	N 7 SAMPLING PROCESS DESIGN	29
7.1	NMOC and SNMOC Canister Samplers	
7.2	VOC and Carbonyl 24-Hour Samplers	
7.3	Carbonyl Only 24-Hour Samplers	
7.4	Hexavalent Chromium Samplers	
7.5	PAMS Sampling	
7.6	HAPs Sampling	

Project No.	0475.00
Element No.	A2
Revision No.	2
Date	August 2023
Page	iv of xiv

SECTIO	N 8 SAMPLING METHOD REQUIREMENTS	40
SECTIO	N 9 SAMPLE HANDLING AND CUSTODY REQUIREMENTS	41
9.1	Canister Sample Custody	41
9.1	,	
9.1	2 Canister Analytical Routing Schedule	47
9.1	3 Canister Cleanup	47
9.2	Carbonyl Sample Custody	49
9.2	.1 Carbonyl Analytical Routing Schedule	49
9.3	HAPs Sample Custody	52
9.4	Invalid Samples	52
9.5	Analytical Data	57
9.6	Sampling Monitoring Data	57
SECTIO	N 10 ANALYTICAL METHODS REQUIREMENTS	58
10.1	Canister Cleanup System	58
10.2	VOC and Concurrent Analytical System	61
10.3	Carbonyl Analytical System	64
10.4	Polycyclic Aromatic Hydrocarbons Analytical Systems	64
10.5	Metals and Lead analysis Using an Inductively Coupled Argon Plasma Mass Sp	ectrometry
Analy	tical System	66
10.6	Hexavalent Chromium Analytical System	67
SECTIO	N 11 QUALITY CONTROL REQUIREMENTS	68
11.1	Sample Canister Integrity Studies	68
11.2	Standard Traceability	68
11.3	Accuracy and Acceptance	69
11.4	Precision	98
11.5	Completeness	98
11.6	Representativeness	99
11.7	Sensitivity (Method Detection Limits)	99
SECTIO	N 12 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE	
<b>REQUIR</b>	EMENTS	106
12.1	SNMOC, VOC, and PAMS	107
12.2	Carbonyls	107
12.3	HAPs	108
SECTIO	N 13 INSTRUMENT CALIBRATION AND FREQUENCY	109
13.1	SNMOC Calibration	109
13.2	VOC Calibration	110
13.3	Carbonyl Calibration	112
13.4	HAPs Calibration	
13.5	Laboratory Support Equipment Calibration	113

Project No.	0475.00
Element No.	A2
Revision No.	2
Date	August 2023
Page	v of xiv

SECTION	N 14 INSPECTION/ACCEPTANCE FOR SUPPLIES AND CONSUMABLES	115
14.1	Purpose	115
14.2	Critical Supplies and Consumables	115
14.3	Acceptance Criteria	115
SECTION	N 15 Non-direct Measurements	119
SECTION	N 16 DATA MANAGEMENT	120
16.1	Data Recording	120
16.2	Data Validation	122
16.3	Data Reduction and Transformation	122
16.4	Data Transmittal	123
16.5	Data Summary	124
16.6	Data Tracking	125
16.7	Data Storage and Retrieval	
ASSESSI	MENT/OVERSIGHT	127
SECTION	N 17 ASSESSMENTS AND RESPONSE ACTIONS	127
17.1	Assessment Activities and Project Planning	127
17.	.1.1 External Technical Systems and Data Quality Audits	
	.1.2 Internal Technical Systems Audits	
	1.3 Proficiency Testing	
	1.4 Data Assessment for Final Report	
17.2	·	
17.	.2.1 TSA, Data Quality Audit, and PT Documentation	
	.2.2 Internal Data Review Documentation	
17.3	Corrective Action	
SECTION	N 18 REPORTS TO MANAGEMENT	136
18.1	Frequency, Content, and Distribution of Reports	
	1.1 Monthly and Annual Reports	
	.1.2 Internal Technical System Audit Reports	
DATA V	ALIDATION AND USABILITY	138
SECTION	N 19 DATA REVIEW AND VERIFICATION	138
19.1	Data Review Design	
19.2	Data Review and Verification	
19.3	Data Reporting	
19.4	Data Validation	
	4.1 PAMS Data Validation	
19.5	Air Quality System	
	.5.1 AQS Flagging and Reporting	

	Element No.	
	Revision No.	2 August 2023
	Page	vi of xiv
SECTION 20 DATA VALIDATION, VERIFICATION METHOD	DS	150
20.1 Process for Validating and Verifying Data		
20.1.1 Verification of Data		
20.1.2 Validation of Data		
20.2 Data Analysis		151
SECTION 21 RECONCILIATION WITH DATA QUALITY OBJ	IECTIVES	152
21.1 Conduct Preliminary Data Review		
21.2 Draw Conclusions from the Data		152
SECTION 22 REFERENCES	•••••	153
APPENDIX A ERG EXEMPTION FROM THE NATTS TAD, R	EVISION 4	156
,		
APPENDIX B 2023/2024 SAMPLING SCHEDULES	•••••	158
APPENDIX C ERG CHANGES FOR 2023/2024 QAPP		163
ADDENDIVE DELEVANTEDO CTANDADO COSTATIVO DO	OCEDI IDEC	46=
APPENDIX D RELEVANT ERG STANDARD OPERATING PR	KOCEDUKES	165
APPENDIX E SUBCONTRACTORS QUALITY ASSURANCE P	PROJECT PLAN	168

0475.00

Project No.

Project No. 0475.00
Element No. A2
Revision No. 2
Date August 2023
Page vii of xiv

# LIST OF TABLES

Table		Page
Table 1-1	Program Organization	
Table 1-2	QC Responsibilities and Review Functions	7
Table 3-1	List of Analytical and Support Services	14
Table 4-1	Measurement Quality Objectives for the National Hazardous Air Pollutants Supp	ort
	Program	20
Table 6-1	Data Documentation and Records	26
Table 8-1	EPA Methods and ERG SOPs for each Sampling System	40
Table 9-1	Example of Canister Pressure Check Spreadsheet	45
Table 10-1	VOC GC/FID/MS Operating Conditions	62
Table 11-1	Summary of SNMOC Quality Control Procedures	70
Table 11-2	Summary of Air Toxics Canister VOC Quality Control Procedures	71
Table 11-3	BFB Key Ion Abundance Criteria	76
Table 11-4	Summary of Carbonyl Quality Control Procedures	
Table 11-5	Summary of Quality Control Procedures for Analysis of SVOC Samples for PAHs .	82
Table 11-6	DFTPP Key Ions and Ion Abundance Criteria	86
Table 11-7	Internal Standards and Associated PAHs	86
Table 11-9	Instrument Mass Calibration & Performance Specifications	90
Table 11-10	Summary of Quality Control Procedures for Hexavalent Chromium	92
Table 11-11	Summary of Quality Control Procedures for Lead Analysis by ICP-MS	93
Table 11-12	Summary of Quality Control Procedures for Lead Analysis by XRF	96
Table 11-13	2023 SNMOC Method Detection Limits	100
Table 11-14	2023 Air Toxics Method Detection Limits	101
Table 11-15	2023 Carbonyl Method Detection Limits (Underivatized Concentration)	102
Table 11-16	2023 PAH Method Detection Limits	103
Table 11-17	2023 Metals Method Detection Limit	104
Table 11-18	Target MDLs for the NATTS Program	105
Table 12-1	Preventive Maintenance in ERG Laboratories	106
Table 13-1	Minimum Relative Response Factor Criteria for Initial Calibration of Common	
	Semivolatile Compounds	
Table 14-1	Critical Supplies and Consumables	116
Table 16-1	Final Report Equations	124
Table 16-2	Data Archive Policies	125
Table 19-1	Qualifier Codes	145
Table 19-2	Null Codes	
Table 19-3	Summary of Quantitation and Detection Limit Flags and Applications	149

Project No. 0475.00
Element No. A2
Revision No. 2
Date August 2023
Page viii of xiv

# **LIST OF FIGURES**

Figure		Page
Figure 1-1	National Monitoring Programs Organizational Chart	6
Figure 3-1	Duplicate/Collocate and Replicate Analysis Schematic	15
Figure 7-1	NMOC, SNMOC, and 3-Hour Air Toxics Sampling System Components	30
Figure 7-2	VOC/Carbonyl Sampler Training Form	31
Figure 7-3	24-Hour Integrated Air Toxics Sampling System Components	35
Figure 7-4	Carbonyl Sampling System Components	
Figure 7-5	Hexavalent Chromium Sampling System Components	38
Figure 9-1	Example NMOC COC	42
Figure 9-2	Example Air Toxics COC	43
Figure 9-3	Example Passive Sample Air Toxics COC	44
Figure 9-4	Example ERG LIMS Login Page	46
Figure 9-5	Canister Tag	46
Figure 9-6	Canister Cleanup Log for the ERG Heated Cleaning System	48
Figure 9-7	Example Carbonyl Compounds COC	50
Figure 9-8	Example PAMS Carbonyl Compounds COC	51
Figure 9-9	Example SVOC Sample COC	53
Figure 9-10	Example Ambient Hexavalent Chromium COC	54
Figure 9-11	Example Metals COC	55
Figure 9-12	Metals Blank COC Record	56
Figure 10-1	Heated Canister Cleanup System Schematic	59
Figure 10-2	Entech Heated Canister Cleanup System Schematic	60
Figure 10-3	VOC GC/MS Systems	63
Figure 10-4	VOC GC/MS/FID System	63
Figure 10-5	HPLC System	65
Figure 13-1	Dynamic Flow Dilution Apparatus	111
Figure 16-1	Data Management and Sample Flow Diagram	121
Figure 17-1	ERG Corrective Action Report Form, Page 1	134
Figure 17-1	ERG Corrective Action Report Form, Continued	135

Project No. 0475.00
Element No. A2
Revision No. 2
Date August 2023
Page ix of xiv

#### SYMBOLS AND ABBREVIATIONS

AAC Atmospheric Analysis and Consulting

AMTIC Ambient Air Monitoring Technical Information Center

AQS Air Quality Subsystem

ASTM American Society for Testing and Materials

Auto-GC Automatic Gas Chromatograph

BFB 4-Bromofluorobenzene

BS/BSD Blank Spike/Blank Spike Duplicate

CAA Clean Air Act

CAL2 Second Calibration Standard

CAR Corrective Action Report

CCB Continuing calibration blank

CCV Continuing calibration verification

CFR Code of Federal Regulations

COC Chain of Custody

cps Counts per second

CSATAM Community Scale Air Toxics Ambient Monitoring

CV Coefficient of Variation

DFTPP Decafluorotriphenylphosphine

DL Detection Limits

DNPH 2,4-Dinitrophenylhydrazine

DPR Daily Performance Check

DQOs Data Quality Objectives

EPA U.S. Environmental Protection Agency

ERG Eastern Research Group, Inc.

ESMB Extraction Solvent Method Blank

FAC Federal Advisory Committee

FB Field Blank

FC-43 perfluorotributylamine

FEM Federal Equivalency Method

FID Flame Ionization Detector

Project No. 0475.00

Element No. A2

Revision No. 2

Date August 2023

Page x of xiv

GC Gas Chromatograph

GPRA Government Performance and Results Act

HAPs Hazardous Air Pollutant(s)

He Helium

H2 Hydrogen

Hg Mercury

HPLC High Performance Liquid Chromatography

HSV High standard verification

IC Ion Chromatography

IC Initial Calibration Standards (for ICP-MS)

ICAL Initial Calibration

ICB Initial Calibration Blank

ICP-MS Inductively Coupled Plasma/Mass Spectrometer

ICSA/IFA Interference Check Standard A

ICSAB/IFB Interference Check Standard B

ICV Initial calibration verification

ID Identification

IR Infrared

IS (or ISTD) Internal Standard

KED Kinetic Energy Discrimination

LCS Laboratory Control Standard

LCV Low Calibration Verification

LIMS Laboratory Information Management System

LOQ Limit of Quantitation

LRB Laboratory Reagent Blank

m Meter(s)

MB (or BLK) Method Blank

MDLs Method Detection Limit(s)

MDLsp MDL absent blank background

mL Milliliter

mm Millimeter

Project No. 0475.00

Element No. A2

Revision No. 2

Date August 2023

Page xi of xiv

mM Millimolar

MQOs Measurement Quality Objective

MS Mass Spectrometer

MS/MSD Matrix Spike/Matrix Spike Duplicate

μg/L Micrograms per liter

μg/mL Micrograms per milliliter

μg/m3 Microgram per cubic meter

μL Microliters

μm Micrometer

N2 Nitrogen

NAAQS National Ambient Air Quality Standard

NATTS National Ambient Toxics Trends Stations

ND Not Detected

NIST National Institute of Standards and Technology

NIOSH National Institute for Occupational Safety and Health

NHAPS National Hazardous Air Pollutant Support

ng Nanogram

ng/m3 Nanogram per cubic meter

nm Nanometer

NMOC Nonmethane Organic Compounds

NOx Oxides of Nitrogen

O3 Ozone

OAQPS Office of Air Quality Planning and Standards

OD Outer Diameter

OSHA Occupational Safety and Health Administration

PAHs Polycyclic Aromatic Hydrocarbons

PAMS Photochemical Assessment Monitoring Stations

PCBs Polychlorinated biphenyls

PDF Portable Document Format

PDFID Preconcentration Direct Flame Ionization Detection

PDS Post digestion spike

Project No. 0475.00
Element No. A2
Revision No. 2
Date August 2023
Page xii of xiv

PE Performance Evaluation

POC Parameter Occurrence Code

ppbC Parts per Billion as Carbon

ppbV Parts per Billion by volume

ppmC Parts per Million as Carbon

ppmV Parts per Million by volume

psig Pounds per square inch gauge

PT Proficiency Testing

PUF Polyurethane Foam

QA Quality Assurance

QAPPs Quality Assurance Project Plan(s)

QC Quality Control

QL Quantitation Limit

RD Raw Data

RE Relative Error

RF Response Factor

RfCs Reference Concentration

RPD Relative Percent Difference

RRF Relative Response Factor

RRTs Relative Retention Times

RSD Relative Standard Deviation

RTS Retention Time Standards

RT Retention Time

RTP Research Triangle Park

SCV Secondary Source Calibration Verification

SIM Selected Ion Monitoring

SIP State Implementation Plan

SMB Solvent Method Blank

SNMOC Speciated Nonmethane Organic Compounds

SOPs Standard Operating Procedure(s)

SQL Sample Quantitation Limit

Project No. 0475.00
Element No. A2
Revision No. 2
Date August 2023
Page xiii of xiv

SRD Serial dilution

SRM Standard Reference Material

SSQC Second Source Quality Control

STI Sonoma Technology, Inc.

SVOC Semivolatile Organic Compounds

TAD Technical Assistance Document

TNI The National Environmental Laboratory Accreditation Conference Institute

TSAs Technical System Audits

TSP Total Suspended Particulate

UAM Urban Airshed Model

UATMP Urban Air Toxics Monitoring Program

UPS United Parcel Service of America

UREs Unit Risk Estimate

UV Ultraviolet

VOCs Volatile Organic Compound

XRF X-ray fluorescence

Project No. 0475.00

Element No. A2

Revision No. 2

Date August 2023

Page xiv of xiv

#### **DISTRIBUTION LIST**

Copies of this plan and all revisions will be provided to:

- Jeff Yane, Work Assignment Manager, U.S. EPA, RTP, NC
- Xi (Doris) Chen, Delivery Order Manager, U.S. EPA, RTP, NC
- Greg Noah, AT QA Technical Lead, U.S. EPA, RTP, NC
- Trisha Curran, AT TO QA Lead, U.S. EPA, RTP, NC

U.S. EPA Regional contacts may obtain a copy of the QAPP by contacting the ERG Program Manager. It is the responsibility of each Regional contact to make copies of the plan for appropriate State personnel or to refer them to ERG Program Manager. The ERG staff working on this contract will receive a copy of this QAPP and all revisions.

Project No. 0475.00
Element No. Section 1 - A4
Revision No. 2
Date August 2023
Page 1 of 155

#### PROJECT MANAGEMENT

### SECTION 1 PROJECT/TASK ORGANIZATION

# 1.1 Assignment of Program Personnel

Table 1-1 presents the program organization listing the program assignment and responsible person for each aspect of the Environmental Protection Agency (EPA) National Hazardous Air Pollutants Support (NHAPS) Programs. The program organizational chart is presented in Figure 1-1. All Eastern Research Group, Inc. (ERG) staff working on this contract are provided access to a current electronic copy of this signed, EPA approved Quality Assurance Project Plan (QAPP).

ERG's primary support on this contract includes Nonmethane Organic Compounds (NMOC),
Speciated Nonmethane Organic Compounds (SNMOC), Volatile Organic Compounds (VOCs),
Polycyclic Aromatic Hydrocarbons (PAHs), Metals, Hexavalent Chromium, and other Hazardous Air
Pollutants (HAPs). Subcontracting services are extended by:

- Orsat, LLC and Consolidated Analytical Systems (CAS) for onsite technical assistance for Photochemical Assessment Monitoring Stations (PAMS) analysis,
- Sonoma Technology, Inc. (STI) for data validation,
- Enthalpy Analytical for VOCs by Method TO-17, pesticides/Polychlorinated biphenyls (PCBs), anions, diisocyanates, 4,4'-methylenedianiline and reduced sulfurs, and
- RTI International for lead by X-ray fluorescence (XRF) and other metals analysis, in the event of a large workload.

ERG is responsible to the client for the work of the subcontractor and choosing subcontractors that meet the applicable requirements for the methods and contracts. The subcontractor should meet the Data Quality Objectives (DQOs) requirements for the appropriate method. ERG shall maintain a record of subcontractor compliance, including documentation of subcontractor's Method Detection Limits (MDLs), QAPPs, etc. Sample analysis will not begin with the subcontractor until MDLs, QAPPs, Proficiency Testing (PT) samples and/or Technical System Audits (TSAs) if they are available through Office of Air Quality Planning and Standards (OAQPS). If such measures are not available, ERG will

Project No. 0475.00
Element No. Section 1 - A4
Revision No. 2
Date August 2023
Page 2 of 155

request audit reports performed by the subcontract lab and will supply PT audits if requested by the EPA when analysis is contracted with the laboratory. etc., have been approved by EPA and ERG.

## 1.1.1 Program Manager

Ms. Julie Swift, an ERG Vice President, serves as the Program Manager for EPA's NHAPS. In this role, she has the primary responsibility for understanding program level needs, both EPA's and their clients' (i.e., State, Local, and Tribal agencies). Ms. Swift is ultimately accountable for providing timely, cost effective, and high-quality services that meet the needs of the NHAPS efforts. Her responsibility is ensuring EPA/client satisfaction by verifying that all components necessary for effective management are in place and active during the contract performance period. Ms. Swift coordinates with the ERG Quality Assurance (QA) Coordinator, and task leaders to provide EPA/client perspective, communicate technical issues and needs, and ensure the program staff facilitates decisions appropriate to their roles on Contract 68HERH22D0002. As Program Manager, Ms. Swift, is responsible for maintaining and distributing the approved QAPP. She prepares budgetary and schedule information and prepares all information for presentation to EPA at scheduled program meetings. As the Program Manager, Ms. Swift is responsible for the technical operation and the quality of the program on a day-to-day basis. She leads the analytical tasks and provides technical direction and support. She assists in the resolution of technical issues and serves as a resource for Task Leaders regarding any project issues. Ms. Swift also performs an overall review of the data that is reported monthly.

## 1.1.2 Deputy Program Manager

As the Deputy Program Manager, Mr. Randy Bower assists the Program Manager for EPA's NHAPS. He assists the Program Manager in all aspects of the technical operation and the quality of the program on a day-to-day basis. He assists the analytical Task Leaders and provides technical direction and support. He assists in the resolution of technical issues and serves as a resource for Task Leaders regarding project issues. Mr. Bower is also the Network Site Coordinator Task Leader.

Project No. 0475.00
Element No. Section 1 - A4
Revision No. 2
Date August 2023
Page 3 of 155

# 1.1.3 Program Technical Adviser

The Program Technical Adviser, Mr. Mitchell Howell assists in the resolution of technical issues. He communicates with ERG management and the technical staff for discussion of real and potential technical problems. He peer-reviews draft and final technical program report products and provides oversight of efforts to evaluate and characterize data.

### 1.1.4 Program QA Coordinator

Ms. Donna Tedder, the Program and Laboratory QA Coordinator, is responsible for ensuring the overall integrity and quality of project results. Ms. Tedder, or her designee, will do a 10 percent QA review for all sample analyses delivered for reporting by the Program Manager. In the case of subcontracted work, 20 percent of data from subcontractor will be reviewed. The lines of communication between management, the Program QA Coordinator, and the technical staff are formally established and allow for discussion of real and potential problems, preventive actions, and corrective procedures. The key Quality Control (QC) responsibilities and QC review functions are summarized in Table 1-2. On major quality issues, Ms. Tedder reports independently to Ms. Jan Connery, ERG's corporate QA Officer.

# 1.1.5 Deputy Program QA Coordinator

The Deputy Program QA Coordinator, Ms. Jennifer Nash, is responsible for ensuring the integrity and quality of project results. The Deputy QA Coordinator will assist the Program QA Coordinator with the QA review for sample analyses delivered for reporting by the Program Manager. The major QC responsibilities and QC review functions are summarized in Table 1-2. The Deputy QA Coordinator will work closely with the Program QA Coordinator to ensure the overall quality of the Program. Ms. Nash is also the HAPs Support Task Leader and, as such, will not do any of the QA review duties for these tasks.

Project No. 0475.00
Element No. Section 1 - A4
Revision No. 2
Date August 2023
Page 4 of 155

#### 1.1.6 Task Leaders

ERG Task Leaders are responsible for meeting the project objectives, meeting report schedules, and directing the technical staff in execution of the technical effort for their respective task(s). The analytical Task Leaders will review 100 percent of all sample analyses. The Program QA Coordinator will request 10 percent of that data for review prior to data reporting by the Program Manager. The Task Leaders manage the day-to-day technical activities on delivery orders for this program. They assess and report on the project's progress and results (e.g., recordkeeping, data validation procedures, sample turnaround time) and ensure timely, high-quality services that meet the requirements in this QAPP.

Project No. 0475.00
Element No. Section 1 - A4
Revision No. 2
Date August 2023
Page 5 of 155

**Table 1-1 Program Organization** 

Program Assignment	Program Personnel Assigned	Phone Number	Email Address
Program Manager	Julie Swift	(919) 468-7924	julie.swift@erg.com
Deputy Program Manager	Randy Bower	(919) 468-7928	randy.bower@erg.com
Task Leader - Network Site Coordination	Randy Bower	(919) 468-7928	randy.bower@erg.com
Task Leader - Shipping and Receiving	Barnabas Van Devender	(919) 468-7929	barnabas.vandevender@erg.com
Task Leader - Air Toxics	Kyle Rasmus	(919) 468-7923	kyle.rasmus@erg.com
Task Leader - Carbonyl Analysis	Cleveland Corbett	(919) 468-7917	cleveland.corbett@erg.com
Task Leader – Metals & Hexavalent Chromium*	Brodie Clark	(919) 468-7920	brodie.clark@erg.com
Task Leader - NMOC/SNMOC Analysis	Mitchell Howell	(919) 468-7915	mitch.howell@erg.com
Task Leader - Semivolatiles	Chris Kopp	(919) 468-7945	chris.kopp@erg.com
Task Leader - PAMS Support **	Mitchell Howell	(919) 468-7915	mitch.howell@erg.com
Task Leader - HAPs Support ***	Jennifer Nash	(919) 468-7881	jennifer.nash@erg.com
Task Leader - Data Characterization	Regi Oommen	(919) 468-7829	regi.oommen@erg.com
Task Leader – Annual/PAMS Data & AQS Entry	Jaime Hauser	(919) 468-7813	jaime.hauser@erg.com
Program Technical Adviser	Mitchell Howell	(919) 468-7915	mitch.howell@erg.com
Program QA Coordinator	Donna Tedder	(919) 468-7921	donna.tedder@erg.com
Deputy QA Coordinator	Jennifer Nash	(919) 468-7881	jennifer.nash@erg.com
Project Administrator	Kerry Fountain	(919) 468-7962	kerry.fountain@erg.com

<sup>\*</sup>Subcontracting support when requested from RTI (for NAAQS Pb).

<sup>\*\*</sup>Subcontracting support when requested from CAS, Orsat and Sonoma Technologies, Inc. (STI).

<sup>\*\*\*</sup>Subcontracting support when requested from Enthalpy and RTI (miscellaneous HAPs).

Project No. 0475.00
Element No. Section 1 - A4
Revision No. 2
Date August 2023
Page 6 of 155

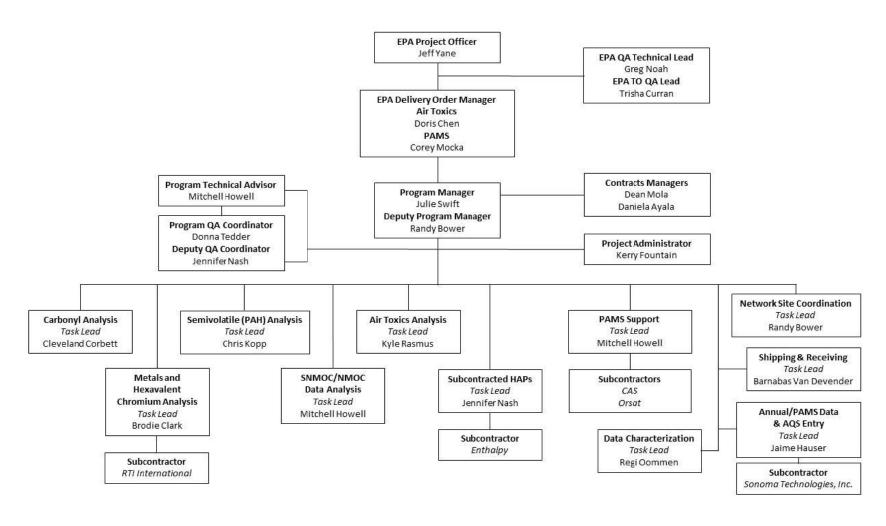


Figure 1-1 National Monitoring Programs Organizational Chart

Project No. 0475.00
Element No. Section 1 - A4
Revision No. 2
Date August 2023
Page 7 of 155

Table 1-2 QC Responsibilities and Review Functions

Responsible Person	Major Responsibilities
Ms. Julie Swift, Program Manager	<ul> <li>Ensure overall timely performance of high-quality technical services</li> <li>Communicate technical issues and needs</li> <li>Assist in the resolution of technical problems</li> <li>Track all management systems and tools</li> <li>Track deliverables and budget performance</li> <li>Ensure appropriate level of staffing and committed resources exist to perform work</li> <li>Communicate daily with the EPA/State/Local/Tribal agencies</li> <li>Ensure data quality</li> <li>Check information completeness</li> <li>Review data completeness and quality before reporting to client</li> <li>Review all reports</li> <li>Report project performance (budget and deliverables) to EPA at scheduled meetings and in monthly progress reports</li> <li>Day-to-day management of task leaders</li> </ul>
Mr. Randy Bower, Deputy Program Manager	<ul> <li>Assist Program Manager where needed</li> <li>Ensure overall timely performance of high-quality technical services</li> <li>Communicate technical issues and needs</li> <li>Assist in the resolution of technical problems</li> <li>Ensure appropriate level of staffing and committed resources exist to perform work</li> <li>Communicate with the EPA/State/Local/Tribal agencies</li> <li>Ensure data quality</li> <li>Check information completeness</li> <li>Review data completeness and quality before reporting to client</li> <li>Day-to-day management of task leaders</li> </ul>
Mr. Mitchell Howell, Program Technical Adviser  Ms. Donna Tedder, Program QA Coordinator	<ul> <li>Assist in the resolution of technical problems</li> <li>Communicate potential technical issues and needs</li> <li>Review draft technical program reports</li> <li>Make QA recommendations</li> <li>Review QAPP</li> <li>Audit laboratory</li> <li>Review QA reports</li> <li>Evaluate the effect of technical issues on data quality</li> <li>Review 10% of all data for reporting</li> <li>Review QA documentation [Standard Operating Procedures (SOPs), reports, etc.]</li> </ul>

Project No. 0475.00
Element No. Section 1 - A4
Revision No. 2
Date August 2023
Page 8 of 155

Table 1-2 QC Responsibilities and Review Functions

Responsible Person	Major Responsibilities
Ms. Jennifer Nash,	Assist QA Coordinator where needed
Deputy Program QA	Make QA recommendations
Coordinator	Review QAPP
	<ul> <li>Assist with laboratory audit(s)</li> </ul>
	<ul> <li>Evaluate the effect of technical issues on data quality</li> </ul>
	<ul> <li>Review 10% of all data for monthly reporting</li> </ul>
	<ul> <li>Review QA documentation (SOPs, reports, etc.)</li> </ul>
Task Leader(s)	Review documentation
	<ul> <li>Review 100% of analytical data generated by analysts</li> </ul>
	Develop analytical procedures
	Propose procedural changes
	Train and supervise analysts
	Meet task report schedules
	<ul> <li>Manage day-to-day technical activities</li> </ul>
	Check information completeness
	<ul> <li>Review instrument and maintenance logbooks</li> </ul>
	Review calibration factor drift
	Perform preventive maintenance

Project No. 0475.00
Element No. Section 2 - A5
Revision No. 2
Date August 2023
Page 9 of 155

# SECTION 2 PROBLEM DEFINITION/BACKGROUND

The Clean Air Act (CAA) Amendments of 1990 required EPA OAQPS to set National Ambient Air Quality Standard (NAAQS) for the "criteria" pollutant ozone (O<sub>3</sub>). In areas of the country where the NAAQS for O<sub>3</sub> was being exceeded, additional measurements of the ambient NMOC were needed to assist the affected States in developing/revising O<sub>3</sub> control strategies. Measurements of ambient NMOC are important to the control of VOCs that are precursors to atmospheric O<sub>3</sub>. Due to previous difficulty in obtaining accurate NMOC concentration measurements, EPA started a monitoring and analytical program in 1984 to provide support to the States. ERG has continuously supported EPA for the NMOC programs since 1984.

In 1987, EPA developed the Urban Air Toxics Monitoring Program (UATMP) to help State, Local and Tribal air monitoring agencies characterize the nature and extent of potentially toxic air pollution in urban areas. Since 1987, several State and local agencies have participated in the UATMP by implementing ambient air monitoring programs. These efforts have helped to identify the toxic compounds most prevalent in the ambient air and indicate emissions sources that are likely to be contributing to elevated concentrations. Studies indicate that a potential for elevated cancer risk is associated with certain toxic compounds often found in ambient urban air<sup>(1)</sup>. As a screening program, the UATMP also provides data input for models used by EPA, State, local and risk assessment personnel to assess risks posed by the presence of toxic compounds in urban areas. The UATMP program is a year-round sampling program, collecting 24-hour integrated ambient air samples at urban sites in the contiguous United States every 6 or 12 days.

The SNMOC program was initiated in 1991 in response to requests by State agencies for more detailed speciated hydrocarbon data for use in  $O_3$  control strategies and Urban Airshed Model (UAM) input.

Title I, Section 182 of the CAA Amendments of 1990 requires States to establish PAMS as part of their State Implementation Plan (SIP) for O<sub>3</sub> nonattainment areas. The rule revises the ambient air quality surveillance regulations to include enhanced monitoring of O<sub>3</sub> and its precursors. The

Project No. 0475.00
Element No. Section 2 - A5
Revision No. 2
Date August 2023
Page 10 of 155

regulations promulgated in 1993 require monitoring of  $O_3$ , oxides of nitrogen ( $NO_x$ ), selected carbonyl compounds, and VOCs. The required monitoring is complex and requires considerable lead time for the agencies to acquire the equipment and expertise to implement their PAMS network. Under the PAMS program, each site may require a different level of support with respect to sampling frequency, sampling equipment, site support, sample analyses, data validation, and report preparation. Presampling, sampling, and analytical activities are performed according to the guidance provided in the Technical Assistance Document (TAD) $^{(2)}$ , for Sampling and Analysis of Ozone Precursors, Revision 2, April 2019. The program objective of PAMS is to provide data that are consistent with the proposed rule for ambient air quality surveillance regulations in accordance with Code of Federal Regulations (CFR) Title 40, Part 58 Appendix D Section 5.

In 1999, EPA expanded this program to provide measurements of additional CAA HAPs to support the Government Performance and Results Act (GPRA). As required under the GPRA, EPA developed a Strategic Plan that includes a goal for Clean Air. The objective of this goal: to improve air quality and reduce air toxics emissions to levels 75 percent below 1993 levels by 2010 to reduce the risk to Americans of cancer and other serious adverse health effects caused by airborne toxics.

In 2001, EPA designed a national network for monitoring air toxics compounds present in ambient air entitled the National Ambient Toxics Trends Station (NATTS). The primary purpose of the NATTS network is tracking trends in ambient air toxics levels to facilitate measuring progress toward emission and risk reduction goals. The monitoring network is intended for long term operation for the principle purpose of discerning national trends in air toxics ambient concentrations.

Beginning in 2003/2004, EPA conducted periodic Community Scale Air Toxics Ambient Monitoring (CSATAM) grant competitions. The resultant 1- to 2-year grants are designed to help State, Local, and Tribal communities identify and profile air toxics sources, characterize the degree and extent of local air toxics problems, and track progress of air toxics reduction

Project No. 0475.00
Element No. Section 2 - A5
Revision No. 2
Date August 2023
Page 11 of 155

activities. Grants have been awarded across the United States, in large, medium, and small communities. The ERG team can offer site support and analysis to any agency for the UATMP, NATTS and CSATAM programs as well as lead analysis for the NAAQS program.

The data obtained by following this QAPP will be used by EPA, State, Local, Tribal and risk assessment personnel to determine prevalent  $O_3$  precursors and air toxics in the urban air. The data collected from the continuous yearly sites gives the data analyst consistent high quality analytical results. Sampling and analytical uncertainties are determined through this program by performing 10 percent sampling duplicate (or collocated) and analytical replicate samples for each of the ambient air sites.

This QAPP defines the preparation, sampling, laboratory analyses and QA/QC procedures conducted by ERG for EPA's NHAPS program to deliver data of sufficient quality to meet the programs' objectives. Many of these procedures described in this QAPP are based on experiences obtained during previous National Program Studies.

Project No. 0475.00
Element No. Section 3 - A6
Revision 2
Date August 2023
Page 12 of 155

#### SECTION 3 PROJECT/TASK DESCRIPTION

This section describes the activities performed under each of the major EPA NHAPS components (NMOC, SNMOC, UATMP, CSATAM, NATTS, PAMS and lead). ERG dedicates passivated canisters, sampling equipment and expendable sampling media to the program to maintain known quality that meets the program objectives. An applicable measurement methods list is presented in Table 3-1. Sampling and analysis are determined when delivery orders are provided by EPA.

### 3.1 PAMS, NMOC and SNMOC

The program objective of PAMS is to provide data that are consistent with the proposed rule for Ambient Air Quality Surveillance in accordance with 40 CFR Part 58 Appendix D Section 5. The ERG team can offer site support to any State that needs to maintain a PAMS site, including carbonyl analysis, auto-GC technical site support, or canister analysis. Canister and/or carbonyl samples are collected on the first sampling day in June through the end of August at each of the designated sites. ERG offers carbonyl and/or canister sample analysis support with data validation and data reporting. ERG also performs certifications of PAMS carbonyl samplers, certification of the PAMS retention time standard (RTS) and calibration standard compressed gas cylinders (or recertification of previously provided standards).

The PAMS program requires collection of three 8-hour ambient air samples over a 24-hour period. This sample collection period occurs every third day. Three sequential 8-hour samples are to be collected on each sampling day, according to the following time schedule, standard local time, unadjusted for daylight saving time:

04:00 to 12:00 p.m. (noon)

12:00 p.m. (noon) to 20:00

20:00 to 04:00

ERG can provide NMOC/SNMOC samplers, sampler training, and any technical assistance needed throughout the monitoring program, for non-PAMS sites needing to collect canister

Project No. 0475.00
Element No. Section 3 - A6
Revision 2
Date August 2023
Page 13 of 155

samples during PAMS season. At least one week before each sample collection episode, ERG ships the necessary clean, certified canisters and/or carbonyl cartridges to the site along with the field chain of custody (COC) forms. The time-integrated ambient samples are then collected and shipped to ERG for analysis.

#### 3.2 UATMP, NATTS and CSATAM

The UATMP program was initiated as an analytical/technical support program focused on ascertaining ambient air levels of organic toxic species. The program has since expanded to provide for the measurement of additional HAPs and the standard sample collection frequency was increased to 1 in 6 days, with some sites continuing at 1 in 12 days.

The NATTS Network is intended for long term operation for the principle purpose of discerning national trends in ambient air toxics levels to facilitate measuring progress toward emission and risk reduction goals. The monitoring network is intended to be able to detect a 15 percent difference (trend) between two successive 3-year annual mean concentrations within acceptable levels of decision error. The standard sample collection frequency is 1 in 6 days.

The program objective of the CSATAM Program is designed to help State, Local, and Tribal communities identify and profile air toxics sources, characterize the degree and extent of local air toxics problems, and track progress of air toxics reduction activities. Grants have been awarded across the entire United States, in large, medium, and small communities. Awarded grants fall into one of three categories: community-scale monitoring, method development/evaluation, and analysis of existing data. The sample collection frequency may be 1 in 6 days or 1 in 12 days. Targeted pollutants generally reflect the NATTS core compounds, criteria pollutants, and/or pollutants related to diesel particulate matter.

The ERG team can offer site support and sample analysis to any State that needs VOC, carbonyl, or other analyses for the PAMS, UATMP, NATTS and CSATAM programs, as shown in Table 3-1. Relevant SOPs are also referenced in the table.

Project No. 0475.00
Element No. Section 3 - A6
Revision 2
Date August 2023
Page 14 of 155

**Table 3-1 List of Analytical and Support Services** 

Analysis	Based on Method	SOP (ERG-MOR-XXX)
Analysis		
Total NMOC	TO-12 <sup>(3)</sup>	-060***
Speciated NMOC/PAMS Hydrocarbons via GC/FID	TAD for Ozone Precursors <sup>(2)</sup>	-005
VOCs via GC/MS	TO-15 <sup>(4)</sup>	-005*
Concurrent SNMOC and VOC via GC/MS/FID	TAD for Ozone Precursors <sup>(2)</sup> /TO-15 <sup>(4)</sup>	-005
Carbonyls via HPLC	TO-11A <sup>(5)</sup>	-024
PM10 HAP Metals via ICP-MS	IO-3.5 <sup>(6)</sup> /EQL-0512-201 <sup>(7)</sup> / EQL-0512-202 <sup>(8)</sup>	-095/-084/-085
TSP Hexavalent Chromium via IC	ASTM D7614 <sup>(9)</sup>	-063
SVOC analysis via GC/MS (SCAN)	TO-13A <sup>(10)</sup> / Method 8270E <sup>(11)</sup>	-044***
PAH analysis via GC/MS (SIM)	TO-13A <sup>(10)</sup> / ASTM D6209 <sup>(12)</sup>	-049
PCB/Pesticides via GC *	TO-4A <sup>(13)</sup>	*
Anions via IC *	NIOSH 7906 – Particulate Fluorides <sup>(14)</sup> NIOSH 7907 – Volatile Acids <sup>(15)</sup> NIOSH 7908 – Non-Volatile Acids <sup>(16)</sup> **	*
VOCs via GC/MS (from cartridge) *	TO-17 <sup>(17)</sup>	*
Diisocyanates *	OSHA Method 42 <sup>(18)</sup>	*
4,4'-Methylenedianiline *	NIOSH Method 5029 <sup>(19)</sup>	*
Landfill gas and natural gas compounds	Modified TAD for Ozone Precursors <sup>(2)</sup>	-005
Reduced sulfurs	ASTM D5504 <sup>(20)</sup>	*
ICP-MS Analysis for NAAQS Lead (Pb)	EQL-0512-201 <sup>(7)</sup> /EQL-0512-202 <sup>(8)</sup>	-084/-114
XRF Analysis of 47mm Teflon filters for Lead (Pb)	40 CFR 50, Appendix Q <sup>(21)</sup>	*
Site Support		
NMOC/SNMOC	TAD for Ozone Precursors <sup>(2)</sup>	-046***
VOC	TO-15 <sup>(4)</sup>	-003
Carbonyls	TO-11A <sup>(5)</sup>	-003 or -047
Hexavalent Chromium	ASTM D7614 <sup>(9)</sup>	-013
PAMS Technical	TAD for Ozone Precursors <sup>(2)</sup>	NA
PAMS Data Validation	TAD for Ozone Precursors <sup>(2)</sup>	113
Other Services		•
Performance Samples for VOC	TO-15 <sup>(4)</sup>	-005 and -061
Performance Samples for Carbonyls	TO-11A <sup>(5)</sup>	-024
Performance Samples for PAH	TO-13A <sup>(10)</sup> / ASTM D6209 <sup>(12)</sup>	-049
Performance Samples for PM10 HAP Metals	IO-3.5 <sup>(6)</sup> /EQL-0512-201 <sup>(7)</sup> / EQL-0512-202 <sup>(8)</sup>	-095/-084/-085
Performance Samples for TSP Hexavalent Chromium	ASTM D7614 <sup>(9)</sup>	-063
Sampler Certification for Carbonyls	TO-11A <sup>(5)</sup>	-100
Sampler Certification for VOC	TO-15 <sup>(4)</sup>	-030
Uniform Calibration Standards	TO-15 <sup>(4)</sup>	NA
Uniform NIST traceable calibration Standards for PAMS AutoGCs	TAD for Ozone Precursors <sup>(2)</sup>	NA
Retention Time Cylinders for PAMS AutoGCs	TAD for Ozone Precursors <sup>(2)</sup>	NA
Humidified canisters with Retention Time gases	TAD for Ozone Precursors <sup>(2)</sup>	NA
AQS Data Entry (per pollutant group)	NA	-098
Report Development/Data Characterization	NA NA	NA

<sup>\*</sup>Will be supplied by subcontractor when analysis is requested.

Project No.	0475.00
Element No.	Section 3 - A6
Revision	2
Date	August 2023
Page	15 of 155

ERG can provide sampler, sampler training, and any technical assistance needed throughout the monitoring program. Canister, carbonyl, and other HAPs samples are collected by State/Local/or Tribal agency personnel every 6 or 12-days at each of the designated sites. ERG ships the necessary clean, certified canisters and/or sampling media to the site along with the field COC forms at least one week before each sample collection episode. The time-integrated ambient samples are then collected and shipped to ERG for analysis.

To determine the overall precision of analysis for the programs, replicate analyses (10 percent of the total number of samples) are used following the schematic shown in Figure 3-1. ERG provides the final data summaries to the associated agencies electronically in Excel® and Adobe® formats. ERG staff finalizes and uploads the data into the Air Quality Subsystem (AQS) database.

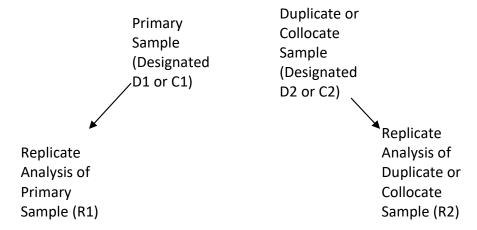


Figure 3-1 Duplicate/Collocate and Replicate Analysis Schematic

<sup>\*\*</sup>NIOSH Method 7903 was replaced with 7906, 7907 and 7908.

<sup>\*\*\*</sup>SOP is currently archived but will be updated if needed for sample analysis and support.

Project No.: 0475.00
Element No.: Section 4 - A7
Revision No.: 2
Date: August 2023
Page: 16 of 155

### SECTION 4 DATA QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

As ERG performs measurement services only, DQOs for defining a toxics network program are not identified in this QAPP. A well-prepared description of the Measurements Quality Objectives (MQOs) can be found in the TAD for the NATTS Program prepared for EPA in October 2016<sup>(22)</sup>. This section will discuss the MQOs of the ERG laboratory analyses, emphasizing the levels of uncertainty the decision maker is willing to allow/accept from the analytical results. The DQOs for the programs – NMOC, NATTS, UATMP, PAMS, and CSATAM – are similar but are not identical. Therefore, the programs are discussed separately.

The NATTS TAD presents the requirements for collecting and reporting data for the NATTS network. Eighteen compounds have been identified as major risk drivers based on a relative ranking performed by EPA and have been designated as NATTS Core or "Tier I" compounds. All other reported compounds, for any NHAPS Programs, are considered compounds of interest, but do not necessitate the NATTS MQOs. The Tier I compounds are acknowledged throughout this document. The EPA-approved ERG exemption from the NATTS TAD is listed in Appendix A.

Once a DQO is established, the quality of the data must be evaluated and controlled to ensure that data quality is maintained within the established acceptance criteria. MQOs are designed to evaluate and control various phases (sampling, preparation, analysis) of the measurement process to ensure that the total measurement uncertainty is within the range prescribed by the DQOs. MQOs can be defined in terms of the following data quality indicators:

<u>Precision</u> - a measure of mutual agreement between individual measurements performed according to identical protocols and procedures. This is the random component of error.

<u>Representativeness</u> - a measure of the degree to which data accurately and precisely represent a characteristic of population, parameter variations at a sampling point, a process condition, or an environmental condition.

<u>Comparability</u> - a measure of the level of confidence with which one data set can be compared to another.

Project No.: 0475.00
Element No.: Section 4 - A7
Revision No.: 2
Date: August 2023
Page: 17 of 155

<u>Bias</u> - the systematic or persistent distortion of a measurement process that causes error in one direction. Bias is determined by estimating the positive and negative deviation from the true value as a percentage of the true value.

<u>Completeness</u> - a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions. Data completeness requirements are included in the reference methods (see Section 3, Table 3-1).

<u>Detectability</u> - the determination of the low range critical value of a characteristic that a method-specific procedure can reliably discern.

Reporting units are consistent for each site across the network to ensure that the data can be reviewed with minimal manipulation. The units for each of the analytes are listed below:

- NMOC parts per million by Carbon (ppmC)
- SNMOC parts per billion by Carbon (ppbC)
- VOCs parts per billion by volume (ppbV)
- Carbonyls microgram per cubic meter (μg/m³) or ppbV
- Metals nanogram per cubic meter (ng/m³)
- Hexavalent Chromium ng/m³
- Semivolatiles ng/m³
- PCB/Pesticides ng/m³
- Anions ppbV
- VOC via cartridge ppbV
- Diisocyanates μg/m³
- 4,4'-Methylenedianaline μg/m³
- Landfill Gas and Natural Gas Compounds ppbC
- Reduced Sulfur Compounds parts per million by volume (ppmV)
- ICPMS for lead μg/filter
- XRF for lead μg/filter

Project No.: 0475.00
Element No.: Section 4 - A7
Revision No.: 2
Date: August 2023
Page: 18 of 155

The determination of analytical and sampling precision has been revised in the TAD, Revision 4<sup>(23)</sup> and will be implemented for the 2023 data reports. If the precision pair (i.e., collocated samples, duplicate samples, replicate analyses, etc.) has one of the pair measurements above and one below the analytical threshold (ex. 3 times the MDL), the RPD is calculated by replacing the value below the threshold with the threshold concentration.

The analytical RPD is calculated by comparing the differences between Replicate analyses (two analyses of the same sample) from the arithmetic mean of the two results as shown below.

Replicate analyses with low variability have a lower Relative Percent Difference (RPD) (better precision), whereas high variability samples have a higher RPD (poorer precision).

$$RPD = \frac{|X_1 - X_2|}{\bar{X}} \times 100$$

Where:

 $X_1$  = Ambient air concentration of a given compound measured in one sample.

X<sub>2</sub> = Concentration of the same compound measured during replicate analysis.

 $\overline{X}$  = Arithmetic mean of  $X_1$  and  $X_2$ .

Method precision is calculated by comparing the concentrations of the duplicates/collocates for each pollutant. The Coefficient of Variation (CV) calculation shown below is ideal when comparing paired values, such as a primary concentration versus a duplicate concentration.

$$CV = 100 \times \sqrt{\frac{\sum_{i=1}^{n} \left[ \frac{(p-r)}{0.5 \times (p+r)} \right]^{2}}{2n}}$$

Where:

p = the primary result from a duplicate or collocated pair.

r = the secondary result from a duplicate or collocated pair.

n = the number of valid data pairs (the 2 adjusts for the fact that there are two values with error).

Bias has been the term frequently used to represent closeness to "truth" and includes a combination of precision and bias error components. Bias is determined through the analysis of PT samples. NATTS PT samples are provided bi-yearly for each of the four sample classes, VOC, carbonyls, metals and semivolatiles. NAAQS lead audit samples are analyzed quarterly and

Project No.: 0475.00
Element No.: Section 4 - A7
Revision No.: 2
Date: August 2023
Page: 19 of 155

reported to the EPA, State, local and regional agencies. The MQOs listed will attempt to separate measurement uncertainties into precision and bias components. Table 4-1 lists the MQOs for pollutants to be measured in all areas of the NHAPS program.

Project No.: 0475.00
Element No.: Section 4 - A7
Revision No.: 2
Date: August 2023
Page: 20 of 155

Table 4-1 Measurement Quality Objectives for the National Hazardous Air Pollutants Support Program

Program	Precision from analysis of Replicate Samples (RPD)	Precision (CV) from collection of Duplicate/Collocate Samples	Representativeness	Comparability/ Based on Method	Bias	Completeness	Sensitivity (MDL)*
NMOC	≤ 10%	≤ 20%	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	GC-PDFID EPA Compendium Method TO-12 <sup>(3)</sup>	± 25%	≥85%	To be determined upon need
SNMOC	≤ 25% for concentrations ≥ 5x MDL	≤ 25% for concentrations ≥ 5x MDL	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	GC-FID TAD for O3 Precursors	± 25%	≥85%	See Table 11-13
VOC	≤ 25% for concentrations ≥ 5x MDL	≤15% for concentrations ≥ 5x MDL	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	GC-FID/MS EPA Compendium Method TO-15 <sup>(4)</sup>	± 25%	≥85%	See Table 11-14 and Table 11-18 for NATTS Tier I
Carbonyls	≤ 10% ≥ 0.5 µg/cartridge	≤15% for concentrations ≥ 0.5 µg/cartridge	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	HPLC EPA Compendium Method TO-11A <sup>(5)</sup>	± 25%	≥85%	See Table 11-15 and Table 11-18 for NATTS Tier I
Metals	≤ 20% for concentrations ≥ 5x MDL	≤15% for concentrations ≥ 5x MDL	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	ICPMS IO-3.5 <sup>(6)</sup> /EQL-0512- 201 <sup>(7)</sup> / EQL-0512-202 <sup>(8)</sup>	± 25%	≥85%	See Table 11-17 and Table 11-18 for NATTS Tier I
Hexavalent Chromium	≤ 20% for concentrations > 5x MDL	≤ 15% for concentrations > 5x MDL	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	IC-UV Detector ASTM D7614 <sup>(9)</sup>	± 25%	≥85%	See Table 11-17
Semivolatiles	≤ 10% for conc. ≥ 0.5 µg/mL or lowest ICAL level, whichever is less	≤15%, for conc. ≥ 0.5 µg/mL or lowest ICAL level, whichever is less	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	GC/MS EPA Compendium Method TO-13A <sup>(10)</sup> and ASTM D6209(12), (or SW-846 Method 8270E <sup>(11)</sup> )	± 25%	≥85%	See Table 11-16 and Table 11-18 for NATTS Tier I
PCB/ Pesticides	≤ 15%	≤ 15%	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	GC EPA Compendium Method TO-4A <sup>(13)</sup>	± 25%	≥85%	To be determined upon need

Project No.: 0475.00
Element No.: Section 4 - A7
Revision No.: 2
Date: August 2023
Page: 21 of 155

Table 4-1 Measurement Quality Objectives for the National Hazardous Air Pollutants Support Program

Program	Precision from analysis of Replicate Samples (RPD)	Precision (CV) from collection of Duplicate/Collocate Samples	Representativeness	Comparability/ Based on Method	Bias	Completeness	Sensitivity (MDL)*
Anions	≤ 15%	≤ 15%	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	IC NIOSH Method 7906 <sup>(14)</sup> , 7907 <sup>(15)</sup> , 7908 <sup>(16)</sup>	± 25%	≥85%	To be determined upon need
VOCs via cartridge	≤ 15%	≤ 15%	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	GC/MS EPA Compendium Method TO-17 <sup>(17)</sup>	± 25%	≥85%	To be determined upon need
Diisocyanates	≤ 15%	≤ 15%	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	HPLC OSHA Method 42 <sup>(18)</sup>	± 25%	≥85%	To be determined upon need
4,4'- Methylene- dianiline	≤ 15%	≤ 15%	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	HPLC NIOSH Method 5029 <sup>(19)</sup>	± 25%	≥85%	To be determined upon need
Landfill gas and Natural gas	≤ 25% for concentrations ≥ 5x MDL	≤ 25% for concentrations ≥ 5x MDL	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	GC-FID TAD for O3 Precursors	± 25%	≥85%	To be determined upon need
Reduced Sulfur							To be determined upon need
Lead Analysis (ICPMS)	± 10% for concentrations ≥ 10x MDL	±20% for concentrations ≥ 10x MDL	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	EQL-0512-201 <sup>(7)</sup> / EQL-0512-202 <sup>(8)</sup>	± 25%	≥85%	See Table 11-17
Lead Analysis (XRF)0.57	± 10% for concentrations ≥ 10x MDL	±20% for concentrations ≥ 10x MDL	24 hours ± 1 hours, flag samples 22-23 hours and 25-26 hours	40 CFR 50, Appendix Q <sup>(21)</sup>	± 15%	≥85%	0.044 ug/filter

Project No. 0475.00
Element No. Section 5 - A8
Revision No. 2
Date August 2023
Page 22 of 155

### SECTION 5 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

The activities of EPA's NHAPS are performed using accepted EPA, National Institute for Occupational Safety and Health (NIOSH), and Occupational Safety and Health Administration (OSHA) sampling and analytical protocols for the field sampling training personnel and analytical laboratory staff.

## **5.1** Field Activities Training Personnel

Field activities training personnel involved in this project have experience in the duties they will be performing in the field. The training of ERG field activities personnel is recorded in the ERG Training Records files. Special certification is not needed for an operator to set up the sampling systems. Each State should document and record the training of their personnel on the field-testing procedures provided by ERG.

The States' field-testing staff will be subject to on-site surveillance by EPA. ERG's Task Leader will provide appropriate corrective action enforcement, if necessary, for the ERG personnel setting up the sampling equipment and the field testing staff. ERG provides on-the-job training in the field on sampler use and maintenance, for supervisors and field site operators. The appropriate SOPs used during training are presented in Appendix D. ERG does not provide SOPs for sampling systems that are not maintained by ERG. Sampling System Training forms used during operator training in the field is presented in Figure 7.2 for VOC/Carbonyl and Carbonyl samplers. The forms will only be provided when new site personnel are trained on the sampling systems. After training is completed and signed in the field, the yellow copy is retained for site records. The original copy is scanned in the laboratory and stored by the QA coordinator.

The sampling equipment that ERG provides for monitoring sites must be maintained inside a sampling building. There are no hazards inherent to the samplers and no special safety training or equipment will be required. Site hazards should be addressed on a site-by-site basis by the site operator's SOPs. All ERG field activities training personnel will follow the ERG Corporate Health and Safety Plan.

Project No. 0475.00
Element No. Section 5 - A8
Revision No. 2
Date August 2023
Page 23 of 155

# 5.2 Analytical Laboratory Personnel

Analytical laboratory personnel involved in this project have been trained in their tasks and have experience in the duties they will be performing in the analytical laboratory. Training of ERG laboratory personnel is recorded in ERG Training Records in an Excel® database and filed as a hardcopy. It is the responsibility of the trainee and the laboratory's Project Administrator to keep the Training Records up to date. It is the responsibility of the Program Manager and Quality Assurance Coordinator to approve analysis training records. Technical training and overview are provided to the analyst by the Task Leader for that analysis. Technical training includes general techniques and specific training based on the appropriate SOP, method, and program QAPP. The trainee first observes the task, then performs the task under supervision of the trainer, then performs the task under supervision of the Task Lead (if the Task Lead is not the trainer). After training, demonstration of each personnel's ability to perform an analytical task involves repeated measurements of a standard, which is described in more detail in each analytical SOP. Currently, no special certifications are needed for the analysis of the ambient samples received for these programs.

ERG maintains appropriate SOPs for each of the analytical methods. These SOPs are presented in Appendix D. All SOPs document equipment and/or procedures required to perform each specific laboratory activity. Laboratory staff will be subject to on-site surveillance by the QA staff and periodic performance evaluation (PE) samples. These audits will assure the program that the appropriate analysts and analytical procedures are being used. The samples involved in this program are generated by monitoring air emissions. Health and Safety training is performed annually. The laboratory personnel will adhere to the ERG Corporate Health and Safety manual.

Project No. 0475.00
Element No. Section 6 - A9
Revision No. 2
Date August 2023
Page 24 of 155

### **SECTION 6 DOCUMENTATION AND RECORDS**

The EPA NHAPS is a collection of individual ambient monitoring programs that generate documents and records that need to be retained/archived. All ERG staff working on this contract are provided access to a current electronic copy of this signed, EPA approved QAPP. Annually, the staff is required to sign a form to document that they read and understood the QAPP. In this QAPP, ERG's reporting package (information required to support the analytical results) includes all data required to be collected as well as support data deemed important by ERG/EPA.

# 6.1 Data Management

ERG has a structured records management system that allows for the efficient archive and retrieval of records. Each laboratory archives the data from the computer systems onto the shared network drive. The laboratory paper copies of all analyses are stored on site in a secured temperature-controlled area for at least five years after the close of the contract. The laboratory also archives the data in the Laboratory Information Management System (LIMS) data server, which is backed up weekly, monthly, and biannually. The Program Manager has final authority for the storage, access to, and final disposal of all records kept for the EPA NHAPS.

# **6.2** Preliminary Monthly Data Reports

Preliminary monthly summary data reports are sent in Adobe Portable Document Format (PDF) and Excel formats to EPA and appropriate State/Local/Tribal agencies. The monthly data reports include analytical results, associated MDL, final units, associated QC samples, and data qualifiers.

# 6.3 Quarterly QA Report

A QA report for each type of data analysis is sent to EPA and appropriate State/Local/Tribal agencies on a quarterly basis in the form of control charts including initial calibration

Project No. 0475.00
Element No. Section 6 - A9
Revision No. 2
Date August 2023
Page 25 of 155

verifications, continuing calibration verifications, method blanks, initial calibration blanks, continuing calibration blanks, and blank spikes.

### 6.4 Annual Summary Reports Submitted to EPA

Beginning with the 2017 sampling year, the written annual report was replaced with a QlikSense® app, which allows users to view and explore data visualizations on their own. The QlikSense app is provided to EPA in the form of a mash-up, or web page developed from the app, and is housed on EPA's server. The intent is for EPA to make the app publicly available such that State/Local/Tribal clients can access the app. The data products are based on data collected from January 1 to December 31 for a given year. The data products contained in the app are similar to those presented in past annual reports and can contain the following information:

- A summary of site- and laboratory-specific happenings affecting data collected in a given year;
- Names of participating sites and corresponding metadata information, including city name, location and the AQS codes;
- Completeness of the monitoring effort for each site;
- General combined and individual site statistical summary of the year's results;
- Risk screening evaluations using toxicity factors [e.g., Unit Risk Estimate (UREs) or Reference Concentration (RfCs)];
- For specific HAPs identified via the risk screening evaluation, summary statistics, time-series plot, bar charts and table presenting annual and quarterly average concentrations, and historical box and whisker plots (where applicable).
- Results of coefficient of variation calculations used to assess precision; and
- Results of audits (e.g., PT audits) conducted during the sampling year.

ERG developed a User Guide and an accompanying Memorandum to help users learn how to maneuver around in the app and understand how the data were treated during the

Project No. 0475.00
Element No. Section 6 - A9
Revision No. 2
Date August 2023
Page 26 of 155

development of the app. If corrections or changes are needed after the app are presented to EPA, they can be made relatively easily.

# 6.5 Records and Supporting Data

All raw data required for the calculation of air toxics concentrations, submission to the EPA/AQS database, and QA/QC data are collected electronically or on data forms that are included in the field and analytical methods sections. All hardcopy information is filled out in indelible ink. Corrections are made by inserting one line through the incorrect entry, initialing the correction (ERG maintains a signature log of project staff), and placing the correct entry alongside the incorrect entry, if this can be accomplished legibly, or by providing the information on a new line. Table 6-1 presents the location of the data records for field and laboratory operations stored at the ERG laboratory.

**Table 6-1 Data Documentation and Records** 

Item	Record	Short Term Location Storage	Long Term Location Storage				
	Field Operations						
Sampling System Training	Sampling System Training Form	ERG	Copy scanned and hardcopy stored by ERG				
сос	ERG COCs	Field gets "pink" copy, ERG gets "yellow" and "white" copy	Copy scanned and stored on ERG LIMS				
QC Sample Records (field blanks, duplicate/ collocated, sample integrity, etc.)	сос	Field	Copy scanned and stored on ERG LIMS				
General Field Procedures	сос	Field	Copy scanned and stored on ERG LIMS				
	Laboratory Records	5					
Sample Prep Data	Bench sheets	Hardcopy filed, LIMS, shared network drive	Hardcopy archived, LIMS, shared network drive				
	Laboratory Operation	ns					
Sample Management Records (sample receipt, handling, storage, etc.)	COCs	LIMS, with sample analytical data	LIMS, with sample analytical data				
Test Methods	SOPs	Hardcopy filed, shared network drive	Shared network drive				

Project No. 0475.00
Element No. Section 6 - A9
Revision No. 2
Date August 2023
Page 27 of 155

Table 6-1 Data Documentation and Records

ltem	Record	Short Term Location Storage	Long Term Location Storage			
QA/QC Reports (General QC records, MDL information, calibration, etc.)	Individual records for each analysis	Hardcopy filed, shared network drive	Hardcopy archived, shared network drive			
Corrective Action Reports	Individual records for each analysis	Hardcopy filed, a copy in data package if appropriate	All copies archived			
Data Reduction, Verification, and Validation						
Electronic Data (used for reporting and AQS)	Excel® and Access®	Shared network drive	Shared network drive			

#### 6.5.1 Notebooks

ERG issues laboratory notebooks upon request. These notebooks are uniquely numbered and associated with the laboratory personnel. Notebooks are archived upon completion for at least 5 years from the end of a project. Although LIMS data entry forms are associated with all routine environmental data operations, the notebooks can be used to record additional information about these operations. The procedures for maintaining notebooks are presented in SOP for Maintaining Laboratory Notebooks (ERG-MOR-039) in Appendix D.

Field Notebooks - Field notebooks are the responsibility of EPA, States, Local or Tribal agencies as ERG is not responsible for the collection of samples.

Laboratory Notebooks - Notebooks are associated with general procedures such as calibration of analytical balances, standard preparation logs, etc., used in this program.

Logbooks are generated and bound by the laboratory's Project Administrator for procedures such refrigerator/freezer temperatures, canister cleaning, etc. Logbook pages have a unique version identifier. Upon completion, logbooks are archived indefinitely, at a minimum at least 5 years from the end of a project.

Project No. 0475.00
Element No. Section 6 - A9
Revision No. 2
Date August 2023
Page 28 of 155

#### 6.5.2 Electronic Data Collection

To reduce the potential for data entry errors, automated systems are utilized (where appropriate) and record the same information that is found on data entry forms. In order to provide a back-up, hardcopy data collected on an automated system will be stored for 5 years after the end of the closed EPA NHAPS contract.

### 6.6 Data Reporting Package Archiving and Retrieval

In general, all the information listed above will be retained for at least 5 years from the date of the end of the closed contract with EPA. However, if any litigation, claim, negotiation, audit, or other action involving the records has been started before the expiration of the 5-year period, the records will be retained until completion of the action and resolution of all issues which arise from it, or until the end of the regular 5-year period, whichever is later. The long-term storage is on-site in a locked climate-controlled file room with limited-access. The Project Administrator keeps a record of documents entering and leaving long-term storage. Access to the facility storage area is limited to authorized personnel only.

# 6.7 Quality System Document Control

To ensure the use of the most current version of quality system documents, all quality documents (QAPP, SOPs, etc.) generated at the ERG Laboratory must be uniquely identified. Original documents shall include the date of issue, revision number, page number, the total number of pages, and appropriate signatures. Copies of quality documents shall be controlled and include a copy control number. When an original quality document is updated, the QA Coordinator or designee will ensure that the copy documents are also updated, and old versions are destroyed. During the project, revised QAPPs will be circulated to appropriate EPA personnel and ERG's laboratory staff. For copies of documents out of the laboratory's control, a stamp or watermark stating "Uncontrolled" or "Draft," if applicable, will be applied. Each approved QAPP will be posted on EPA's Ambient Air Monitoring Technical Information Centers (AMTIC) Website without the associated SOPs.

Project No. 0475.00
Element No. Section 7 - B1
Revision No. 2
Date August 2023
Page 29 of 155

### **MEASUREMENT DATA ACQUISITION**

### SECTION 7 SAMPLING PROCESS DESIGN

Sampling procedures for the NMOC, SNMOC, UATMP, NATTS, and CSATAM programs are discussed in this section. ERG provides site-specific support for the PAMS and HAPs sampling. All parameters listed in this section are necessary for the sampling systems listed below. ERG is not responsible for the collection of samples nor the design of these programs. The 2023 and 2024 6-day and PAMS sampling calendars are presented in Appendix B. All sites are instructed to send collected samples back to the laboratory soon after sampling to meet extraction and/or analytical hold time requirements.

### 7.1 NMOC and SNMOC Canister Samplers

Sampling for NMOC and SNMOC takes place each workday from the beginning of June to the end of September at designated NMOC and SNMOC sites from 0400-1200, 1200-2000, and 2000-0400 local time. Sampling procedures have been discussed in detail in other documents <sup>(1)</sup>, Figure 7-1 is a diagram of the ERG sampling system used for collecting the ambient air samples. Clean, evacuated passivated stainless-steel canisters are shipped daily from ERG's Research Triangle Park (RTP) Laboratory to the NMOC and SNMOC sites. Canisters are connected to the sampling system by local operators. The digital timer automatically activates the pump and solenoid valve to start and stop sample collection. The pump pressurizes air samples during the sampling period to about 15 pounds per square inch gauge (psig), and the flow control valve (variable orifice) ensures a constant sampling rate over the 8-hour period. A 2-micron stainless steel filter is installed in the sampling line to remove particulate from the ambient air that may damage or plug the variable orifice. The sample probe inlet is positioned from 2 to 10 meters (m) above ground level.

ERG installs the sampling systems at the site location and trains associated local operators on site. Operator training is documented on the Sampler Training Form (Figure 7-2). It is the responsibility of the local operators to operate the sampling apparatus and complete the field sample COC form that ERG supplies with each canister. ERG staff maintain telephone and/or

Project No. 0475.00
Element No. Section 7 - B1
Revision No. 2
Date August 2023
Page 30 of 155

email contact throughout the project to provide whatever assistance is needed to resolve technical issues that arise during the sampling program.

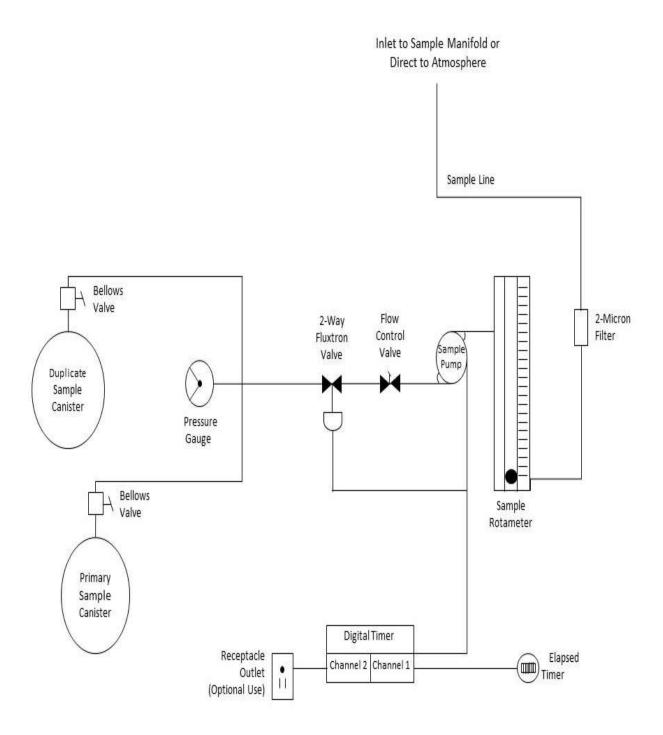


Figure 7-1 NMOC, SNMOC, and 3-Hour Air Toxics Sampling System Components

Project No. 0475.00
Element No. Section 7 - B1
Revision No. 2
Date August 2023
Page 31 of 155

# Sampling System Training

Installation Date:		Trainer:		
Site ID:		Copy of SOP on Site: (Y/N)  Replaced Sampler ID #:		
Installed Sampler ID #:	5-4 			
Time Set:	-37	Carb Line Replaced:	(Y/N)	
Timer Set:		VOC Line Replaced:	(Y/N)	
Trainee:	Signature:	Dat	te:	
4	* *	40 90	-	
(c)		#: #8 #2 16	<i>S</i>	
ā			<del></del>	
48			7	
NOTES:	<del></del>			
St. (2007)			-	
75 56			9 9	
<u> </u>				
<u> </u>				
-				
			3	

ERG assumes no personal and/or property liability realized by the user from the use of ERG provided equipment. The user, by virtue of accepting the ERG equipment for use, undertakes any/all personal and/or property liabilities that could be associated with its use (including operational, housing, and/or safety).

L:\samplers\Sampling System Training Form rev 11-14.pdf

Figure 7-2 VOC/Carbonyl Sampler Training Form

Project No. 0475.00
Element No. Section 7 - B1
Revision No. 2
Date August 2023
Page 32 of 155

For a 3-hour ambient air sample, NMOC, SNMOC, and VOC measurements may all be performed from the same canister. Refer to Section 7.2 for sampler certification.

## 7.2 VOC and Carbonyl 24-Hour Samplers

ERG provides the sites with a sampling schedule each year. A total of 31 sampling days will be scheduled per site for a 12-day sampling schedule and 61 sampling days for the 6-day sampling schedule. Days for duplicate (or collocated) sampling and field blank (if applicable) sampling will also be designated. The 2023 and 2024 Sampling calendars are presented in Appendix B.

Prior to installation of an ERG sampler at a UATMP, NATTS or CSATAM site, the sampler is certified at the ERG laboratory. Certification establishes that the system is functioning correctly and provides for the appropriate level of specified compound recovery and cleanliness. To certify the sampling system, cleaned, humidified air is first flushed through the sampler for at least 12 hours to remove the potential for organic contaminants in the manifold and system. The canister sub-system of the samplers is then challenged with a mixture of representative VOCs at known concentrations to qualify the sampler recovery characteristics (as recommended in the NATTS TAD)<sup>(22)</sup>. After the sampler has been challenged, it is flushed again with humidified air for at least 12 hours. A Sampling System Blank, with clean humidified air, is then collected in canisters and on carbonyl cartridges and is analyzed based on EPA Compendium Method TO-15<sup>(4)</sup> and Method TO-11A<sup>(5)</sup> to verify that the system meets the required cleanliness criteria for Tier 1 NATTS compounds, 0.03 ppbV or 3 x MDL above the reference canister, whichever is lower, (as required by the NATTS TAD<sup>(22)</sup>) with non-Tier 1 compound criteria of 0.1 ppbV or 3 x MDL above the reference canister, whichever is higher (with upper limit of 0.2 ppbV) and with acetonitrile criteria of < 0.2 ppbV above the reference canister. These results are documented in a file specific to each sampler by system identification number. The certification procedures are presented in SOP for Canister Sampling System Certification Procedures (ERG-MOR-030) and SOP for Carbonyl System Certification Procedures (ERG-MOR-100) in Appendix D.

Project No. 0475.00
Element No. Section 7 - B1
Revision No. 2
Date August 2023
Page 33 of 155

Integrated ambient air samples are collected in 6-liter passivated stainless-steel canisters (SUMMA, Silonite®, TO-Can, etc.) and carbonyl cartridges for a 24-hour period beginning at midnight for each scheduled sampling event. Carbonyl cartridges are shipped cold, and the cleaned, quality-controlled canisters are shipped under vacuum to the site from the ERG laboratory. After sampling, the final pressure in the canister should ideally be between 2 to 8 inches of Mercury ("Hg) vacuum. The sampling assembly for the sample collection is shown in Figure 7-3.

The physical mechanism for filling the canister is vacuum displacement. The vacuum pump shown in Figure 7-3 is used to purge the mass flow controller and the sample inlet lines. A second vacuum pump is used to draw ambient air through the carbonyl sampling probe and cartridges. Ozone is removed from the sample stream prior to collection on the 2,4-Dinitrophenylhydrazine (DNPH) sampling cartridge. To accomplish O<sub>3</sub> removal, the sample stream (ambient air) is drawn through a potassium iodide-coated denuder O<sub>3</sub> scrubber which is an internally integrated component of the sampler. Carbonyl samplers can be collected with or without canister samples.

# 7.3 Carbonyl Only 24-Hour Samplers

Carbonyl samples are collected using DNPH-impregnated sampling cartridges with an integrated sampling system (e.g., vacuum pump, critical orifices, and  $O_3$  scrubber), shown in Figure 7-4. Ambient air is drawn through the cartridges via a separate sampling probe. A potassium iodide-coated denuder  $O_3$  scrubber is an internally integrated component of the sampler that removes  $O_3$  from the sample stream prior to the DNPH sampling cartridge.

Prior to installation of an ERG sampler at a UATMP, NATTS or CSATAM site, the sampler is certified at the ERG laboratory. Certification establishes that the system is functioning correctly and provides for the appropriate level of cleanliness. To certify the sampling system, cleaned, humidified  $N_2$  is first flushed through the sampler for at least 12 hours to remove the potential contaminants from the manifold and system. A Sampling System Blank and a reference blank

Project No. 0475.00
Element No. Section 7 - B1
Revision No. 2
Date August 2023
Page 34 of 155

are then collected on carbonyl cartridges and are analyzed based on EPA Compendium Method TO-11A<sup>(5)</sup> to verify that the system meets the required cleanliness criteria and can produce non-biased samples as required by the NATTS TAD<sup>(22)</sup>. These results are documented in a permanent

Project No. 0475.00
Element No. Section 7 - B1
Revision No. 2
Date August 2023
Page 35 of 155

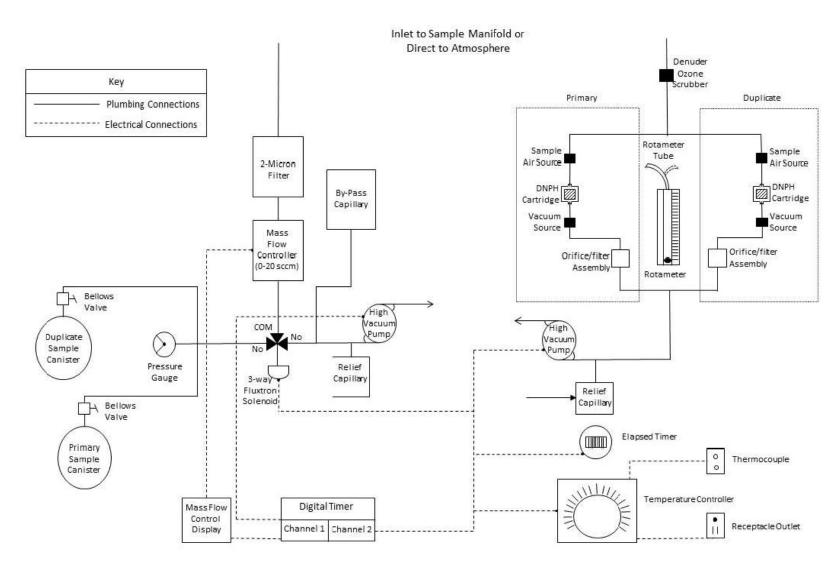
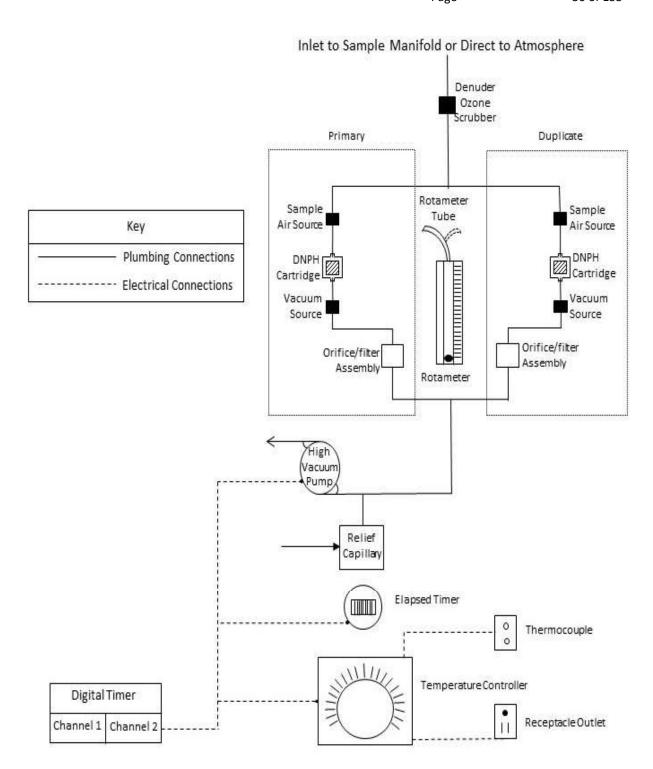


Figure 7-3 24-Hour Integrated Air Toxics Sampling System Components

Project No. 0475.00
Element No. Section 7 - B1
Revision No. 2
Date August 2023
Page 36 of 155



**Figure 7-4 Carbonyl Sampling System Components** 

Project No. 0475.00
Element No. Section 7 - B1
Revision No. 2
Date August 2023
Page 37 of 155

file specific to each sampler by system identification number. The certification procedure is presented in the SOP for Carbonyl Sampling System Certification (ERG-MOR-100) in Appendix D.

A total of 31 sampling cartridges for a 12-day sampling schedule and 61 sampling cartridges for a 6-day sampling schedule will be collected and analyzed per site. Duplicate (or collocated) samples and field blanks will be collected monthly and are designated in the 2023 Sampling calendar presented in Appendix B.

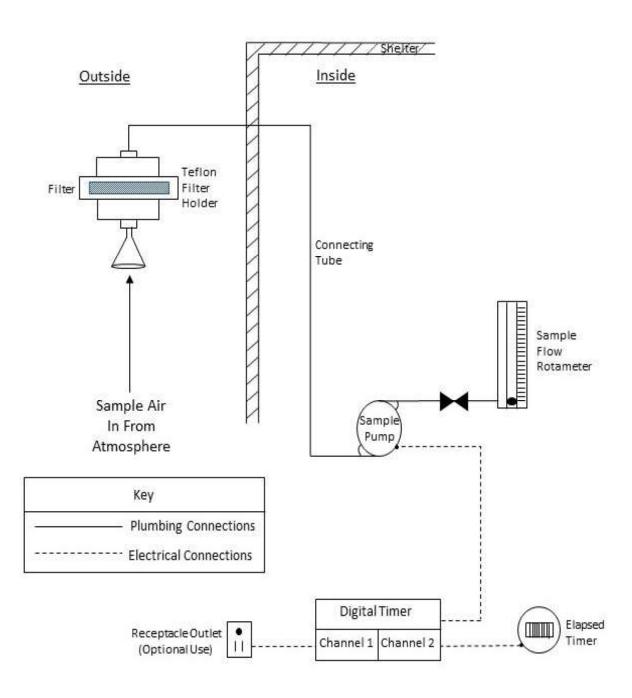
# 7.4 Hexavalent Chromium Samplers

Sodium bicarbonate-impregnated cellulose filters are connected to the Hexavalent Chromium sampler as shown in Figure 7-5 and ambient air is drawn through the filters through a glass sampling probe using Teflon sampling lines. Prepared filters are shipped to each site for the hexavalent chromium sampling. ERG ships the bicarbonate-impregnated sodium cellulose filters to each site in coolers (chilled with blue ice packs). The samples are collected for a 24-hour period. Disposable polyethylene gloves are used by the field operators when handling the filters to reduce background contamination. After sampling, the filters are removed from the sampling apparatus, sealed, and returned to the ERG laboratory in the coolers and ice packs in which they were received. Additional qualifying information for the hexavalent chromium sampling and analysis techniques is presented in the American Society for Testing and Materials (ASTM) D7614<sup>(9)</sup> method and specific details are provided in ERG's SOP for the Preparation and Analysis of Ambient Air for Hexavalent Chromium by Ion Chromatography (ERG-MOR-063) presented in Appendix D.

### 7.5 PAMS Sampling

PAMS sampling is performed completely by the PAMS sites in accordance with the Ozone Precursors TAD<sup>(2)</sup> with ERG only supplying support as requested [e.g., sampling system for automated gas chromatograph (GC) systems]. The need for support of automated GC systems is site specific. ERG ships cleaned canisters and prepared carbonyl cartridges to the PAMS sites on

Project No. 0475.00
Element No. Section 7 - B1
Revision No. 2
Date August 2023
Page 38 of 155



**Figure 7-5 Hexavalent Chromium Sampling System Components** 

Project No. 0475.00
Element No. Section 7 - B1
Revision No. 2
Date August 2023
Page 39 of 155

the appropriate schedule to support the sampling program, and the samples are shipped to the ERG laboratory for analysis.

# 7.6 HAPs Sampling

HAPs sampling is performed by the sites in accordance with the methods listed in Table 3-1, with the exception of hexavalent chromium sampling (see Section 7.4). ERG provides the hexavalent chromium sampling systems and media and receives the samples from the sites for analysis.

Project No. 0475.00
Element No. Section 8 - B2
Revision No. 2
Date August 2023
Page 40 of 155

### **SECTION 8 SAMPLING METHOD REQUIREMENTS**

The sampling methods that are used in this program are described in this Section. Since there are four separate sampling systems and subsequently four separate analytical techniques, each of the sampling methods is different.

As ERG is not responsible for actual execution of the field sampling in this program, the ERG SOPs list general sampling guidelines needed for the NMOC, UATMP, Carbonyl, and Hexavalent Chromium sampling for sites using ERG sampling systems. Table 8-1 identifies the different methods and SOP numbers for operation of each type of sampler ERG provides. Some HAPs sampling is not addressed in the NHAPS Support contract (Metals, PAHs, etc.), and are not discussed in this QAPP.

Table 8-1 EPA Methods and ERG SOPs for each Sampling System

Sampling System	Based on Applicable Method	ERG SOP Number
NMOC	EPA Compendium Method TO-12 <sup>(3)</sup>	ERG-MOR-046
voc	EPA Compendium Method TO-15 <sup>(4)</sup>	ERG-MOR-003
Carbonyl	EPA Compendium Method TO-11A <sup>(5)</sup>	ERG-MOR-047
Hexavalent Chromium	ASTM D7614 Method <sup>(9)</sup>	ERG-MOR-013

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 41 of 155

### **SECTION 9 SAMPLE HANDLING AND CUSTODY REQUIREMENTS**

Similar sample custody procedures are followed for all monitoring programs. However, program-specific differences exist because the analytical requirements for the programs vary. As these activities are conducted under one EPA contract, United Parcel Service of America (UPS) with Next and 2<sup>nd</sup> Day Air delivery will handle all shipping to and from the sites. Unless specified below, samples taken in the field should not require any extra special precautions for shipping.

The Shipping and Receiving Task Leader will ensure that sample media that leaves and field samples that are received in the laboratory follow all procedures listed in this QAPP and the individual SOPs. The Task Leader will also advise the Project Manager of any issues or obstacles regarding sample shipping, receipt, login, and storage. The sample custodian working under the Shipping and Receiving Task Leader will ship sample media to the field and receive custody of samples, complete COC receipt information, document sample receipt, and enter COC information into LIMS to create a work order.

### 9.1 Canister Sample Custody

# 9.1.1 Canister Custody

A color-coded, three-copy canister sample COC form (Figures 9-1, 9-2 and 9-3) is shipped with each 6-liter canister for the NMOC, SNMOC, UATMP, NATTS, CSATAM, or PAMS sites. If duplicate or collocated samples are to be taken, two canisters and two COC forms are sent in the shipping container(s) to the site. When a sample is collected, the site operator fills out the form, detaches the pink copy to be retained on-site and sends the remaining copies with the canister in the shipping container to ERG's laboratory.

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 42 of 155

1 Keystone	ven Rever to Grup. no. 9 Park Drive, Suite 700, Morrisville, NC 27560	
Lab Pre-Sampling	Site Code: City/State: AQS Code: Collection Date: Options NMOC (Y/N): SNMOC (Y/N): TOXICS (Y/N):	Date Can. Cleaned:  Cleaning Batch # :  Duplicate Event (Y/N):  Duplicate Can # :
Field Setup	Operator:Sys. #:Setup Date:Field Initial Can. Press. ("Hg):	Elapsed Timer Reset (Y/N):  Canister Valve Opened (Y/N):
Field Recovery	Recovery Date: Field Final Can. Press. (psig):	Sample Duration (3 or 24 hr):
Lab   Recovery	Received by: Date: Status: Valid Void (Circle If void, why:	e one)
NMOC	Analyst:	Database entry by:Date:Date:
SNMOC   Option	Analyst: Batch ID	Date:
Toxics Option	Analyst: Batch ID	Date:
ommen	ts:	

Figure 9-1 Example NMOC COC

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 43 of 155

ØE	RG	ERG LIMS ID #
Keystone	Park Drive, Suite 700, Morrisville, NC 27560  AIR TOXICS SAMPL	E CHAIN OF CUSTODY
	Site Code:	Canister Number:
	City/State:	Collection Date:
<b>D</b>	AQS Code:	Lab Initial Can. Press. ("Hg):
Lab Pre-Sampling		Cleaning Batch #:
Lab	Options:	Date Can. Cleaned:
ė	SNMOC (Y/N):	
	TOXICS (Y/N):	
	METHANE (Y/N):	Duplicate Can # :
	Relinquished by:	
	Received by:	
	Operator:	
모음	System #:	Elapsed Timer Reset (Y/N):
Field Setup	Channel #:	Canister Valve Opened (Y/N):
	Setup Date:	
	Field Initial Can. Press.:	
	Recovery Date:	
- C	Operator:	Elapsed Time:
Field Recovery	Field Final Can. Press.:	
_ &	Status: VALID VOID (Circle	one) Canister Valve Closed (Y/N):
	Relinquished by:	Date:
>	Received by:	Date:
Lab	Lab Final Can. Press.:	psig "Hg (Circle one)
Rec	Status: VALID VOID (Circle	vac. one) Gauge: 1 3 Assessment (Circle one)
	If void, why:	
		Samples stored in Air Tox Lab (Room 130)
		03-20
mmen	ts:	

Figure 9-2 Example Air Toxics COC

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 44 of 155

Keystone Pi	art Drive, Suite 700, Morrisville, NC 27560  PASSIVE SAMPLE C	
		HAIN OF CUSTODY
	Site:	Canister Number:
8	City/State:	Lab Initial Can. Press. ("Hg):
Lab Pre-Sampling	Options:	Cleaning Batch #:
Sami	SNMOC (Y/N):	Date Can. Cleaned:
é	TOXICS (Y/N):	Duplicate Event (Y/N):
	METHANE (Y/N):	Duplicate Can #:
	Relinquished by:	Date:
	Received by:	
	Operator:	
	Probe ID #:	
Field	Timer Used (Y/N):	Timer ID #:
	Leak Check: PASS FAIL (Circle Or	ne) Leak Check Rate: (If applicable)
	Field Initial Can Proce	"Hg Canister Valve Opened (Y/N):
	Pied Illiai Gal. Fless.	ng Canster valve Opened (1714).
	Recovery Date:	
d eny	Operator:	End Time:
Field Recovery	Field Final Can. Press.:	The state of the s
æ		e) Canister Valve Closed (Y/N):
	Relinquished by:	Date:
7	Received by:	Date:
Lab Recovery	Lab Final Can. Press.: "H	lg .
Rec	Status: VALID VOID (Circle on	e) Gauge: 1 2 3 (Circle one)
	If void, why:	
		Samples stored in Air Tox Lab (Room 130)

Figure 9-3 Example Passive Sample Air Toxics COC

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 45 of 155

Upon receipt, the sample canister vacuum/pressure is measured and compared against the field documented vacuum/pressure to ensure the canister remained airtight during transport. If the receiving vacuum differs from the field vacuum more than 3"Hg, the program manager is notified, and sample canister may be voided. Because there are potential differences in barometric pressures and temperatures between the sampling site and the receiving laboratory (such as those sites at high altitudes), and different accuracies for different types of pressure gauges, there can be a consistent difference in final field pressure and lab receipt pressure for canister samples. This difference and other parameters are considered to determine the validity of the canister samples. These are monitored daily, and the pressures are logged into an Excel spreadsheet. This allows the laboratory the ability to determine if the canister leaked en route or if the difference is typical for that site. A sample of the spreadsheet is presented in Table 9-1.

Table 9-1 Example of Canister Pressure Check Spreadsheet

Date Received	Site	Field Pressure Reading	Lab Pressure Reading	Difference
2/10/22	NBIL	8.5 "Hg	10 "Hg	-1.5 "Hg
2/16/22	NBIL	7 "Hg	7.5 "Hg	-0.5 "Hg
2/16/22	NBIL	7 "Hg	7.5 "Hg	-0.5 "Hg
2/22/22	NBIL	8.1 "Hg	9 "Hg	-0.9 "Hg
2/10/22	CSNJ	5 "Hg	5.5 "Hg	-0.5 "Hg
2/16/22	CSNJ	5 "Hg	6 "Hg	-1.0 "Hg
2/16/22	CSNJ	5 "Hg	5 "Hg	0 "Hg
2/22/22	CSNJ	4 "Hg	5.5 "Hg	-1.5 "Hg

The canister should be cleaned no more than 30 days before sampling. If the canister is older than 30 days, a note will be made in LIMS and a flag will be added to the sample results in AQS. More detailed sample receipt procedures and sample acceptance policies are presented in the SOP for Sample Receipt at the ERG Chemistry Laboratory, ERG-MOR-045 in Appendix D. The sample specific information from the COC is then entered into LIMS (example login page is shown in Figure 9-4) following the SOP for Sample Login to the Laboratory Information

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 46 of 155

Management System, ERG-MOR-079 found in Appendix D. The sample is given a unique LIMS identification (ID) number and tagged (see Figure 9-5), noting the desired analysis, site location and the sample collection date.

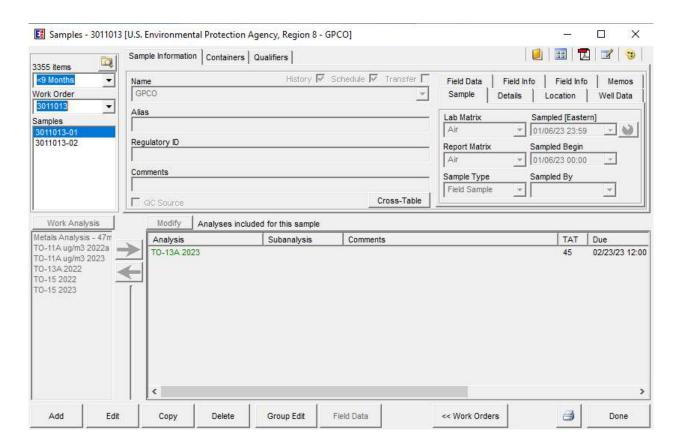


Figure 9-4 Example ERG LIMS Login Page

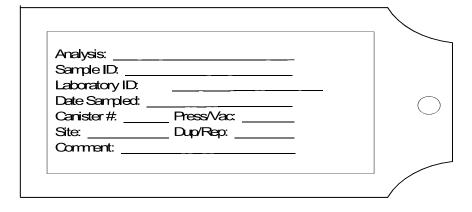


Figure 9-5 Canister Tag

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 47 of 155

The LIMS ID number is recorded on all ERG copies of the COC. The remaining copies of the canister sample COC are separated. The white copy is scanned (the PDF is stored in the LIMS system) and is kept with the canister sample until analysis is complete. After sample analysis, the white copy goes into the data package with the sample data. The yellow copy is stored chronologically in a designated file cabinet for 6 months. The file cabinet is in Room 102 in the Laboratory building.

# 9.1.2 Canister Analytical Routing Schedule

Each canister has a unique identification number inscribed on the canister. This number is used during can cleaning, field collection, laboratory receipt, and laboratory sample analysis, and is included on the individual Toxics/SNMOC COCs and entered into the LIMS.

The canister sample analysis hold time is 30 days from the sampling date. The canister samples are sent to the ERG Air Toxics Laboratory for VOC and SNMOC/PAMS analysis via GC/Flame Ionization Detector/Mass Spectrometer (FID/MS) or GC/MS, as applicable. The canister sample is analyzed and kept in the laboratory until after the analyst reviews the relevant analytical data.

### 9.1.3 Canister Cleanup

All canisters are cleaned prior to reuse following SOP ERG-MOR-105 (SOP for Sample Canister Cleaning using Wasson TO-Clean Automated System) as shown in Appendix D. The canisters are cleaned using the procedure described in Section 10.1.1. One of 8 cleaned canisters (or 2 of 12 cleaned canisters) is brought to  $\leq$  5 psig and analyzed. The canister cleanliness criteria is <3x MDL or 0.03 ppbV, whichever is lower (and Tier  $1 \leq$  MDL MQO), and 20 ppbC for Total SNMOC. If the canister fails the Blank criteria, it is returned to the cleaning system bank with the other canisters that were cleaned along with it and all canisters are put through an additional Vacuum and Pressure cycle. The same canister is analyzed again. All canisters, whether used for NMOC, SNMOC, UATMP, NATTS, CSATAM, or PAMS, are cleaned by the same procedure and are

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 48 of 155

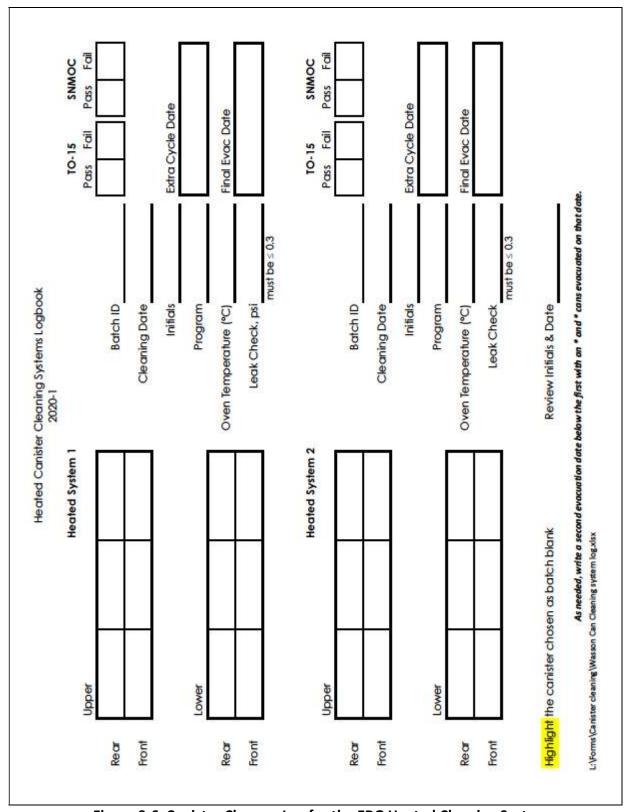


Figure 9-6 Canister Cleanup Log for the ERG Heated Cleaning System

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 49 of 155

entered into the canister cleanup log, shown in Figure 9-6. In instances where canisters have to be used pollutant compounds that fail are noted in LIMS and AQS with the appropriate flags, see Section 11.

# 9.2 Carbonyl Sample Custody

Figure 9-7 shows the color-coded, three-copy COC form used for all NATTS, UATMP, CSATAM type carbonyl sampling documentation. Figure 9-8 shows the color-coded, three copy COC form used for all PAMS carbonyl sampling. One PAMS COC can be used for each sampling day, 3-samples per day. A COC is shipped to the site with each of the carbonyl cartridges. After sampling, the COC form is completed by the site operator and the pink copy is retained for site records. The carbonyl sample cartridges and remaining COC copies are shipped to ERG's analytical laboratory.

When samples are received, they are logged into the LIMS database and given a unique LIMS ID number following the SOP for Sample Login to the Laboratory Information Management System, SOP ERG-MOR-079, found in Appendix D. The remaining copies of the COC are separated. The white copy of the COC is scanned (the PDF is stored in the LIMS system) and is labeled with the LIMS ID number, site code, sampling date, individual sample designations, and date of receipt and initials of receiving personnel and put into a bag. The sample bag is stored in a refrigerator designated for carbonyl samples only. The yellow copy is stored chronologically in a designated file cabinet for 6 months. The file cabinet is in Room 102 in the Laboratory building. More detailed sample receipt procedures and sample acceptance policies are presented in the SOP for Sample Receipt at the ERG Chemistry Laboratory, ERG-MOR-045.

### 9.2.1 Carbonyl Analytical Routing Schedule

The carbonyl cartridge samples are extracted within 14 days of the sampling day and analyzed within 30 days after extraction. The extracts are kept in the designated extract refrigerator until after the analyst and the Task Leader reviews all the relevant analytical data.

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 50 of 155

Field	Received by:  Set-Up Date:  City/State:		Date: Operator: AQS Code:	
		Sample P D1 C1 (Circle One)	Duplicate/Collocate D2 C2 (Circle One)	Field Blank
	ERG Lab ID #			
	Cartridge #			
	Sample Date			
_	Elapsed Time			
_ ii	Pre Sampling Reading			
Field ormati	Post Sampling Reading			
Field Information	Total Volume (std Liters)			
_	System #			
	Channel #			
	Cartridges Capped (Y/N)		1	
	Valid/Void (Circle one)		Valid Void	Valid Void
	Recovery Date:		Operator:	
	Relinquished by:	Date	2:	
	Received by:		Date:	
Lab Recovery	Status: VALID	VOID (Circle one)		
Lab Sov	If void, why:		Corrected Temperatu	ure: °C
_ &	and the same of th		IR Gun: 1 2	3 (Circle one)
			Samples stored in	CNACO CONTROL DIRECTOR AND CONTROL OF PARTY

**Figure 9-7 Example Carbonyl Compounds COC** 

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 51 of 155

	RG			ERG	Lab ID#		
Keystone	PAMS C		L COMP	OUNDS	CHAIN OF	CUSTOD	Y
Lab Pre-Samp.	Site Code:  Duplicate Event ( Field Blank Event Relinquished	(Y/N): t (Y/N): by:			Collection Date:		
Field	Set-Up Date:		Oper	ator:	Sy		
Field	Recovery Date: Operator: Cartridges Cappe Relinquished	ed (Y/N):		-	Status: VAI	n (8 or 24 hr): LID VOID (	(Circle one)
Lab	Received by: Status: VA If void, why:	LID VO	ID (Circle o	ne) Uncorrec	ted Temperature ed Temperature IR Gun	<u> </u>	°C °C (Circle one
		Sample Time	Sample Duration	Sample Volume	Cartridge Pouch#	Sample ID	Lab ID
	Sample Date			19		18 19	
PAMS	Sample Date						
SWY							.00-2

Figure 9-8 Example PAMS Carbonyl Compounds COC

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 52 of 155

#### 9.3 HAPs Sample Custody

Samples collected on prepared sample media (i.e., XAD-2®, Polyurethane Foam (PUF), hexavalent chromium filters, etc.) use supplied three-copy COC forms to document sample collection. Field testing personnel will record applicable collection data (such as time, date, location, meteorological parameters) on the appropriate COC forms (Figures 9-9 and 9-10) and keep the pink copies for site records. The COCs are then shipped to ERG with the collected samples.

Because the sites supply the filters used for metal analysis, COC forms are normally supplied by the State, Local or Tribal agency for these samples. If needed, however, COC forms can be supplied by ERG electronically inputting multiple filters for metal analysis (Figure 9-11 and 9-12). Samples are received at ERG's laboratory as presented in the SOP for Sample Receipt at ERG Chemistry Laboratory, ERG-MOR-045.

All HAPs samples received at the ERG laboratory will be logged into the LIMS as described in the SOP for Sample Login to the Laboratory Information Management System, ERG-MOR-079.

#### 9.4 Invalid Samples

The sample COC form may indicate that the sample sent from a site is invalid. The sample can be determined invalid at the site or in the laboratory. SOP ERG-MOR-045 describes the sample receiving procedure and sample acceptance. Individual sites will be contacted if there are any questions about the samples upon receipt. When a sample is designated as invalid, the assigned LIMS ID number is notated as a void and is invalidated on the individual respective COC form. Another sample media will be sent to the site with the COC designated to make up on non-standard sampling days. If the site has repeated invalid samples, normally three voids in a row, the ERG site coordinator Task Leader will work with the site personnel to diagnose and correct the problem. The sites will also be notified in the monthly analytical reports of any invalid samples.

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 53 of 155

	Site Code:		Conta	ainer#:			
g l				ction Date:			
_ <u>≅</u>	AQS Code:			cated Event (Y/N):			
Lab Samp							
Lab Pre-Sampling	Cartridge Certification I	Date:		XAD Lot:			
₽				PUF Lot:			
	Relinquished by:		Date:	Filter Lot:			
흑	Received by:						
sett.				m #:			
Field Setup	According Books		- Language	ed Timer Reset (Y/N			
Fie							
	Recovery Date: Collection System Information:						
		30535		Magnehelic	Flowrate	,	
	Elapsed Time	e Temp (°C)	Barometric ("Hg)	("H <sub>2</sub> O)	(std. m³/mi	in)	
/ery	Start						
00	End						
8	Average						
ield Recovery	Total Collection Time (	Minutes)	Total	Collection Volume (s	etd m <sup>3</sup> )		
		200		perator:	20.		
	Relinquished by:						
	Received by:		Date:	_ Container #:			
			December 19 anning 19			°C	
<u>ن</u>		Void (Circle		rected Temperature:	1		
ab overy		Void (Circle	e one) Uncor	rected Temperature: rected Temperature:		°C	
Lab Recovery	Status: Valid	Void (Circle	e one) Uncor			(A)	
Lab Recovery	Status: Valid	Void (Circle	e one) Uncor	rected Temperature:		°C	

Figure 9-9 Example SVOC Sample COC

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 54 of 155

11 Keystone	Park Drive, Suite 700, Mornsville, NC 27560	ERG Lab ID#
	AMBIENT HEXAVALENT CHROMIUI	M CHAIN OF CUSTODY FORM
9	Site Code:	Collection Date:
Lab Sampling	City/State:	Primary Event (Y/N):
	1	Collocated Event (Y/N):
P		Batch I.D. No.:
-	Relinquished by:	Date:
	Received by: Date:	
Field Setup	Site Operator:	
	Set-Up Date:	
	Collection Date:	
	Initial Rotameter Setting (C.O. B.):	(After 5 minutes warm-up)
	Programmed Start Time:	Programmed End Time:
	Recovery Date:	Recovery Time:
>	Site Operator:	
Field	Final Rotameter Reading (C.O.B.):	
E 5	Elapsed Time:	
	Relinquished by:	
11200		Container #:
)	Status: Valid Void (Circle one)	The Particle Control of the Particle State Control of the Control
Recovery	If void, why:	
Ω	Collection Time (Minutes):	IR Gun. 1 2 3
2	Avg. Flowrate (L/min):	(Circle one)
	Total Volume of Air Sampled (m³):	
		Samples stored in Freezer # 11
comment	S:	

Figure 9-10 Example Ambient Hexavalent Chromium COC

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 55 of 155

01 Keystone	Park Drive, Suite 700, Moi			ERG Lab		TODY	
Lab Pre-Samp.	Site Code:  City/State:  AQS Code:  Relinquished	by:		Du Date:	llection Date: plicate Event	(Y/N):	
Field Setup	Received by:			Date:			
Field Recovery	Recovery Date: Status: Val Relinquished	id Vo	oid (Circ	Sa	mple Duratio		
Lab Recovery	Received by:Status: Val	id Vo	oid (Circ	Date:		d in ICP-MS Lab	
METALS	Sample Date	Start Time	End Time	Total Time	System #	Total Vol (m³)	Lab ID
PM <sub>10</sub> / TSP METALS	Sample Date	Start Time	End Time	Total Time  Avg Flow (L/min)	System #	Total Vol (m³)	Lab ID
Comment	s:						7-20

Figure 9-11 Example Metals COC

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 56 of 155

			- 1		Relinquished by:		
AQS Code	AQS Code				Date/Time:		
Submitter:	<u> </u>				Received by:		
	Filter ID	Poto	TSP (ICPMS)	PM10 (ICPMS)	Date/Time: Sample Volume (m³)	Comments	ERG LIMS ID
1	Filler ID	Date		_	(111)	Comments	(Lab use only)
2							
3							
4							
5							
6							
7							
8							
9			_				
10			_				
11			+				
12			_	_	+		
13			+		<del>                                     </del>		-
14			+				
15			+				
17							1
18							
19							
20							
21							
22							
23							
24							
25							

Figure 9-12 Metals Blank COC Record

Project No. 0475.00
Element No. Section 9 - B3
Revision No. 2
Date August 2023
Page 57 of 155

#### 9.5 Analytical Data

After analysis, the laboratory will provide narratives describing any anomalies and modifications to analytical procedures, data and sample handling records, and laboratory notes for inclusion in the final report. All laboratory electronic records will be stored for archive on the shared network drive. The shared network has limited access. Raw data will be stored on the shared network for at least 5 years after the end of the closed contract.

All records generated include physical or electronic signatures of the person performing the work and are reviewed by an appropriate Task Leader.

# 9.6 Sampling Monitoring Data

All COC forms from the monitoring sites will be stored with the analytical results. The forms are also scanned and stored in the LIMS as described in the SOP for Sample Login to the Laboratory Information Management System, SOP ERG-MOR-079. The COC forms will be reviewed by the sample custodian(s), Task Leaders, and Program Manager. The laboratory will contact the individual site if necessary information is not completed on the COC forms. The original field data will remain in ERG custody and will eventually be stored on file with the final report until 5 years after the end of the closed contract.

Project No. 0475.00
Element No. Section 10 - B4
Revision No. 2
Date August 2023
Page 58 of 155

#### **SECTION 10 ANALYTICAL METHODS REQUIREMENTS**

Analytical procedures are program-specific because the instrumentation and the target compounds of the programs differ. The primary analytical instrument is GC/FID/MS or GC/MS for VOCs; GC/FID/MS or GC/FID for SNMOC, and PAMS hydrocarbons; High Performance Liquid Chromatography (HPLC) for carbonyls (24-hour samples and PAMS time integrated samples); GC/MS for Semivolatiles (SVOC); Inductively Coupled Plasma/Mass Spectrometer (ICP-MS) for Metals and Pb-TSP; X-ray Fluorescence (XRF) for Pb-PM<sub>10</sub>; and Ion Chromatography (IC) for Hexavalent Chromium. Corrective action for analytical system failures realized at time of analyses is initiated by the Analyst and supported by the Task Leader for that method. All analytical method SOPs are provided in Appendix D. The methods used for NMOC and other individual HAPs analysis not currently discussed will be added to this QAPP when the individual States request the analyses. Samples will not be analyzed until ERG receives approval from EPA.

The analytical SOPs for each method are reviewed annually and updated as necessary. The QA Coordinator, Program Manager and Writer/Editor will review, sign and date SOPs before distributing to the laboratories satellite file areas. The previous copies will be replaced with the revised edition. The original, and all previously revised edits, are stored in a historical file maintained by ERG's Project Administrator.

# **10.1** Canister Cleanup System

Canisters are cleaned using the Wasson TO-Clean Model TO 0108 and the Entech 3100D Canister heated systems. A bulk liquid Nitrogen ( $N_2$ ) dewar is located external to the ERG laboratory facility. This dewar continuously produces a volume of ultrapure gaseous  $N_2$  from its headspace area (~100 psig) that is more than adequate to accommodate all in-lab gaseous  $N_2$  applications. Ultrapure gaseous  $N_2$  is extracted from the dewar headspace and delivered to the cleaning systems. Transport of the gas is accomplished through a 3/8" outer diameter (OD) precleaned stainless-steel tubing.

The heated canister cleaning systems are commercially available systems manufactured by Wasson-ECE (Figure 10-1) and Entech (Figure 10-2). These systems can clean up to twelve

Project No. 0475.00
Element No. Section 10 - B4
Revision No. 2
Date August 2023
Page 59 of 155

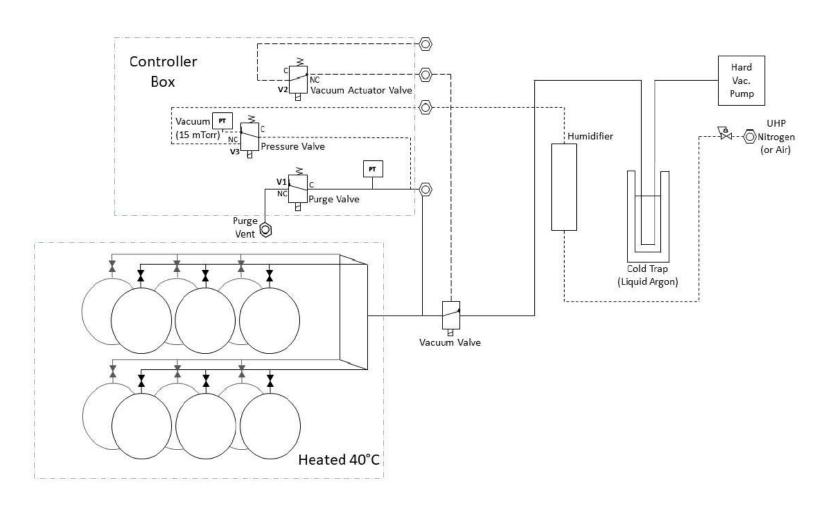


Figure 10-1 Heated Canister Cleanup System Schematic

Project No. 0475.00
Element No. Section 10 - B4
Revision No. 2
Date August 2023
Page 60 of 155

# Entech 3100D Canister Cleaning System Flow Path

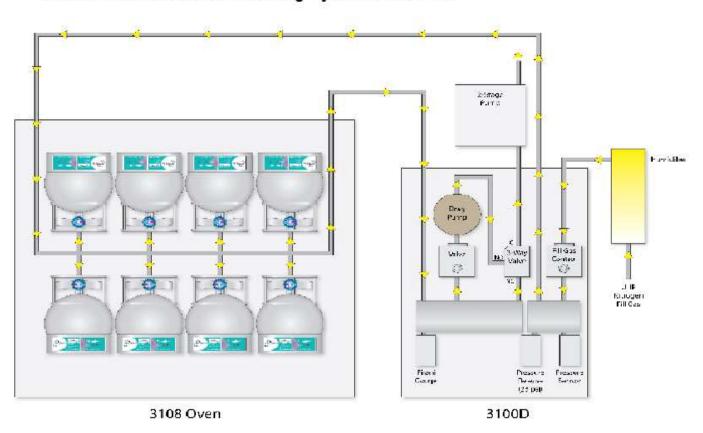


Figure 10-2 Entech Heated Canister Cleanup System Schematic

Project No. 0475.00
Element No. Section 10 - B4
Revision No. 2
Date August 2023
Page 61 of 155

canisters per system at a selected temperature from ambient to  $100^{\circ}$ C. Each system consists of an oven that holds the canisters, a vacuum pump, a humidification chamber for the dilution gas, and a control unit. The cleaning system ovens have enough capacity to clean up to 12 canisters at a time. Two racks hold up to six canisters each. Canisters are connected to a 12-port, two-level manifold with compression fittings and flexible stainless-steel tubing. Ultra-pure  $N_2$  is the dilution gas and is applied to the manifold via an electrically actuated valve. Vacuum is applied to the manifold through a pneumatically actuated vacuum valve. The oven is heated to  $40^{\circ}$ C during the cleaning cycles. The procedure for cleaning canisters is in the SOP for Sample Canister Cleaning using the Wasson-ECE, ERG-MOR-105, and the SOP for Sample Canister Cleaning using the Entech 3100D/3108, ERG-MOR-115 in Appendix D.

# 10.2 VOC and Concurrent Analytical System

The VOC GC/MS analyses are performed on a 250-milliliter (mL) sample from the canister with an Agilent 8890 GC, equipped with an Agilent 5977 MS with Selected Ion Monitoring (SIM) using a 60 m by 0.32-millimeter (mm) Inner Diameter and a 1.8-micrometer ( $\mu$ m) film thickness Restek Rxi-624Sil capillary column.

The VOC GC/FID/MS, for concurrent analyses, are performed on a 250 mL sample from the canister with an Agilent 6890 GC/FID and an Agilent 5975 MS with SIM using a 60 m by 0.32 mm inner diameter and a 1 µm film thickness Restek Rxi-1ms capillary column followed by a Y-union connector that splits the mobile phase between the MS and the FID. Instrument optimization is ongoing, and the most up-to-date operating conditions will be presented in the analytical SOP for the Concurrent GC/FID/MS Analysis of Canister Air Toxic Samples using EPA Compendium Method TO-15 and EPA Ozone Precursor Method (ERG-MOR-005) presented in Appendix D. Table 10-3 shows the operating conditions for the VOC GC/MS analytical system and

Project No. 0475.00
Element No. Section 10 - B4
Revision No. 2
Date August 2023
Page 62 of 155

Table 10-1 VOC GC/FID/MS Operating Conditions

Parameter	GC/MS/FID System (Concurrent VOC/SNMOC analysis)	GC/MS/FID System (Concurrent VOC/ SNMOC analysis)
Sample Volume	250 mL	250 mL
Restek Capillary Column:	Rxi-624Sil	Rxi-1ms
Length:	60 m	60 m
Inside diameter:	0.32 mm	0.25 mm
Film thickness:	1.8 μm	1.0 μm
	-50°C for 4 minutes, 10°C/min to -	-50°C for 5 minutes, 15°C/min to
Oven temperature:	10°C then 4°C/min to 16°C, then	0°C then 5°C/min to 150°C, then
	8°C/min to 180°C, then 25°C/min to	25°C/min to 220°C for 2.5 minutes
	250°C, 250°C for 1 minute, then -	then 25°C/min to 150°C for
	25°C/min to 180°C, 180°C for 6	3.57 minutes
	minutes	
Temperatures:		
Front Inlet Temperature:	220°C	300°C
MS Quad Temperature:	200°C	220°C
MS Source Temperature:	350°C	200°C
FID:	NA	350°C
Gas Flow Rates:		
Column Carrier Gas (Helium (He)):	2 mL/min	2 mL/min
FID Make-up (He):	NA	29 mL/min
FID (Hydrogen (H₂)):	NA	30 mL/min
FID (Air):	NA	300 mL/min
Entech Sample Interface Conditions:		
Module 1 - Blank Trapping	-40°C	-40°C
Temperature:		
Module 2 - Tenax® Trapping	-70°C	-77°C
Temperature:		
Module 3 - Cryofocuser Temperature:	-190°C	-180°C

Figure 10-3 shows the GC/MS systems and Figure 10-4 shows the GC/MS/FID arrangement.

Canister samples must be analyzed within 30 days from sample collection.

Project No. 0475.00
Element No. Section 10 - B4
Revision No. 2
Date August 2023
Page 63 of 155

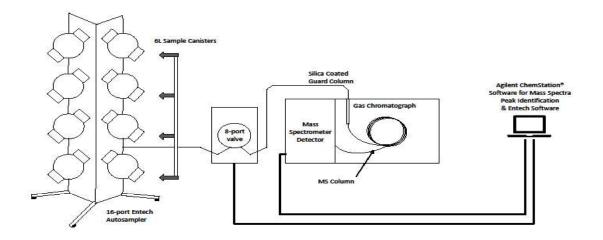


Figure 10-3 VOC GC/MS Systems

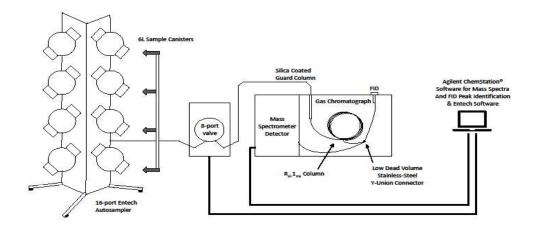


Figure 10-4 VOC GC/MS/FID System

Project No. 0475.00
Element No. Section 10 - B4
Revision No. 2
Date August 2023
Page 64 of 155

#### 10.3 Carbonyl Analytical System

Carbonyl samples are stored in the refrigerator after they are received from the field prior to analysis. The carbonyl cartridge samples are extracted within 14 days of the sampling day and analyzed within 30 days after extraction. Samples are prepared using an extraction manifold by which 5 mL of acetonitrile is vacuum-filtered through a cartridge into a 5 mL Cass A volumetric flask and filled to volume. The extract is transferred to a 2 mL autosampler vial fitted with a Teflon-lined, self-sealing septum and a 4 mL vial with a Teflon-lined cap. Both vials are stored in a dedicated refrigerator at 4°C or below until analysis.

The analytical separation of carbonyls is performed using a Waters HPLC configured with a reverse-phase 250 mm by 4.6 mm C-18 silica analytical column with a 5-micron particle size. A typical HPLC system is shown in Figure 10-4 ERG's system uses an Agilent HPLC chromatographic data software system. Typically, 9-microliters (µL) of sample extract are injected with an automatic sample injector. A mobile phase gradient of HPLC water, acetonitrile, and methanol is used to perform the analytical separation at a flow rate of 1.0 mL/minute. The multiwavelength Ultraviolet (UV) detector operates at 360 nanometer (nm) and 300 nm concurrently. The complete SOP for Preparing, Extracting, and Analyzing DNPH Carbonyl Cartridges by Method TO-11A (ERG-MOR-024) is presented in Appendix D. Sample and waste disposal procedures are outlined in ERG-MOR-033, the SOP for Hazardous Waste.

# 10.4 Polycyclic Aromatic Hydrocarbons Analytical Systems

After PAH sampling, sampling modules containing PUF/XAD-2®, petri dishes containing glass microfiber filters, tweezers and completed COC forms will be shipped to the ERG laboratory from the field. Before shipment, each filter should be folded in quarters, placed inside the cartridge (with the PUF/XAD), and capped by field operator. Upon receipt at the laboratory, samples will be logged into the LIMS system and stored in the refrigerator. Sample preparation and analysis procedures are based on EPA Compendium Method TO-13A<sup>(10)</sup> and the ASTM D6209 (12) method. Hold times are 14 days after sampling for extraction and 40 days after extraction for analysis.

Project No. 0475.00
Element No. Section 10 - B4
Revision No. 2
Date August 2023
Page 65 of 155

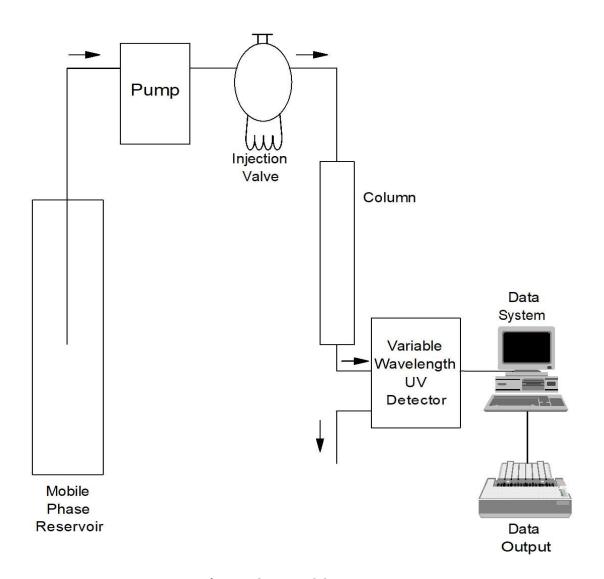


Figure 10-5 HPLC System

Sample extracts will be analyzed for PAHs using GC/MS in SIM mode. The MS will be tuned and mass-calibrated as required using perfluorotributylamine (FC-43), per the analytical procedures presented in the SOP for analysis of Semivolatile Organic Compounds (Polynuclear Aromatic Hydrocarbons) Using EPA Compendium Method TO-13A and ASTM D6209 (ERG-MOR-049) (see Appendix D). Sample and waste disposal procedures are outlined in ERG-MOR-033, the SOP for Hazardous Waste.

Project No. 0475.00
Element No. Section 10 - B4
Revision No. 2
Date August 2023
Page 66 of 155

# 10.5 Metals and Lead analysis Using an Inductively Coupled Argon Plasma Mass Spectrometry Analytical System

Upon receipt from the field, the samples are checked against the COC forms and then logged into the LIMS system. Each sample component is examined to determine if damage occurred during travel. Abnormal color, appearance, and other sample particulars are noted when necessary. Sample preparation and analysis procedures are based on EPA Compendium Methods IO-3.1<sup>(23)</sup> and IO-3.5<sup>(6)</sup>, respectively for the Determination of Metals in Ambient Particulate Matter using ICP-MS techniques. Quartz and glass fiber sample filters are carefully folded end over end for digestion. A complete description of the preparation procedures is presented in the SOPs for quartz and glass fiber (8x10") filter prep (ERG-MOR-084) and for Teflon 47mm filter prep (ERG-MOR-085) and all sample types are analyzed by ICP-MS (ERG-MOR-095). These procedures were approved as NAAQS Federal Equivalency Methods (FEM) for the analysis of Lead for Total Suspended Particulate (TSP) on quartz and glass fiber filters (EQL-0512-201<sup>(7)</sup>) and for PM10 on Teflon filters (EQL-0512-202<sup>(8)</sup>). Analysis hold time for all metals filters is 180 days.

The ICP-MS consists of an inductively coupled plasma source, ion optics, a quadrupole MS, a recirculating chiller, autosampler, and a desktop computer for operational control. The MS is mass-calibrated and resolution checked before each analysis. Resolution across the mass range is indicated by the following isotopes 7Li; 24, 25, and 26Mg; 59Co; 115In; 206, 207, and 208Pb; and 238U. Instrument stability must be demonstrated by running 10 replicates of a tuning (daily performance check) solution [1 micrograms per liter ( $\mu$ g/L) of barium, bismuth, cerium, cobalt, indium, lead, lithium and uranium, and 15  $\mu$ g/L of magnesium] with a resulting Relative Standard Deviation (RSD) of absolute signals for all analytes less than 2 or 5 percent, depending on element and instrument acquisition mode. Sample and waste disposal procedures are outlined in ERG-MOR-033, the SOP for Hazardous Waste.

Project No. 0475.00
Element No. Section 10 - B4
Revision No. 2
Date August 2023
Page 67 of 155

#### 10.6 Hexavalent Chromium Analytical System

Hexavalent chromium filter samples are stored in the freezer after they are received from the field prior to analysis. Internal studies have shown that the hexavalent chromium does not degrade for up to 21 days if the samples are stored in the freezer before extraction. Upon receipt from the field, the samples are checked against the COC forms and then logged into LIMS. Due to oxidation/reduction and conversion between the trivalent and hexavalent chromium, the extraction is performed immediately prior to analysis. Therefore, it is important that the IC be equilibrated, calibrated and ready for analysis before filters are extracted. Sample preparation is performed by removing the filter from the filter holder and placing it into a 14 mL polystyrene tube. The filters are extracted in 10 mL of a 20 millimolar (mM) sodium bicarbonate solution. The tubes are shaken for 45 minutes using a wrist action shaker before a 2.5 mL aliquot is removed for analysis on the IC. All analysis is completed within 24 hours of the filter extraction.

The analytical separation for the hexavalent chromium is performed using a Dionex-600 IC or Dionex ICS-5000 with a Dionex LC 20 Chromatography Enclosure with a post-column reagent delivery device and an advanced gradient pump configured with an IonPac AS7 analytical column and an IonPac NG1 guard column. Both of ERG's ICs use the Dionex Chromeleon® data system. For the Dionex-600 IC, samples are injected using a Dionex AS40 autosampler. The samples analyzed with the Dionex ICS-5000 are injected using an AS-DV autosampler. A mobile phase is used to perform the analytical separation at a flow rate of 1.0 mL/min, and a post-column reagent flow rate of 0.3 mL/min. The multiwavelength UV detector is set at 530 nm. The samples are prepped and analyzed following ASTM D7614<sup>(9)</sup> method and the SOP for the Preparation and Analysis of Ambient Air for Hexavalent Chromium by Ion Chromatography (ERG-MOR-063) that is presented in Appendix D. Sample and waste disposal procedures are outlined in ERG-MOR-033, the SOP for Hazardous Waste.

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 68 of 155

#### SECTION 11 QUALITY CONTROL REQUIREMENTS

This section describes the quality control requirements for each of the major program components (NMOC, SNMOC, VOC, Carbonyls, PAMS, HAPs – SVOC, Metals, Lead and Hexavalent Chromium). The 2023 MDLs are also presented in this section.

As there is not a current need for some of the HAPS (SVOC analysis following TO-13A<sup>(10)</sup>/SW 846 Method 8270E<sup>(11)</sup>, PCB/Pesticides<sup>(13)</sup>, inorganic acids<sup>(14, 15, 16)</sup>, etc.), this information is not provided in this QAPP. As soon as these analyses are requested by EPA or States, however, the QAPP will be modified to include the applicable quality control requirements, and a current set of MDLs will be completed and presented to EPA.

## 11.1 Sample Canister Integrity Studies

Before any VOC samples are collected for a program, all stainless-steel sample canisters are certified via leak tightness and bias checks. To test for leaks, the canisters are evacuated to less than 25" Hg. The canister vacuum, measured on a Heise gauge, and the barometric pressure are recorded. After 7 days, the canister vacuum and barometric pressure is remeasured. The canisters are considered leak-free if there is no more than 1" Hg difference in vacuum (adjusting for differences in the barometric pressure). Canisters that pass the leak check are cleaned using the procedure described in Section 10 and set aside for a specified number of days before analysis for positive bias check. The canister is then injected with low level standard and analyzed for negative bias check. The procedure for canister certification is described in more detail in SOP ERG-MOR-110.

#### 11.2 Standard Traceability

The standards used for all analytes are vendor-supplied National Institute of Standards and Technology (NIST) standards or vendor-supplied standards referenced to a NIST standard. All analytical methods are also certified by comparison to a second source NIST-traceable standard. The ERG-MOR-022 SOP for the Preparation of Standards in the ERG Laboratory, provides direction for preparing standards from solid or liquid chemicals. The SOP used to

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 69 of 155

prepare canister standards is SOP for Standard Preparation Using Dynamic Flow Dilution System, ERG-MOR-061 (Appendix D).

## 11.3 Accuracy and Acceptance

As ambient air measurements encompass a range of compounds and elements whose individual concentrations are unknown, defining absolute accuracy is not possible. Each instrument calibration is discussed by method in Section 13 of this QAPP. For the duration of an instrument's calibration, accuracy is monitored through continuous analysis of QC samples. All analytical methods require the analysis of field and analytical blank QC samples to characterize method bias, while some analytical methods also require internal standards to adjust for instrument drift. Given the diverse nature of the instrumentation used for each major program component, each system requires specific QC to verify that analytical instruments are operating within acceptable limits. The specific QC acceptance criteria are described in the following tables. More detailed discussion, including definitions and concentration levels, for each of the QC sample types can be found in the applicable ERG SOP.

Program Component	QA/QC Table Number
SNMOC	Table 11-1
TO-15/TO-15A	Table 11-2 and 11-3
Carbonyls/TO-11A	Table 11-4
SVOC/TO-13A	Table 11-5, 11-6, and 11-7
Metals/IO-3.5	Table 11-8 and 11-9
Hexavalent Chromium	Table 11-10
NAAQS Pb	Table 11-9 and 11-11
XRF	Table 11-12

Note: The qualifiers in these tables are LIMS qualifiers, the AQS qualifiers are directed by the NATTS TAD<sup>(22)</sup>.

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 70 of 155

**Table 11-1 Summary of SNMOC Quality Control Procedures** 

QC Check	Frequency	Acceptance Criteria	Corrective Action
Multiple point calibration (5 points	Upon CCV failure, not to exceed 1	Average Response Factor (RF) curve fit	1) Repeat individual Initial
minimum); propane and benzene,	year.	with RF RSD within ±20%	Calibration (ICAL) standard analysis
bracketing the expected sample		ICV Recovery for selected hydrocarbons	2) Repeat ICAL
concentration. Laboratory Control		70-130%	3) Reprepare ICAL standards and
Standard (LCS) (or Initial Calibration			analyze
Verification (ICV))			
Continuing calibration verification	Daily, prior to sample analysis	Recovery for 10 selected hydrocarbons	1) Repeat analysis
(CCV)		spanning the carbon range 70-130 %	2) Reprepare CCV and analyze
			3) Repeat calibration curve
Method Blank Analysis	Daily, following calibration check	< 20 ppbC	1) Repeat analysis
			2) Reprepare Method Blank (MB)
			and analyze
			3) Check system for
			leaks/contamination

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 71 of 155

**Table 11-2 Summary of Air Toxics Canister VOC Quality Control Procedures** 

QC Check	Frequency	Acceptance Criteria	Corrective Action
BFB Instrument Tune Performance Check	Daily, prior to sample analysis	Evaluation criteria presented in Section 16.1.1 of the SOP and Table 11-3 of this QAPP.	1) Retune 2) Clean ion source and/or quadrupole
ICAL consisting of at least 5 points bracketing the expected sample concentration.	Following any major change, repair, or maintenance or if daily QC is not acceptable.  Recalibration not to exceed three months.	1) % RSD of Response Factors < 30.1% RSD (with two exceptions of up to ± 40% for non-NATTS compounds only) 2) Internal Standard (IS) response < ±40.1% of mean curve IS response 3) Each calibration standard concentration must be < ±30.1% of nominal	1) Repeat individual ICAL standard analysis within 24 hours of original 2) Flag non-Tier 1 failing compounds with LJ and void Tier 1 failing compounds in curve and sample data. 3) Repeat ICAL if Tier 1 fails 4) Reprepare ICAL standards and analyze
LCS ({ICV} Second source calibration verification check)	Following the calibration curve	RRF < ±30.1% Deviation from calibration curve average RRF	1) Repeat ICV 2) Reprepare ICV and analyze 3) Repeat calibration curve 4) Flag ICAL, ICV and sample data for failing non-Tier 1 compounds with LK or LL and void for failing Tier 1 compounds
CCV of approximately mid- point of the calibration curve using a Certified Standard	At beginning and end of sequence on the days of sample analysis (on days ICAL is run, do not need a beginning CCV)	RRF < ±30.1% Deviation from the calibration curve average RRF	1) Repeat CCV once 2) Flag non-Tier 1 failing compounds with LK or LL and void Tier 1 failing compounds in CCV and sample data 3) Rerun sequence if Tier 1 compounds fail

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 72 of 155

**Table 11-2 Summary of Air Toxics Canister VOC Quality Control Procedures** 

QC Check	Frequency	Acceptance Criteria	Corrective Action
Method Blank Analysis	Daily, following BFB and	1) ≤ MDL MQO (NATTS workplan) and ≤3x MDL or 0.03	1) Repeat analysis if Tier 1
(Zero Air Sample Check)	calibration check; prior to sample analysis	ppbV, whichever is lower 2) IS area response <± 40.1% of avg ICAL IS	compounds fail 2) Flag non-Tier 1 compounds in MB and sample data with QB-01 and void Tier 1 failing compounds in MB and sample data 3) Check system for leaks, contamination and reanalyze blank 4) Rerun sequence if Tier 1 compounds fail
Duplicate Samples	Duplicate and Collocate field samples	<25.1% RPD for compounds with at least one result ≥ 5 x MDL (for precision check: 5 x MDL substitution for the paired result < 5 x MDL)	1) Repeat sample analysis 2) Flag data with D-F in Duplicate/Collocate pair
Replicate Analysis	1 per sequence and Duplicate/Collocate sample	<25.1% RPD for compounds with at least one result in pair ≥ 5 x MDL (for precision check: 5 x MDL substitution for the paired result < 5 x MDL)	1) Repeat sample analysis 2) Flag data with R-F in Duplicate/Collocate and associated Replicate and sequence for failed compound
Retention Time (RT) in samples	All qualitatively identified compounds	Target and IS RT within 2 seconds of most recent initial calibration average RT	Compound not identified unless based on analyst experience
Internal Standards in samples	All samples	IS area response < $\pm$ 40.1% and IS RT within $\pm$ 0.33 min. (20 seconds) of most recent calibration average IS response	Repeat analysis     Flag data associated with failed IS with LJ

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 73 of 155

**Table 11-2 Summary of Air Toxics Canister VOC Quality Control Procedures** 

QC Check	Frequency	Acceptance Criteria	Corrective Action
Preconcentrator Leak Check	Each standard and sample canister connected to the preconcentrator/ autosampler	≤ 0.2 psi change/minute	1) Retighten and re-perform leak check     2) Perform maintenance and Re-perform leak check test
Dilution Blank (DB)	One canister analyzed per air cylinder installed on dilution system	≤ MDL MQO (NATTS workplan) and ≤3x MDL or 0.03 ppbV, whichever is lower	Examine effect on sample dilution or calibration curve and perform additional scrubbing or cleanup on dilution gas to eliminate such background
Canister Cleaning Batch Blank	One canister analyzed on the Air Toxics system per batch of 8 (2 per batch of 9 to 12)	≤ MDL MQO (NATTS workplan) and ≤3x MDL or 0.03 ppbV, whichever is lower	1) Reclean batch of canisters or choose a 2 <sup>nd</sup> batch blank (if passes, then only 1 <sup>st</sup> batch blank canister must be recleaned) 2) Flag sample data associated with batch blank for failing compounds with CC and LK. Note: consider the target compound for specialized projects

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 74 of 155

**Table 11-2 Summary of Air Toxics Canister VOC Quality Control Procedures** 

QC Check	Frequency	Acceptance Criteria	Corrective Action
Canister Cleaning Certification	Every canister, at least	Leak check: ≤ 0.1 inches Hg over 7 days	Leak check failure:
	every 3 years		Repair canister
		Cleanliness: ≤ MDL MQO (NATTS workplan) and ≤3x MDL or 0.03 ppbV, whichever is lower	Cleanliness: Flag sample data collected in the canister for failing compounds with CF and LK; if Tier 1 compounds > 5 x MDL invalidate
		Known Standard Check: $< \pm 30.1\%$ of the theoretical concentration	Known STD Check: Flag sample data collected in the canister for failing compounds with CF and LK or LL
Sampler Certification - Standard Challenge with a reference can and a Zero Check with a reference can	Annual	Challenge: < ±15.1% of the concentration in the reference canister (< ±25.1% for acrolein and carbon tetrachloride)	Challenge: 1) Repeat (a requirement for Tier I compounds) 2) Flag failing compound data
		Zero: <3x MDL or 0.03 ppbV (whichever is lower) higher than the reference canister for all Tier I compounds. Non-Tier I compounds up to 0.1 ppbV or	in samples collected with sampler with SB and LK or LL
		3x MDL (whichever is higher, with the upper limit of 0.2 ppbV) higher than the reference canister.  Acetonitrile < 0.2 ppbV higher than reference canister.	Zero: 1) Repeat (a requirement for Tier I compounds) 2) Flag failing compound data in samples collected with sampler with SB and LK; if Tier 1 compounds > 5 x MDL
			invalidate

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 75 of 155

Table 11-2 Summary of Air Toxics Canister VOC Quality Control Procedures

QC Check	Frequency	Acceptance Criteria	Corrective Action
Hold Time	All samples	Analyze within 30 days from collection	Flag with O-04
Sampling Period	All samples	24 hours ± 1 hour	1) Notify Program Manager and
			Invalidate and re-sample for sample < 23 and > 25hours

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 76 of 155

Table 11-3 BFB Key Ion Abundance Criteria

Target Mass	Rel. To Mass	Lower Limit %	Upper Limit %
50	95	8	40
75	95	30	66
95	95	100	100
96	95	5	9
173	174	0	2
174*	95	50	120
175	174	4	9
176	174	93	101
177	176	5	9

<sup>\*</sup> alternate base peak

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 77 of 155

**Table 11-4 Summary of Carbonyl Quality Control Procedures** 

Parameter	QC Check	Frequency	Acceptance Criteria	Corrective Action
HPLC Efficiency	Analyze Second Source QC (SSQC) sample	Once per 12 hours or less	<ol> <li>Resolution between acetone and propionaldehyde ≥ 1.0</li> <li>Column efficiency &gt; 5,000 plate counts</li> </ol>	1) Eliminate dead volume 2) Back flush 3) Replace the column repeat analysis
DNPH Peak	All samples	Every chromatogram from an extracted cartridge (field sample, method blank, lot blank, and BS/BSD)	DNPH must be $\geq$ 50% of the DNPH in the MB. Samples with DNPH < 1,000 area counts are invalid.	1) Sample concentration will be flagged with DNPH in LIMS and DN in AQS
Sampler Certification	Zero Challenge cartridge with a reference cartridge	Annual	Each compound must be ≤ 0.2 ppbV above the reference cartridge	1) Repeat certification of samplers, a requirement for Tier I compounds 2) Flag failing compound data in samples collected with sampler with SB and LK; if > 5 x MDL invalidate
ICAL	Run a 5-point calibration curve	At setup or when CCV is out of acceptance criteria (at least every 6 months)	1) Correlation coefficient at least 0.999, relative error for each level against calibration curve < 20.1% 2) The absolute value of the intercept/slope of the calibration curve must be less than the MDL absent blank background (MDLsp) for each compound	1) Check integration 2) Reanalyze 3) Reprepare standards and recalibrate 4) Flag non-Tier 1 failing compounds with LJ and void Tier 1 failing compounds in ICAL and sample data
ICV	Analyze SSQC sample	After calibration in triplicate	Within ± 15.1% of nominal	1) Check integration 2) Recalibrate 3) Reprepare standard 4) Flag non-Tier 1 failing compounds with LJ and void Tier 1 failing compounds in ICV, ICAL and sample data

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 78 of 155

**Table 11-4 Summary of Carbonyl Quality Control Procedures** 

Parameter	QC Check	Frequency	Acceptance Criteria	<b>Corrective Action</b>
Retention Time	Analyze SSQC	Once per 12 hours or less	Each target compound within ± 2.0% of the mean calibration standards RT (set in Agilent® software)	<ol> <li>Check integration,</li> <li>Check for plug in LC</li> <li>Check column temperature in LC</li> <li>Recalibrate if RT is stable</li> </ol>
ccv	Analyze SSQC sample	Once per 12 hours or less	Within ± 15.1% of nominal	1) Check integration 2) Reanalyze, reprepare standard, or recalibrate 3) Reanalyze samples not bracketed by acceptable standards 4) Flag non-Tier 1 failing compounds with LJ and void Tier 1 failing compounds in CCV and sample data
Solvent Blank (aka Continuing calibration blank (CCB), System Blank, or Laboratory Reagent Blank (LRB))	Analyze acetonitrile	Prior to ICAL and Bracket sample batch, 1 at beginning and 1 at end of batch	Measured concentration must be < MDLsp for each compound	1) Locate and eliminate contamination 2) Flag non-Tier 1 failing compounds with QB-04 and void Tier 1 failing compounds in sequence sample data
Lot Blank Check	Analyze blank for new lots received	Analyze 1.0 % of total lot or a minimum of 3 cartridges, whichever is greater	Compounds must be less than values listed: Formaldehyde <0.15 μg/cartridge (0.03 μg/mL) Acetaldehyde <0.10 μg/cartridge (0.02 μg/mL) Acetone <0.30 μg/cartridge (0.06 μg/mL) Others <0.10 μg/cartridge (0.02 μg/mL)	1) Reanalyze an additional set of cartridges from the new lot 2) If a manufacturer defect is suspected, notify the vendor if lot blank continues to fail and acquire new lot if possible 3) Invalidate data associated with bad lot Note: With client permission, may flag data associated with bad lot with QB-03

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 79 of 155

**Table 11-4 Summary of Carbonyl Quality Control Procedures** 

Parameter	QC Check	Frequency	Acceptance Criteria	Corrective Action
Extraction Solvent Method Blank (ESMB)	Aliquot of extraction solvent prepared with samples during extraction		All target compounds must be < MDLsp	1) Check integration 2) Reanalyze 3) Locate and resolve contamination in extraction glassware/solvent 4) Flag batch data with A-01 5) Re-perform ESMB with the next extraction to verify resolution.
Field Blank (FB)	Field blank samples collected in the field	Monthly (if provided by site)	Underivatized compound concentrations must be less than values listed: Formaldehyde   <0.3 µg/cartridge (0.06 µg/mL) Acetaldehyde   <0.4 µg/cartridge (0.08 µg/mL) Acetone   <0.75 µg/cartridge (0.15 µg/mL) Others   <7.0 µg/cartridge (1.4 µg/mL)	1) If FB fails, notify site coordinator, schedule another FB. Additional FBs are collected until the problem is corrected and data are acceptable. 2) Flag FB and associated sample FB-01; samples from the last acceptable FB to the next passing FB will be flagged when input in AQS
Trip Blank (TB)	Trip blank if collected	if provided	Compounds must be less than values listed: Formaldehyde <0.15 µg/cartridge (0.03 µg/mL) Acetaldehyde <0.10 µg/cartridge (0.02 µg/mL) Acetone <0.30 µg/cartridge (0.06 µg/mL) Others <0.10 µg/cartridge (0.02 µg/mL)	1) If TB fails, notify site coordinator, schedule another TB 2) Flag TB and associated sample(s) with B6

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 80 of 155

**Table 11-4 Summary of Carbonyl Quality Control Procedures** 

Parameter	QC Check	Frequency	Acceptance Criteria	Corrective Action
Duplicate or Collocate Samples	Analysis of Duplicate and Collocated samples	As collected (10% of sampling schedule)	< 20.1% RPD for concentrations ≥ 0.5 µg/cartridge	<ol> <li>Check integration</li> <li>Check instrument function</li> <li>Reanalyze Duplicate samples</li> <li>Flag Duplicate/Collocate data with D-F</li> </ol>
Replicate Analyses	Replicate injections	One per batch of 20 samples at a minimum. Performed on every Duplicate and Collocate sample or if not available on a field sample	< 10.1% RPD for concentrations ≥ 0.5 µg/cartridge	1) Check integration 2) Reanalyze Duplicate/Collocate and Replicate 3) Flag Primary, Replicate and sequence data R-F
MB (BLK)	Analyze MB	One per batch of 20 samples	Underivatized compound concentrations must be less than values listed: Formaldehyde   <0.15 µg/cartridge (0.03 µg/mL) Acetaldehyde   <0.10 µg/cartridge (0.02 µg/mL) Acetone   <0.30 µg/cartridge (0.06 µg/mL) Others   <0.10 µg/cartridge (0.02 µg/mL)	1) Reanalyze MB 2) Check extraction procedures 3) Flag MB and extraction batch data with QB-01
Blank Spike/Blank Spike Duplicate, (BS/BSD or LCS/LCSD)	Analyze BS/BSD (or LCS/LCSD)	One BS/BSD (LCS/LCSD) per batch of 20 samples	79.9-120.1% recovery for Formaldehyde and 69.9-130.1% for all other compounds. BSD (LCSD) precision <20.1% RPD of BS (LCS)	<ol> <li>If a chromatographic issue is suspected, reanalyze BS/BSD</li> <li>Check calibration if CCVs also fail.</li> <li>Check extraction procedures</li> <li>Flag BS or BSD and extraction batch data if either the BS and BSD fail with GC-BS</li> </ol>

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 81 of 155

**Table 11-4 Summary of Carbonyl Quality Control Procedures** 

Parameter	QC Check	Frequency	Acceptance Criteria	Corrective Action
Hold Time	All samples	All samples	Extract within 14 days of collection and analyze extracts within 30 days of extraction	Flag missed extract hold time with O-05; Flag missed analysis hold time with O-04
Receipt Temperature	All samples	All samples	≤ 4C	Flag affected sample data with TT
Sampling Period	All samples	All samples	24 hours ± 1 hours	Notify Program Manager     Invalidate samples < 23 hours     and > 25 hours and re-sample

Note: Crotonaldehyde tautomerizes into two chromatographically separate peaks after it is spiked onto the DNPH cartridge. The best analytical recovery is determined when both peaks are integrated together for all samples and QC. Breakthrough cartridges are not submitted or analyzed as specified by Compendium Method TO-11A.

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 82 of 155

Table 11-5 Summary of Quality Control Procedures for Analysis of SVOC Samples for PAHs

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
DFTPP instrument tune	Daily prior to calibration check and	Evaluation criteria presented in	1) Re-analyze tune check standard
check	sample analysis; every 12 hours during	Section 11, Table 11-5	2) Prepare new tune check standard;
	instrument operation		analyze
			3) Re-tune instrument; reanalyze
			4) Clean ion source; re-tune instrument;
			reanalyze
Initial Calibration (five-	Following any major change, repair, or	< 30.1% RSD of the RRFs for each	1) Check integrations and calculations
point minimum) (ICAL)	maintenance and if daily quality control	compound; Avg Relative Response	2) Repeat individual calibration standard
	check is not acceptable. Minimum	Factor (RRF) above or equal to	analyses
	frequency every six weeks	minimum RRF limit for each	3) Prepare new calibration standards and
		pollutant; < 30.1% of nominal	repeat analysis
			4) Perform maintenance on GC, especially
		RRTs within ± 0.06 RRT units of	leak check and repeat analysis
		mean RRT of calibration	5) Clean ion source and repeat analysis
			6) Flag non-Tier I compounds with LJ
		IS RT within ± 20.0 sec of mean RT	(invalidate Tier I compounds) for samples
		of calibration	using ICAL
Secondary Source	Immediately after each ICAL	≤ 30% Difference for each	1) Check integrations and calculations
Calibration Verification		compound RRF compared to the	2) Repeat SCV analysis
(SCV)		mean RRF of the calibration curve.	3) Prepare a new SCV standard and repeat
			analysis
			4) Perform maintenance on GC, especially
			leak check; reanalyze
			5) Recalibrate; reanalyze
			6) Clean ion source; recalibrate
			7) Flag non-Tier 1 failing compounds with LJ
			in SCV, ICAL and sample data (invalidate
			Tier 1 compounds)

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 83 of 155

Table 11-5 Summary of Quality Control Procedures for Analysis of SVOC Samples for PAHs

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration	Daily; every 12 hours during instrument	Above or equal to RRF minimum	1) Check integrations and calculations
Verification (CCV)	operation	and ≤ 30% Difference for each	2) Repeat CCV analysis
Standard		compound RRF compared to the	3) Prepare a new CCV standard and repeat
		mean RRF of the calibration curve.	analysis
			4) Perform maintenance on GC, especially
			leak check; reanalyze
			5) Recalibrate; reanalyze
			6) Clean ion source; recalibrate
			7) Flag non-Tier 1 failing compounds with LJ
			in CCV and samples bracketed by CCV
			(invalidate Tier 1 compounds)
SB (without IS) aka IBL	Prior to ICAL and daily beginning CCV	All target, surrogate, and IS	Flag sequence for failing compounds with
		compounds not qualitatively	LB
		detected	
Solvent Method Blank	One with every extraction batch of 20 or	All target compounds not	1) Check integrations and calculations
(SMB) extraction solvent	fewer field-collected samples	qualitatively detected.	2) Reanalyze
check (with IS) aka Batch			3) Flag extraction batch for failing
ССВ			compounds with QB-04
			4) Consider removing solvent lot from use
Method Blank (MB)	With every extraction batch ≤ 20	All analytes < 2x MDL	1) Repeat analysis
	samples.		2) Flag sample data with QB-01
Blank Spike (BS) or (LCS)	One BS (or LCS) with every extraction	59.9-120.1% recovery of nominal	1) Repeat analysis
BSD (or LCSD)	batch ≤ 20 samples.	for all compounds	2) Flag sample data with GC-BS
	BSD (or LCSD) once per quarter.	< 20.1% RPD compared to BS (or	
		LCS)	
ССВ	Following CCVs as needed for NELAP	All analytes < MDL	1) Repeat analysis
	requirement of blank following each		2) Flag associated analytical sample data
	CCV (can use MB, or Batch CCB instead)		with QB-04

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 84 of 155

Table 11-5 Summary of Quality Control Procedures for Analysis of SVOC Samples for PAHs

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Surrogate compound recoveries: Laboratory surrogates fluorene-d10 pyrene-d10 Field Surrogates fluoranthene-d10 benzo(a)pyrene-d12	Every sample/blank/BS	59.9-120.1% Recovery	1) Check integrations and calculations 2) Repeat analysis 3) Flag surrogate compounds 4) Flag compounds in sample if both field or both lab surrogates fail with S-GC 5) Invalidate sample if three or more surrogates fail
Internal Standard naphthalene-d8 acenaphthylene-d10 phenanthrene-d10 chrysene-d12 perylene-d12	Every sample/blank/BS/Calibration Standard	Response: Within 50% to 200% of the ISs in the most recent initial calibration CAL4  RT: Within ± 20 seconds of avg ICAL RT	1) Repeat analysis 2) Invalidate or flag IS and associated sequence data with LJ
Sample Retention Time (RT)	All qualitatively identified compounds	RT within ± 10 seconds of RT of most recent CCV	Compound not identified, without experienced analyst's opinion.
Cartridge Lot Blank	One cartridge (and filter) for each batch of prepared cartridges for a particular sample date.	Naphthalene ≤ 200 ng/cartridge or ≤ 10% of the 5th percentile concentration for the site from the previous 3 years, whichever is higher All other individual target PAHs ≤ 10 ng/cartridge or ≤ 10% of the 5th percentile concentration for the previous 3 years, whichever is higher	Repeat analysis     Flag sample data if unable to reanalyze prior to cartridge shipment with QB-03

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 85 of 155

Table 11-5 Summary of Quality Control Procedures for Analysis of SVOC Samples for PAHs

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Field Blank	Monthly (or as provided by site)	Target compounds ≤ 3x MDL	1) If FB fails, notify Program Manager 2) Flag FB, associated sample FB-01;
			samples from the last acceptable FB to the
			next passing FB will be flagged when input
			in AQS
			Note: if it appears the filter part of media
			of FB and sample was inadvertently
			swapped void FB and sample
Replicate Analysis	Replicate sample, on each collocate or	< 10.1% RPD for compounds with at	1) Check integrations and calculations
	at a minimum one per sequence	least one result in pair ≥ 0.5 ng/μL	2) Repeat analysis
		(for precision check: 0.5 ng/μL	3) Flag data with R-F in Collocate and
		substitution for the paired result <	associated Replicate and sequence for
		0.5 ng/μL)	failed compound
Collocate Samples	Collocated samples, 10% of field	< 20.1% RPD for compounds with at	1) Check integrations and calculations
	samples, or as collected	least one result ≥ 0.5 ng/μL (for	2) Verify Collocate/Primary sample results
		precision check: 0.5 ng/μL	agree with their Replicate within the
		substitution for the paired result <	Replicate Analysis Criteria
		0.5 ng/μL)	3) Flag Collocate samples with D-F
Hold Time	All samples	Extract within 14 days of collection	Flag missed extract hold time with O-05;
		and analyze extracts within 40 days	Flag missed analysis hold time with O-04
		of extraction	
Receipt temperature	All samples	≤ 4C	Flag with TT
Sampling Period	All samples	24 hours ± 1 hours	1) Notify Program Manager
			2) Invalidate samples < 23 hours and > 25
			hours and re-sample

NOTE: Matrix Spikes are not performed as required by Compendium Method TO-13A. Matrix spikes are not required by ASTM D2609.

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 86 of 155

Table 11-6 DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	10 to 80% of base peak
68	< 2% of mass 69
69	Present
70	< 2% of mass 69
127	10 to 80% of base peak
197	< 2% of mass 198
198	Base peak (100% relative abundance) or >50% of mass 442
199	5 to 9% of mass 198
275	10 to 60% of base peak
365	> 1.0% of mass 198
441	Present but < 24% of mass 442
442	Base peak, or >50% of mass 198
443	15 to 24% of mass 442

Note: All ion abundances must be normalized to the nominal base peak, 198 or 442. This criterion is based on the tune criteria for Method 8270D.

**Table 11-7 Internal Standards and Associated PAHs** 

Internal Standard	Associated Compound	
Naphthalene-d <sub>8</sub>	Naphthalene	
Acenaphthelene-d <sub>10</sub>	Acenaphthylene Acenaphthene	Fluorene
Phenanthrene-d <sub>10</sub>	Anthracene Fluoranthene	Phenanthrene
Chrysene-d <sub>12</sub>	Benz(a)anthracene Chrysene Pyrene	
Perylene-d <sub>12</sub>	Benzo(a)pyrene Benzo(e)pyrene Benzo(k)fluoranthene Dibenz(a,h)anthracene Perylene	Benzo(b)fluoranthene Benzo(g,h,i)perylene Coronene Indeno(1,2,3-cd)pyrene

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 87 of 155

**Table 11-8 Summary of Quality Control Procedures for Metals Analysis** 

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Daily Performance Check (DPR) STD Mode	Before each analysis	See Table 11-9	1) Repeat analysis of DPR 2) Re-optimize instrument tuning parameters 3) Reprepare DPR standard 4) Perform instrument maintenance
Daily Performance Check (DPR) KED Mode	Before each analysis	See Table 11-9	1) Repeat analysis of DPR 2) Re-optimize instrument tuning parameters 3) Reprepare DPR standard 4) Perform instrument maintenance
Initial Calibration Standards (IC)	At least 5 non-zero calibration points and a blank before each analysis	Correlation of Determination of $(R^2) \ge 0.995 \& RSD \le 10\%$ .	1) Repeat analysis of calibration standards 2) Reprepare calibration standards and reanalyze 3) Flag non-Tier 1 failing elements with QX and LJ and void Tier 1 failing elements in ICAL and sample data
Initial Calibration Verification (ICV)	Immediately after calibration	Recovery 89.9-110.1%	1) Repeat analysis of ICV 2) Reprepare ICV standard 3) Recalibrate and reanalyze 4) Flag non-Tier 1 failing elements with QX and LJ and void Tier 1 failing elements in ICV, ICAL and sample data
Initial Calibration Blank (ICB)	Immediately after ICV	Absolute value must be < s*K (the component of the FAC MDL that does not include the mean)	Locate and resolve contamination problems before continuing     Reanalyze or recalibrate failing elements for the entire analysis when appropriate
High standard verification (HSV)	After ICB and before ICS	Recovery from 94.9-105.1%	Repeat analysis of HSV     Reprepare HSV
Interference Check Standard (ICSA/IFA)	Following the HSV	None	None
Interference Check Standard (ICSAB/IFB)	Following the ICSA	Recovery 79.9-120.1% of nominal after IFA correction (if IFA > 3x s*K)	1) Repeat analysis of ICSAB 2) Reprepare ICSAB and analyze 3) Flag failing associated elements with ICS-01 and LK or LL
Continuing Calibration Verification (CCV)	Analyze before samples, after every 10 samples, and at the end of each run	Recovery 89.9-110.1%	1) Reanalyze CCV 2) Reprepare CCV 3) Recalibrate and reanalyze samples since last acceptable CCV 4) Flag non-Tier 1 failing elements with QX and LJ and void Tier 1 failing elements in CCV and sample data

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 88 of 155

**Table 11-8 Summary of Quality Control Procedures for Metals Analysis** 

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Low Calibration Verification (LCV) Lead Only	At the beginning and end of each analysis, between the CCV and CCB	Recovery 70-130% for Pb only	Reanalyze LCV     Reprepare LCV     Recalibrate and reanalyze samples since last acceptable LCV
Continuing Calibration Blanks (CCB)	Analyzed after each CCV	Absolute value must be < s*K	1) Reanalyze CCB 2) Flag associated failing elements for all samples before and after failing CCB QB-04
Laboratory Reagent Blank (LRB/BLK1)	1 per batch of ≤ 20 samples	Absolute value < MDL	Reanalyze for verification     Plag associated failing elements for all samples in extraction batch B
Method Blank (MB/BLK2)	1 per batch of ≤ 20 samples	Absolute value < MDL	Reanalyze for verification     Flag associated failing elements for all samples in extraction batch QB-01
Standard Reference Material (SRM) Lead Only	1 per batch of ≤ 20 samples	Recovery 80-120% for Pb only	1) Reanalyze 2) Flag sample data SRM-01
Reagent Blank Spike (RBS)	1 per batch of ≤ 20 samples	Recovery 79.9-120.1%	1) Reanalyze 2)Flag associated failing elements for all samples in extraction batch QX and LJ
Laboratory Control Sample (LCS/BS/BSD)	1 BS/BSD per batch of ≤ 20 samples	Recovery 79.9-120.1%  BSD < 20.1% RPD compared to BS	1) Reanalyze 2)Flag data for all samples in extraction batch GC-BS 3) Re-prepare sample batch if recovery for most elements fail criteria (quartz only)
Duplicate (DUP1) (Laboratory Duplicate)	1 per batch of ≤ 20 samples	< 20.1% RPD for quartz/glass fiber sample, <10.1% RPD for Teflon samples, and for element pairs with at least one result ≥ 5 x MDL (for precision check: 5 x MDL substitution for the paired result < 5x MDL	1) Check for matrix interference 2) Repeat duplicate analysis if necessary 3) Flag data, "D-F" for quartz/glass, "R-F" for Teflon
Replicate Analysis (Analytical Duplicate)	1 per batch of ≤ 20 samples, ensuring 6 per site per year	< 10.1% RPD for element pairs with at least one result ≥ 5 x MDL (for precision check: 5 x MDL substitution for the paired result < 5x MDL	1) Repeat replicate analysis if analysis issue suspected 2) Flag sequence data for failing element(s), "R-F"

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 89 of 155

Table 11-8 Summary of Quality Control Procedures for Metals Analysis

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Collocated Samples (C1/C2)	10% of samples annually (for sites that conduct collocated sampling)	< 20.1% RPD for element pairs with at least one result ≥ 5 x MDL (for precision check: 5 x MDL substitution for the paired result < 5x MDL	1) Repeat C1 and/or C2 analyses if necessary. 2) Flag C1 and C2 results, if necessary, "D-F"
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	1 MS/MSD per batch of ≤ 20 samples Not applicable to Teflon method	Quartz/Glass Fiber Recovery 79.9-120.1%when the parent sample concentration is less than 4 times the spike concentration; Sb recovery should be 74.9-125.1%	1) Flag failing elements in MS/MSD and parent 2) Reanalyze if analysis issue suspected 3) Diluting the MS/MSD may resolve heavy metal matrix effects 4) Flag fail elements with appropriate QM-## flag
Post Digestion Spike (PDS)	1 per batch of ≤ 20 samples	Recovery 74.9%-125.1%	<ol> <li>Flag failed elements PS-01 for parent sample and PDS</li> <li>Reprepare PS if preparation issue is suspected reason for failure.</li> </ol>
SRD	1 per batch of ≤ 20 samples	<±10.1% RPD of undiluted sample if the element concentration is ≥ 25x MDL	1) Re-prepare dilution if preparation issue is suspected reason for failure.     2) Flag failed analytes in parent sample and SRD, SRD-01
Field Blank	As received	< MDL in ng/m3	1) Flag failed elements in FB and field sample with FB-01; samples from the last acceptable FB to the next passing FB will be flagged when input in AQS
Internal Standards (ISTD)	Every Calibration, QC and Field Sample	Recovery 60-125% of the measured intensity of the calibration blank	1) If drift suspected, stop analysis and determine cause, recalibrate if necessary 2) Reanalyze sample 3) If recovery > 125% due to inherent ISTD, dilute sample and reanalyze 4) Flag failed elements in sequence data QX and LJ
Hold Time	All samples	Digestion and analysis within 180 days from collection	Flag O-05 for missed digestion hold time Flag O-04 for missed analysis hold time
Digestion Temperature	Every digestion batch	Within ±5°C of 78.0 °C	Flag failed elements in batch LJ
Sampling Period	All samples	24 hours ± 1 hours	Notify Program Manager     Nouse of the samples of the sample of th

Note: antimony tends to adhere to quartz media, which causes low recoveries for BS/MS/MSD; associated data is qualified.

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 90 of 155

**Table 11-9 Instrument Mass Calibration & Performance Specifications** 

Parameter	Peak Width	Sensitivity/Criteria*	RSD
iCAP-Q Criteria		, , , , , , , , , , , , , , , , , , , ,	
Standard Mode			
Bkg4.5	NA	< 1.0 cps	N/A
7Li	0.65-0.85	> 50,000 cps	< 2% RSD
24Mg	0.65-0.85	> 500,000 cps	< 2% RSD
25Mg	0.65-0.85	> 70,000 cps	< 2% RSD
26Mg	0.65-0.85	> 80,000 cps	< 2% RSD
59Co	0.65-0.85	> 100,000 cps	< 2% RSD
115ln	0.65-0.85	> 220,000 cps	< 2% RSD
206Pb	0.65-0.85	> 70,000 cps	< 2% RSD
207Pb	0.65-0.85	> 60,000 cps	< 2% RSD
208Pb	0.65-0.85	> 100,000 cps	< 2% RSD
238U	0.65-0.85	> 300,000 cps	< 2% RSD
140Ce16O/140Ce	NA	< 0.02	N/A
137Ba++/137Ba+	NA	< 0.03	N/A
Bkg220.7	NA	< 2.0 cps	N/A
Analyzer Pressure	NA	< 10-6 mbar	NA
KED Mode†			
Bkg4.5	NA	< 0.5 cps	N/A
24Mg	0.65-0.85	> 3,000 cps	< 5% RSD
25Mg	0.65-0.85	> 500 cps	< 5% RSD
26Mg	0.65-0.85	> 600 cps	< 5% RSD
59Co	0.65-0.85	> 30,000 cps	< 2% RSD
115ln	0.65-0.85	> 30,000 cps	< 2% RSD
206Pb	0.65-0.85	> 60,000 cps	< 2% RSD
207Pb	0.65-0.85	> 50,000 cps	< 2% RSD
208Pb	0.65-0.85	> 80,000 cps	< 2% RSD
238U	0.65-0.85	> 80,000 cps	< 2% RSD
140Ce16O/140Ce	NA	< 0.01	N/A
59Co/35Cl16O	NA	> 18.0	N/A
Bkg220.7	NA	< 2.0 cps	N/A
iCAP-RQ Criteria			
Standard Mode			
Bkg4.5	NA	< 1.0 cps	N/A
7Li	0.65-0.85	> 55,000 cps	< 2% RSD
24Mg	0.65-0.85	> 500,000 cps	< 2% RSD
25Mg	0.65-0.85	> 80,000 cps	< 2% RSD
26Mg	0.65-0.85	> 100,000 cps	< 2% RSD
59Co	0.65-0.85	> 100,000 cps	< 2% RSD
115ln	0.65-0.85	> 240,000 cps	< 2% RSD
206Pb	0.65-0.85	> 80,000 cps	< 2% RSD
207Pb	0.65-0.85	> 70,000 cps	< 2% RSD
208Pb	0.65-0.85	> 160,000 cps	< 2% RSD
238U	0.65-0.85	> 330,000 cps	< 2% RSD

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 91 of 155

**Table 11-9 Instrument Mass Calibration & Performance Specifications** 

Parameter	Peak Width	Sensitivity/Criteria*	RSD
140Ce16O/140Ce	NA	< 0.02	N/A
137Ba++/137Ba+	NA	< 0.03	N/A
Bkg220.7	NA	< 2.0 cps	N/A
Analyzer Pressure	NA	< 10-6 mbar	NA
KED Mode†			
Bkg4.5	NA	< 0.5 cps	N/A
24Mg	0.65-0.85	> 10,000 cps	< 5% RSD
25Mg	0.65-0.85	> 2,000 cps	< 5% RSD
26Mg	0.65-0.85	> 3,000 cps	< 5% RSD
59Co	0.65-0.85	> 30,000 cps	< 2% RSD
115ln	0.65-0.85	> 35,000 cps	< 2% RSD
206Pb	0.65-0.85	> 100,000 cps	< 2% RSD
207Pb	0.65-0.85	> 90,000 cps	< 2% RSD
208Pb	0.65-0.85	> 200,000 cps	< 2% RSD
238U	0.65-0.85	> 85,000 cps	< 2% RSD
140Ce16O/140Ce	NA	< 0.01	N/A
59Co/35Cl16O	NA	> 18.0	N/A
Bkg220.7	NA	< 2.0 cps	N/A

<sup>\*</sup>cps – Counts per second

<sup>† –</sup> There are no vacuum requirements for KED mode

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 92 of 155

Table 11-10 Summary of Quality Control Procedures for Hexavalent Chromium

QC Check	Frequency	Acceptance Criteria	Corrective Action
Initial 6-point calibration	Before every sequence	Correlation coefficient ≥ 0.995;	1) Repeat analysis of calibration standards
standards		Relative Error (RE) < 20%	2) Reprepare calibration standards and reanalyze
ICV	Before every sequence,	Recovery 90-110%	1) Repeat analysis of initial calibration verification
	following the initial calibration		standard
			2) Repeat analysis of calibration standards
			3) Reprepare calibration standards and reanalyze
ICB	One per batch, following the	Analyte must be < MDL	1) Reanalyze
	ICV		2) Reprepare blank and reanalyze
			3) Correct contamination and reanalyze blank
			4) Flag data of all samples in the batch
CCV	Every 10 injections and at the	Recovery 90-110%	1) Repeat analysis of CCV
	end of the sequence		2) Reprepare CCV
			3) Flag data bracketed by unacceptable CCV
Laboratory Control Sample	Two per sample batch of ≤ 20	Recovery 90-110%	1) Reanalyze
(LCS/LCSD)	samples		2) Flag data of all samples since the last acceptable
			LCS
МВ	One per batch	Analyte must be ≤ MDL	Reanalyze
			Flag data for all samples in the batch
Replicate Analysis	Duplicate, Collocate, BS/BSD	RPD ≤ 20% for concentrations greater	1) Check integration
	and/or replicate samples only	than 5 x the MDL	2) Check instrument function
			3) Flag samples
ССВ	After every CCV and at the end	Analyte must be < MDL	1) Reanalyze
	of the sequence		2) Reprepare blank and reanalyze
			3) Correct contamination and reanalyze blank
			4) Flag data of all samples in the batch
Retention Time (RT)	For identification of analyte	RT must be within 5% window of the	1) Check integration/identification
, ,	,	average RT of initial calibration	2) Reanalyze
		standards	

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 93 of 155

Table 11-10 Summary of Quality Control Procedures for Hexavalent Chromium

QC Check	Frequency	Acceptance Criteria	Corrective Action
Sampling Duration	All samples		1) Notify Program Manager 2) Flag samples 22-23 hours and 25-26 hours in AQS with a "Y" flag 3) Invalidate and re-sample for > 24±2 hours

# Table 11-11 Summary of Quality Control Procedures for Lead Analysis by ICP-MS

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Daily Performance Report (DPR) KED Mode	Before each analysis	See Table 11-9	Repeat analysis of DPR     Re-optimize instrument tuning parameters     Reprepare DPR standard     Perform instrument maintenance
Initial Calibration Standards (IC)	Daily, at least 5 non-zero calibration points before each analysis	Correlation coefficient determination (R²) ≥ 0.998 & replicate %RSD ≤ 10. RSDs ≤ 20% are acceptable for lead in the CAL2 standard (at Limit of Quantitation (LOQ) concentration).	Repeat analysis of calibration standards     Reprepare calibration standards and reanalyze
ICV	Immediately after calibration	Recovery 90-110%	Repeat analysis of ICV     Recalibrate ICV standard     Recalibrate and reanalyze
ICB	Immediately after ICV	Absolute value must be < MDL	Locate and resolve contamination problems before continuing     Reanalyze or recalibrate or flag failing result for the entire analysis when appropriate
HSV	After ICB and before ICS	Recovery from 95-105%	1) Repeat analysis of HSV 2) Reprepare HSV

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 94 of 155

Table 11-11 Summary of Quality Control Procedures for Lead Analysis by ICP-MS

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
ccv	Analyze before samples, after every 10 samples, and at the end of each run	Recovery 90-110%	Reanalyze CCV     Reprepare CCV     Recalibrate and reanalyze samples since last acceptable CCV
LCV	After the first and last CCV	Recovery 70-130%	Reanalyze LCV     Reprepare LCV     Recalibrate and reanalyze samples since last acceptable LCV
ССВ	Analyzed after each CCV	Absolute value must be < MDL	Reanalyze CCB     Reanalyze samples since last acceptable CCB
Laboratory Reagent Blank (LRB)/Blank (BLK1)	1 per 20 samples, a minimum of 1 per batch	Absolute value must be < MDL	<ol> <li>Reanalyze for verification</li> <li>Flag failing batch QC and samples</li> <li>When enough sample filter remains (for glass fiber filters), a re-extraction and analysis of the batch should be considered</li> </ol>
Standard Reference Material (SRM)	1 per 20 samples, a minimum of 1 per batch	Recovery 80-120%	1) Reanalyze 2) Flag sample data
Duplicate (DUP1) (Laboratory Duplicate)	1 per 20 samples, for Glass fiber filters only	≤ 20% RPD for sample and duplicate values ≥ 5x MDL	1) Check for matrix interference. 2) Repeat duplicate analysis, if necessary, per ERG-MOR-114 3) Flag primary and DUP1, "D-F"
Replicate Analysis (Analytical Duplicate)	1 per batch of ≤ 20 samples	≤ 10% RPD for sample and replicate values ≥ 5x MDL	Repeat replicate analysis, if necessary, per ERG-MOR-114     Plag primary and replicate, "R-F"
Collocated Samples (C1/C2)	10% of samples annually (for sites conducting collocated sampling)	≤ 20% RPD of samples and collocated values ≥ 5x MDL	1) Flag C1 and C2 data if associated replicate analyses verify results     2) Repeat analysis if replicate analysis fails, per ERG-MOR-114

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 95 of 155

Table 11-11 Summary of Quality Control Procedures for Lead Analysis by ICP-MS

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	1 per 20 samples per sample batch	Recovery 80-120% when the parent sample concentration is less than 4 times the spike concentration	1) Flag data if recovery for fails criteria, or when a matrix interference is confirmed by SRD and/or PDS results 2) Reanalyze
		MS/MSD RPD ≤20%	3) Reprepare sample batch if contamination is evident
Post Digestion Spike (PDS)	1 per batch of ≤ 20 samples	Recovery 75%-125%	Flag parent sample and PDS     Reprepare PDS if preparation issue is suspected reason for failure
Serial Dilution (SRD)	1 per batch of ≤ 20 samples	10% RPD of undiluted sample if the concentration is ≥ 25x MDL	Reprepare dilution if preparation issue is suspected reason for failure     Plag failed analytes
Field/Trip/Lot Blanks	All client-generated blank samples	None.	None.
Internal Standards (ISTD)	Every Calibration, QC and Field Sample	Recovery 60-125% of the measured intensity of the calibration blank	1) If drift suspected, stop analysis and determine cause, recalibrate if necessary 2) Reprepare sample 3) If recovery > 125% due to inherent ISTD, dilute sample and reanalyze
NAAQS Audit strips	Quarterly – 3 high level and 3 low level	≥ ±10% difference of true Lead value	<ol> <li>Investigate</li> <li>Reanalyze</li> <li>Analyze another audit if available</li> </ol>

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 96 of 155

Table 11-12 Summary of Quality Control Procedures for Lead Analysis by XRF

Item	Inspection Frequency	Performed by	Inspection Parameter	Acceptance Criteria	Corrective Action
Energy Adjustment	Daily, performed prior to analysis	XRF Manager	Automated process of peaks appear at the correct energy in the spectrum	Instrument gain setting exceeds >20	Investigate for instability within the system or temperature controlled room. Service call, if needed
Calibration	As needed	XRF Manager	Use of Micromatter thin film standards	±10% through the calibration verification process	Re-calibrate
Minimum Detection Limit	Initially on method set-up and after any major instrument component change	Analyst / XRF Manager	10 lot blanks (unexposed filters) analyzed through the approved method	Ensuring the change across the elements does not exceed the historical limits	Investigate and run additional filters with change of lot
Calibration verification	Weekly, if samples are present	Analyst	Percentage of recovery of lead on thin-film National Institutes of Standards and Technology reference materials and Micromatter thin film standards	±10%	Re-analysis of calibration verification samples and if still outside of limits may require calibration
	Monthly, EPA audit samples, if samples are present	Analyst	Samples previously characterized by independent laboratories with Pb concentrations within specific ranges.		Re-analysis of calibration verification samples and if still outside of limits may require calibration
Ongoing calibration verification	Run with every tray of samples	Analyst	Micromatter multi-element sample containing Ti, Fe, Cd, Se, Pb, and SiO deposits of 5- 10µg/cm <sup>2</sup>	±10%	Re-check instrument calibration and adjust if necessary; re-analyze samples

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 97 of 155

Table 11-12 Summary of Quality Control Procedures for Lead Analysis by XRF

	Inspection	Performed by			
Item	Frequency		Inspection Parameter	Acceptance Criteria	Corrective Action
Replicate Analysis	10% of field samples	Analyst / PM	Reproducibility of analysis	RPD calculated when the original value is >10x the uncertainty. >10x uncertainty ±50%	Re-analysis of samples and inspect filter deposition
Audit strips	When analyzing samples - Quarterly	Analyst	Percentage of recovery of lead on Audit prepared for EPA	≥ ±10% difference of true Lead value	<ol> <li>Investigate</li> <li>Reanalyze</li> <li>Analyze another audit if available</li> </ol>

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 98 of 155

#### 11.4 Precision

Analytical precision is estimated by repeated analysis of approximately 10 percent of the samples. The second analysis is performed in the same analytical batch as the first analysis. Duplicate and collocated samples are reanalyzed once each to determine overall method precision, including sampling and analysis variability.

Precision estimates are calculated in terms of absolute percent difference. Because the true concentration of the ambient air sample is unknown, these calculations are relative to the average sample concentration. Refer to the tables provided in Section 11.3 for detailed criteria by analytical method.

Precision is determined as the RPD using the following calculation:

$$RPD = \frac{\left| X_1 - X_2 \right|}{\overline{X}} \times 100$$

Where:

 $X_1$  is the ambient air concentration of a given compound measured in one sample;

X<sub>2</sub> is the concentration of the same compound measured during duplicate/collocate/replicate analysis; and

 $\overline{X}$  is the arithmetic mean of  $X_1$  and  $X_2$ .

# 11.5 Completeness

Completeness, a quality measure, is calculated at the end of each year. Percent completeness is calculated as the ratio of the number of valid samples received to the number of scheduled samples (beginning with the first valid field sample received through the last field sample received). This quality measure is presented in the final report and can be expressed as completeness in terms of the number of samples collected or in terms of valid results per parameter. The completeness criteria for all parameters were previously presented in Table 4-1.

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 99 of 155

Completeness is determined using the following calculation:

$$Completeness = \frac{Number\ of\ valid\ samples}{Total\ expected\ number\ of\ samples}\ x\ 100$$

### 11.6 Representativeness

Representativeness measures how well the reported results reflect the actual ambient air concentrations. This measure of quality can be enhanced by ensuring that a representative sampling design is employed. This design includes proper integration over the desired sampling period and following siting criteria established for each task. The experimental design for sample collection should ensure samples are collected at proper times and intervals for their designated purpose per the data quality objectives. ERG is not responsible for the sampling design; therefore, representativeness is beyond the scope of this QAPP. The state and local areas should designate the representativeness following EPA guidelines, however a copy of the 2023 EPA sampling schedule is presented in Appendix B.

## 11.7 Sensitivity (Method Detection Limits)

The NATTS program has adopted two MDL procedures, a modified Definition and Procedure for the Determination of the Method Detection Limit<sup>(24)</sup> described in the NATTS TAD, revision  $4^{(23)}$ , and the Federal Advisory Committee (FAC) Single Laboratory Procedure (v2.4)<sup>(25)</sup>.

The modified EPA method detection limit procedure is used to determine MDLs for SNMOC, TO-15, TO-11A, and TO-13A. The initial MDL determinations are performed by comparing MDLs calculated using a spiked sample study to MDLs calculated from method blank sample data; the largest value of the two is the determined MDL. Every quarter, additional spiked and blank MDL samples are prepared as a part of standard laboratory operations; this data is evaluated at least every 13 months against the determined MDLs to verify the initial MDL concentrations.

The MDL for NMOC has not been determined in 2023. If this method is needed, a detection limit study will be performed before analysis begins. The MDLs for the SNMOC are listed in

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 100 of 155

Table 11-13, for VOCs in Table 11-14, and carbonyl compounds (based on a sample volume of 1000 L) in Table 11-15. The PAH MDLs, based on a sampling volume of 300 m<sup>3</sup>, are presented in Table 11-16.

The Sample Quantitation Limit (SQL) is also reported in the MDL tables. The SQL is defined as the lowest concentration an analyte can be reliably measured within specified limits of precision and bias during routine laboratory operating conditions. The SQL is defined by EPA as a multiplier (3.18) of the MDL and is considered the lowest concentration that can be accurately measured, as opposed to just detected. ERG submits this data into AQS using flags to show where the data is in respect to the detection level.

Table 11-13 2023 SNMOC Method Detection Limits

	MDL	SQL		MDL	SQL
Target Compound	(ppbC)	(μg/m³)	Target Compound	(ppbC)	(μg/m³)
1,2,3-Trimethylbenzene*	0.129	0.224	Cyclopentene	0.0189	0.0335
1,2,4-Trimethylbenzene*	0.124	0.215	Ethane*	0.103	0.201
1,3,5-Trimethylbenzene*	0.109	0.189	Ethylbenzene*	0.0443	0.0764
1,3-Butadiene*	0.0291	0.0512	Ethylene*	0.110	0.200
1-Butene*	0.0510	0.0930	Isobutane*	0.0261	0.0493
1-Dodecene	0.0400	0.0729	Isobutene	0.0840	0.153
1-Heptene	0.0329	0.0600	Isopentane*	0.0423	0.0794
1-Hexene*	0.0271	0.0506	Isoprene*	0.0171	0.0302
1-Nonene	0.222	0.405	Isopropylbenzene*	0.0679	0.118
1-Octene	0.0610	0.111	<i>m,p</i> -Xylene*	0.101	0.174
1-Pentene*	1.19	2.18	<i>m</i> -Diethylbenzene*	0.124	0.216
1-Tridecene	0.0640	0.117	Methylcyclohexane*	0.0356	0.0649
1-Undecene	0.137	0.249	Methylcyclopentane*	0.0198	0.0362
2,2,3-Trimethylpentane	0.0307	0.0571	<i>m</i> -Ethyltoluene*	0.0839	0.146
2,2,4-Trimethylpentane*	0.0178	0.0330	<i>n</i> -Butane*	0.0210	0.0397
2,2-Dimethylbutane*	0.0282	0.0526	<i>n</i> -Decane*	0.104	0.193
2,3,4-Trimethylpentane*	0.0444	0.0823	<i>n</i> -Dodecane*	0.0717	0.132
2,3-Dimethylbutane*	0.0157	0.0293	<i>n</i> -Heptane*	0.0195	0.0363
2,3-Dimethylpentane*	0.100	0.187	n-Hexane*	0.0355	0.0663
2,4-Dimethylpentane*	0.0724	0.116	<i>n</i> -Nonane*	0.0436	0.0807
2-Ethyl-1-butene	0.0367	0.0669	n-Octane*	0.0378	0.0701
2-Methyl-1-Butene	0.0141	0.0257	<i>n</i> -Pentane*	0.0217	0.0406

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 101 of 155

Table 11-13 2023 SNMOC Method Detection Limits

	MDL	SQL		MDL	SQL
Target Compound	(ppbC)	(μg/m³)	Target Compound	(ppbC)	(μg/m³)
2-Methyl-1-Pentene	0.0322	0.0601	<i>n</i> -Propylbenzene*	0.0746	0.130
2-Methyl-2-Butene	0.0263	0.0479	<i>n</i> -Tridecane	0.0630	0.116
2-Methylheptane*	0.0400	0.0743	<i>n</i> -Undecane*	0.170	0.314
2-Methylhexane*	0.0848	0.158	o-Ethyltoluene*	0.0739	0.128
2-Methylpentane*	0.0167	0.0312	o-Xylene*	0.0664	0.114
3-Methyl-1-Butene	0.0244	0.0444	<i>p</i> -Diethylbenzene*	0.139	0.243
3-Methylheptane*	0.0197	0.0365	<i>p</i> -Ethyltoluene*	0.0784	0.136
3-Methylhexane*	0.861	1.60	Propane*	0.126	0.241
3-Methylpentane*	0.250	0.466	Propylene*	0.0650	0.119
4-Methyl-1-Pentene	0.0502	0.0916	Propyne	0.0181	0.0314
Acetylene*	0.0222	0.0376	Styrene*	0.115	0.195
Benzene*	0.0252	0.0426	Toluene*	0.0490	0.0838
cis-2-Butene*	0.0170	0.0310	trans-2-Butene*	0.147	0.268
cis-2-Hexene	0.0197	0.0368	trans-2-Hexene	0.0226	0.0421
cis-2-Pentene*	0.0222	0.0416	trans-2-Pentene*	0.105	0.197
Cyclohexane*	0.0251	0.0459	α-Pinene*	0.0413	0.0732
Cyclopentane*	0.0329	0.0599	<i>β</i> -Pinene*	0.0723	0.128

<sup>\*</sup> PAMS compounds

**Table 11-14 2023 Air Toxics Method Detection Limits** 

	MDL	SQL		MDL	SQL
Target Compounds	(μg/m³)	(μg/m³)	Target Compounds	(μg/m³)	(μg/m³)
1,1,1-Trichloroethane	0.0519	0.165	Dibromochloromethane	0.163	0.519
1,1,2,2-Tetrachloroethane	0.130	0.414	Dichlorodifluoromethane	0.0299	0.0949
1,1,2-Trichloroethane	0.0218	0.0694	Dichloromethane	0.0812	0.258
1,1-Dichloroethane	0.0573	0.182	Dichlorotetrafluoroethane	0.0368	0.117
1,1-Dichloroethene	0.0626	0.199	Ethyl Acrylate	0.0319	0.101
1,2,4-Trichlorobenzene	0.587	1.87	Ethyl <i>tert</i> -Butyl Ether	0.0373	0.119
1,2,4-Trimethylbenzene	0.157	0.500	Ethylbenzene	0.0565	0.180
1,2-Dibromoethane	0.0507	0.161	Ethylene Oxide *	0.0636	0.202
1,2-Dichloroethane	0.0470	0.149	Hexachloro-1,3-Butadiene	0.387	1.23
1,2-Dichloropropane	0.0995	0.316	<i>m,p</i> -Xylene	0.181	0.576
1,3,5-Trimethylbenzene	0.155	0.494	m-Dichlorobenzene	0.222	0.705
1,3-Butadiene *	0.0486	0.154	Methyl Isobutyl Ketone	0.237	0.752
Acetonitrile	0.0554	0.176	Methyl Methacrylate	0.135	0.429
Acetylene	0.0158	0.0502	Methyl <i>tert</i> -Butyl Ether	0.0363	0.116
Acrolein *	0.189	0.602	<i>n</i> -Octane	0.120	0.383

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 102 of 155

Table 11-14 2023 Air Toxics Method Detection Limits

	MDL	SQL		MDL	SQL
Target Compounds	(μg/m³)	(μg/m³)	Target Compounds	(μg/m³)	(μg/m³)
Acrylonitrile	0.0252	0.0802	<i>o</i> -Dichlorobenzene	0.219	0.695
Benzene *	0.0322	0.103	<i>o</i> -Xylene	0.102	0.324
Bromochloromethane	0.0479	0.152	<i>p</i> -Dichlorobenzene	0.220	0.699
Bromodichloromethane	0.0596	0.189	Propylene	0.0459	0.146
Bromoform	0.112	0.355	Styrene	0.116	0.370
Bromomethane	0.0830	0.264	tert-Amyl Methyl Ether	0.0528	0.168
Carbon Disulfide	0.0521	0.166	Tetrachloroethylene *	0.0583	0.185
Carbon Tetrachloride *	0.0472	0.150	Toluene	0.0571	0.181
Chlorobenzene	0.0377	0.120	trans-1,2-Dichloroethylene	0.0167	0.0530
Chloroethane	0.0360	0.114	trans-1,3-Dichloropropene	0.0373	0.119
Chloroform *	0.0655	0.208	Trichloroethylene *	0.0365	0.116
Chloromethane	0.0200	0.064	Trichlorofluoromethane	0.0534	0.170
Chloroprene	0.0445	0.141	Trichlorotrifluoroethane	0.161	0.511
cis-1,2-Dichloroethylene	0.0520	0.165	Vinyl Chloride *	0.0131	0.0417
cis-1,3-Dichloropropene	0.0161	0.0513			

<sup>\*</sup>NATTS Tier I compounds

**Table 11-15 2023 Carbonyl Method Detection Limits (Underivatized Concentration)** 

Compound	MDL (μg/m3)	SQL (μg/m3)
2-Butanone (Methyl Ethyl Ketone)	0.107	0.340
Acetaldehyde*	0.0613	0.195
Acetone	0.304	0.966
Benzaldehyde	0.0205	0.0651
Butyraldehyde	0.0233	0.0742
Crotonaldehyde	0.0151	0.0480
Formaldehyde*	0.0561	0.178
Hexaldehyde	0.0356	0.113
Propionaldehyde	0.0348	0.111
Valeraldehyde	0.00812	0.0258

NOTE: Assumes 1000 L sample volume.

<sup>\*</sup>NATTS Tier I compounds

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 103 of 155

Table 11-16 2023 PAH Method Detection Limits

	MDL	SQL
Compounds	(ng/m³)	(ng/m³)
Acenaphthene	0.0194	0.0617
Acenaphthylene	0.00586	0.0187
Anthracene	0.0132	0.0419
Benz(a)anthracene	0.00719	0.0229
Benzo(a)pyrene*	0.0101	0.0320
Benzo(b)fluoranthene	0.0106	0.0338
Benzo(e)pyrene	0.00865	0.0275
Benzo(g,h,i)perylene	0.00944	0.0300
Benzo(k)fluoranthene	0.00498	0.0158
Chrysene	0.00684	0.0217
Coronene	0.00513	0.0163
Dibenz(a,h)anthracene	0.00320	0.0102
Fluoranthene	0.0260	0.0827
Fluorene	0.0432	0.138
Indeno(1,2,3-cd)pyrene	0.00587	0.0187
Naphthalene*	1.14	3.63
Perylene	0.00434	0.0138
Phenanthrene	0.112	0.356
Pyrene	0.0118	0.0376

NOTE: Assumes a 300 m<sup>3</sup> sample volume.

Metals analysis used the FAC MDL procedures for MDL determination. In this method, historic blank sample data is used to determine the MDL. Because all ICP-MS data is non-zero, it is not necessary to prepare a spiked MDL study for metals analysis. Each filter type, (quartz, Teflon, and glass) is evaluated separately, resulting in three separate MDLs for metals analysis. Glass fiber filters are only used for Pb NAAQS monitoring. The metals MDLs are shown in Table 11-17 and are based on a sampling volume of 1627 m³ for the quartz, 2000 m³ for glass fiber, and 24.04 m³ for the Teflon filters. The hexavalent chromium MDL is also included in Table 11-18 and is based on a sampling volume of 21.6 m³.

<sup>\*</sup>NATTS Tier I compounds

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 104 of 155

The NATTS Program requires sampling and analysis for 18 target air toxic analytes. Hexavalent chromium is no longer required by the NATTS program but was given a target MDL in the latest NATTS TAD<sup>(22)</sup> and the NATTS Work Plan Template (April 2019)<sup>(26)</sup>. The NATTS program uses sensitivity to assess quantification from a monitoring site with the appropriate level of certainty. In order to meet this objective, target MDLs have been established for the NATTS Program and are compared to the current 2023 ERG MDLs in Table 11-19. Note at the time of this QAPP, two NATTS Tier I compounds, ethylene oxide and acrolein, are above the NATTS MDL MQO. The EPA is aware, understanding the difficulty with these analytes.

Table 11-17 2023 Metals Method Detection Limit

	47 mm Tefl	on	8x10" Quar	tz	8x10" Glass	Fiber
Element	MDL (ng/m³)	SQL (ng/m³)	MDL (ng/m³)	SQL (ng/m³)	MDL (ng/m³)	SQL (ng/m³)
Antimony *	0.0822	0.262	0.0441	0.140	, 0, ,	, 0, ,
Arsenic *	0.0240	0.0764	0.00955	0.0304		
Beryllium *	0.00707	0.0225	0.00332	0.0106		
Cadmium *	0.00759	0.0241	0.109	0.345		
Chromium *	6.43	20.4	2.03	6.46		
Cobalt *	0.0193	0.0615	0.0156	0.0495		
Lead *	0.0645	0.205	0.276	0.879	0.980	3.12
Manganese *	0.508	1.62	1.19	3.77		
Mercury	0.0121	0.0386	0.00660	0.0210		
Nickel *	0.292	0.929	0.801	2.55		
Selenium *	0.0424	0.135	0.0110	0.0351		
Hexavalent Chromium MDL (47mm Cellulose)						
Hexavalent Chromium	0.00625	0.0199				

NOTE: For total metals: Assumes total volume of 24.04 m<sup>3</sup> for Teflon filters and 1627 m<sup>3</sup> for Quartz filters. For hexavalent chromium: Assumes total volume of 21.6 m<sup>3</sup>.

<sup>\*</sup>NATTS Tier I Compounds

Project No. 0475.00
Element No. Section 11 – B5
Revision No. 2
Date August 2023
Page 105 of 155

Table 11-18 Target MDLs for the NATTS Program

Pollutant	NATTS Target MDL (μg/m³) er I VOC HAPs	ERG 2023 MDL (µg/m³)	Is ERG MDL < Target MDL?		
Acrolein	0.090	0.189	No	_	
Benzene	0.13	0.0322	Yes	_	
1,3-Butadiene	0.10	0.0486	Yes	-	
Carbon Tetrachloride	0.17	0.0472	Yes		
Chloroform	0.50	0.0655	Yes		
Ethylene Oxide	0.054	0.0636	No		
Tetrachloroethylene	0.17	0.0583	Yes	-	
Trichloroethylene	0.20	0.0365	Yes	-	
Vinyl Chloride	0.11	0.0131	Yes		
NATTS Tier I	Carbonyl HAPs		•		
Acetaldehyde	0.45	0.0613	Yes		
Formaldehyde	0.080	0.0561	Yes		
Pollutant	NATTS Target MDL (ng/m³)	ERG 2023 MDL (ng/m³)	Is ERG MDL < Target MDL?		
NATTS Tie	er I PAH HAPs	, 0, ,	l .		
Benzo(a)pyrene	0.91	0.0101	Yes		
Naphthalene	29	1.14	Yes		
·	29 I Metal HAPs	1.14	Yes		
·		1.14 (Low Vol	I	(High Vo	ol PM10)
·			I	(High Vo	ol PM10) Yes
NATTS Ties	I Metal HAPs	(Low Vol	PM10)		
NATTS Ties  Arsenic (PM10)	0.23	(Low Vol 0.0240	<b>PM10)</b> Yes	0.00955	Yes
Arsenic (PM10)  Beryllium (PM10)	0.23 0.42	(Low Vol 0.0240 0.00707	PM10) Yes Yes	0.00955 0.00332	Yes Yes
Arsenic (PM10) Beryllium (PM10) Cadmium (PM10)	0.23 0.42 0.56	(Low Vol 0.0240 0.00707 0.00759	PM10) Yes Yes Yes	0.00955 0.00332 0.109	Yes Yes Yes

NOTE: Target MDL's were obtained from the NATTS Work Plan Template<sup>(26)</sup>, Section 3.1 and the NATTS TAD<sup>(22)</sup>

Project No. 0475.00
Element No. Section 12 - B6
Revision No. 2
Date August 2023
Page 106 of 155

# SECTION 12 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

To ensure the quality of the sampling and analytical equipment, ERG conducts performance checks for all equipment used in each of the programs. ERG checks the sampling systems annually and makes repairs as needed. ERG analysts track the performance of the analytical instrumentation to ensure proper operation each day samples are analyzed. ERG also maintains a spare parts inventory to shorten equipment downtime. Table 12-1 details the maintenance items, how frequently they will be performed, and who is responsible for performing the maintenance. All checks, testing, inspections, and maintenance done on each instrument are recorded in the appropriate Maintenance Logbook or LIMS Instrument Maintenance Logs for each instrument.

**Table 12-1 Preventive Maintenance in ERG Laboratories** 

Item	Maintenance Frequency	Responsible Party
Analytical Systems		
Replace GC/LC/IC Column	As necessary (i.e., observe peaks tailing, retention time shifts, increased baseline noise, etc.)	Analyst
Detector Maintenance	As necessary	Analyst
Computer Backup	Biweekly, Daily preferred	Analyst
Accelerated Solvent Extractor		
Piston Rinse Seal	Quarterly, or as needed	Analyst
Standard Rinse Seal	Quarterly, or as needed	Analyst
High Performance Liquid Chromatog	raphy	
In-line filter/Guard Column	As necessary (when pressure increases above 4000 psi)	Analyst
Inspect Delivery System Motor	Annually	Service Technician
Replace Teflon Delivery Tubing	Annually	Service Technician
Ion Chromatography		
Rinse Post Column Reagent lines with methanol	As necessary	Analyst
Rinse Eluent Lines with Deionized water	After every sequence	Analyst
Sonicate Inlet and Outlet Check Valves	As necessary	Analyst

Project No. 0475.00
Element No. Section 12 - B6
Revision No. 2
Date August 2023
Page 107 of 155

**Table 12-1 Preventive Maintenance in ERG Laboratories** 

Item	Maintenance Frequency	Responsible Party
Rinse Autosampler Injector	As necessary	Analyst
Inorganic Laboratory		
Flush system for 5 minutes with the plasma on with a rinse blank	After every sequence	Analyst
Cleaning cones, torch, injector, spray chamber	Quarterly, or as needed for analysis quality	Analyst
Change Roughing Pump Oil	Annually	Service Engineer
Replace Air Filters	Annually	Service Engineer
Sampling Field Equipment (UATMP,	Carbonyl, NMOC/SNMOC, and Hexava	lent Chromium)
Inspect/Replace vacuum pump diaphragms and flapper valves	At each system certification effort	ERG
Inspect Sampler (overall)	At each system certification effort and prior to each scheduled collection event	ERG/Field Operator
Inspect/Replace Cartridge Connectors	Prior to each collection event, replace as needed	ERG/Field Operator
Replace Ozone Scrubber	At each system certification effort	ERG
MFM Check or Flow check	At each system certification effort	ERG
Inspect/Replace Fans	At each system certification effort	ERG

# 12.1 SNMOC, VOC, and PAMS

The GC/ MS systems are maintained under a service agreement. ERG personnel perform minor maintenance, such as filament changes, carrier gas filter replacements, column maintenance, and source cleaning. The following spare parts should be kept in the lab: traps, filament, column, and split for the column. All procedures, checks, and scheduled maintenance checks for VOC GC/FID/MS analysis are provided in ERG's SOP (ERG-MOR-005) presented in Appendix D.

# 12.2 Carbonyls

The carbonyl HPLC analytical systems are maintained under a service agreement. ERG personnel perform minor maintenance, such as column and detector maintenance, on an as-needed basis. The following spare parts should be kept in the lab: solvent frit, column, in-line

Project No. 0475.00
Element No. Section 12 - B6
Revision No. 2
Date August 2023
Page 108 of 155

filter and guard column. All procedures, checks, and scheduled maintenance checks are provided for carbonyl HPLC analysis in ERG's SOP (ERG-MOR-024) presented in Appendix D.

#### 12.3 HAPs

The GC/MS systems for PAH and VOC analysis are maintained under the same service agreement. ERG personnel perform minor maintenance as needed. The following spare parts are kept in the lab: injector sleeve, filament, and column.

For the HAPs sample analyses performed on the ICP-MS and IC, routine preventive maintenance is performed by the Analyst or Task Lead. ERG personnel perform minor maintenance on an as-needed basis, such as cleaning/replacing sample introduction components, chromatography columns, or detectors. Contracted service agreements are in place for non-routine maintenance. Spare peristaltic pump tubing, sample and skimmer cones, nebulizers, torches, injectors, and o-rings are kept in the lab for the ICP-MS. A spare guard and analytical column, piston seals, reaction coil, and reservoir frits are kept in the lab for the IC. More procedures, checks, and scheduled maintenance checks are provided in ERG's SOP (ERG-MOR-049) for PAH analysis by GC/MS, ERG-MOR-095 for metals analysis by ICP-MS, and ERG-MOR-063 for hexavalent chromium by IC presented in Appendix D.

Project No. 0475.00
Element No. Section 13 - B7
Revision No. 2
Date August 2023
Page 109 of 155

#### SECTION 13 INSTRUMENT CALIBRATION AND FREQUENCY

The programs are discussed separately in this section because the requirements for analytical system calibrations differ. Analytical instruments and equipment are calibrated when the analysis is set up, when the laboratory takes corrective action, following major instrument maintenance, or if the continuing calibration acceptance criteria have not been met.

Appropriate standards are prepared by serial dilutions of pure substances or accurately prepared concentrated solutions. In preparing stock solutions of calibration standards, great care is exercised in measuring weights and volumes, since analyses following the calibration are based on the accuracy of the calibration.

Each calibration analysis is stored, electronically and hardcopy, with traceability for the samples analyzed using that calibration. Each of the analytical systems is calibrated for all reported target analytes, except for the NMOC and SNMOC calibrations. The NMOC calibration is based on propane and the SNMOC calibration is based on propane and benzene average response factors. NMOC calibration will be discussed in more detail when the analysis is requested by a State.

#### 13.1 SNMOC Calibration

For the SNMOC method, average carbon response factors are obtained annually (at a minimum) based on the analysis of humidified calibration standards prepared in canisters. The Dynamic Flow Dilution System (SOP Number ERG-MOR-061, Appendix D) is used to dilute certified standards into clean, evacuated SUMMA®- treated canisters. The gas standards are traceable via the gravimetric preparation using NIST-traceable weights. These gas standards are recertified annually or at manufacturer specified expiration date. HPLC grade water is used to humidify the standard to approximately 50 percent. The standard is diluted with scientific-grade air to achieve the desired concentrations for the calibration. The response factors generated from the calibration are used to determine concentrations of detected compounds, on the assumption that FID response is linear with respect to the number of carbon atoms present in the compound.

Project No. 0475.00
Element No. Section 13 - B7
Revision No. 2
Date August 2023
Page 110 of 155

At least five calibration standards are prepared in ranges from approximately 5 to 400 ppbC concentrations. The average response factors for propane and benzene are determined using the response correlated to concentration. Individual sample concentrations for the C2 through C13 compounds detected on the FID are calculated using one of the two response factors, with a similar Carbon number. The calibration is considered representative if the average RF RSD for the curve is within ±20 percent. Immediately following calibration, an ICV standard from a second vendor source (such as Air Environmental gas standard), is analyzed to verify the concentration of the calibration standards. Ten selected hydrocarbons, ranging from C2 through C10, from the ICV standard are compared to the calculated theoretical concentrations. A percent recovery of 70-130 percent is considered acceptable showing the analytical system is in control.

A blank of cleaned, humidified air or nitrogen is analyzed after the ICV and before any samples are analyzed. The system is considered in control if the total NMOC concentration for the blank is less than 20 ppbC.

## 13.2 VOC Calibration

Calibration of the GC/ MS is accomplished quarterly (at a minimum) by analyzing humidified calibration standards prepared in canisters generated from NIST-traceable Linde or Air Environmental (or equivalent) gas standards. The certified gas standards contain the VOC target compounds at approximately 500 ppbV. These gas standards are used within expiration and are recertified annually or at manufacturer specified expiration date. If a gas standard must be used after expiration, the procedure in NATTS TAD, revision 4 Section 4.2.6.1.3 will be followed. Calibration standards are prepared with a dynamic flow dilution apparatus (Figure 13-1, see Standard Operating Procedure ERG-MOR-061, Appendix D). The gases are mixed in a SUMMA®-treated mixing sphere and bled into evacuated canisters. One dilution air stream is humidified by routing it through a SUMMA®- treated bubbler containing HPLC-grade water; the other stream is not humidified. The dilution air streams are then brought together for mixing with the streams from the certified cylinders. Flow rates from all streams are gauged and controlled by

Project No. 0475.00
Element No. Section 13 - B7
Revision No. 2
Date August 2023
Page 111 of 155

mass flow controllers. The split air dilution streams are metered by "wet" and "dry" rotameters (~50 percent relative humidity) from the humidified and unhumidified dilution air streams, respectively.

The system is evacuated with a vacuum pump while the closed canister is connected. The lines leading to the canister and to the mixing sphere are flushed for at least 20 minutes with standard gas before being connected to the canister for filling. A precision pressure gauge measures the canister pressure before and after filling.

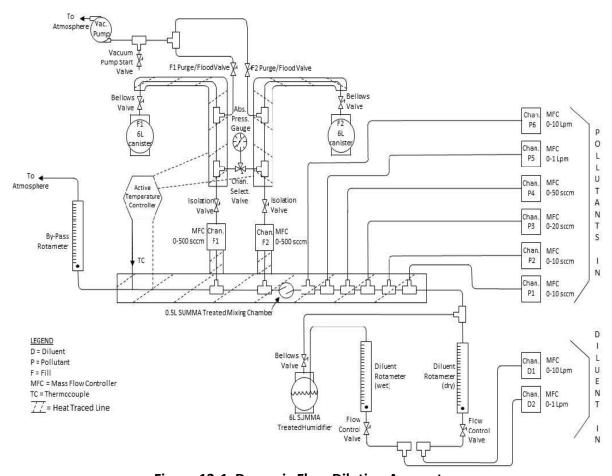


Figure 13-1 Dynamic Flow Dilution Apparatus

Initial calibration standards are prepared at approximate nominal concentrations of 0.125, 0.25, 0.5, 1, 2.5, 5, and 10 ppbV for each of the target compounds (a minimum of 5 levels are required). All standards and samples are analyzed with the following internal standards: n-hexane-d14, 1,4-difluorobenzene, and chlorobenzene-d5. The calibration requires average

Project No. 0475.00
Element No. Section 13 - B7
Revision No. 2
Date August 2023
Page 112 of 155

response factors, based on the internal standard, of  $\pm$  30.1 percent RSD. The nominal recovery at each calibration level must be within  $\pm$ 30.1%. Immediately following calibration, an ICV standard from a second vendor source at an average concentration of 2.5 ppbV is analyzed. The calibration is considered acceptable if those criteria are met and the ICV RRFs are within  $\pm$  30.1% of the mean initial calibration RRFs. If the secondary standard, ICV, recovery fails high for ethylene oxide, the primary standard, ICAL, may have degraded and any data from the ICAL is suspect.

# 13.3 Carbonyl Calibration

For the carbonyl analyses, the HPLC instrument is calibrated using an acetonitrile solution containing the derivatized targeted compounds. The calibration curve consists of six concentration levels ranging from 0.01 to 3.0 microgram per milliliter ( $\mu g/mL$ ) (underivatized), and each level is analyzed in triplicate. The standard linear regression analysis performed on the average of the triplicate data for each analyte must have a correlation coefficient greater than or equal to 0.999. The Relative Error (RE) for each compound at each level against the calibration curve must be  $\leq 20.1$  percent. Additionally, the absolute value of the y-intercept divided by the slope must be less than the determined MDLsp. To verify the calibration standards and check HPLC column efficiency, an ICV standard containing target carbonyl compounds from a second vendor source at a known concentration is analyzed after every calibration curve, with an 84.9-115.1 percent recovery criterion.

#### 13.4 HAPs Calibration

The GC/MS system in SIM mode is calibrated for PAH analysis at a minimum every six weeks. The average calibration RRF must be greater than or equal to the minimum RRF presented in Table 13-1. For the other HAPs sample analyses, calibration is performed on the ICP-MS and IC. Minimum calibration requirements for the HAPs analytical methods are in Tables 11-7, 11-9 and 11-10; for more details see the analytical SOPs that cover the ICP-MS and IC analyses.

Project No. 0475.00
Element No. Section 13 - B7
Revision No. 2
Date August 2023
Page 113 of 155

Table 13-1 Minimum Relative Response Factor Criteria for Initial Calibration of Common Semivolatile Compounds

Semivolatile Compounds	Minimum RRF	
Naphthalene	0.700	
Acenaphthylene	1.300	
Acenaphthene	0.800	
Fluorene	0.900	
Phenanthrene	0.700	
Anthracene	0.700	
Fluoranthene	0.600	
Pyrene	0.600	
Benz(a)anthracene	0.800	
Chrysene	0.700	
Benzo(b)fluoranthene	0.700	
Benzo(k)fluoranthene	0.700	
Benzo(a)pyrene	0.700	
Indeno(1,2,3-cd)pyrene	0.500	
Dibenz(a,h)anthracene	0.400	
Benzo(g,h,i)perylene	0.500	
Perylene	0.500	
Coronene	0.700	
Benzo(e)pyrene		

Note – The ASTM method includes no minimum RRF criteria, therefore none are listed here for the ASTM (12) compounds.

# 13.5 Laboratory Support Equipment Calibration

Analytical balances are serviced and calibrated annually with NIST traceable weights by a vendor service technician. The same vendor also verifies the Class 1 weights annually. The balance calibrations are checked daily on days of use with Class 1 weights and recorded. The data loggers used for temperature/humidity/pressure have calibration checks annually performed by the vendor. The infrared (IR) thermometers are annually vendor calibrated with NIST-traceable standards. Thermometers requiring a calibration check will be checked against a thermometer with an annual NIST traceable vendor calibration. The pressure gauges used for measuring sample canister pressure at receipt are calibrated annually by a certified vendor. Other pressure gauges, used in canister cleaning or canister sample dilution, are checked

Project No. 0475.00
Element No. Section 13 - B7
Revision No. 2
Date August 2023
Page 114 of 155

against a "transfer standard" gauge that is calibrated annually by a certified vendor. MFCs used in the canister dynamic dilution standard system are calibrated annually.

Pipette calibrations are checked and recorded quarterly. If a pipette fails a calibration check, they are rechecked. If it continues to fail, it is sent back to the manufacturer for recalibration. If recalibration is not possible it will be repaired or replaced with a new pipette. Syringe calibrations are checked and recorded annually. If a syringe fails the calibration check, it will be replaced with a new one. Class A volumetric glassware is used throughout the laboratory for bringing sample extracts up to final volume.

Project No. 0475.00
Element No. Section 14 - B8
Revision No. 2
Date August 2023
Page 115 of 155

# SECTION 14 INSPECTION/ACCEPTANCE FOR SUPPLIES AND CONSUMABLES

# 14.1 Purpose

The purpose of this element is to document a system for inspecting and accepting all supplies and consumables that may directly or indirectly affect the quality of the NHAPS program. By having documented inspection and acceptance criteria, consistency of the supplies can be assured. This section details the supplies/consumables, their acceptance criteria, and the required documentation for tracing this process.

#### 14.2 Critical Supplies and Consumables

Table 14-1 details the various components for the field and laboratory operations.

## 14.3 Acceptance Criteria

Acceptance criteria for supplies/consumables must be consistent with overall project technical and quality criteria. As requirements change, so do the acceptance criteria. Knowledge of laboratory equipment and experience are the best guides to acceptance criteria. It is the laboratory analyst's responsibility to update the criteria for acceptance of consumables and the testing, inspection, and maintenance of equipment. The acceptance criteria for sample media, such as carbonyl lot blank and TO-13A PUF/XAD, are listed in the respective SOPs. Other acceptance criteria such as observation of damage due to shipping can only be performed once the equipment has arrived on site.

All supplies and consumables are inspected and accepted or rejected upon receipt in the laboratory. The ERG employee who ordered the supply is responsible for verifying that the order is acceptably delivered, stored, and dispersed upon receipt in the laboratory. The recipient's signature on the packing slip indicates the received goods were received and are acceptable. Some supplies or consumables listed in Table 14-1 must be deemed acceptable through testing or blanking, such as with the carbonyl DNPH cartridges. Any changes in type of standards, sample media, etc. must be shown to meet the acceptance criteria outlined in

Project No. 0475.00
Element No. Section 14 - B8
Revision No. 2
Date August 2023
Page 116 of 155

Section 11 for that particular method. Such testing and blanking data is stored with the sample data. Staff should not use supplies or consumables of different model numbers or grades without first discussing it with the Program Manager and specific Task Leader and testing the supply or consumable. Staff should keep any certificate of analysis or documentation pertaining to cleanliness that arrives with the supply/consumable on file. For specific information on reagents and standards used, see applicable method SOP.

**Table 14-1 Critical Supplies and Consumables** 

Area	Item	Description	Vendor	Model Number			
Field Supplies and Consumables (Fabrication Lab)							
All Samplers	Various Swagelok® fittings	All Samplers	Swagelok	Various			
NMOC Sampler	Pump	Metal Bellows	KNF Newberger	UN 05-SV.91			
VOC Sampler	Vacuum Pump	VOC System	Thomas	2107VA20			
	Canisters	VOC Canisters	Entech	6-liter Silonite® Canisters			
Carbonyl Sampler	DNPH Cartridges	DNPH coated plastic cartridges	Waters	WAT 037500			
Hexavalent Chromium Sampler	Pump	High Vacuum	Thomas	VA-2110			
Laboratory Supplies and Consumables (Laboratories listed below)							
All Laboratories	Powder Free Gloves	Polyethylene	VWR	32915-246			
All Laboratories	Gloves	Nitrile	Expotech, ThermoFisher, VWR	1461558 (Expotech)			
Liquid Chromatography	Guard column	Waters VanGuard (3.9 mm x 5 mm) 2.7 micron	Waters	186007684			
Liquid Chromatography	Chromatographic Column	Waters CORTECS (4.6 mm ID x 15 cm) 2.7 micron	Waters	186007378			
Liquid Chromatography	UV Lamp	For 2489 detector	Waters	WA 5081142			
GC/MS – VOC	Chromatographic Column	0.32 x 1 μ - 60 m column	Restek	Rxi-lms			
GC/MS – SVOC	Chromatographic Column	0.25 mm x 0.25 μm - 30 m column with 10 m deactivated	Restek	Rxi®-5Sil MS Integra-Guard			
GC/MS – SVOC	Chromatographic Column	0.25 mm x 0.10 μm - 60 m column	Restek	Rxi-PAH			
GC/MS – SVOC	Inject seal	Injection port seal	Expotech	2264837			
GC/MS – SVOC	Liner	Injection port liner	Expotech	2377232			
GC/MS & Liquid Chromatography	Helium	Carrier Gas	Air Gas	UHP			

Project No. 0475.00
Element No. Section 14 - B8
Revision No. 2
Date August 2023
Page 117 of 155

# **Table 14-1 Critical Supplies and Consumables**

Area	Item	Description	Vendor	Model Number
GC/MS – VOC	Chromatographic	0.32 x 1 μ - 60 m	Restek	Rxi-lms
CC/NC VOC	Column	column	5	D : C2 45:1
GC/MS – VOC	Chromatographic Column	1.8-micrometer (µm) film thickness	Restek	Rxi-624Sil
CC/NAC			Air Coo	capillary
GC/MS	Hydrogen Gas	FID Gas	Air Gas	UHP
GC/MS	Liquid Nitrogen	Coolant Gas	Air Gas	Bulk
GC/MS	Liquid Argon	Coolant Gas	Air Gas	Bulk
GC/MS	Air	FID Gas	Air Gas	Zero
GC/MS	Traps	Glass bead/Tenax Trap	Entech	01-04-11340
GC/MS	Trap Heater	Sample Trap Heater	Entech	01-09-13010
GC/MS	Cryogenic Valve	Cryogenic Valve	Entech	01-01-71760
ICP-MS	Liquid Argon	Coolant Gas	Air Gas	Bulk
ICP-MS	Acid	High Purity Nitric	Fisher/SCP Science	A200- 212/Plasma Pure Plus
ICP-MS	Acid	Hydrochloric Acid	Fisher/SCP Science	A466-1/Plasma Pure Plus
ICP-MS	Hydrogen Peroxide	Hydrogen Peroxide, 30%	SCP Science	Plasma Pure Plus
ICP-MS	Whatman 8"x11" Quartz/Glass Fiber Filters	Filters	GE Healthcare Life Sciences & MTL	1851-8531 1882-8532
	MTL 47mm Teflon™ Filters			PT47-EP
IC	Reaction Coil	Knitted Reaction Coil	ThermoFisher	042631
IC	Guard Column	Dionex Ion Pac NG1	ThermoFisher	039567
IC	Analytical Column	Dionex Ion Pac AS7	ThermoFisher	035393
IC	Methanol	Solvent	Expotech, Fisher, VWR	HPLC grade
IC	Sample vials 14 mL, polystyrene with caps	Sample containers	ThermoFisher	352057
IC	Whatman Filters	Filters–47mm ashless cellulose	Expotech, Fisher	09-850H
Prep	Water Filter	Ultrapure Ion Exchange Cartridge	Expotech	1425973
Prep	Water Filter	Cartridge submicron	Expotech	1425977
Prep	Water Filter	Pretreatment Cartridge	Expotech	1426051
Prep	Whatman Filters	Filters–110mm GFA	Expotech	1422153
Prep	PUF	Pre-cleaned PUF	Cen-Med,	824-20038,
•			Expotech	2256468
Prep	XAD®	XAD®	Expotech	2255045
Prep	Petri Dish	Filter container	Expotech	1426833
Prep	Tweezers	Tweezers	VWR	100499-866
Prep	Acetonitrile	Solvent	Expotech, Fisher,	HPLC grade

Project No. 0475.00
Element No. Section 14 - B8
Revision No. 2
Date August 2023
Page 118 of 155

**Table 14-1 Critical Supplies and Consumables** 

Area	Item	Description	Vendor	Model Number
			VWR	
Prep	Methylene Chloride	Solvent	Expotech, Fisher, VWR	Optima grade
Prep	Methanol	Solvent	Expotech, Fisher, VWR	Optima grade
Prep	Hexane	Solvent	Expotech, Fisher, VWR	95% (Optima grade)
Prep	Toluene	Solvent	Expotech, Fisher, VWR	Optima grade
Prep	Water	Solvent	Expotech, Fisher, VWR	HPLC grade
Prep	Nitrogen	Evaporation gas	Air Gas	UHP (or Bulk)
Prep	Amber glass bottles 250 mL	Sample containers	Expotech	2373176
Prep	30mm glass fiber filters	Extraction filters	Expotech	2262135
Prep	Extraction cells	Sample containers	Thermo Electron	068077
Prep	Ottawa sand	Extraction filler	Expotech	2262138
Prep	Seals	ASE Vespel Seals	Fisher	056776
Prep	O-rings	Extraction cell o-rings	Expotech	2374568
Prep	Disposable pipets	Disposable pipets	Expotech	1405717
Prep	2 mL amber sample vials	Sample containers	Sigma-Aldrich	27000
Prep	4 mL amber sample vials	Sample containers	Expotech, Fisher, VWR	66030-734 (VWR)
Prep	4 mL sample Teflon lined caps	Sample containers	Expotech, Fisher, VWR	66030-771 (VWR)
Prep	Autosampler snap- it vials	Sample containers	Waters	WAT 094220
Prep	Autosampler snap- it caps	Sample containers	Waters	18000303

Consumables and supplies with special handling and storage needs must be handled and stored as suggested by the manufacturer. Consumables with expiration dates, such as solvents and standards, must be labeled with a receipt date, date opened, and the initials of the person that opened the consumable and standard expiration dates must be entered into the standards section of LIMS. To decrease waste, the oldest supplies or consumables should be used first.

Project No. 0475.00
Element No. Section 15 – B9
Revision No. 2
Date August 2023
Page 119 of 155

# **SECTION 15 Non-direct Measurements**

Not applicable to this project. This project is based on direct measurements.

Project No. 0475.00
Element No. Section 16 – B10
Revision No. 2
Date August 2023
Page 120 of 155

#### SECTION 16 DATA MANAGEMENT

# 16.1 Data Recording

Data management for sample data is presented in Figure 16-1. The sample data path is shown from sample origination to data reporting and storage. The LIMS allows the laboratory to manage and track samples, instrument workflow, and reporting. The LIMS administrator is responsible for ensuring the LIMS processes the data correctly. The LIMS stores the raw instrument data and performs the conversion calculations to put the data into final reporting units. These LIMS calculations are reviewed biannually by the QA coordinator and documentation is kept in the QA files in Room 102. The monthly data reviews also verify that the LIMS calculations are being performed correctly. The LIMS procedures are described in the SOP for the Laboratory Information Management System (ERG-MOR-099). The main functions of the LIMS system include, but are not limited to:

- Sample login;
- Sample scheduling, and tracking;
- Sample processing and quality control; and
- Sample reporting and data storage.
- All LIMS users must be authorized by the LIMS Administrator and permitted specified privileges. The following privilege levels are defined:
- Data Entry Privilege The individual may see and modify only data within the LIMS that he or she has personally entered.
- Reporting Privilege Without additional privileges.
- Data Administration Privilege Data Administrators for the database are allowed to change data as a result of QA screening and related reasons. Data Administrators are responsible for performing the following tasks on a regular basis:
  - Merging/correcting the duplicate data entry files;
  - Running verification/validation routines, correcting data as necessary.

Project No. 0475.00
Element No. Section 16 – B10
Revision No. 2
Date August 2023
Page 121 of 155

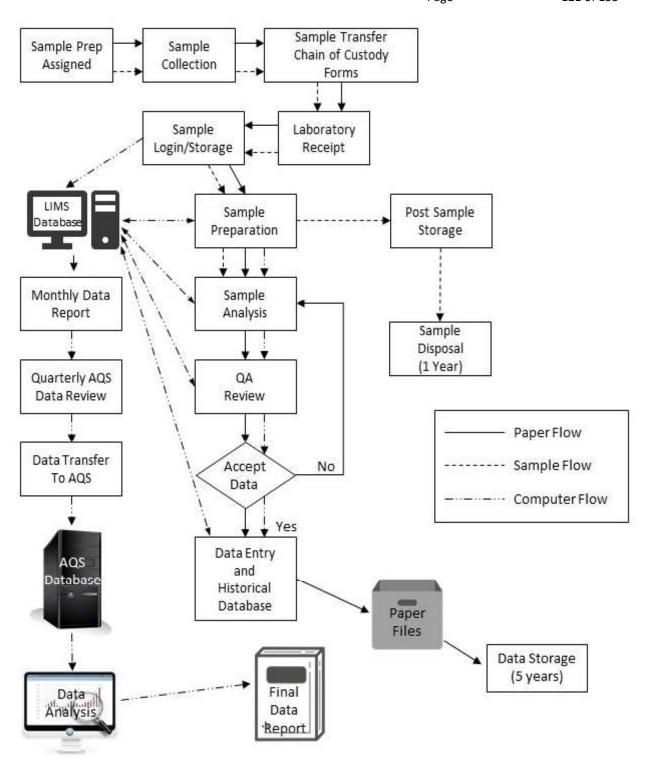


Figure 16-1 Data Management and Sample Flow Diagram

Project No. 0475.00
Element No. Section 16 – B10
Revision No. 2
Date August 2023
Page 122 of 155

#### 16.2 Data Validation

Data validation is a combination of checking that data processing operations have been carried out correctly and of monitoring the quality of the field operations. Data validation is confirmed by examination of objective evidence that the requirements for a specific intended use are fulfilled as presented in Section 4. This data validation is performed prior to the annual final report. The data reported monthly are considered preliminary until the data is validated by the AQS Task Leader or designee, entered into the AQS database, and reported in the annual final report. Data validation is discussed in more detail in Section 18.4.

#### 16.3 Data Reduction and Transformation

Data generated on an instrument is reduced by the analyst via instrument chromatographic software. Any manual integration to chromatographic data follows SOP ERG-MOR-097, the SOP for Manual Integration of Chromatographic Peaks. The analyst is responsible for reviewing the data before the data goes to the Task Leader. The Task Leader is responsible for reviewing the data before the data goes to the QA staff. The data reviews are documented using the QA review checklist located in individual analytical SOPs. Designees trained to review analytical data may be assigned for any level of review, however that designee must not have performed the previous data review task(s).

Specific equations used by the instrument chromatographic software to calculate concentration are documented in the individual analytical SOPs found in Appendix D. The equations for transforming raw data are set up to automatically calculate to final concentrations in the LIMS system. The initial and final reporting units for SNMOC are ppbC. The initial units for the Carbonyl Compounds analysis are  $\mu g/mL$ , while the final reporting units are in either ppbV or  $\mu g/m^3$ , per site request, however the NATTS sites are to be reported in  $\mu g/m^3$  per the NATTS TAD<sup>(22)</sup>. The initial units for VOC are ppbV and the LIMS data reports are in ppbV and  $\mu g/m^3$ . The PAH initials units are  $ng/\mu L$  with final reporting units of  $ng/m^3$ . The initial units for metals are ng/L with final reporting units of  $ng/m^3$ . The initial units for the hexavalent chromium analysis

Project No. 0475.00
Element No. Section 16 – B10
Revision No. 2
Date August 2023
Page 123 of 155

are ng/mL with final reporting units of ng/m<sup>3</sup>. The associated MDLs are reported in final reporting units with the final concentrations. MDLs are adjusted for dilution, actual prep volumes, and sample collection volume where applicable, before reporting.

The electronic data file is uploaded onto a network server (which is backed-up daily) and into the LIMS. Once the data is in LIMS, the Task Leader reviews it following the QA review checklists presented in the SOPs using instrument software, quality control procedure tables in Section 11, and the method-specific control limits set up in LIMS. Ten percent of all data is reviewed by the QA Coordinator or designee following the checklist and method specific acceptance criteria in the summary quality control procedure tables outlined in Section 11. After data has successfully completed both reviews and the checklists have been initialed, it is available for reporting by the Program Manager.

Reporting formats are designed to fulfill the program requirements and to provide comprehensive, conventional tables of data. The LIMS data reporting format includes any required data qualifiers, footnotes, detection limits for each analyte, and appropriate units for all measurements. The LIMS can produce Adobe and Excel data reports, which is standard for this program. Each report is reviewed by the Program Manager or designee before it is sent to the client.

#### 16.4 Data Transmittal

Data transmittal occurs when data are transferred from one person or location to another or when data are copied from one form to another. Some examples of data transmittal are copying raw data from a notebook into a LIMS bench sheet and electronic transfer of raw chromatographic data to a LIMS data entry table. Checks directed by the QA review checklist and data validation assist in finding transcription errors due to data transmittal.

ERG reports ambient air quality data and associated information to AQS as specified by the documentation at the following website https://www.epa.gov/aqs/aqs-manuals-and-guides. Such air quality data and information will be fully screened and validated by the AQS TL or

Project No. 0475.00
Element No. Section 16 – B10
Revision No. 2
Date August 2023
Page 124 of 155

designee and will be submitted directly to the AQS database via electronic transmission, in AQS format, and in accordance with required quarterly schedule. The SOP for the Preparation of Monitoring Data for AQS Upload is presented in Appendix D (SOP ERG-MOR-098).

## 16.5 Data Summary

ERG's AQS Task Leader implements the data summary and analysis for the NHAPS in the final annual report. The following specific summary statistics will be tracked and reported for the network:

- Single sampler bias or accuracy (based on laboratory audits if available);
- Analytical precision (based on analytical replicates);
- Sampler precision (based on duplicate and collocated data);
- Network-wide bias and precision; and
- Data completeness.

Equations used for these reports are given in Table 16-1.

Table 16-1 Final Report Equations

Criterion	Equation
Coefficient of Variation (CV)- p and r are concentrations from the primary and duplicate samplers, respectively. This equation is also used for collocated samples and replicate analysis.	$CV = 100 \times \sqrt{\frac{\sum_{i=1}^{n} \left[\frac{(p-r)}{0.5 \times (p+r)}\right]^{2}}{2n}}$
Percent Completeness - Where, N valid is the number of valid samples collected and analyzed in the sampling year and N expected is the number of valid samples that are expected within that same sampling year.	$Completeness = \frac{N_{valid}}{N_{expected}} \times 100$

Project No. 0475.00
Element No. Section 16 – B10
Revision No. 2
Date August 2023
Page 125 of 155

## 16.6 Data Tracking

The ERG LIMS database contains the necessary input functions and reports appropriate to track and account for the status of specific samples and their data during processing operations. The following input locations are used to track sample and sample data status:

## Sample Control

- Sample collection information (by Work Order);
- Sample receipt/custody information;
- Unique sample number (LIMS ID);
- Storage location;
- Required analyses;

## Laboratory

- Batch/bench assignment;
- Sequence assignment (if applicable);
- Data entry/review;
- Query/update analysis status;
- Standards/calibration information.

## 16.7 Data Storage and Retrieval

Data archival policies for hardcopy records are shown in Table 16-2. Task Leaders are responsible for retaining hardcopy laboratory records.

**Table 16-2 Data Archive Policies** 

Data Type	Medium	Location	Retention Time	Final Disposition
Laboratory notebooks	Hardcopy	Laboratory	5 years after close of contract	N/A
LIMS Database	Electronic (on-line)	Laboratory	Backup media after 5 years	Backup retained indefinitely

Project No. 0475.00
Element No. Section 16 – B10
Revision No. 2
Date August 2023
Page 126 of 155

All data are stored on the ERG LIMS server. This system has the following specifications:

• Operating System: Windows Server 2019 Standard

• Memory: 16G RAM

• Hard Drives: 1.5T hard drive space

 Security: Network login password protection on all workstations; Additional password protection applied by application software.

Security of the data in the database is ensured by the following controls:

- Password protection on the data base that defines three levels of access to the data;
- Logging of all incoming communication sessions, including the originating telephone number, the user's ID, and connect times; and
- Storage of media, including backup tapes, in an alternate location that is at a locked, restricted access area.

Project No. 0475.00
Element No. Section 17 - C1
Revision No. 2
Date August 2023
Page 127 of 155

## ASSESSMENT/OVERSIGHT

#### SECTION 17 ASSESSMENTS AND RESPONSE ACTIONS

An assessment is defined as an evaluation process used to measure the performance or effectiveness of the quality system or the establishment of the monitoring network and sites and various measurement phases of the data operation.

The results of QA assessments indicate whether the control efforts are adequate or need to be improved. Documentation of all QA and QC efforts implemented during the data collection, analysis, and reporting phases are important to data users, who can then consider the impact of these control efforts on the data quality. Both qualitative and quantitative assessments of the effectiveness of these control efforts will identify those areas most likely to impact the data quality. ERG will perform the following assessments to ensure the adequate performance of the quality system.

## 17.1 Assessment Activities and Project Planning

## 17.1.1 External Technical Systems and Data Quality Audits

A TSA is a thorough and systematic on-site qualitative audit, where facilities, equipment, personnel, training, procedures, subcontractor systems, and record keeping are examined for conformance to the QAPP. The TSAs will be performed by EPA, or its designee, at the ERG Laboratory. The TSAs for the contract are conducted approximately every 2 to 3 years. ERG will participate in any data quality audits by EPA or designee at the discretion of the EPA QA Coordinator.

The EPA audit team will prepare a brief written summary of findings for the Program Manager and Program QA Coordinator. Problems with specific areas will be discussed and an attempt made to rank them in order of their potential impact on data quality. ERG will work with EPA to determine the required corrective actions. As part of corrective action and follow-up, an audit finding response letter will be generated by the Program Manager and Program QA

Project No. 0475.00
Element No. Section 17 - C1
Revision No. 2
Date August 2023
Page 128 of 155

Coordinator. The audit finding response letter will address what actions are being implemented to correct the finding(s) of the TSA. EPA will determine if the proposed corrective actions are appropriate. The TSA findings summary from EPA and the following response from ERG are filed in the QA/QC file in Room 102. The findings and the follow-up corrective actions are discussed in the annual QA Management Systems Review.

As part of the ongoing accreditation under The National Environmental Laboratory

Accreditation Conference Institute (TNI), TSAs are performed at ERG through the Florida

Department of Health by an auditing contractor every two years. A summary of findings is sent to ERG, specifically the QA Coordinator. The QA Coordinator sends a response of corrective actions which is either accepted or denied by Florida Department of Health. This documentation is stored in the QA/QC file in Room 102. The findings and the follow-up corrective actions are discussed in the annual QA Management Systems Review.

## 17.1.2 Internal Technical Systems Audits

An internal TSA is performed examining facilities, equipment, personnel, training, procedures, and record keeping for conformance to the individual SOPs and this QAPP. The TSAs will be performed by the Program QA Coordinator and/or Deputy Program QA Coordinator and will be conducted at least once per year. The checklists for the internal TSAs are based on the NATTS TSA or the 2016 TNI Environmental Laboratories Standards Accreditation checklists with additional areas addressing the individual method SOPs and this QAPP. The content of the checklists vary episode to episode to ensure comprehensive in-depth coverage of procedures over time. Such elements will be included in the checklists:

- Criteria listed in Section 11 of this QAPP
- SOP specifications
- Method specifications
- Supporting equipment specifications
- Other laboratory wide QA systems in place (ex. Satellite SOP notebooks)

Project No. 0475.00
Element No. Section 17 - C1
Revision No. 2
Date August 2023
Page 129 of 155

The Program QA Coordinator will report internal audit findings to the Program Manager within 30 days of completion of the internal audit in the form of a report. The EPA Delivery Order Manager will be informed if issues from the internal audit impact the quality of this program. The report is filed in the QA/QC file in Room 102. All corrective actions are addressed and implemented as soon as they are determined, as applicable. The findings and the follow-up corrective actions are discussed in the annual QA Management Systems Review to assess effectiveness of the corrective actions.

## 17.1.3 Proficiency Testing

The PT is an assessment tool for the laboratory operations. 'Blind' samples are sent to the laboratory, where they follow the normal handling routines that any other sample follows. The results are sent to the Program Manager and Program QA Coordinator for final review and reporting to the auditing agency. The auditing agency prepares a PT report and sends a copy of the results to the Program Manager, Program QA Coordinator, and the EPA QA Office(s). Any results outside the acceptance criteria are noted in the PT report. Two repeated analyte failures in a row are investigated to determine the root cause and documented on a Corrective Action Report (CAR). If the determined root cause also affects samples, the affected analytes are flagged. The PT reports are filed in the QA/QC file in Room 102. The performance on these audits is discussed in the annual QA Management Systems Review.

There are three PT audit programs supported by this contract. The NATTS PT program provides PTs for carbonyl, metals, VOC, and PAH analyses. Currently, PAMS PTs for carbonyl analysis are satisfied with the NATTS carbonyl PT. The NAAQS audit program provides quarterly lead audit strips for the lead-only analysis. These PT audits are provided to ERG from EPA (or an EPA contractor) throughout the year. The acceptable limits are provided on the annual reports presented to the participating States and EPA.

ERG participates in round robin studies, such as Regional EPA round robin studies, when available for VOC, metals, carbonyls, and SNMOC. In these studies, each participating

Project No. 0475.00
Element No. Section 17 - C1
Revision No. 2
Date August 2023
Page 130 of 155

laboratory result is compared against the calculated average. Reports from these studies are kept in the QA/QC file in Room 102. The performance on these studies is discussed in the annual QA Management Systems Review.

## 17.1.4 Data Assessment for Final Report

A data quality assessment is the statistical analysis of environmental data to determine whether the quality of data is of adequate quality, based on the MQOs. The data assessment in the final report is presented to EPA and State agencies and includes the following:

- Review of the MQOs of the program, which includes completeness, precision, and accuracy.
- Present the results of the data quality assessment using summary statistics, plots and graphs while looking for and discussing any patterns, relationships, or anomalies.
- Qualify the data that does not meet the MQO for completeness for each monitoring site and for site-specific summary statistics.

## 17.2 Documentation of Assessments

## 17.2.1 TSA, Data Quality Audit, and PT Documentation

All reports from EPA or designated contractors regarding ERG's performance on TSAs, Data Quality Audits, and PTs are filed in the QA/QC file in Room 102. PT reports are dispersed and discussed with contributing staff.

Reports from internal TSAs are prepared and discussed with the contributing staff and Program Manager and filed in the QA/QC file in Room 102.

Project No. 0475.00
Element No. Section 17 - C1
Revision No. 2
Date August 2023
Page 131 of 155

#### 17.2.2 Internal Data Review Documentation

Internal data review is performed on 100 percent of the data by the Task Leader and 10 percent of the data by the Program QA Coordinator or designee against the criteria in the individual method SOPs and this QAPP prior to being reported each month. The assessment is documented on the data review checklist, which is returned to the Task Leader for minor correction action and inclusion in the data package. The checklists used for analyses are shown in their respective SOPs (Appendix D) as follows:

- VOC ERG-MOR-005, SOP for the Concurrent GC/FID/MS Analysis of Canister Air Toxic Samples using EPA Compendium Method TO-15 and EPA Ozone Precursor Method.
- Carbonyl ERG-MOR-024, SOP for Preparing, Extracting, and Analyzing DNPH
   Carbonyl Cartridges by Method TO-11A.
- SVOC/PAH ERG-MOR-049, SOP for Analysis of Semivolatile Organic Compounds (Polynuclear Aromatic Hydrocarbons) Using EPA Compendium Method TO-13A & ASTM D6209.
- Metals ERG-MOR-095, SOP for the Analysis of High-Volume Quartz, Glass Fiber
   Filters, and 47 mm Filters for Metals by ICP-MS using Method IO 3.5 and FEM
   Method EQL-0512-201 and FEM Method EQL-0512-202.
- Hexavalent chromium ERG-MOR-063, SOP for the Preparation and Analysis of Ambient Air for Hexavalent Chromium by Ion Chromatography.
- SNMOC ERG-MOR-005, SOP for the Concurrent GC/FID/MS Analysis of Canister Air Toxic Samples using EPA Compendium Method TO-15 and EPA Ozone Precursor Method.

During the internal data review, major QC problems identified are brought to the attention of the Program Manager and are documented on a CAR. The final project report also addresses QA considerations for the whole project.

Project No. 0475.00
Element No. Section 17 - C1
Revision No. 2
Date August 2023
Page 132 of 155

#### 17.3 Corrective Action

The CAR will be filed whenever a problem is found such as an operational problem, or a failure to comply with procedures that affects the quality of the data. A CAR is an important ongoing report to management because it documents primary QA activities and provides valuable records of QA actions. A CAR can be originated by anyone on the project but must be sent to the Program QA Coordinator and Program Manager for review and signature. Any problem that affects the quality of the overall program will be discussed with EPA.

On the numbered CAR, the description of the problem, the cause of the problem, the corrective action, and the follow-up are documented. CARs will be handled in a timely manner, with follow-up within 45 days. The follow-up assists the QA coordinator in determining if the corrective action was successful and if it was handled in a timely manner. The CAR is recorded on a form, the original copy goes into the QA file (Room 102), and if applicable, a copy goes into the data package. An example of the ERG CAR Form is shown in Figure 17-1.

The Program QA Coordinator will verify that the corrective action has been implemented. A summary of the past years' CARs is discussed during the annual QA Management Systems Review.

The following actions are taken by the laboratory QA Coordinator and Program Manager when any aspect of the testing work, or the results of this work, does not conform to the requirements of the quality system or testing methods:

- Identify nonconforming work and take actions, as needed, such as halting of work or withholding test reports;
- Evaluate of the impact of nonconforming work on quality and operations;
- Take remedial action and make decision about the acceptability of the nonconforming work (resample, use as is with qualification, or unable to use);
- Document in a CAR, as needed.

Project No. 0475.00
Element No. Section 17 - C1
Revision No. 2
Date August 2023
Page 133 of 155

ERG and its subcontractors are responsible for implementing the analytical phase of this program and are not responsible for the overall field site-specific program DQOs. Therefore, this QAPP tries to ensure that analytical results are of known and adequate quality to ensure the achievement of the various program DQOs.

Project No. 0475.00
Element No. Section 17 - C1
Revision No. 2
Date August 2023
Page 134 of 155

DE	RG
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CAR Number: Click or tap here to enter text.

Corrective Action Report Version 2021-01

CAR Initiator: Click or tap here to enter text. Initiation Date:

Area/Procedure Affected: Click or tap here to enter text.

Is Immediate Stop of Work Required? Choose an item.

#### Non-Conformance

Date of Discovery:

Description of Non-Conformance: What happened? How is this a non-conforming event?

Click or tap here to enter text.

Investigation of Non-Conformance: How was the non-conformance discovered?

Click or tap here to enter text.

Impact Assessment: What is affected by the nonconformance? If no impact, provide documentation to that proves this to be the case whenever possible.

Click or tap here to enter text.

Root Cause Analysis: What caused the nonconformance?

Click or tap here to enter text.

Further Analysis: Could this nonconformance be evident in other areas? Are other CARs related to this nonconformance?

Click or tap here to enter text.

#### Corrective Action

Due Date for Remedial Action Completion:

Immediate and/or Long-Term Remedial Corrective Actions Taken: Include both successful and unsuccessful corrective actions used.

Click or tap here to enter text.

Assessment of Corrective Action Effectiveness: This section may be left blank if follow-up is required.

Click or tap here to enter text.

	Signatures	
	Signature & Date	Comments
QA Officer:		Click or tap here to enter text.
Project Manager:		Click or tap here to enter text.
Initiator:		Click or tap here to enter text.
2021-01 Revision		Page 1 of 2

Figure 17-1 ERG Corrective Action Report Form, Page 1

Project No. 0475.00
Element No. Section 17 - C1
Revision No. 2
Date August 2023
Page 135 of 155

CAR Number: Click or tap here to enter text. Follow-up If applicable, reference or attach documentation that demonstrates the return to conformance or Click or tap here to enter text. Follow-up Auditor: Click or tap here to enter text. Date Completed: Were corrective action procedures effective? Click or tap here to enter text. 2021-01 Revision Page 2 of 2

Figure 17-1 ERG Corrective Action Report Form, Continued

Project No. 0475.00
Element No. Section 18 - C2
Revision No. 2
Date August 2023
Page 136 of 155

#### **SECTION 18 REPORTS TO MANAGEMENT**

This section describes the quality-related reports and communications to management necessary to support monitoring network operations and the associated data acquisition, validation, assessment, and reporting. Important benefits of regular monthly reports to EPA provide the opportunity to alert of data quality problems, to propose viable solutions to problems, and to procure necessary additional resources.

Effective communication among all personnel is an integral part of a quality system. Regular, planned quality reporting provides a means for tracking the following:

- Adherence to scheduled delivery of data and reports;
- Documentation of deviations from approved QA and test plans, and the impact of these deviations on data quality; and
- Analysis of the potential uncertainties in decisions based on the data.

## 18.1 Frequency, Content, and Distribution of Reports

Frequency, content, and distribution of reports for monitoring are shown below.

## 18.1.1 Monthly and Annual Reports

Analytical data reports prepared by the Program or Deputy Program Manager are sent to EPA, State, Local and Tribal agencies monthly. These reports include the analytical data for each sample collected monthly including sample results, MDLs, sample information (canister ID, sample volume, etc.) and a QA report (could include duplicates, MB, CCB, CCV, MS/MSD, etc., depending on the analysis). Quarterly QA reports are distributed which include a summary of analyte specific quality control charts (ICV, ICB, CCB, CCV, BLK, BS/BSD, etc.). An annual data report, containing a summary of the yearly assessment of the air toxics data, is reported to EPA and State agencies by the Program Manager after the conclusion of a sampling year via a QlikSense application (app) housed on EPA's server. This app provides some statistical analysis

Project No. 0475.00
Element No. Section 18 - C2
Revision No. 2
Date August 2023
Page 137 of 155

and quality assessments for the measurement data and, in some cases, whether the data objectives for the program were met.

The annual report app includes the quality information for each participating air toxics monitoring site. The app includes a Program and update and select Method Quality Objectives for measurement data and associated data quality assessment:

- Completeness determinations;
- Estimates for method and analytical precision and bias; and
- PTs that were performed during the sampling year, if applicable.

## 18.1.2 Internal Technical System Audit Reports

The Program QA Coordinator or designee performs an internal technical system audit at least once a year for the monitoring network for EPA, State, and NATTS contracts. The findings are listed in reports which are presented to the Program Manager and filed in the QA/QC storage file cabinet located in Room 102. These reports are available to EPA personnel during their TSA. More detail on internal TSAs is provided in Section 17.

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 138 of 155

#### DATA VALIDATION AND USABILITY

#### SECTION 19 DATA REVIEW AND VERIFICATION

Data review and verification are performed to determine if the sampling and analytical data collection process is complete, is consistent with the DQOs discussed in this QAPP and associated method SOPs, and meets the program requirements. The data are reviewed for accuracy by checking for transcription, calculation, and reduction errors. The analyst is responsible for reviewing the data before it goes to the Task Leader. The Task Leader is responsible for reviewing the data before it goes to the QA staff. The data reviews use the QA review checklist located in individual analytical SOPs. The data is also verified to be complete and accepted, meeting the quality requirements of the program. Designees trained to review analytical data may be assigned for any level of review, however that designee must not have performed the previous data review task(s).

## 19.1 Data Review Design

The analysts, Task Leaders, and QA staff review the data prior to reporting. Information used to perform these reviews, include, but are not limited to:

- Sample COCs, holding times, preservation methods.
- Multi-point calibrations the multipoint calibrations are used to establish proper initial calibration and can be used to show changes in instrument response.
- Standards certifications, identification, expiration dates.
- Instrument logs all activities and samples analyzed are entered into the LIMS logs (batches, sequences, etc.) to track the samples throughout the measurement procedures.
- Supporting equipment identification, certifications, calibration, if needed.
- Blank, CCVs, replicate and spike results these QC indicators can be used to ascertain whether sample handling or analysis is causing bias in the data set.
- Review Checklists these record data quality reviews performed on all data by Task

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 139 of 155

Leader and on 10 percent of the data by the QA Coordinator or designee. The checklists used to review data is presented in the method SOPs.

• Summary Reports – monthly summary data reports present the preliminary data to EPA and respective State/Local/Tribal representatives including data qualifiers.

#### 19.2 Data Review and Verification

Prior to reporting, the data are reviewed by analyst, Task Leader, and QA staff for accuracy, starting with checking the COC forms to ensure accurate transcription. Other records are examined, such as notes of unusual circumstances during analysis (e.g., power loss or fluctuations, temporary leaks or adjustments, operator error) on the LIMS bench sheets, etc. The data are scrutinized daily to ensure the collection of valid data. Any issues identified in the QA staff review are reported by the QA staff to the Task Leader or analyst as applicable for resolution.

QC samples and procedures performed during the monitoring program are checked against those described in Section 11 of the QAPP. If QC is found unacceptable, corrective actions are implemented (as described in the same section). Prior to reporting, 100 percent of the data is reviewed by the Task Leader(s). To verify accuracy, at least 10 percent of the data is checked by the QA Coordinator or designated reviewer. Items checked can include required QC, original raw data, COCs, checks of all calculations (from calibration to sample analysis), and data transfers. As the data are checked, corrections are made to the database as errors or omissions are encountered. If major errors are found in the data review process, a greater percent of the data is checked to verify data quality.

The data are also critically reviewed to locate and isolate spurious values. A spurious value, when located, is not immediately rejected. All questionable data, whether rejected or not, are maintained along with rejection criteria and any possible explanation. Such a detailed approach can be time-consuming but can also be helpful in identifying sources of error and, in the long run, save time by reducing the number of outliers.

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 140 of 155

The analytical procedures performed during the monitoring program will be checked against those described in the QAPP and the SOPs for the UATMP, PAMS, and NMOC support included in Appendix D. Deviations from the QAPP will be classified as acceptable or unacceptable, and critical or noncritical. During review and assessment, qualifiers will be applied to the data as needed; data found to have critical flaws (such as low spike for surrogate recoveries, contaminated blanks, etc.) will be invalidated and a CAR filled out and implemented, if needed.

## 19.3 Data Reporting

The Program Manager reviews all data before they are reported to EPA and/or the State/Local/Tribal agencies. ERG prepares a final report containing all aspects of the individual programs including data summaries, QA, QC, and data analysis results for EPA, and distributes site-specific summaries of the final data to designated personnel.

Monthly site-specific data reports for the NHAPS program are distributed to the participating EPA technical staff, administrators, and to the administrators of the State/Local/Tribal agencies involved in the study. NATTS, CSATAM, and UATMP data consists of any toxics including VOC, SNMOC, carbonyl, or other HAPs (metals, semivolatiles, etc.) requested by the program participants. Each report is prepared after 45 days from the end of the sampling month. Cumulative listings are periodically generated upon request. This timely turnaround of data assists in planning, preliminary modeling, and program development for the participating State/Local/Tribal agencies. Any changes made in the preliminary data because of subsequent data validation processes performed by EPA and/or State/Local/Tribal agencies are noted in the separate data reports for each specific sampling site. The data reports include:

- Site code;
- Sample identifications;
- Sample dates;
- Target compound list;
- Concentrations (ppbV, ppbC, ng/m³ and/or μg/m³); and

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 141 of 155

#### Method detection limits.

Values below the MDL for QC sample results in the Adobe reports are recorded as not detected (ND), however the actual values are reported in the accompanying Excel report with a "U" flag (Under the detection limit). Preliminary monthly data reports are emailed to the program participants. These data reports are considered preliminary until the data is validated and entered into the AQS database, as detailed in Section 19.5.

#### 19.4 Data Validation

Data validation is confirmed by examination of objective evidence that the requirements for a specific intended use are fulfilled as presented in Section 4. Intended use deals with data of acceptable quality to permit making decisions at the correct level of confidence. Ongoing data review and adherence to the data quality objectives keeps the data quality consistent, while data validation ensures the data quality. This data validation is performed prior to inclusion in the QlickSense® app by the AQS Task Leader or designee. Any anomalies are conveyed to the appropriate Task Leader for investigation.

To assess precision, a CV is calculated for each site and compound using the equation in Table 16-1, and across each method. Method precision is based on measurements from the collection and analysis of duplicate (inter-system) and collocated (intra-system) samples while analytical precision is based on replicate analysis only. The overall CVs for each method are compared against the precision DQO for the NATTS program and provided in the QlickSense® app.

As discussed in Section 11.6, ERG is not responsible for the sampling design; therefore, representativeness is beyond the scope of this QAPP. Comparability is based on the measure of confidence with one data set compared to another. Ongoing data review and adherence to the data quality objectives keeps the data quality consistent and therefore comparable over the project. Comparability is an ongoing data quality review followed by a data assessment prior to inclusion in the QlikSense® app.

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 142 of 155

Completeness is determined by comparing the number of valid samples reported to the number of valid samples expected. The number of expected samples is determined by counting the number of scheduled samples based on the sampling calendar for that site. Eighty-five percent completeness is the DQO/MQO for all programs. This is an ongoing assessment used to facilitate make-up sampling in the same quarter when possible.

To ensure that the data is reliable in the ranges of concern, the minimum detection limit targets are those specified for the NATTS program, even though the other programs may be less stringent. This is an ongoing assessment since detection limits are determined annually.

#### 19.4.1 PAMS Data Validation

To process and validate PAMS Auto-GC data, data will be handled in the following manner. The SOP for PAMS autoGC data validation, ERG-MOR-112, is listed Appendix D.

- Received data will be imported into a database and queries will be run to
  quantitatively perform the screening checks identified in Table 10-1 of the PAMS
  TAD. An initial summary of data failing screening checks will be provided to the
  PAMS Site.
- Data will be pulled into a customized QlikSense app and so that data can be
  reviewed using pre-populated data visualizations (e.g., time series, bar charts, etc.).
  Any questionable data identified during this review will also be summarized and
  provided to the PAMS site. The QlikSense app will be made available to the PAMS
  Site such that the app can support their own data review processes.
- Data will be revised per the PAMS Site instruction. Steps 1-3 may be repeated based on additional feedback and collaboration.
- Finalized data will be coded for AQS and undergo the same quality assurance steps outlined in Section 19.5.

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 143 of 155

## 19.5 Air Quality System

ERG submits data collected for the NMOC, UATMP, NATTS, CSATAM, PAMS, and other air toxics programs to the AQS database.

Prior to ERG's submittal of data to AQS, the State/Local/Tribal agency submits, at a minimum, Basic Site Information transactions (Type AA) for each sampling site, and transaction Types AB through AE, if necessary, to the AQS database. ERG then submits monitor transactions (Types MA through MX, as applicable) to prepare the AQS database for data upload. Data that are uploaded into AQS include Raw Data transactions (Type RD), QA transactions (Type Duplicate, Replicate, PAMS Carbonyl sampler audits, and Pb Analysis Audit) and Blank transactions (Type RB). ERG follows the NATTS<sup>(22)</sup> and PAMS<sup>(2)</sup> TADs to code data for the AQS database.

The submittal process involves the following steps:

- The raw data are formatted into pipe-delimited ( | ) coding that is accepted by AQS.
   Raw data, data generated by single sample episodes, by the primary sample (D1) of a duplicate episode, or by collocates (C1 and C2), are submitted using RD transactions.
- Precision data, data generated by Duplicate and Replicate samples (R1, D2, and/or R2), are submitted using QA transactions, specifically Duplicate and Replicate transactions. Accuracy data, generated for lead-FEM audit results, are also submitted using QA transactions.
- Blank data are submitted using RB transactions, specifically for field blanks, trip
  blanks, and lot blanks. Per the NATTS TAD, lot blank data will be submitted as the
  average lot blank concentration per analyte for each lot of cartridges (or other
  media) for all monitoring sites supported by ERG. For analytes that exhibit both
  numerical and non-detect values in lot blanks, zero will be substituted for nondetects to determine the average concentration.

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 144 of 155

- The RD QA (specifically Duplicate, Replicate and Pb Analysis Audit), and RB coding are generated and reviewed following procedures specified in the SOP for the Preparation of Monitoring Data for AQS Upload (ERG-MOR-098) to ensure that the monitor ID (including state, county, site, parameter, and Parameter Occurrence Code (POC) codes), sampling duration, units, method, sample date, start time, and sample values, and MDLs are correct. The transactions are stored as text files for upload into the AQS database.
- Transaction files are primarily loaded under the Monitoring and Quality Assurance screening group.
- Transactions are edited to correct any errors found by AQS and then resubmitted.
   This step is repeated until the transactions are free of errors.
- AQS performs a statistical check on the data submitted to validate the data and determines if there are any outliers based on past data.
- Raw data (RD) transactions are then posted into the AQS database.

## 19.5.1 AQS Flagging and Reporting

Air toxics data submittals may be submitted to AQS with flags to indicate additional information related to the sample. There are two qualifier flag types that may be applied: Null codes and Qualifier codes.

- Null Code assigned when a scheduled sample is not usable (e.g., canister leaked, sample damaged in shipment, etc.).
- Qualifier Code used to note a procedural or quality assurance issue that could possibly affect the concentration value or the uncertainty of the result. These flags can also be applied to indicate atypical field conditions (e.g., nearby fires, construction in the area).

Qualifier Codes can be used in combination, with up to 10 possible codes applied. If a Null code is used, no other flag is normally used since no results are reported. Table 19-1 presents the

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 145 of 155

Qualifier codes and Table 19-2 presents the Null codes available to AQS users, however more flags are listed on the AQS website. These flags are applicable to the various steps of sample collection and analysis such as field operations, chain of custody, and laboratory operations.

Blank issue flags are qualifier flags used if reported blank values are above the limits set by the method SOPs or QAPP. If high blank values are associated with samples, the sample values are reported but appropriately flagged as described in the NATTS TAD<sup>(22)</sup>. Samples will not be invalidated due to high blank values. Blank issue flags are included in Table 19-1.

**Table 19-1 Qualifier Codes** 

Qualifier Code	Qualifier Description			
1	Deviation from a CFR/Critical Criteria Requirement			
1V	Data reviewed and validated			
2	Operational Deviation			
3	Field Issue			
4	Lab Issue			
5	Outlier			
6	QAPP Issue			
7	Below Lowest Calibration Level			
9	Negative value detected - zero reported			
СВ	Values have been Blank Corrected			
CC	Clean Canister Residue			
CF	Canister Bias: NATTS/UATMP Data for compounds that have failed certification for the			
	canister.			
CL	Surrogate Recoveries Outside Control Limits			
DI	Sample was diluted for analysis			
DN	DNPH peak less than NATTS TAD requirement, reported value should be considered an estimate			
EH	Estimated; Exceeds Upper Range			
FB	Field Blank Value Above Acceptable Limit			
FX	Filter Integrity Issue			
HT	Sample pick-up hold time exceeded			
IA	African Dust			
IB	Asian Dust			
IC	Chemical Spills & Industrial Accidents			
ID	Cleanup After a Major Disaster			
IE	Demolition			
IF	Fire – Canadian			
IG	Fire - Mexico/Central America			
IH	Fireworks			
II	High Pollen Count			
IJ	High Winds			

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 146 of 155

# **Table 19-1 Qualifier Codes**

Qualifier Code	Qualifier Description			
IK	Infrequent Large Gatherings			
IL	Other			
IM	Prescribed Fire			
IN	Seismic Activity			
10	Stratospheric Ozone Intrusion			
IP	Structural Fire			
IQ	Terrorist Act			
IR	Unique Traffic Disruption			
IS	Volcanic Eruptions			
IT	Wildfire-U. S.			
J	Construction			
LB	Lab blank value above acceptable limit			
П	Identification of Analyte Is Acceptable; Reported Value Is an Estimate			
LK	Analyte Identified; Reported Value May Be Biased High			
LL	Analyte Identified; Reported Value May Be Biased Low			
MD	Value less than MDL			
MS	Value reported is ½ MDL substituted			
MX	Matrix Effect			
ND	No Value Detected, Zero Reported			
NS	Influenced by nearby source			
PQ	Value Between PQL and MDL			
QP	Pressure Sensor Questionable			
QT	Temperature Sensor Questionable			
QX	Does not meet QC criteria			
SB	Sampler Bias: NATTS/UATMP Data for compounds that have failed certification for the sampler			
SP	NATTS/UATMP data with Spike Recovery outside acceptance limits			
SQ	Values Between SQL and MDL			
SS	Value substituted from secondary monitor			
SX	Does Not Meet Siting Criteria			
Т	Multiple PM2.5 Validity Flags			
ТВ	Trip Blank Value Above Acceptable Limit			
TT	Transport Temperature is Out of Specs			
V	Validated Value			
VB	Value below normal; no reason to invalidate			
W	Flow Rate Average out of Spec.			
Х	Filter Temperature Difference or Average out of Spec.			
Υ	Elapsed Sample Time out of Spec.			

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 147 of 155

## Table 19-2 Null Codes

Null Code	Qualifier Description				
1C	A 1-Point QC check exceeds acceptance criteria but there is compelling evidence that the				
	analyzer data is valid				
IF	No 1 Point QC but need to count for completeness				
AA	Sample Pressure out of Limits				
AB	Technician Unavailable				
AC	Construction/Repairs in Area				
AD	Shelter Storm Damage				
AE	Shelter Temperature Outside Limits				
AF	Scheduled but not Collected				
AG	Sample Time out of Limits				
AH	Sample Flow Rate or CV out of Limits				
AI	Insufficient Data (cannot calculate)				
AJ	Filter Damage				
AK	Filter Leak				
AL	Voided by Operator				
AM	Miscellaneous Void				
AN	Machine Malfunction				
AO	Bad Weather				
AP	Vandalism				
AQ	Collection Error				
AR	Lab Error				
AS	Poor Quality Assurance Results				
AT	Calibration				
AU	Monitoring Waived				
AV	Power Failure				
AW	Wildlife Damage				
AX	Precision Check				
AY	QC Control Points (zero/span)				
AZ	QC Audit				
ВА	Maintenance/Routine Repairs				
ВВ	Unable to Reach Site				
ВС	Multi-point Calibration				
BD	Auto Calibration				
BE	Building/Site Repair				
BF	Precision/Zero/Span				
BG	Missing ozone data not likely to exceed level of standard				
ВН	Interference/co-elution/misidentification				
BI	Lost or damaged in transit				
BJ	Operator Error				
ВК	Site computer/data logger down				
BL	QA Audit				
BM	Accuracy check				
BN	Sample Value Exceeds Media Limit				
BR	Sample Value Below Acceptable Range				
CS	Laboratory Calibration Standard				

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 148 of 155

Table 19-2 Null Codes

Null Code	Qualifier Description
DA	Aberrant Data (Corrupt Files, Aberrant Chromatography, Spikes, Shifts)
DL	Detection Limit Analyses
EC	Exceeds Critical Criteria
FI	Filter Inspection Flag
MB	Method Blank (Analytical)
MC	Module End Cap Missing
QV	Quality Control Multi-point Verification
SA	Storm Approaching
SC	Sampler Contamination
ST	Calibration Verification Standard
SV	Sample Volume out of Limits
TC	Component Check & Retention Time Standard
TS	Holding Time or Transport Temperature Is Out Of Specs.
XX	Experimental Data

ERG submits data to AQS using qualifier flags to show where the data are with respect to the detection level. A variety of terms and acronyms are used for defining the lowest level that can be detected for each analytical method. These terms and applications are derived from EPA's TAD for the NATTS program and are presented below:

- Quantitation Limits (QL) the lowest level at which the entire analytical system
   must provide a recognizable signal and acceptable calibration point for the analyte.
- Detection Limits (DL) the minimum concentration of an analyte that can be measured above instrument background.
- MDL the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in each matrix containing the analyte (Part 136, App. B).
- SQL the lowest concentration of an analyte reliably measured within specified limits of precision and accuracy during routine laboratory operating conditions. Per the NATTS TAD, the SQL is a multiplier of the method detection limit (3.18 times the MDL) and is considered the lowest concentration that can be accurately measured, as opposed to just detected.

Project No. 0475.00
Element No. Section 19 - D1
Revision No. 2
Date August 2023
Page 149 of 155

The qualifier flags associated with quantitation and detection limits are also included in Table 19-1, while Table 19-3 summarizes how they are applied to the data.

**Table 19-3 Summary of Quantitation and Detection Limit Flags and Applications** 

If Concentration is:	Value to Report	Flag Applied
≥ SQL	Value	None
≥ MDL and < SQL	Value	SQ
< MDL	Value	MD
Not Detected	0	ND

Project No. 0475.00
Element No. Section 20 - D2
Revision No. 2
Date August 2023
Page 150 of 155

## **SECTION 20 DATA VALIDATION, VERIFICATION METHODS**

Many of the processes for verifying and validating the measurement phases of the data collection operation have previously been discussed in Section 19. If these processes are followed, and the sites are representative of the boundary conditions for which they were selected, one would expect to achieve the DQOs. However, exceptional field events may occur, and field and laboratory activities may negatively affect the integrity of samples. In addition, it is expected that some of the QC checks will fail to meet the acceptance criteria. This section will outline how ERG will take the data to a higher level of quality analysis by performing software tests, plotting, and other methods of analysis. Verification and validation procedures are described in more detail in the SOP for the Preparation of Monitoring Data for AQS Upload (ERG-MOR-098). Data resolutions will be brought to the attention of the Program Manager who will contact the sites to resolve any issues.

## 20.1 Process for Validating and Verifying Data

#### 20.1.1 Verification of Data

For the analytical data, the entries are reviewed to reduce the possibility of entry and transcription errors. Once the data are transferred to the ERG LIMS database, the data will be reviewed for routine data outliers and data outside acceptance criteria. These data will be flagged appropriately. Prior to reporting, 100 percent of the data is reviewed by the Task Leaders and 10 percent of the database is checked by the QA Coordinator or designated reviewer via the review checklist. The PM also reviews the data prior to the preliminary report. After a preliminary reporting batch is completed, a review of 10 percent of the data will be conducted for completeness and manual and electronic data entry accuracy by the Annual Report/AQS Task Leader.

Project No. 0475.00
Element No. Section 20 - D2
Revision No. 2
Date August 2023
Page 151 of 155

#### 20.1.2 Validation of Data

Data validation is performed by examination of objective evidence that the requirements for a specific intended use are fulfilled as presented in Section 4. Data is examined for comparability, completeness, precision, and bias. This data validation, some of it performed with summary statistical analysis, is performed prior to the annual final report. Data validation is discussed in more detail in Section 19.5.

## 20.2 Data Analysis

Data analysis refers to the process of interpreting the data that are collected. Although there are a large number of parameters to analyze, many of these parameters present similar characteristics, (i.e., VOC, SVOC, and particulate metals, grouped according to their physical and chemical properties).

ERG will employ software programs, described below, to help analyze the data.

- Spreadsheet Select ERG employees perform analysis on the data sets using Excel® spreadsheets (analysts, Task Leaders, QA reviewers, and AQS Task Leader) and Access® databases (AQS data entry). Spreadsheets and databases allow the user to input data and statistically analyze, graph linear data. This type of analysis will allow the user to see if there are any variations in the data sets. In addition, various statistical tests such as tests for linearity, slope, intercept, or correlation coefficient can be generated between two strings of data.
- QlikSense The AQS Task Leader performs time series plots, compound-specific plots, and control charts can help identify the following trends:
  - Large jumps or dips in concentrations;
  - o Periodicity of peaks within a month or quarter; and
  - Expected or unexpected relationships among species.

Project No. 0475.00
Element No. Section 21 - D3
Revision No. 2
Date August 2023
Page 152 of 155

## **SECTION 21 RECONCILIATION WITH DATA QUALITY OBJECTIVES**

The project management team, QA Coordinator, and sampling and analytical team members are responsible for ensuring that all measurement procedures are followed as specified and that measurements data meet the prescribed acceptance criteria. Prompt action is taken to correct any problem that may arise.

## 21.1 Conduct Preliminary Data Review

A preliminary data review will be performed as discussed in Sections 17 and 19 to uncover potential limitations to using the data, to reveal outliers, and generally to explore the basic structure of the data. The next step is to calculate basic summary statistics, generate graphical presentations of the data, and review these summary statistics and graphs to determine if the program requirements in Section 4, representativeness, comparability, completeness, precision, bias, and sensitivity, were met. These steps are discussed in more detail in Section 19.4. Comparability is based on method measure of the level of confidence with which one data set can be compared to another. Completeness is measured by the amount of valid sample data obtained compared to what was expected. Precision is determined from replicate analyses for a given method. Laboratory bias is demonstrated through PT samples and second source standards. Sensitivity is demonstrated through minimum detection limits.

#### 21.2 Draw Conclusions from the Data

If the sampling design and statistical tests conducted during the final reporting process show results that meet acceptance criteria, it can be assumed that the network design and the uncertainty of the data are acceptable. This conclusion can then be reported to EPA and the States/Local/Tribal agencies, who then decide whether to perform risk assessments and analyze the data to determine whether these data can be used to address health effects.

Project No. 0475.00
Revision No. 2
Date August 2023
Page 153 of 155

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Project No. 0475.00
Revision No. 2
Date August 2023
Page 154 of 155

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Project No. 0475.00
Revision No. 2
Date August 2023
Page 155 of 155

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# ERG EXEMPTION FROM THE NATTS TAD, REVISION 4

	NATTS	NATTS			
	TAD Rev 4	TAD Rev 3	QC		EPA
Analyte	Reference	Reference	Parameter	ERG Exception	Approval/Decision
PAH	4.5.5.2	new exemption	Working standards and open ampules of stock standards must be stored protected from light at ≤ -10°C in amber vials with PTFE-lined lids in a storage unit separate from sampled cartridges and sample extracts.	ERG would like to store standards according to manufacturers' recommendations. It should be noted that some TO-13A compounds crystallize in standard solutions at freezer temperatures, potentially causing inadequate transfer of those compounds.	Approved. We agree standards should be stored in accordance with the manufacturer's recommendations.



# 2023 6-Day Sampling Calendar

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M Makeup Duplicate Collection

Standard Sample Collection

FB Field Blank Collection

or normal sample

# 2023 3-Day PAMS Sampling Calendar (with 1 duplicates 2 FB)

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# Standard Sample Collection

FB Field Blank Collection

# 2024 6-Day Sampling Calendar

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Standard Sample Collection

FB Field Blank Collection

Makeup Duplicate Collection or normal sample

# 2024 3-Day PAMS Sampling Calendar (with 1 duplicates 2 FB)

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25	26	27	FB	29	30	31

# Standard Sample Collection

FB Field Blank Collection



#### ERG Changes/Comments for 2023/2024 QAPP

Listed are the major changes to the 2023 QAPP to prepare the 2023/2024 QAPP. The other changes are insignificant and editorial in nature.

#### Section 1

- Updated Figure 1-1.
- Noted Table 1-1 changes in Shipping and Receiving Task leads.

#### Section 7

• Added sampler certification blank check criteria to Section 7.2.

#### Section 9

• Updated the canister cleaning batch blank criteria and required number of batch blanks per batch of cleaned canisters.

#### Section 10

• Added metals sample filter fold technique in Section 10.5.

#### Section 11

- Updated canister blank certification procedure in Section 11.1.
- Any changed procedures adopted from TAD Revision 4 are updated in Tables 11-2 through 11-5, and Table 11-8.

#### Section 13

Updated Section 13.2 to include integrity checks for gas standards from NATTS TAD,
 Revision 4.

## Exemptions Table - Appendix A

• Updated the ERG exemption to the NATTS TAD, Revision 4.

#### 2023/2024 Sampler Calendar – Appendix B

- Added 2024 sampling calendar.
- Added 2024 3-month PAMS calendar that includes 2 Field Blanks and 1 Duplicate sample per month.

Appendix D Relevant ERG	Standard Operating Procedures	
The information contained herein is confidential a without the express written	nd proprietary and may not be used in any manner or form permission of the Program Manager.	
The information contained herein is confidential a without the express written	nd proprietary and may not be used in any manner or form permission of the Program Manager.	
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The information contained herein is confidential a without the express written	nd proprietary and may not be used in any manner or form permission of the Program Manager.	

ERG-MOR-003D	Field Procedure for Collecting Ambient Air Toxics and Carbonyl Compounds Samples Using the ERG:AT/C Sampling System (with O3 Denuder Scrubber and Mass Flow Meter)
ERG-MOR-005	Standard Operating Procedure for the Concurrent GC/FID/MS Analysis of Canister Air Toxic Samples using EPA Compendium Method TO-15 and EPA Ozone Precursor Method
ERG-MOR-013	Field Procedure for Collecting Ambient Air Hexavalent Chromium Samples Using the ERG:CR6 Sampling System
ERG-MOR-017	Standard Operating Procedure for Developing, Documenting, and Evaluating the Accuracy of Spreadsheet Data
ERG-MOR-022	Standard Operating Procedure for the Preparation of Standards in the ERG Laboratory
ERG-MOR-024	Standard Operating Procedure for Preparing, Extracting, and Analyzing DNPH Carbonyl Cartridges by Method TO-11A
ERG-MOR-030	Standard Operating Procedure for Canister Sampling System Certification Procedures
ERG-MOR-033	Standard Operating Procedure for Hazardous Waste
ERG-MOR-039	Standard Operating Procedure for Maintaining Laboratory Notebooks
ERG-MOR-044*	Standard Operating Procedure for Method 8270C – GC/MS Analysis of Semivolatile Organics
ERG-MOR-045	Standard Operating Procedure for Sample Receipt at the ERG Chemistry Laboratory
ERG-MOR-046*	Field Procedure for Collecting Speciated and/or Total Nonmethane Organic Compounds Ambient Air Samples Using the ERG:S/NMOC Sampling System
ERG-MOR-047B	Field Procedure for Collecting Ambient Carbonyl Compounds Samples Using the ERG:C Sampling System
ERG-MOR-049	Standard Operating Procedure for analysis of Semivolatile Organic Compounds (Polynuclear Aromatic Hydrocarbons) Using EPA Compendium Method TO-13A & ASTM D 6209-13
ERG-MOR-057	Standard Operating Procedure for Project Peer Review
ERG-MOR-060	Standard Operating Procedure for PDFID Sample Analysis by Method TO- 12
ERG-MOR-061	Standard Operating Procedure for Standard Preparation Using Dynamic Flow Dilution System
ERG-MOR-062	Standard Operating Procedure for Sample Canister Cleaning
ERG-MOR-063	Standard Operating Procedure for the Preparation and Analysis of Ambient Air for Hexavalent Chromium by Ion Chromatography

ERG-MOR-079	Standard Operating Procedure for Sample Login to the Laboratory Information Management System
ERG-MOR-084	Standard Operating Procedure for the Preparation and Extraction of High Volume Quartz and Glass Fiber Filters for Metals by ICP-MS using Method IO 3.1 and FEM Method EQL-0512-201
ERG-MOR-085	Standard Operating Procedure for the Preparation and Extraction of 47mm Filters for Metals by ICP-MS using Method IO 3.1 and FEM Method EQL-0512-202
ERG-MOR-095	Standard Operating Procedure for the Analysis of High Volume Quartz, Glass Fiber Filters, and 47mm Filters for Metals by ICP-MS using Method IO-3.5, FEM Method EQL-0512-201, and FEM Method EQL-0512-202
ERG-MOR-097	Standard Operating Procedure for Manual Integration of Chromatographic Peaks
ERG-MOR-098	Standard Operating Procedure for the Preparation of Monitoring Data for AQS Upload
ERG-MOR-099	Standard Operating Procedure for the Laboratory Information Management System
ERG-MOR-100	Standard Operating Procedure for Carbonyl System Certification
ERG-MOR-105	Standard Operating Procedure for Sample Canister Cleaning using the Wasson TO-Clean Automated System
ERG-MOR-108	Standard Operating Procedure for the Calibration and Verification of Support Equipment and Reference Standards
ERG-MOR-110	Standard Operating Procedure for the Management of Canister Certification and Inventory
ERG-MOR-111	Standard Operating Procedure for Corrective Action Reports
ERG-MOR-113	Standard Operating Procedure for PAMS AutoGC Data Validation
*These SOPs are not curr	rent because they are not in need. Once EPA/State/Local or Tribal agency requests this

<sup>\*</sup>These SOPs are not current because they are not in need. Once EPA/State/Local or Tribal agency requests this work, the SOP will be updated and provided to the EPA before work begins.

# **Appendix E Subcontractors Quality Assurance Project Plan RTI Laboratories** Will be provided when work is initiated. The information contained herein is confidential and proprietary and may not be used in any manner or form without the express written permission of the Program Manager.



# ERG PROPRIETARY INFORMATION

**Standard Operating Procedure** 

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 1 of 26

# **ENGINEERING AND SCIENCE DIVISION**

Standard Operating Procedure for th Extraction of High Volume Quartz Fibe by ICP-MS using modified Method IO	er Filters for Metals JAN 3 1 2023
ERG-MOR-099, ERG-MOR-108, ERG	G-MOR-045, ERG-MOR-010, ERG-MOR-095, Health and Safety Manual, EPA Compendium Document for the National Air Toxics Trends
SATELLITE FILES: ICP-MS Laboratory	
[ · ·	NATTS TAD revision; Added LOQ subsection 14 and 16; Minor edits based on Revision 4 of Quartz Extraction Record
WRITER/EDITOR: NAME/DATE  Brothi Claud 1-30-23	PROJECT MANAGER/TECHNICAL DIRECTOR: NAME/DATE  July 1 31 23
QUALITY ASSURANCE COORDINATOR: NAME/DATE  Dom Telle 1/30/23	NEXT SCHEDULED REVIEW: March, 2024



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 2 of 26

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# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 3 of 26

#### 1.0 IDENTIFICATION AND PURPOSE

This standard operating procedure (SOP) provides the sample preparation and extraction procedure for suspended particulate matter collected on quartz filters (PM<sub>10</sub> or TSP) for total metals determination by Inductively Coupled Plasma - Mass Spectrometer (ICP-MS).

#### 2.0 MATRIX OR MATRICES

This procedure applies to the preparation of ambient particulate matter samples collected with quartz filters ( $PM_{10}$  or TSP) for total metals.

#### 3.0 METHOD DETECTION LIMIT

Method detection limit procedures for Metals are discussed in SOPs ERG-MOR-095 and ERG-MOR-010.

#### 4.0 SCOPE AND APPLICATION

## 4.1 Scope

This procedure details the acid extraction of ambient air samples for analysis using an inductively coupled plasma-mass spectrometer (ICP-MS). The extraction procedures are suitable for high-volume ambient air samples collected on quartz membrane filters, sized 8 x 10". Different size filters of these matrices (e.g. circular) can be extracted using this procedure but caution needs to be exercised during extraction and calculating final concentrations.

# 4.2 Applicability

This SOP is applicable to the extraction of suspended particulate matter collected with quartz filters. Acid digestion and filtration is required prior to analysis of quartz filter extracts. The procedure is applicable to, but not limited to, the 28 metals listed below. Eleven of these elements are target metals for EPAs Urban Air Toxics Monitoring Program (UATMP) and ten for the National Air Toxics Trends Station (NATTS) program. Analytes for which ERG has demonstrated the acceptability of this method are listed below.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 4 of 26

Element	Symbol	CASRN
Aluminum	Al	7429-90-5
Antimony*	Sb	7440-36-0
Arsenic*	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium*	Be	7440-41-7
Cadmium*	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium*	Cr	7440-47-3
Cobalt*	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead*	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese*	Mn	7439-96-5
Mercury*	Hg	7439-97-6
Molybdenum	Mo	7439-98-7
Nickel*	Ni	7440-02-0
Phosphorus	P	7723-14-0
Potassium	K	7440-09-7
Rubidium	Rb	7440-17-7
Selenium*	Se	7782-49-2
Sodium	Na	7440-23-5
Strontium	Sr	7440-24-6
Thallium	T1	7440-28-0
Thorium	Th	7440-29-1
Uranium	U	7440-61-1
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

<sup>\*</sup> Target elements for EPA UATMP/NATTS program.

#### 5.0 METHOD SUMMARY

This SOP describes the multi-elemental extraction procedure for determination of total metals by ICP-MS in ambient air samples collected on quartz filters following guidelines in EPA method IO-3.1, the NATTS TAD (Rev 4). An 8"x 1" portion is cut from the exposed filter and placed in an extraction tube. The filter is digested in a HotBlock<sup>TM</sup> for 2.5 hours using an extraction fluid containing 1.5% hydrochloric acid (HCl), 5.55% nitric acid (HNO<sub>3</sub>) and 1 mg/L Au (for mercury stabilization). An aliquot of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) is added after 1.5 hours and 2.0 hours of extraction and allowed to effervesce. Once cooled, the extract is brought to final volume, filtered and ready for analysis by ICP-MS.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 5 of 26

#### 6.0 DEFINITIONS AND ABBREVIATIONS

# 6.1 <u>Definitions</u>

- 6.1.1 **Duplicate (DUP).** A second aliquot of a sample extract that is analyzed from the original sample to determine the precision of the method. This sample is also referred to as a replicate. There are multiple types of duplicate samples in this method, see section 16.4 for more information.
- 6.1.2 **Blank (BLK).** An analytical sample designed to assess specific sources of contamination. In this method, there are two BLKs, the Laboratory Reagent Blank (LRB), which is always reported as BLK1; and the Method Blank (MB), which is typically reported as BLK2.
- 6.1.3 **Blank Spike (BS).** A quality control sample (QCS) that contains a quartz filter strip spiked with a known quantity of analytes that is carried through the entire extraction process. This sample is synonymous with the Laboratory Control Sample (LCS).
- 6.1.4 **Reagent Blank Spike (RBS).** A spiked aliquot of LRB with no filter material which is used as a quality control sample (QCS) to demonstrate spike recovery.
- 6.1.5 **Field Blank (FB).** A blank quartz filter sample intended to assess field conditions during sampling and laboratory sources of contamination. The blank sample matrix is briefly exposed to the sampling environment.
- 6.1.6 **Interferents.** Substances (atoms, ions, polyatomic ions, etc.) which may affect the analytical result for the element of interest.
- 6.1.7 **Laboratory Reagent Blank (LRB).** An aliquot of ASTM Type I water acidified with the same acid matrix as is present in the calibration standards that is treated exactly as a sample; including exposure to all labware, equipment, solvents, reagents and internal standards that are used with other samples. This sample is always reported as BLK1. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or apparatus.
- 6.1.8 **Matrix Spike and Matrix Spike Duplicate (MS/MSD).** Aliquots of a batch sample (using separate filter strips) which are spiked prior to digestion/extraction with known quantities of analytes and carried through the entire analytical process to determine whether the sample matrix contributes bias through spike recoveries and precision through comparison of MS and MSD.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 6 of 26

- 6.1.9 **Method Blank (MB).** An aliquot of LRB with a blank quartz filter strip that is carried through the entire preparation and extraction process to demonstrate background contamination contribution from the filter and extraction process. This sample is typically reported as BLK2, but may use a different identifier in the event of a double batch.
- 6.1.10 **Performance Evaluation (PE) Sample.** A sample of known composition provided by a source outside the laboratory for preparation and analysis that evaluates the laboratory's analytical performance.
- 6.1.11 **Trip Blank.** A blank quartz filter sample that is transported with field samples without being exposed to sampling procedures. This blank sample assesses contamination introduced during shipping and field handling procedures.

## 6.2 Abbreviations

ASTM American Society for Testing and Materials CASRN Chemical Abstract Services Registry Number

COC Chain of Custody

DI Deionized

DQO Data Quality Objective

HNO<sub>3</sub> Nitric Acid

ICP-MS Inductively Coupled Plasma - Mass Spectrometry

L Liter(s)

LDPE Low Density Polyethelyene

LIMS Laboratory Information Management System

MΩ Megohm m Meter(s)

m<sup>3</sup> Cubic Meter(s) mg Milligram(s)

mg/L Milligram(s) per liter(s)

min Minute(s)
mL Milliliter(s)
mm Millimeter(s)
ms Millisecond(s)
N/A Not Applicable
ng Nanogram(s)

ng/L Nanogram(s) per liter(s)
ng/mL Nanogram(s) per milliliter(s)

NIST National Institute of Standards and Technology

QC Quality Control SD Standard Deviation



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 7 of 26

SOP Standard Operating Procedure

Std. Standard

μg/L Microgram(s) per liter

μg/m<sup>3</sup> Microgram(s) per cubic meter(s) μg/mL Microgram(s) per milliliter(s)

μL Microliter (s) μm Micrometer

v/v Volume per volume ratio

#### 7.0 INTERFERENCES

**Note:** The background level of metals on a given lot of quartz filters can vary. Any background levels found on blanks should be documented for all the filters from the corresponding lot when available. It is recommended to consult 40 CFR Part 50, Appendix G for guidance.

# 7.1 <u>Laboratory Interferences</u>

- 7.1.1 Wear powder-free nitrile or neoprene gloves when handling unexposed or exposed filters.
- 7.1.2 Maintain and clean all equipment used for metals sample preparation in a manner consistent with good laboratory practices. Regularly clean the fume hood and filter prep area to prevent sample contamination from dust or previous samples. Clean out the wells of the HotBlock as needed to prevent contamination during extractions. See Section 20 in this SOP and Section 14.3.1 in SOP ERG-MOR-031 for glassware cleaning procedures.
- 7.1.3 Use ASTM Type I DI water or equivalent, with a resistivity  $\geq 18 \text{ M}\Omega$ , for sample extraction and standard preparation. Record the water resistivity in the "Deionized Water Treatment Log" prior to use.

## 7.2 Chemical Interferences

Pay close attention to the nature of solutions intended to be introduced to the ICP-MS.

7.2.1 Nitric acid must be less than 2% (v/v) for ICP-MS analysis to minimize the damage to the interface and to minimize isobaric molecular interferences.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 8 of 26

#### 8.0 SAFETY

- 8.1 Personal protection should be used for all work performed in the inorganic laboratory, (e.g., gloves, safety glasses, laboratory coats, etc.).
- 8.2 Make sure that sample vials are kept capped and in racks to prevent spills.
- 8.3 All personnel should be trained in the handling and extraction of acid samples for inorganic sample preparation.
- 8.4 Strong acids must not be stored with organic solvents or samples.
- 8.5 Follow normal laboratory safety procedures as outlined in the ERG Health and Safety Manual and the site-specific laboratory SOP.

## 9.0 EQUIPMENT

# 9.1 Digestion System

Environmental Express HotBlock<sup>TM</sup> Digestion System or equivalent system capable of maintaining a temperature of 95°C within  $\pm$  2°C. This temperature will heat the samples to a target temperature of ~78.0°C ( $\pm$ 5°C).

## 9.2 Labware Drying Box

AirClean Systems PCR Workstation. Used for drying all inorganics labware. Provides clean drying area to prevent contamination.

# 9.3 <u>Vacuum and Manifold</u>

12 position manifold and 20 L/min vacuum pump for filtering samples after extraction.

#### 10.0 MATERIALS

- 10.1 Graduated polypropylene sample extraction vials with screw caps, 50 mL volume (certified to be within  $\pm$  0.2mL).
- 10.2 Pipettes with adjustable volumes ranging from 0.5 μL to 10 mL and disposable tips. Repeatable, mechanical pipettes, such as Eppendorf Research®, may be used and their accuracy and precision should be verified on a quarterly basis according to Section 16.0 of ERG SOP 091.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 9 of 26

- 10.3 Reflux caps and DigiFilter<sup>™</sup> 1 µm Teflon<sup>®</sup> filters.
- 10.4 Miscellaneous: powder-free nitrile or neoprene gloves; disposable laboratory wipes; self-adhesive labels.
- 10.5 Storage bottles. Wide and narrow mouth, Teflon® FEP (fluorinated ethylene propylene) with Tefzel ETFE (ethylene tetrafluoroethylene) screw closure: 50, 100, 250, 500, 1,000 and 2,000 mL capacities.
- 10.6 Wash bottles made of LDPE and Teflon® having 500 mL and 1 L capacities.
- 10.7 Plastic or Teflon® coated tweezers and Teflon stirring rods.
- 10.8 Filter cutting apparatus consisting of a 2-part Plexiglas board and plastic rotary cutter
- 10.9 Quartz filters of 8" x 10" dimensions.
- 10.10 Thermometers with serial numbers for monitoring extraction temperatures. See SOP ERG-MOR-108 for details about annual thermometer certification using a NIST traceable thermometer.

# 11.0 CHEMICALS, REAGENTS, STANDARDS AND THEIR PREPARATION

**Note:** In general, chemicals, reagents and commercial stock standards expire when specified by the manufacturer. If the manufacturer does not provide an expiration date, then they shall expire one year from the opened date. Standards and other solutions prepared in-house expire no later than the earliest manufacturer's expiration date of any standard or reagent used for preparation, with the exception of gold as it is added to standards for the stability of mercury and does not directly impact analytical measurements. Examples of approved commercial vendors are Fisher Scientific and SCP Science. Proper disposal of hazardous waste is discussed in detail in the Solid and Hazardous Wastes SOP (ERG-MOR-033).

11.1 <u>High Purity Acids</u> - ultrapure and concentrated stored in Teflon<sup>®</sup> Bottles. These reagents are used for the preparation of sample extraction fluid and all standards.

**Note:** Concentrated high purity reagents are not necessarily 100% of the specified reagent. All percentages in this SOP are expressed in terms of volume per volume (v/v) rather than true percentages of reagent in solution.

- 11.1.1 Nitric Acid (HNO<sub>3</sub>), 60-70%
- 11.1.2 Hydrochloric Acid (HCl), 32-35%
- 11.1.3 Extraction fluid (1.5% (v/v) HCl, 5.55% (v/v) HNO<sub>3</sub>, and 1 mg/L Au). See Table 24-2 for preparation.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 10 of 26

- 11.2 <u>Hydrogen Peroxide  $(H_2O_2)$ </u> ultrapure and concentrated (30-32%) stored in Teflon<sup>®</sup> bottles. This reagent is used in the extraction procedure.
- 11.3 <u>Nitric Acid</u>-Trace Metal Grade in 2.5 L glass for acid bath and labware cleaning.
  - 11.3.1 10–15% (v/v) HNO<sub>3</sub> acid bath for labware cleaning

To prepare a 10% acid bath solution, add 2.5L of concentrated trace metal grade HNO<sub>3</sub> to 22.5 L of ASTM Type I DI water in a clean 42 L polypropylene acid bath tank. The acid bath must be stored in a fume hood.

- 11.4 ASTM Type I deionized water with a minimum resistivity of  $\geq$  18 M $\Omega$ .
- 11.5 <u>Single-Element Stock Standard Solutions</u> Commercially prepared NIST traceable standards from ultra-high-purity grade chemicals or metals (99.99 99.999% pure) designed for use with ICP-MS instruments (e.g., Mercury Std.). Examples of some approved commercial vendors are High Purity and Inorganic Ventures.
- 11.6 <u>Blank Spike (BS) Standard</u> The BS standard is used to spike the RBS/BS/BSD (See Section 16.3). This is the same as the matrix spike standard. See Table 24-4 for preparation.
- 11.7 <u>Matrix Spike (MS) Standard</u> The MS standard is used to spike the BS/LCS and MS/MSD. See Table 24-4 for preparation.
- 11.8 <u>Citranox® Acid Cleaner and Detergent Bath</u> Prepare a 2% solution by adding 240 mL of Citranox® to 11.7 L of warm hot tap water for labware cleaning and decontamination. This detergent bath should be changed about once every month, depending on use.
- 11.9 <u>Limit of Quantitation(LOQ)</u> The lowest concentration of an analyte that produces a signal/response that is sufficiently greater than the signal/response of lab reagent blanks to enable reliable detection and quantification during routine lab operating conditions. The LOQ is determined in compliance with the 2016 TNI standard as described in SOP ERG-MOR-010. An LOQ verification sample is prepared and carried throughout the extraction process as a normal sample. An LOQ check is performed once per quarter.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 11 of 26

# 12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

# 12.1 <u>Collection and Handling of Filters</u>

Whenever the filter is handled use clean disposable nitrile or neoprene gloves and if necessary, clean Teflon® coated (no exposed metal surfaces) or plastic tweezers. Never touch the particulate laden surface of the filter and take care not to puncture or damage the filter with the tweezers. The filter should be folded in half lengthwise to prevent loss of particulate. Upon retrieval from field sampling during humid or rainy conditions, be certain the filter is not moisture laden. Damp filters may stick to the sample transport container causing damage and potentially invalidating the sample. See Section 16.1 for more information about filter condition.

# 12.2 Preservation and Storage of Filters

Samples do not have a preservative and can be stored for up to 180 days in ambient conditions. Once the filter arrives at the laboratory, a unique LIMS identification number is assigned and written on the COC, a label placed on the sample envelope and/or container for tracking and storage purposes. Extraction and analysis of samples must take place within the hold time, 180 days from the sample date. In the event this is not possible, samples are flagged with the appropriate qualifier.

## 12.3 Shipment of Filters

When filters are shipped to or from the laboratory follow proper handling instructions in Section 12.1 and take proper precautions when packing such that filters are not exposed to contaminants or damaged during shipment. If the sample is found to be moisture-laden allow the membrane to dry before enclosing in the transport container.

# 13.0 CALIBRATION AND STANDARIZATION

N/A

#### 14.0 PROCEDURE

#### 14.1 LIMS Batch Procedure

**Note:** Please perform the following procedure the same day that as the extraction. If something happens and the extraction cannot be performed, edit the bench sheet with the correct extraction date, both in the LIMS and on the hardcopy bench sheets.

Follow the instructions in ERG-MOR-099 Section 14.1 to create a bench sheet in the LIMS with the following metals-specific procedures.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 12 of 26

#### 14.1.1 Metals Batch Procedure

- Department: Inorganics
- Preparation Method: ICP-MS Extraction
- In the reagents box be sure to set the current Hydrogen peroxide and Extraction fluid ID.
- Bench sheet comments should include:
  - o Extraction tube manufacturer, lot number and box number
  - o Digifilter manufacturer, lot number and box number
  - ID of any pipettes used during the extraction process(one for BS/MS solution, one for extraction fluid, one for hydrogen peroxide)
  - Ensure all information is correct before moving on the Bench Sheet.

#### 14.1.2 Metals Bench Sheet Procedure

- Add in all samples desired for the current batch (maximum of 20 samples per normal batch). For all samples update the comments to include the site code and any specific sample designation (NBIL C1, OCOK FB, etc.). Update the initial volume based on the sample volume listed on the chain of custody. Field Blanks/Trip Blanks should use the same volume as the sample with the closest sample date.
- Duplicates: Due to the large number of collocated sites for metals analysis, duplicates are not required for every C1/C2 pair. Instead, at least two duplicates are added per batch, with preference given to collocated samples in the batch. If there is a DUP added to a C1 sample, there must also be a DUP on the corresponding C2 sample. Update the initial volume to match the source sample, and include the source sample site ID (e.g., S4MO) in the comment for every DUP sample. If there are no collocated samples in the batch, one sample should be selected as the DUP1 which is a second strip duplicate, and another sample should be selected for the DUP2 which is an analytical replicate, not a second strip sample. Every batch is required to have one second strip duplicate and one analytical replicate sample.

**Note:** DUP selections should be monitored throughout the year to ensure each site has the contract required number of duplicates analyzed annually (*i.e.*, 10% of samples). A file is located on the network which should be used to keep track of DUP selections.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 13 of 26

- Blanks: Two BLK samples (a reagent and method blank) must be included for every 20 samples. If over 20 samples are selected there must be 4 blanks (two reagent and two method) included.
  - o Add a second BLK sample using the "Add" menu.
  - Rename the BLK1 "Reagent Blank" and the BLK2 "Method Blank" by right clicking on the individual QC sample and selecting "Name."
  - The filter number must be included in the comments for the BLK2.

# • Spiked Samples:

- O Update the name of the Reagent Blank Spike to "RBS" to distinguish it from the BS (which is labeled as LCS in the LIMS). The RBS is spiked with 500μL of the Matrix Spike solution into an empty tube. The RBS will typically be set up as BS2.
- The filter number must be included in the comments for the BS1.
   The BS1 sample is spiked with 500μL of the Matrix Spike solution.
- O The source selected for the MS/MSD should be the same as the source for the DUP1 sample. Update the initial volume to match the source sample, and include the source sample site ID in the comments for both spikes. Both MS/MSD are spiked with 500 μL of the Matrix Spike solution
- O The source selected for the PS should be the same as the source for the DUP1 sample. Update the initial volume to match the source sample, and include the source sample site ID in the comments for the post spike. The PS should be set with 50μL of the appropriate PS solution. This sample is created during the analysis and is not a separate extracted sample.
- Edit the "Spike ID" column for the each spike sample to the appropriate LIMS standard ID used for each of these QC samples.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 14 of 26

- Ensure all spikes are set with the type "Pre-Prep." If this or any other spike information is not properly set, the LIMS may not correctly calculate the results from analysis.
- Once the Bench Sheet is printed, the "Extraction Reviewed by" line should be signed by the analyst who performed the extraction.
- Print two sets of sample extraction tube labels. One for the initial extraction and another for the filtering step. Label and cap all tubes needed for the extraction. Sample order should be BLK1, BLK2, BS1, BS2, QC source sample, DUP1, MS1, MSD1, SRD1, PS1, and then all other samples in the order listed on the bench sheet. Take care to not cover key volume lines when labeling tubes.

## 14.2 Filter Extraction Procedure

- 14.2.1 Prior to sample processing, be sure to turn on the HotBlock <sup>TM</sup> and select the "FEM IO3.5 TEFLON AND QUARTZ" method program and initiate to allow the HotBlock<sup>TM</sup> to warm to extraction temperature. Be sure to allow the HotBlock<sup>TM</sup> interface to establish connection with the block itself (shown on the screen as "Please Wait") prior to beginning any extraction method. Failure to do this will cause the HotBlock<sup>TM</sup> to heat to higher than intended temperatures.
- 14.2.2 Wipe the filter-cutting apparatus and plastic rotary-cutter with a Kimwipe<sup>®</sup> prior to use. Ensure the work area and all tools are clean and free of any visible debris.
- 14.2.3 When preparing samples, be sure to check that the sample information on the tube matches the sample, including checking the physical filter number on the filter. If the filter is not already folded in half, carefully fold the filter in half along the 10" length with the side containing the particulates facing on the inside of the fold. If the filter is incorrectly or poorly folded, it may be necessary to refold it to ensure a sample respective of the whole filter is taken. No sample area should be visible when the filter is folded properly. Inspect filter for damage, irregularities or lack of/improper folding and insert a note in the sample comments section of the LIMS bench sheet. If possible, avoid any irregularities when selecting a strip. When inspecting filter for DUP1/MS1/MSD1 ensure all 4 strips will be homogenous to avoid analysis issues. Select a different filter for these QC samples if the filter appears nonhomogeneous. Using the filter-cutting apparatus, align the 10" folded edge of the filter along



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 15 of 26

the dashed line. Cut a 4" by 1" section of the folded filter using the rotary-cutter.

14.2.4 Carefully fold the 4" by 1" section end over end into a size just larger than the mouth of the extraction vial. Gently fold the corners in to make a cup shape. Place the folded filter into the labeled extraction vial and push to the bottom using a pre-cleaned Teflon® stirring rod for each sample to be sure the filter is below the 10 mL line. Before cutting the next filter strip, wipe the filter cutting assembly and the rotary cutting blade with a Kimwipe®. If necessary, wet the Kimwipe® with deionized water from wash bottle to help gather any residual particulate.

Once all filters have been placed into the extraction tubes move the samples to the hood to prepare the spikes. Once all spikes have been prepared, use a pipette to add 10mL of extraction fluid to all samples. Ensure the extraction fluid fully surrounds the filter by gently rotating or swirling the tube, as air pockets may form below the filter.

**Note**: Do not place the filter flush with the bottom of the sample tube, as this will prevent proper acid flow through the tube during extraction. It is only necessary to make sure the filter is below the 10 mL line.

- 14.2.5 The Reagent blank (BLK1) and Reagent Blank Spike (RBS) tubes will have no filter included. The Method blank (BLK2) and BS samples should have a blank quartz filter strip placed in the tube. While working in the hood, add 0.5 mL of Matrix Spike Standard to the BS, MS, and MSD samples with a mechanical pipette.
- 14.2.6 Print out a copy of the Quartz Extraction Record from L:\Metals Lab (see Table 24-1). Record the batch ID and extraction date. Refer to the HotBlock<sup>TM</sup> log file to determine which well to place the thermometer tube in. The well position for the thermometer should progress by one spot with each extraction, to ensure that every well is monitored over time. Record the thermometer sample position on the extraction log sheet.
- 14.2.7 Place all samples in a plastic HotBlock<sup>TM</sup> rack and add a reflux cap to each sample tube before placing in the HotBlock<sup>TM</sup>. Record the time samples were placed in the HotBlock<sup>TM</sup> and the temperature displayed on the HotBlock<sup>TM</sup> control box on the extraction record sheet. Samples will be extracted for a total time of 2.5 hours at a HotBlock<sup>TM</sup> temperature of 95°C, and an approximate sample target temperature of 78.0°C (±5.0°C).
- 14.2.8 To monitor sample temperature during extraction, a thermometer tube should be set up. Refer to the extraction log sheet for the serial number



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 16 of 26

and correction factor of the proper thermometer. Be sure to check that the serial number of the thermometer used matches what is written on the extraction record. Place the thermometer in a tube with enough extraction fluid to go slightly above the immersion line of the thermometer and place it according to the position on the extraction log sheet. The temperature read from the thermometer will be recorded as the uncorrected temperature (if a correction factor is required, record uncorrected and corrected temperatures, see SOP ERG-MOR-108). Record these temperatures in the HotBlock<sup>TM</sup> log file.

- 14.2.9 After 1.5 hours of extraction record the sample temperature from the thermometer, and then add 1.8 mL of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) to all samples (excluding the thermometer tube) and allow to effervesce for 0.5 hours. Then add an additional 1.8 mL of H<sub>2</sub>O<sub>2</sub> and allow to effervesce again for the final 0.5 hours. This completes the extraction process. Record the time both aliquots of H<sub>2</sub>O<sub>2</sub> were added on the Quartz Extraction Record.
- 14.2.10 Check the filters occasionally during extraction. If a filter floats out of the acid, use a clean Teflon stirring rod to push the filter below the extraction fluid.
- 14.2.11 Immediately before removing samples from HotBlock<sup>TM</sup>, record the temperatures indicated by both the thermometer and HotBlock<sup>TM</sup> UI on the Quartz Extraction Record. After extraction time is complete, remove the rack of samples and allow them to cool to room temperature (~30-45 minutes) inside the fume hood. Record the time the samples were removed from the HotBlock<sup>TM</sup> for cooling.
- 14.2.12 Using a wash bottle, add DI water to slightly below the 50mL fill line, cap, and then shake vigorously until the filter is thoroughly broken down. Once this is done allow the extracts to sit for 0.5 hours. This step is critical and must not be omitted as it allows the acid to diffuse from the filter into the rinse.
- 14.2.13 After this time period bring the sample volume to the 50 mL line on the vial with a wash bottle filled with ASTM Type I DI water prior to filtering. Record the time the samples were brought to final volume on the Quartz Extraction Record.

**Note:** If the final volume exceeds 50 mL, the amount exceeded can be measured using an appropriate pipette (usually the 1 or 5 mL). Using the pipettor, pull sample from the vial incrementally until the sample line is level with the 50 mL line on the vial. Determine the amount of liquid removed in mL and then return the liquid to the vial. Record the



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 17 of 26

final volume as the sum of exceeded amount measured over 50mL. This information must be entered into the LIMS so that the final sample concentration is calculated and reported correctly. This overfilling may be avoided by using fine streamed wash bottles while adding the D.I. water.

14.2.14 Prepare for filtering by labeling a second set of new 50mL extraction tubes. Attach a DigiFilter to each tube so that the actual filter in the housing is closest to the new tube and plug the hole furthest from the new tube with one of the blue plugs included with the filters.

Place the vacuum manifold in the hood and turn on the vacuum pump. Keep the pump in the hood as well to remove any potential fumes. All the sample valves on the manifold should be in the closed position.

**Note**: Verify the final volume in sample tube is correct before filtering. Once the extract has been filtered the volume **cannot** be altered or checked so it is important to ensure they are at proper volume before they are filtered.

Homogenize the sample by shaking vigorously and then attach the filter tube to the appropriate sample tube. Take care to ensure that the label on each tube matches before connecting them. Place the connected tubes onto the manifold with the sample tube above the empty tube, and make sure the vacuum valve slots into the unplugged hole in the filter.

Once the tubes are in position and connected to the vacuum system, open the valve to allow the vacuum to pull the sample down, this will take a few minutes to fully filter. Continue loading samples until all positions on the manifold are occupied. Once the extract has been fully filtered into the new tube close the valve and remove the tubes. The now empty sample, tube with the filter remnants can be disposed of, and the new extract tube free of filter material, should be capped and placed back in the sample rack. Continue this process until all samples are filtered.

#### 15.0 CALCULATIONS

N/A

#### 16.0 QUALITY CONTROL

In addition to inspecting sampled filters for potential issues, the lab technician must prepare several quality control samples, the analytical results which are used to determine if the Data Quality Objectives (DQO) criteria are met. The DQO criteria are summarized in SOP 095.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 18 of 26

# 16.1 <u>Sample Collection Quality Control</u>

- 16.1.1 Filters which are dropped or become contaminated with any foreign matter (i.e., dirt, finger marks, ink, liquids, etc.) may be invalid. Check with Program Manager and/or QA/QC Department on how to proceed with dropped filters or filters that may have become contaminated.
- 16.1.2 Filters with tears or pinholes that occurred before or during sampling are invalid.
- 16.1.3 A power failure during a field sample collection event may invalidate the sample collected during that event. See SOP ERG-MOR-045 for more information about the sample acceptance criteria.

#### 16.2 Blanks

- 16.2.1 Laboratory Reagent Blank (LRB/BLK1) extraction fluid carried through the entire preparation and extraction process.
- 16.2.2 The Method Blank (MB/BLK2) is prepared as an LRB but with the addition of a blank Quartz Fiber filter.

#### 16.3 Spikes

- 16.3.1 Reagent Blank Spike (RBS): An RBS is prepared and carried through the entire sample digestion process. At least one RBS is analyzed for every 20 samples, if more than 20 are selected an additional RBS is required. The RBS is represented by the BS2 sample typically. It is prepared by spiking an empty extraction tube and carrying it through the entire preparation and extraction procedure.
- 16.3.2 Laboratory Control Sample (LCS/BS): An LCS is prepared and carried through the entire sample digestion process. One LCS is analyzed for every 20 samples included in a batch, if more than 20 are selected an additional LCS is required. The LCS is essentially a MB with a spike added and is also referred to as a Blank Spike (BS).
- 16.3.3 Matrix Spike and Matrix Spike Duplicate (MS/MSD): Per batch, two extra filter strips are cut from the selected source sample filter and spiked. They are carried through the entire preparation and extraction procedure as described in Section 14.2. If a batch exceeds 20 samples, an additional MS/MSD pair must be included.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 19 of 26

16.3.4 Post Digestion Spike (PDS, also known as PS): The PDS is an analyzed aliquot of an extracted sample, preferably the same parent sample used for DUP1, that is spiked with the PDS standard (See SOP ERG-MOR-095 for more information on this standard). Preferably, the same sample that is used for the MS/MSD is also spiked for the PDS. The PDS spike addition should produce a minimum concentration level of 10 times and a maximum of 100 times the QL. If a batch exceeds 20 samples, an additional PS must be included.

# 16.4 <u>Duplicates</u>

There are three types of duplicates found in this procedure:

- 16.4.1 Matrix spike duplicate (MSD): A spiked laboratory duplicate of a separate strip of the parent (also called source) sample filter used to prepare the matrix spike.
- 16.4.2 Laboratory duplicate (DUP1): This duplicate is prepared with an additional strip of the parent (or source) sample filter. One of this type of duplicate is required with every batch. Preference is given to collocated samples when selecting a source sample for the DUP1. If there are no collocated samples in the batch, a regular field sample should be selected for the DUP1 source.
- 16.4.3 Analytical duplicate (or replicate): A second aliquot of an extracted sample analyzed using the same analytical method. These are often performed on collocated field samples, usually corresponding to the DUP1 sample. If a collocated sample is not in the batch, a field sample should be chosen to perform the replicate analysis, one per 20 samples. One of this type of duplicate is required per batch, in addition to the DUP1.

#### 16.5 Performance Evaluation (PE) Samples

Performance evaluation samples are obtained as available from independent sources and prepared as a routine sample. PE samples are prepared in the same way as field samples.

#### 16.6 Initial Demonstration of Capability

Each lab technician must demonstrate initial proficiency for sample preparation by preparing samples that generate data of acceptable accuracy and precision for four LCSs. This demonstration is repeated whenever new staff receives training or



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 20 of 26

significant changes in extraction procedure are made. These are maintained in the staff training files.

## 16.7 Ongoing Demonstration of Capability

To evaluate the continued performance of an analyst annually, it is acceptable to use CCVs, blank spikes, external audit samples, MDLs, or internal blind samples (blind samples at a concentration from LOQ to CCV level). Acceptable percent recovery is within the range of 75-125%. These are evaluated at the beginning of each year.

# 16.8 Decontaminating/Cleaning Labware

Procedures for proper cleaning and removal of trace metals from labware are found in Section 14.3.1 in SOP ERG-MOR-031. Detergent bath preparation instructions can be found in Section 11.8 of this SOP. Transport/dip baths of DI water ("To" and "From" acid baths) should be labeled with the date they are filled with fresh DI and changed about once every month to minimize contaminants being brought to the acid bath and being reintroduced to clean labware. This will not only extend the life of the acid bath, but it will help reduce hazardous waste production. The acid bath can be monitored by preparing a 5x dilution that can be analyzed as necessary, or whenever contamination issues are suspected.

## 17.0 PREVENTION

When possible, minimize the amount of chemicals used in the preparation of the metals filters to reduce waste.

#### 18.0 CORRECTIVE ACTION

N/A

#### 19.0 WASTE MANAGEMENT

Hazardous waste disposal is discussed in detail in SOP ERG-MOR-033. Sample digestates are retained for a period of at least 6 months from the sample date. After this time, sample digestates are disposed of by pouring them off in the satellite waste containers located in the laboratory. Used sample vials are disposed of in the laboratory waste bin. Although some clients may request that the remaining filter be sent back to them, quartz samples are stored for at least 1 year from the sampled date and then disposed.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 21 of 26

#### **20.0 MAINTENANCE**

- 20.1 Pre-filters in the AirClean System PCR Workstation should be changed roughly every 6 months, or whenever they appear dirty enough to hinder good air flow. Use the iCAP Q maintenance log to record the date of installation on the pre-filter. If the pre-filter becomes too clogged, air is forced to bypass the pre-filter by going around, making the HEPA filter do all the work, which will shorten the life of this very expensive filter.
- 20.2 The HotBlock™ is monitored at a different well location during every extraction to ensure it is working normally and evenly across all wells. The wells should be cleaned out with the small shop vacuum as needed to prevent sample contamination and the vial racks should be rinsed off with warm water to remove any dust as needed.
- 20.3 The level of the acid bath should be maintained with DI water throughout the year. The level will gradually go down due to evaporation and loss from transfer. Keep the bath at the working level marked on the container.

#### 21.0 SHORTHAND PROCEDURE

The flow chart shown in Figure 24-1 shows the procedural steps for extraction of Quartz filter samples.

#### 22.0 DOCUMENTATION AND DOCUMENT CONTROL

22.1 All information concerning sample preparation, standard preparation, etc., must be documented in the appropriate binders (i.e., Extraction Log, Standards Log, etc.) and/or electronically in the LIMS.

#### 23.0 REFERENCES

Code of Federal Regulations – 40 CFR Part 50, Appendix G – Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air – January 1, 2011.

Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP/MS). Compendium Method IO-3.5, *In:* Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air. Center for Environmental Research Information, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH, 45268, June 1999.

ERG's Corporate Health and Safety Manual.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-084

Revision Number: 20

Revision Date: January 27, 2023

Page: 22 of 26

ERG-MOR-031. Standard Operating Procedure for Cleaning Glassware and Syringes for Organic Analysis. Revision Number 6, Effective: October 20, 2017.

ERG-MOR-033. Standard Operating Procedure for Hazardous Waste. Revision Number 5, Effective: February 8, 2016.

ERG-MOR-045. Standard Operating Procedure for Sample Receipt at the ERG Chemical Laboratory. Revision Number 9, Effective: March 6, 2017.

ERG-MOR-099. Standard Operating Procedure for use of the Laboratory Information Management System and Data Progression and Data Reporting. Revision Number 4, Effective: February 20, 2018.

ERG-MOR-108. SOP for the Calibration and Verification of Reference Standards. Effective: April 2018.

Federal Advisory Committee on Detection and Quantitation Approaches and Uses In Clean Water Act Programs. Appendix D: DQ FAC Single Laboratory Procedure v2.4, 8/30/2007.

Selection, Preparation and Extraction of Filter Material. Compendium Method IO-3.1, *In:* Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air. Center for Environmental Research Information, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH, 45268, June 1999.

Technical Assistance Document for the National Air Toxics Trends Stations Program, Revision 4, July 2022



Donne Tedda

# ERG PROPRIETARY INFORMATION

Standard Operating Procedure Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 1 of 67

# ENGINEERING AND SCIENCE DIVISION

TITLE: **EFFECTIVE** DATE: Standard Operating Procedure for the Analysis of High SEP 1 9 2023 Volume Quartz and 47mm Filters for Metals by ICP-MS using Method IO 3.5 REFERENCES: ERG-MOR-010, ERG-MOR-031, ERG-MOR-033, ERG-MOR-045, ERG-MOR-084, ERG-MOR-85, ERG Health and Safety Manual, 40 CFR, Part 136, Appendix B, EPA Compendium Method IO-3.5, Technical Assistance Document for the National Air Toxics Trends Stations Program, Revision 4 SATELLITE FILES: ICP-MS Laboratory, Inorganic II REVISIONS: Removed mercury from target list and gold from solutions; Updated some QC sample criteria and qualification based on NATTS TAD, Version 4; Updated Figures 24-1 and 24-2; Updated Table 24-3. PROJECT MANAGER/TECHNICAL DIRECTOR: WRITER/EDITOR: NAME/DATE NAME/DATE Julie L. Suift 9/19/23 9-18-23 **OUALITY ASSURANCE COORDINATOR:** NEXT SCHEDULED REVIEW: NAME/DATE March, 2024



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 2 of 67

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# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 3 of 67

#### 1.0 IDENTIFICATION AND PURPOSE

This standard operating procedure (SOP) provides the analysis procedures for suspended particulate matter collected on quartz or Teflon® 47mm filters for total metals determination by Inductively Coupled Plasma - Mass Spectrometer (ICP-MS).

#### 2.0 MATRIX OR MATRICES

This procedure applies to the analysis of ambient particulate matter samples collected with quartz or Teflon® 47mm filters for total metals.

#### 3.0 METHOD DETECTION LIMIT

## 3.1 Method Detection Limits (MDL)

- 3.1.1 The method detection limit (MDL) for each isotope is determined according to the requirements of EPA in the NATTS TAD, Revision 4. These requirements allow the use of Appendix D: DQ FAC Single Laboratory Procedure v2.4, 8/30/2007. MDL values are determined from historic method blank (BLK2) data following the procedures outlined in the documents above.
- 3.1.2 Use the same internal standards, calibration standards, instrument method and settings (sweeps and dwell) for the MDL study and field sample analysis.
- 3.1.3 For the MDL determination in units of ng/m³, assume 1627 m³ per sample for quartz filters or 24.04 m³ per sample for Teflon® filters.
- 3.1.4 The MDLs are calculated once per year from the two years' previous method blank data. For the current quartz fiber filter MDLs, refer to Table 24-1. The current Teflon® filter MDLs are presented in Table 24-2.

## 4.0 SCOPE AND APPLICATION

# 4.1 Scope

This procedure details the trace elemental analysis of ambient air sample extracts to determine total metals concentrations using an inductively coupled plasma-mass spectrometer (ICP-MS). The analysis procedures are suitable for low and high-volume ambient air samples. Quartz filters, sized 8" x 10", are used for high volume sampling, while Teflon® membrane filters, sized 47 millimeters in diameter, are used for low-volume sampling. Different size quartz filters (e.g.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6 Revision Date: August 31, 2023

Page: 4 of 67

circular) can be analyzed using this procedure but caution needs to be exercised during extraction and calculating final concentrations.

## 4.2 Applicability

This SOP is applicable to the analysis of suspended particulate matter collected with quartz or Teflon® 47mm filters. Acid digestion and filtration (quartz method only) is required prior to analysis of quartz and Teflon® filter extracts. The procedure is applicable to, but not limited to, the 28 metals listed below. Eleven of these elements are target metals for EPAs Urban Air Toxics Monitoring Program (UATMP) and ten for the National Air Toxics Trends Station (NATTS) program. Analytes for which ERG has demonstrated the acceptability of this method are listed below. See Table 24-6 for a list of isotopes used for quantitation and monitoring.

Element	Symbol	CASRN	Tier
Aluminum	Al	7429-90-5	
Antimony*	Sb	7440-36-0	II
Arsenic*	As	7440-38-2	I
Barium	Ba	7440-39-3	
Beryllium*	Be	7440-41-7	I
Cadmium*	Cd	7440-43-9	I
Calcium	Ca	7440-70-2	
Chromium*	Cr	7440-47-3	II
Cobalt*	Со	7440-48-4	II
Copper	Cu	7440-50-8	
Iron	Fe	7439-89-6	
Lead*	Pb	7439-92-1	I
Magnesium	Mg	7439-95-4	
Manganese*	Mn	7439-96-5	I
Molybdenum	Mo	7439-98-7	
Nickel*	Ni	7440-02-0	I
Phosphorus	P	7724-13-0	
Potassium	K	7440-09-7	
Rubidium	Rb	7440-17-7	
Selenium*	Se	7782-49-2	II
Sodium	Na	7440-24-5	
Strontium	Sr	7440-24-6	
Thallium	T1	7440-28-0	
Thorium	Th	7440-29-1	
Uranium	U	7440-61-1	
Vanadium	V	7440-62-2	



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 5 of 67

Zinc	Zn	7440-66-6	
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<sup>\*</sup> Target elements for the EPA NHAPS program.

#### 5.0 METHOD SUMMARY

This SOP describes the multi-elemental analysis of total metals by ICP-MS in ambient air samples collected on 8" x 10" quartz or 47mm Teflon® filters following guidelines in EPA Method IO-3.5 and NATTS TAD Revision 4. Field samples are first processed following ERG ambient particulate matter digestion methods, which are outlined in SOPs ERG-MOR-084 and -085. These sample preparation methods dissolve the target metals into an acidic solution, which can then be introduced into the ICP-MS for analysis. Before samples are analyzed, the ICP-MS quadrupole is mass calibrated, and the detector is cross calibrated to achieve a linear transition from pulse to analog acquisition modes. Performance reports are generated for relevant analysis modes (STD/KED) and if any performance specifications fail, the instrument is tuned or undergoes maintenance until the performance report(s) pass. Following the analytical procedures outlined in this SOP, the ICP-MS is then calibrated, and the samples are loaded onto an autosampler for analysis. The data are collected using the manufacturer's software and the digitally exported data is uploaded to ERG's LIMS for review and reporting.

#### 6.0 DEFINITIONS AND ABBREVIATIONS

### 6.1 Definitions

- 6.1.1 **Analytical Duplicate (DUP).** A second aliquot of a sample extract that is analyzed from the original sample to determine the precision of the method. This sample is also referred to as a replicate. See Section 16.4 for further elaboration on duplicates.
- 6.1.2 **Blank (BLK).** An analytical sample designed to assess specific sources of contamination, the Laboratory Reagent Blank (LRB), which is always reported as BLK1; and the Method Blank (MB), which is typically reported as BLK2.
- 6.1.3 **Blank Spike (BS or LCS).** There are two types of Blank Spikes in this method. The Reagent Blank Spike (RBS, typically reported as BS2) is a quality control sample containing no filter material which is spiked with a known quantity of analytes that is carried through the entire extraction process. The Lab Control Sample (LCS, typically reported as BS1) is a quality control sample that contains a quartz filter strip, or Teflon<sup>®</sup> filter spiked with a known quantity of analytes that is carried through the entire extraction process.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 6 of 67

- 6.1.4 **Blank Spike Duplicate.** An additional quality control sample for the Teflon® method to demonstrate spike recoveries and precision. This is prepared in the same way as the LCS which contains a filter spiked with a known quantity of analytes that is carried through the entire extraction process. This sample is typically reported as the BSD1.
- 6.1.5 **Calibration Blank.** A blank solution containing all the reagents in the same concentration as those used in the analytical sample preparation when brought to final volume. This blank is not subject to the preparation method but contains the same matrix (i.e., the same amounts of reagents and/or preservatives) as the sample preparations to be analyzed.
- 6.1.6 **Calibration Standards.** A series of known standard solutions used by the analyst for calibration of the instrument (*i.e.*, preparation of the analytical curve). The solutions are not subject to the preparation method but contain the same matrix (i.e., the same amounts of reagents and/or preservatives) as the sample preparations to be analyzed.
- 6.1.7 **Continuing Calibration Blank (CCB).** A volume of ASTM Type I water acidified with the same acid matrix as is present in the calibration standards run after every CCV. This is used to verify that the instrument blank checks are reading < the absolute value of the s\*K portion of the MDL.
- 6.1.8 **Continuing Calibration Verification (CCV).** A multi-element standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV is the original calibration standard, whose concentration is at the midpoint of the calibration curve that is re-analyzed as a quality control (QC) sample.
  - This standard check is critical for Tier I elements (See section 4.2) so any failures must be reanalyzed. This standard is operational for Tier II elements or non-Tiered elements so failures may not require reanalysis, the CCV and associated samples may be qualified with "QX" and either "LL" or "LK", as appropriate.
- 6.1.9 **Field Blank (FB).** A blank Teflon® or quartz filter sample collected in the field intended to assess field sources of contamination. The blank sample matrix is briefly exposed to the sampling environment and returned to the laboratory with a corresponding field sample.
- 6.1.10 **High Standard/Calibration Verification (HSV/HCV).** The HSV is the highest calibration standard that is reanalyzed after the ICB and before the



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 7 of 67

analysis of samples to verify the accuracy of the calibration curve at that concentration.

For Tier I elements, any failures must be reanalyzed. For Tier II elements or non-Tiered elements failures may not require reanalysis, the HSV and associated samples may be qualified with "QX" and either "LL" or "LK".

- 6.1.11 **Initial Calibration Blank (ICB).** The ICB is a re-analysis of the calibration blank, which is analyzed after the Initial Calibration Verification (ICV). This check is used to verify that the instrument blank checks read < the absolute value of the s\*K portion of the MDL.
- 6.1.12 **Initial Calibration Verification (ICV).** A solution prepared from a stock standard solution obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration.

For Tier I elements, any failures must be reanalyzed. For Tier II elements or non-Tiered elements failures may not require reanalysis, the ICV and associated samples may be qualified with "QX" and either "LL" or "LK".

- 6.1.13 Interference Check Standard (ICS). A solution that may contain only interfering elements (ICSA) or both interfering elements and analytes of interest (ICSAB) in known concentrations. This standard is used to check for polyatomic interferences and verify interference removal techniques, such as Collision Cell Technology (CCT), Kinetic Energy Discrimination (KED) mode, and correction equations are working.
- 6.1.14 **Interferents.** Substances (atoms, ions, polyatomic ions, etc.) which may affect the analytical result for the element of interest.
- 6.1.15 **Internal Standard (ISTD).** A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance and correcting for drift.
- 6.1.16 **Limit of Quantitation (LOQ).** The lowest concentration of an analyte that can be reported with a specified degree of confidence. The LOQ is determined as described in SOP ERG-MOR-010.
- 6.1.17 **Linear Dynamic Range (LDR).** The concentration range over which the analytical working curve generated from the calibration standards is proven



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 8 of 67

to remain linear, which is performed optionally as an alternative to diluting samples which exceed the calibration range. See Section 13.9 for more information on the LDR.

- 6.1.18 **Matrix Interference/Effect.** In general, the interference and/or effect that matrix constituents may cause during sample processing and/or analysis. Matrix effects may be determined to exist from the careful interpretation of QC samples and criteria. Examples of observed effects include but are not limited to poor recoveries of spikes/ISTD and poor percent differences.
- 6.1.19 Matrix Spike and Matrix Spike Duplicate (MS/MSD). For quartz samples, aliquots of a batch sample (using separate filter strips) are spiked prior to digestion/extraction with known quantities of analytes and carried through the entire analytical process to determine whether the sample matrix contributes bias through spike recoveries and precision through comparison of MS and MSD. See Table 24-12 for preparation.
- 6.1.20 **Method Detection Limit (MDL).** The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.
- 6.1.21 **Minimum Reporting Level (MRL) Check.** A sample prepared quarterly at or below the established LOQ concentration for each element to verify the reported LOQ. These samples are spiked and carried through the entire extraction procedure prior to analysis.
- 6.1.22 **Performance Evaluation (PE) Sample.** A sample of known composition provided by a source outside the laboratory for analysis that evaluates the laboratory's analytical performance.
- 6.1.23 **Post Digestion Spike (PS).** A spiked aliquot of an already digested sample which is used to demonstrate spike recoveries in the sample matrix during analysis. The analytical results of the PS may help determine if poor MS/MSD recoveries were a result of the digestion process or are matrix related. Comparison of the PS recoveries to MS/MSD recoveries, in conjunction with the DUP and SRD results, is a useful tool to verify matrix interference when the MS/MSD fail to meet criteria.
- 6.1.24 **Serial Dilution (SRD).** The dilution of a sample by a factor of five. The SRD may indicate the influence of physical or chemical interferents due to sample matrix. The SRD is an analytical QC, therefore one is required per twenty field samples analyzed.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 9 of 67

6.1.25 **Stock Standard Solution.** A commercially prepared standard solution (traceable to NIST or other certified standard sources), which can be diluted to derive other standards.

- 6.1.26 **Trip Blank.** A blank quartz filter sample that is transported with field samples without being exposed to sampling procedures. This blank sample assesses contamination introduced during shipping and field handling and transport procedures.
- 6.1.27 **Tuning Solution.** A solution used to determine acceptable instrument performance prior to calibration and sample analyses. This solution is used for mass calibration, nebulizer optimization, lens optimization, and daily performance reports.

### 6.2 Abbreviations

amu Atomic Mass Units

ASTM American Society for Testing and Materials CASRN Chemical Abstract Services Registry Number

CCT Collision Cell Technology

COC Chain of Custody cps Counts Per Second

DI Deionized

DQO Data Quality Objective

HNO<sub>3</sub> Nitric Acid

ICP-MS Inductively Coupled Plasma - Mass Spectrometry

KED Kinetic Energy Discrimination

kW Kilowatts L Liter(s)

LDPE Low Density Polyethylene

LIMS Laboratory Information Management System

 $M\Omega$  Megohm

MCA Multichannel Analyzer
MQO Method Quality Objectives

m Meter(s)

m<sup>3</sup> Cubic Meter(s)

mbar Millibar mg Milligram(s)

mg/L Milligram(s) per liter(s)

min Minute(s)
mL Milliliter(s)
mm Millimeter(s)
ms Millisecond(s)



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 10 of 67

ng Nanogram(s)

ng/L Nanogram(s) per liter(s) ng/mL Nanogram(s) per milliliter(s)

NIST National Institute of Standards and Technology

QC Quality Control RBS Reagent Blank Spike

RSD Relative Standard Deviation RSE Relative Standard Error RPD Relative Percent Difference

SD Standard Deviation

SOP Standard Operating Procedure

Std. Standard

STD Standard Mode

μg/L Microgram(s) per liter

μg/m<sup>3</sup> Microgram(s) per cubic meter(s) μg/mL Microgram(s) per milliliter(s)

μL Microliter (s) μm Micrometer

v/v Volume per volume ratio

#### 7.0 INTERFERENCES

**Note:** The background level of metals on a given lot of quartz and Teflon<sup>®</sup> filters can vary. Any background levels found on blanks should be documented for all the filters from the corresponding lot when available. It is recommended to consult 40 CFR Part 50, Appendix G for guidance.

#### 7.1 Laboratory Interferences

- 7.1.1 Wear powder-free nitrile or neoprene gloves when handling unexposed or exposed filters.
- 7.1.2 Maintain and clean all equipment used in the sample preparation and analysis in a manner consistent with good laboratory practices for metals analysis (See Section 20 in this SOP for instrument maintenance and Section 14.3.1 in SOP ERG-MOR-031 for glassware cleaning).
- 7.1.3 Use ASTM Type I DI water or equivalent, with a resistivity  $\geq 18 \text{ M}\Omega$ , for sample extraction and standard preparation. Record the water resistivity in the "Deionized Water Treatment Log" prior to use.

### 7.2 Chemical Interferences

Pay close attention to the nature of solutions introduced to the ICP-MS.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 11 of 67

- 7.2.1 Total acid concentrations should be less than 2% (v/v) for ICP-MS analysis to minimize the damage to the interface and to minimize isobaric molecular interferences. Although higher acid concentrations may be aspirated for short periods of time to help clean sample introduction components, this time should be minimized to help reduce the wear effects it may have on susceptible components. The use of platinum cones and other acid-resistant sample introduction components can be used for more aggressive acid matrices.
- 7.2.2 The final dilutions of sample extracts must match the acid content of the calibration standards to prevent potential interferences.
- 7.2.3 The concentrations of dissolved solids in analysis solutions should be less than 2% to protect the sample interface on the instrument and prevent signal suppression. Higher concentrations may plug the sample and/or skimmer cone orifices.

**Note:** Protect the channel electron multiplier from high chemical concentrations (high ion currents). The channel electron multiplier suffers from fatigue after being exposed to high ion currents. This fatigue can last from several seconds to hours depending on the extent of exposure. During this period, response factors are constantly changing, which causes instrument instability that invalidates the calibration curve, and thereby, invalidates all associated sample results. A sodium bicarbonate (NaHCO<sub>3</sub>) sample matrix is known to cause this problem.

### 7.3 <u>Instrument Interferences</u>

- 7.3.1 Molecular ion interferences are caused by polyatomic ions (e.g., the contribution of 40Ar35Cl+ on the 75As signal), while double-charged ion interferences are caused by doubly charged element isotopes whose mass is twice the mass of the analyte isotope of interest (example, 138Ba++on 69Ga).
- 7.3.2 Isobaric interferences result from the presence of non-target elements whose isotopes have a very similar mass as the analyte of interest (e.g., 82Se and 82Kr) that cannot be differentiated by low resolution quadrupole mass spectrometers.
- 7.3.3 Transport interferences are a specific physical interference associated with the sample nebulization and transport process through the instrument. These usually result from sample matrix components that influence the aerosol formation or cause a change in the surface tension or viscosity. Changes in the matrix composition can cause observed signal suppression or enhancement.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 12 of 67

- 7.3.4 Matrix interferences may be caused by elemental chemical and physical properties in the samples. For matrices of known composition, match the composition of the calibration and QC standards to that of the samples. For matrices of unknown composition, use an ISTD that has been matched to the analytes' chemical and physical properties (i.e., ionization potential, ±50 amu) so that the ISTD and element of interest behave similarly during the analytical process.
- 7.3.5 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sample and skimmer cones, spray chamber, peristaltic pump tubing and the type of nebulizer all affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interferences.
- 7.3.6 Lead values are reported from isotope 208; however, all three isotopes must be used to quantitate lead to allow for the variability of lead isotopes in nature. The following correction equation must be applied to isotope 208:

$$(1.000) (^{206}Pb) + (1.000) (^{207}Pb) + (1.000) (^{208}Pb)$$

### 7.4 Matrix Interferences

7.4.1 Antimony has a tendency to adhere to the quartz media filters, which is a known issue for blank spikes (BS/LCS) and matrix spikes(MS/MSD) samples. For this reason, antimony recovery failures in the quartz method for these QC samples are common. All analyses that show low recoveries for antimony in the blank spike and/or matrix spikes must be qualified with an "SL" flag to indicate the sample concentration is biased low.

### 8.0 SAFETY

- 8.1 Personal protection should be used for all work performed in the inorganic laboratory, (e.g., gloves, safety glasses, laboratory coats, etc.).
- 8.2 The compressed gas cylinders must be stored and handled according to relevant safety codes outlined in the corporate health and safety manual. In use, the cylinders must be secured to an immovable structure and moved using a gas cylinder cart.
- 8.3 Make sure that sample vials are kept capped and in racks to prevent spills.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 13 of 67

- 8.4 All personnel should be trained in the handling, extraction, and analysis of acidic samples for inorganic analysis. Extra precautions should be taken when working with hydrofluoric acid.
- 8.5 Strong acids must not be stored with organic solvents or samples.
- 8.6 Follow normal laboratory safety procedures as outlined in the ERG Health and Safety Manual and the site-specific laboratory SOP.

### 9.0 EQUIPMENT

### 9.1 ICP-MS

The Thermo Scientific iCAP Q and iCAP RQ ICP-MS instruments consist of an inductively coupled plasma source, ion optics, a collision cell chamber, a quadrupole mass spectrometer, a computer that controls the instrument, data acquisition and data handling software (Thermo Scientific Qtegra Software, Versions 2.10.4345.236 and 2.14.5122.306), a printer, an autosampler (CETAC ASX-520 for the iCAP Q and ESI 2DX for the iCAP RQ) and a chiller. The quadrupole mass spectrometer has a mass range of 2 to 270 amu. Examples of typical operating conditions are listed below.

#### **Typical Operating Conditions**

Plasma forward power 1.55 kW Plasma/Coolant argon flow rate 14.0 L/min Auxiliary argon flow rate 0.8 L/min Nebulizer flow rate 1.08 L/min Solution uptake rate 0.4 mL/min Spray chamber temperature 2.7 °C Detector mode Dual (Pulse counting/Analog) Replicate integrations Mass range (collected data) 4.6 - 235 amu Dwell time 0.03 to 0.2 mins

Number of MCA channels

Number of scan sweeps

1

Total acquisition time 6.22 min/sample

#### 9.2 Labware Drying Box

AirClean Systems PCR Workstation. Used for drying all inorganics labware. Provides clean drying area to prevent contamination.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 14 of 67

9.3 Branson 8510, sonication bath with heating capability.

#### 10.0 MATERIALS

- 10.1 Graduated polypropylene sample extraction vials with screw caps, 50 mL volume (certified to be within  $\pm$  0.2mL).
- 10.2 Pipettes with adjustable volumes ranging from 0.5 μL to 10 mL and disposable tips. Repeatable, mechanical pipettes, such as Eppendorf Research®, may be used and their accuracy and precision should be verified on a quarterly basis according to Section 16.0 of ERG SOP 091.
- 10.3 Miscellaneous: powder-free nitrile or neoprene gloves; disposable laboratory wipes; self-adhesive labels.
- 10.4 Volumetric flasks. Teflon<sup>®</sup>, Class A: 50, 100, 250 and 500 mL capacities.
- 10.5 Storage bottles. Wide and narrow mouth, Teflon® FEP (fluorinated ethylene propylene) with Tefzel ETFE (ethylene tetrafluoroethylene) screw closure: 50, 100, 250, 500, 1,000 and 2,000 mL capacities.
- 10.6 Wash bottles made of LDPE or Teflon® having 500 mL and 1 L capacities.
- 10.7 Graduated polyproplene autosampler tubes with stopper caps, 15 mL volume

### 11.0 CHEMICALS, REAGENTS, STANDARDS AND THEIR PREPARATION

**Note:** In general, chemicals, reagents and commercial stock standards expire when specified by the manufacturer. If the manufacturer does not provide an expiration date, then they shall expire one year from the opened date. Standards and other solutions prepared in-house expire no later than the earliest manufacturer's expiration date of any standard or reagent used for preparation. Examples of approved commercial vendors are Fisher Scientific and SCP Science. Standards procured from new vendors should be tested for accurate concentrations prior to use for analyses that include field samples. Proper disposal of hazardous waste is discussed in detail in the Solid and Hazardous Wastes SOP (ERG-MOR-033).

11.1 <u>High Purity Acids</u> - ultrapure and concentrated stored in Teflon<sup>®</sup> Bottles. These reagents are used for the preparation of sample extraction fluid and all standards.

**Note:** Concentrated high purity reagents are not necessarily 100% of the specified reagent. All percentages in this SOP are expressed in terms of volume per volume (v/v) rather than true percentages of reagent in solution.

- 11.1.1 Nitric Acid (HNO<sub>3</sub>), 60-70%
- 11.1.2 Hydrochloric Acid (HCl), 32-35%



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 15 of 67

- 11.1.3 Hydrofluoric Acid (HF), 47-51%
- 11.1.4 **Quartz** Reagent Blank/Standard solvent (0.3% (v/v) HCl, 1.11% (v/v) HNO<sub>3</sub> with 0.2 mg/L Au) This solution is used for the Calibration Blank (CAL1/ICB/CCB), as well as the base solution for calibration standards. See Appendix I for preparation.
- 11.1.5 **Teflon**® Reagent Blank/Standard solvent (0.3% (v/v) HCl, 1.11% (v/v) HNO<sub>3</sub>, 0.10% (v/v) HF with 0.2 mg/L Au). This solution is used for the Calibration Blank (CAL1/ICB/CCB), as well as the base solution for calibration standards. See Appendix II for preparation.
- 11.1.6 Rinse blank (2% (v/v) HNO3, 0.5% (v/v) HCl with 0.2 mg/L Au). See Table 24-14 for preparation.
- 11.2 ASTM Type I deionized water with a minimum resistivity of 18 M $\Omega$ .
- 11.3 <u>Argon gas</u> Purity > 99.996%, Oxygen < 5 mg/L, Hydrogen < 1 mg/L, Nitrogen < 20 mg/L and Water < 4 mg/L.
- 11.4 <u>Calibration Standards</u> A series of 5 non-zero calibration standards are used to calibrate the ICP-MS. Two different stock standards prepared in-house make these 5 standards. One prepares the LOQ standard and the second prepares the remaining four standards. These standards consist of CAL2 (LOQ), CAL3, CAL4, CAL5 (CCV), and CAL6 (HSV). See Appendices I and/or II as applicable, depending on analysis, for the preparation of these stock and working calibration standard solutions.
- 11.5 <u>Single-Element Stock Standard Solutions</u> Commercially prepared NIST traceable standards from ultra-high-purity grade chemicals or metals (99.99 99.999% pure) designed for use with ICP-MS instruments. Examples of some approved commercial vendors are High Purity Standards and Inorganic Ventures.
- 11.6 <u>Interference Check Standard</u> Commercially prepared multi-element standard that is diluted to prepare ICSA and ICSAB interferent checks. See Appendices I and/or II, depending on analysis, for quartz and Teflon® analysis preparation, respectively. An example approved commercial vendor is Spex CertiPrep.
- 11.7 <u>Tune Solution</u> The tuning solution for this SOP may be purchased through Inorganic Ventures (Thermo-4AREV) or prepared in-house using single-element standards to contain 15 μg/L of Mg and 1μg/L of Ba, Bi, Ce, Co, In, Pb, Li, and U



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 16 of 67

in 2.5% (v/v) HNO<sub>3</sub> and 0.5% (v/v) HCl. See Table 24-9 and 24-10 for preparation of stock and working solutions.

- 11.8 <u>Detector Cross Calibration Solution</u> Refer to Section 13.3 for more information about this solution. Although custom cross calibration solutions can be used, it may be purchased through Inorganic Ventures (Thermo 5A Setup Solution) or prepared in-house using single-element standards. See Tables 24-7 and 24-8 for preparation of stock and working solutions.
- 11.9 <u>Post Digestion Spike (PS) Standard</u> The PS standard is used to spike the PS source sample. This post digestion spike is used to help determine if poor matrix spike recoveries are due to interferents. Spike 1 µL per 1 mL of sample analyzed. See Table 24-13 for preparation.
- 11.10 <u>Second Source Standards</u> A commercially prepared single- or multi-element standard from a secondary source (different manufacturer from the multi-element calibration standard). These NIST traceable calibration standards are used to produce the ICV, which is run as a verification of the instrument's calibration for accuracy and precision. A second source multi-element stock standard is prepared in-house to create the working ICV solution. This set of standards is also used to prepare the PS standard. See Appendices I and/or II as applicable, depending on analysis, for the ICV preparation, and Table 24-13 for PS preparation.
- 11.11 <u>Internal Standards (ISTD)</u> Section 13.6 discusses internal standardization. See Appendix I or II, depending on analysis, for preparation.
- 11.12 <u>Citranox® Acid Cleaner and Detergent Bath</u> Prepare a 2% solution by adding 240 mL of Citranox® to 11.7 L of warm hot tap water for labware cleaning and decontamination. This detergent bath should be changed about once every month, depending on use.

### 12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

N/A

#### 13.0 CALIBRATION AND STANDARDIZATION

#### 13.1 Daily Optimization Procedures

Daily optimization is performed as necessary through the software's Autotune Wizard. Refer to Tables 24-4 and 24-5 for Optimization Procedures and Performance Report Specifications, respectively. For more detailed information,



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 17 of 67

the Thermo Scientific Qtegra software guide is available for reference as a PDF file on the instrument computer.

### 13.2 Mass Calibration and Resolution

Before performing any kind of calibration or optimization, allow a period of at least 30 minutes for instrument warm-up. After the warm-up, the mass calibration and resolution may be optimized using the "Thermo 5A Setup Solution" (see Section 11.8) by running a mass calibration and resolution optimization through the Mass Calibration Wizard. Resolutions for low to high masses are indicated by the following isotopes: 7 Li; 24, 25, and 26 Mg; 59 Co; 115 In; 206, 207, and 208 Pb; and 238 U. Peaks for each isotope may be viewed in the performance reports. The automated mass calibration wizard will adjust the spectrometer to  $\pm$  0.03 amu or better and the resolution to produce a peak width of 0.75  $\pm$  0.1 amu at 10% peak height. See Table 24-5 for acceptance criteria. Repeat the mass calibration wizard if the performance report mass calibration verification fails.

### 13.3 Detector Cross Calibration

The detector cross calibration is used to provide a smooth transition from the pulse counting mode to the analog mode, which extends the linear dynamic range of the detector. Thermo Scientific's "Setup Solution" (Thermo 5A) with various elements and concentrations is used for this calibration. This cross calibration must be performed daily before each analysis to ensure linearity when switching from pulse counting to analog mode.

### 13.4 Daily Performance

- 13.4.1 The daily performance report for both STD and KED modes must be generated and meet specifications before the start of each analysis.
- 13.4.2 Instrument stability must be demonstrated by running a daily performance check using the tuning solution. A minimum of 10 replicates with resulting relative standard deviations of absolute signals of less than 2% (or 5% for the 3 Mg isotopes in KED mode) is required prior to calibration. Performance specifications for the daily performance are listed in Table 24-5.

**Note:** The daily performance criteria differ between the two instruments. Be sure the correct criteria are followed for the instrument in use.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 18 of 67

### 13.5 Calibration

- 13.5.1 Prior to initial calibration, set up proper instrument software routines for quantitative analysis (i.e., autosampler table, QC sample names etc.). The instrument must be calibrated using a minimum of a calibration blank and five non-zero calibration standards. A minimum of three replicate integrations are required.
  - a. RSDs should be < 10.1% for all non-zero calibration standards for Tier I elements. All other elements may be reported with RSDs > 10% using "LJ" and "OX" qualifiers.
  - b. The calibration curve must have a coefficient of determination  $\geq 0.995$ .
  - c. The Relative Standard Error (RSE) for the measured concentrations of the first non-zero calibration point (CAL2) and the midpoint (CAL5) of the calibration curve must be within  $\pm 30\%$  or  $\pm 10\%$  of the nominal concentrations of the standards, respectively. See Section 15.5 for calculation.
- 13.5.2 Use the average of the integrations for instrument calibration and data reporting. The resulting curve must then be verified with high-level (HSV) and mid-level (ICV and CCV) calibration standards as described in Section 16.6 before the analysis of samples.
- 13.5.3 Use an ISTD that has been matched to the analytes chemical and physical properties (i.e., ionization potential,  $\pm 50$  amu) so that the ISTD and element of interest behave similarly during the analytical process.
- 13.5.4 The rinse blank should flush the system between solution changes for blanks, standards, and samples. Allow sufficient rinse time (≥ 1 min) to remove traces of the previous sample. Solutions should aspirate for at least 30 seconds prior to the acquisition of data to establish equilibrium.
- 13.5.5 Refer to Appendices I and/or II as applicable, depending on analysis, for the preparation of standard solvent. Once prepared, all calibration standards must be stored in Teflon® bottles, flasks can be used but only for temporary storage until clean Teflon® bottles become available.
- 13.5.6 Refer to the quality control requirements presented in Table 24-3 for calibration acceptance criteria.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 19 of 67

### 13.6 Internal Standardization

- 13.6.1 Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. Internal standard quality control requirements as described in Section 16.7 must be followed.
- 13.6.2 Internal standards for this method are <sup>6</sup>Li, <sup>45</sup>Sc, <sup>73</sup>Ge, <sup>89</sup>Y, <sup>115</sup>In, <sup>159</sup>Tb, <sup>165</sup>Ho and <sup>209</sup>Bi for analytes beginning with mass 6 and ending with mass 238. On the iCAP Q internal standards are added in-line with a mixing "T", on the iCAP RQ internal standards are manually spiked into each sample tube and calibration standard at a ratio of 2µl ISTD per 1 mL of solution.
- 13.6.3 Concentrations of the internal standards for this method are determined by the concentration of each element that will produce an intensity that is sufficiently stable. Typical intensities are between 10,000 and 1,000,000 cps; however, ideal intensities may exceed 1,000,000 cps.
- 13.6.4 Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. However, be aware that internal standards themselves may be responsible for polyatomic and/or doubly charged interferences.

#### 13.7 Instrument Performance

- 13.7.1 After instrument calibration, an ICV and ICB must be analyzed for initial verification of the calibration curve. Refer to Sections 16.6.1 for ICV, 16.2.2 for ICB, and Table 24-3 for specific QC criteria.
- 13.7.2 To verify that the instrument is properly calibrated on a continuing basis, analyze a CCV and CCB before the analysis of samples and after every 10 samples.

### 13.8 <u>Limit of Quantitation (LOQ) and Minimum Reporting Level (MRL) Check</u>

The LOQ is defined as 3.18 times the MDL, the level defined by the EPA as the Sample Quantitation Limit (SQL) and for which all field sample concentrations between the MDL and SQL are qualified prior to upload into AQS.

The LOQ is determined by spiking blank Teflon filters or quartz filter strips, and then extracting and analyzing as normal samples. Initially a 7 spike study must be performed then ongoing quarterly checks are done. These 7 spikes must be split among separate extractions and analyses. The average analyzed concentration between the seven spiked samples must be within 30% of the known concentration for the LOQ level to be confirmed.



## ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 20 of 67

The MRL check is prepared quarterly to verify the established LOQ. This check is performed by spiking a blank Teflon filter or quartz filter strip, and then extracting and analyzing as a normal sample. Quarterly MRL samples must be checked immediately upon analysis. The MRL concentration should be within 30% of known concentration.

For more information about the determination of the LOQ levels see SOP ERG-MOR-10.

### 13.9 <u>Linear Dynamic Range (LDR)</u>

While dilutions are the preferred course of action to account for sample results above the calibration range, a Linear Dynamic Range (LDR) may be used as well. This is done by analyzing one or more standards above the standard calibration range. Standards must recover within 10% of the nominal value to be accepted. Sample concentrations below 90% of the LDR concentration do not need to be diluted even if they exceed the highest calibration standard.

#### 14.0 PROCEDURE

### 14.1 <u>LIMS Sequence Procedure</u>

- 14.1.1 Log into the LIMS Software.
- 14.1.2 From the "Laboratory" menu, select "Sequence."
- 14.1.3 Click "Add" in the top right corner and select either "Randy FEM Teflon," or "Randy FEM Quartz," as the Template ID depending on which analysis is being performed, then click done. This will automatically add all the calibration standards and QC samples for a typical analysis sequence. The software will automatically fill in the instrument name, be sure to check that the appropriate instrument is selected.
- 14.1.4 Click the pull-down menu for "Source Batch" at the upper middle part of the screen and select the batch desired for analysis. Then click the "Add" button and select "Batch QC Sample." When the list of QC samples appears click the first sample and holding the shift button double-click the last sample and it will insert them into the sequence. Then click "Add" again and select "Batch Sample." Add all the samples listed as done previously for the Batch QC Samples.
- 14.1.5 Add in two Tune Samples to the beginning of the sequence. TUN1 will represent the 5A solution used during setup, TUN2 will represent the Tune solution used for tuning/performance reports. Make sure these TUN



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 21 of 67

samples are properly labeled with the correct standard ID and name ("5A" & "Tune/Performance report"). Arrange all the samples following the method sample list template in the Qtegra software.

	Quartz	Teflon®
Autosampler	Sample Name	Sample Name
Rack/Position		
	1611018-TUN1	1611018-TUN1
	1611018-TUN2	1611018-TUN2
Standard 10	1611018-CAL1	1611018-CAL1
Standard 9	1611018-CAL2	1611018-CAL2
Standard 8	1611018-CAL3	1611018-CAL3
Standard 7	1611018-CAL4	1611018-CAL4
Standard 6	1611018-CAL5	1611018-CAL5
Standard 5	1611018-CAL6	1611018-CAL6
Standard 4	1611018-ICV1	1611018-ICV1
Standard 10	1611018-ICB1	1611018-ICB1
Standard 5	1611018-HCV1	1611018-HCV1
Standard 2	1611018-IFA1	1611018-IFA1
Standard 1	1611018-IFB1	1611018-IFB1
Standard 6	1611018-CCV1	1611018-CCV1
Standard 10	1611018-CCB1	1611018-CCB1
Sample 1/1	B0B1707-BLK1	B0B1707-BLK1
Sample 1/2	B0B1707-BLK2	B0B1707-BLK2
Sample 1/3	B0B1707-BS1	B0B1707-BS1
Sample 1/4	B0B1707-BS2	B0B1707-BSD1
Sample 1/5	0020993-01	B0B1707-BS2
Sample 1/6	B0B1707-DUP1	0020993-01
Sample 1/7	B0B1707-MS1	B0B1707-DUP1
Sample 1/8	B0B1707-MSD1	B0B1707-SRD1
Sample 1/9	B0B1707-SRD1	B0B1707-PS1
Sample 1/10	B0B1707-PS1	0020993-02
Standard 6	1611018-CCV2	1611018-CCV2
Standard 10	1611018-CCB2	1611018-CCB2
Standard 6	1611018-CCV3	1611018-CCV3
Standard 10	1611018-CCB3	1611018-CCB3

**Note**: Autosampler positions may vary, this template serves as a typical example. Verify listed positions match actual sample/standard placement.

14.1.6 Right-click the SRD sample and choose the appropriate source sample from the batch to ensure the LIMS correctly calculates the RPD from the source sample. Preference should be given to the parent sample on which batch QC is performed (DUP/MS/MSD).



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 22 of 67

For sequences with more than twenty field samples, the second SRD should be assigned to the parent sample for the second batch of samples; if none then choose any non-blank field sample with sufficient extract.

- 14.1.7 Using the shift key, select all samples in the sequence (excluding the TUN samples) and right-click to choose "Internal Standard ID" then select the ISTD being used for this sequence.
- 14.1.8 Right-click each calibration standard and QC sample and select "Standard ID" to set the current standard being used for each solution. If these IDs are not properly set the LIMS may not correctly calculate recoveries.
- 14.1.9 In the comments section of the sequence be sure to include pipette serial numbers for SRD and PS, as well as the LIMS ID of reagent used for the SRD.
- 14.1.10 Click "Save" and print a double-sided copy in landscape format to include with the data package. Click "edit" and then use the "export" button to create a CSV file which will be used to create the labbook in the instrument software.

### 14.2 Sample Analysis Procedure

- 14.2.1 Prior to analyzing samples, run the mass calibration and detector cross calibration using the 5A solution, and check the instrument performance by analyzing the tuning solution through the Performance Report Wizard. The performance specifications that must be met are in Table 24-5. If the performance check fails, repeat or follow the optimization procedures in Table 24-4.
- 14.2.2 Before starting the calibration, be sure to flush the sample introduction system with sufficient rinse blank and be certain the rinse blank bottle has enough solution for the analysis. Use the previously created CSV file and the appropriate template (Teflon or quartz) to create a unique labbook for each sequence. The labbook should be named using the format of "Sequence number, Batch number, month, day and year, followed by either a Q for quartz, or T for Teflon®" (e.g., 1512013\_B5L0402\_12082015Q) Enter all sequence information (sample and QC sample names) into the Sample List in the Qtegra software and save.
- 14.2.3 *Prepare standards:* Standards are typically prepared in 100mL amounts, and then run from 10mL pour offs in sample tubes. For standards which are run multiple times, such as the CCV, it is advisable to prepare multiple tubes



## ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 23 of 67

so that they do not run empty. Once all standard tubes have been labeled and prepared, they should be placed in the autosampler, and ensure the positions match what is set in the instrument software, taking care to update autosampler positions for multiple tubes of standard.

**Note:** For standards used on the iCAP RQ, standards are spiked with ISTD immediately after pouring off to ensure proper ISTD concentration. For a 10 mLsample tube, add  $20\mu L$  of ISTD and homogenize.

- 14.2.4 *Prepare Field Samples:* Label autosampler tubes with a black marker or printed labels with the LIMS ID. For samples run on the iCAP Q, pour off  $\sim 5.0$  mL of homogenized sample to each of the autosampler tubes and place in appropriate autosampler location for analysis. For samples run on the RQ, tubes are first spiked with  $10\mu L$  of ISTD and then 5 mL of sample is pipetted into the appropriate tube.
- 14.2.5 *Prepare the SRD*: The SRD sample is a 5x dilution prepared for the iCAP Q by diluting 1 mL of sample with 4 mL of reagent blank. When preparing an SRD to run on the RQ, 10µL of ISTD is spiked, followed by 1mL of sample and 4mL of reagent blank without ISTD spiked into it as for the calibration blank.
- 14.2.6 *Prepare the PS*: The PS sample is prepared by adding 1.0 μL of spike solution to 1.0 mL of sample to be analyzed (i.e., 5 μL of PS to 4.995 mL of sample). When preparing a PS to run on the iCAP RQ, 10μl of ISTD is spiked, then 5μL of PS, and 4.995mL of sample.
- 14.2.7 Begin instrument analysis.
- 14.2.8 Samples with analyte concentrations greater than the highest calibration concentration must be diluted and re-analyzed or flagged if there is not a valid LDR.

#### 14.3 Unexpected Instrument Shutdown

If the ICP-MS shuts down during an analysis the proper procedure to be followed by the analyst is:

14.3.1 If the analyst was present during the loss of the plasma and the instrument has not significantly cooled, then a shorter time for warm-up may be sufficient. Restart the instrument and allow it to warm-up for a minimum of 15 minutes. If the instrument has been inoperable overnight and is at room temperature warm-up for 30 minutes.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 24 of 67

- 14.3.2 After the instrument has been thermally stabilized a new daily performance report (DPR) should be analyzed with the operating conditions being used for the analysis.
- 14.3.3 If the DPR passes the analysts must check the calibration by analyzing a continuing calibration verification (CCV) and a continuing calibration blank (CCB) to be sure the calibration is still valid. If the CCV & CCB pass the analyst may proceed with where the analysis left off. Any samples that did not complete their analysis should be repeated.
- 14.3.4 If the DPR, CCV or CCB do not pass the analysis must be terminated and any samples not bracketed by valid CCV & CCB checks must be reanalyzed with a new analysis/calibration.
- 14.3.5 The DPR, CCV and CCB checks must be kept for documentation. The analyst should document the event briefly in the sequence narrative, so the reviewer is aware of the instrument shutdown.

### 14.4 LIMS Data Upload Procedure

- 14.4.1 When a data package is complete, the analyst will transfer the data from the instrument computer to the appropriate network server. An xls data file and pdf report should be exported from the instrument software to a network folder. These will be used for data upload and review.
- 14.4.2 To begin upload into the LIMS, open the "Element" software. Go to the laboratory menu and click on "Data Entry/Review." In this window, select "Sequence" in the top left corner, making sure that "Inorganics" is selected from the drop-down menu. Highlight the correct sequence and click on "Create" in the Data Entry box in the top right corner. Once the spreadsheet is created in the LIMS, select "DataTool" in the Data Entry box and save the file as the sequence name in the same location as other sequence files.
- 14.4.3 The DataTool interface should open to the "Select Data System Files" window. In this window, check to make sure the correct file information is selected:
  - 1) File Type: Thermofisher ICAP Qtegra v2.2XLS (\*.xls, \*.xml)
  - 2) Drives: y:\Air Toxics
- 14.4.4 In the box below Drives: select the folder the data is stored in for the sequence. In the Y drive, select the ICP-MS DATA folder, then the corresponding year, and finally the folder for the data the sequence was run.



## ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 25 of 67

- 14.4.5 All of the data files for that sequence will appear in the lower right-hand box. Double-click on the appropriate data file and click "Auto Select" for each file that needs to be included in the sequence. Pay attention as samples that were re-run in analysis may have duplicate entries in this section, ensure the correct sample run is selected.
- 14.4.6 Click "Done" when all sample and QC files have been selected to return to the main window. Click "Merge Files" at the bottom of the window. DataTool will merge the files and show the data in the Data Transfer window.

**Note:** Confirm that the number of samples on each side matches. Other than a few specific cases, the number of samples listed in the Instrument Data File should match the number of files from the Element Data Entry Table. Review the content of the top windows in the Data Transfer window for red text. If there is any, the DataTool cross table requires editing. Seek the advice of the LIMS administrator to correct this.

- 14.4.7 Update the analyst and instrument ID, click "Save" and save the spreadsheet in the same folder as other analysis documentation. Close DataTool.
- 14.4.8 In Element, go back to the Data Entry/Review window. The newly merged data should appear in the window. Click "Save" to save the files to Element and then "Query" in the Data Review box. Element will perform all necessary calculations at this point.
- 14.4.9 In the Data Entry/Review window, samples and QC can be reviewed for pass/fails. Any data that does not pass its assigned criteria will have red text. Use appropriate data qualifiers to flag data that does not meet criteria.

#### 14.5 LIMS Dilution Data Upload Procedure

- 14.5.1 Browse to source batch and add a re-extract of the diluted sample to the same batch. Include a comment about the type and reason for dilution (5x Dilution for Pb).
- 14.5.2 Open Element and go to the Data Entry/Review window. Select the sequence that the diluted sample was originally analyzed on. Click on "Query."

**Note**: If the dilution was performed on a later analysis then the re-extract will be included in that sequence, not the original. Data query can also be performed by Batch, in which case the original and dilution will be in the same table.

14.5.3 In the Data Entry/Review window, unselect the reporting box on the original sample for any analytes requiring dilution. Only mark the desired



## ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 26 of 67

analytes for reporting on the re-extract. Update the dilution value in the LIMS, the software will perform any recalculations required based on the dilution factor.

- 14.5.4 Right click on the sample & analyte that has dilution information and select "Qualifiers" -> "Quick Analyte Qualifiers." Select qualifier flag "D" for dilutions.
- 14.5.5 Repeat steps 1 through 4 for any additional dilution data that needs to be input for the sequence. If any QC data was altered (for example, Dups) the LIMS will prompt you to click on "Re-calc" and then "Save." If only sample information was altered, click "Save."
- 14.5.6 Re-run the query to verify all dilution data was saved to the sequence.

**Note:** In the LIMS, the dilution factor is applied to the associated MDL as well as to the sample concentration.

#### 14.6 Data Review

All instrument data should be first reviewed by the analyst and then a secondary reviewer, usually the project Task Lead for metals analysis. Both the analyst and secondary review must use the "Quality Control Requirements for Metals Analysis" checklist to complete data review (see Figures 24-1 or 24-2 as applicable). Reviewers must initial and date each parameter check on the review form to verify that each meets the established acceptance criteria.

#### 14.6.1 Initial Calibration

In addition to the requirements outlined in Figures 24-1 and 24-2 (as applicable), the analyst and secondary reviewer must also verify that the intensities measured for reportable analytes in the calibration blank are acceptable and will not interfere with the sensitivity. A review of previously analyzed calibration blanks can demonstrate acceptable intensity values. The intensities of the internal standards in the calibration standard should be monitored relative to the intensities seen in the calibration blank.

#### 14.6.2 Internal Standards

Internal standards must be monitored for each sample throughout a sequence; the measured intensities must stay between 60 and 125% of the measured intensity of internal standard in the calibration blank; however, dramatic changes in internal standard recovery during an analysis should be investigated and possibly warrant reanalysis of affected samples even if



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 27 of 67

recoveries stay within criteria. See Section 16.7 for corrective actions to remedy internal standard intensities that are measured outside of this range.

### 14.6.3 Relative Standard Deviations (RSDs)

Follow the prescribed acceptance criteria for RSDs of calibration standards as listed in Table 24-3. Sample RSDs should also be monitored throughout analysis. High RSDs (greater than 20%) for concentrations above the MDL can indicate memory interference from previous samples, as well as other instrumentation issues that may need to be corrected before analysis can be continued.

#### 14.6.4 Element/LIMS Data

The analyst and second reviewer must verify that quantities imported into the LIMS reflect the raw data. This can be accomplished by checking a few analytes for random client samples and QC samples. Hand entered data (i.e., dilution) should be verified by the second reviewer to be certain the values, dilution factors and flags are properly inserted. The LIMS calculated final value should also be checked to ensure the system is correctly using the method's custom equation.

#### 14.6.5 Multiple Isotopes

If an element has more than 1 monitored isotope, examine the concentration calculated for each isotope, or isotope ratios, to detect possible interferences. Consider both primary and secondary isotopes when evaluating the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes; therefore, differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes.

#### 15.0 CALCULATIONS

### 15.1 Quartz Filter Analyte Concentration

15.1.1 Metal concentration in **ng/m<sup>3</sup>** should be calculated as follows:

$$C = \frac{[C_i \times V_f \times 9]}{V_{std}}$$

Where:

 $C = concentration, ng/m^3$ 



### ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 28 of 67

 $C_i$  = metal concentration determined from Section 14.2, ng/L.

 $V_{\rm f}$  = final sample extraction volume from extraction procedure (i.e., 0.05 L).

9 = [Usable filter area  $(8"\times9")$ ] / [Exposed area of one strip  $((4"\times1")\times2$  (representing a strip that has been folded)]

 $V_{std}$  = standard air volume pulled through the filter,  $m^3$ 

15.1.2 Metal concentration in µg/filter should be calculated as follows:

$$C = \frac{[C_i \times V_f \times 9]}{1000}$$

Where:

 $C = concentration, \mu g/filter$ 

 $C_i$  = metal concentration, ng/L.

 $V_{\rm f}$  = final sample extraction volume from extraction procedure (i.e., 0.05 L).

9 = [Usable filter area  $(8"\times9")$ ] / [Exposed area of one strip  $((4"\times1")\times2$  (representing a strip that has been folded)]

1000 = conversion factor, ng/µg

### 15.2 Teflon® Filter Analyte Concentration

Metal concentration in **ng/m**<sup>3</sup> should be calculated as follows:

$$C = \frac{(C_i \times V_f)}{V_{std}}$$

Where:

 $C = concentration, ng/m^3$ 

 $C_i$  = metal concentration, ng /L.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 29 of 67

 $V_{\rm f}$  = final sample extraction volume from extraction procedure (i.e., 0.05 L).

 $V_{std}$  = standard air volume pulled through the filter,  $m^3$ 

Metal concentration in  $\mu$ g/filter should be calculated as follows:

$$C = \frac{(C_i \times V_f)}{1000}$$

Where:

 $C = concentration, \mu g/filter$ 

 $C_i$  = metal concentration, ng /L.

 $V_{\rm f}$  = final sample extraction volume from extraction procedure (i.e., 0.05 L).

 $1000 = \text{conversion factor}, \text{ng/}\mu\text{g}$ 

### 15.3 Method Detection Limits

The MDL is calculated as follows using the DQ FAC MDL calculation method:

$$MDL = (K) \times (SD)$$

Where:

K = K Value as prescribed in Appendix D: DQ FAC Single Laboratory Procedure v2.4, 08/30/2007

SD = standard deviation of the historical BLK2 analysis.

### 15.4 Relative Percent Difference (RPD)

The RPD is calculated as follows:

$$RPD = \frac{R_1 - R_2}{(R_1 + R_2)/2} \times 100$$

Where:

 $R_1$ ,  $R_2$  = values that are being compared (i.e., duplicate and replicate analysis data)



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 30 of 67

### 15.5 Relative Standard Error (RSE)

The RSE is calculated as follows:

$$RSE = \frac{R_1 - R_2}{R_1} \times 100$$

Where:

 $R_1$  = Nominal concentration value (ng/L)

 $R_2$  = Measured concentration value (ng/L)

### 15.6 Percent Recovery

Percent Recovery is calculated as follows:

$$Percent \ Recovery = \frac{Analytical \ Result}{Theoretical \ Result} \times 100$$

### 15.7 Relative Standard Deviation (RSD)

RSD is calculated as follows:

$$RSD = \frac{Standard\ Deviation}{Average} \times 100$$

### 15.8 <u>Limit of Quantitation (LOQ)</u>

The LOQ is defined as 3.18 multiplied by the MDL and expressed in ng/L.

$$LOQ(ng/L) = MDL_{fac} * 3.18$$

### 15.9 Internal Standard Correction and Interpolation

This method makes use of internal standard correction and interpolation, which adjusts the sample concentration result based on the variance of the two ISTDs that bracket the element over the course of the analysis. This is done automatically in software as data is analyzed. ISTD correction adjusts the sample result based on the variance of the ISTD intensity per sample when compared to the first blank which is established as the 100% baseline. Interpolation makes use of each ISTD that brackets each analyte to weigh the correction based on which ISTD is a closer mass to the analyte.



### ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 31 of 67

Every measured intensity *i* will be corrected with the appropriate internal standard correction factor *gISC*. Using the reference sample, *gISC* is calculated by:

$$gISC = \frac{st}{sr}$$

Where *sr* and *st* are the reference and target sensitivity of the internal standard isotope:

$$sr = \frac{ir}{cr}$$
 and  $st = \frac{it}{cs}$ 

with *ir* as the average intensity in the reference sample over all runs, *it* as the target intensity in the corrected sample, and *cr* and *cs* as the known concentration of the reference and the corrected sample, respectively. The target sensitivity is calculated for every run in every sample differing from the reference.

For every none-standard isotope with an internal standard defined, the measured run intensity will be corrected according to:

$$icorr = \frac{i}{gISC}$$

#### Interpolation corrections

Additionally, a refined internal standard correction is offered. Let gISC1, gISC2 be the internal standard correction factors for two consecutive Internal Standards with masses m1, m2.

An isotope x with intensity ix and mass mx with  $m1 \le mx \le m2$  will be corrected with gISC given by the linear interpolation:

$$gISC = gISC1 + \frac{gISC2 - gISC1}{m2 - m1} * (mx - m1)$$
 and  $ixcorr = \frac{ix}{gISC}$ 

#### 16.0 QUALITY CONTROL

The analyst must perform the quality control checks listed in Table 24-3 and meet the requirements in this section. Data Quality Objectives (DQO) and data assessment criteria are determined from the results of the quality control samples. The DQO criteria are summarized in Table 24-3.

### 16.1 Sample Collection Quality Control

16.1.1 Filters which are dropped or become contaminated with any foreign matter (i.e., dirt, finger marks, ink, liquids, etc.) are invalid.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 32 of 67

16.1.2 Filters with tears or pinholes that occurred before or during sampling are invalid.

16.1.3 A power failure during a field sample collection event invalidates the sample collected during that event. See SOP ERG-MOR-045 for more information about the sample acceptance criteria.

### 16.2 Blanks

- 16.2.1 The Rinse Blank must be used to flush the system between standards and samples. Refer to Table 24-14 for preparation details.
- 16.2.2 Initial Calibration Blank (ICB) is analyzed immediately following the ICV. The absolute value of the instrument response should be less than the s\*K portion of the MDL. If the ICB fails it may be re-analyzed once. If it fails again, a fresh ICB solution from the same LIMS ID used for the calibration blank (CAL1) may be poured and analyzed. If the ICB still does not pass, the analysis should be terminated, the problem corrected and the ICV and ICB must be verified again before the analysis can continue. The calibration itself should be scrutinized at this point and sample introduction components suspected of causing the issue should be examined.
  - If the CAL1 prior to the calibration standards fails, the analysis must be reanalyzed from the calibration blank. If the ICB analyzed after the ICV fails, the analysis can be reanalyzed from the ICV.
- 16.2.3 Continuing Calibration Blanks (CCB) are analyzed following each continuing calibration verification sample. The absolute value of the CCB should be <the s\*K portion of the MDL. If the CCB fails it may be reanalyzed once. If it fails again, a fresh CCB solution from the same LIMS ID used for the calibration blank (CAL1) may be poured and analyzed. If the CCB continues to exceed criteria, all samples not bracketed by passing CCBs must be qualified with the "LL" or "LK", for negative or positive failures, respectively, and "QX" flags for the failing element; however CCB failures suspected to be caused by analysis issues should be investigated and associated samples should be reanalyzed if possible.
- 16.2.4 Laboratory Reagent Blanks (LRB/BLK1) are analyzed immediately following opening instrument QC. If the absolute value of the analyte concentration of the LRB is < MDL (See Tables 24-1 or 24-2 as applicable), no further action is required. If the BLK1 fails it may be reanalyzed once. If it fails reanalysis, the BLK1 and all associated batch samples are flagged.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 33 of 67

If the element fails positively, then all batch samples are flagged with the "B" flag.

See Section 16.10 for guidance about negative analyte results.

16.2.5 The Method Blank (MB/BLK2) is analyzed immediately following the BLK1. The absolute value of the measured concentrations should be less than the MDL, and if not, the elements >MDL in BLK2 should be flagged with the "B-02" qualifier. Resulting BLK2 data is recorded and used to determine FAC MDL values.

Note: It is important to appropriately change QC sample names on the bench sheet.

### 16.3 Spikes

- 16.3.1 Reagent Blank Spike (RBS) is analyzed immediately following the BLK2 (as the BS2). The results of the spike recovery for target analytes should be within 79.9-120.1% of actual values. If they fail, they can be reanalyzed for verification or all associated batch samples are flagged as appropriate with an "LJ" (biased) and "QX" flag.
- 16.3.2 Laboratory Control Samples (LCS/BS) are analyzed immediately following the BLK2. The results of the spike recovery for target analytes must be within 79.9% to 120.1% of actual values. If they fail, they can be reanalyzed for verification or all associated batch samples are flagged as appropriate with an SL (biased low) or SH (biased high) flag. It is known that spiked antimony (Sb) recoveries are poor in quartz BSs. For quartz analyses where Sb is <80% in the BS, Sb in this QC sample and all batch samples should be flagged with the "SL" flag.

**Note:** In cases where the LCS failure can be traced to background contamination known to be present in the filter media, the LCS and data will not be flagged. If the LCS failure cannot be traced to the background contamination in the filter media, it must be flagged appropriately. See Section 22.8.2 for more information about reporting/flagging BSs when background from blank filter strips is present.

16.3.3 The Matrix Spike and Matrix Spike Duplicate (MS/MSD) are analyzed immediately following the parent sample and extraction duplicate. These samples are expected to recover within 79.9% to 120.1% of the true value and have <20.1% RPD. Any qualifiers used on the MS/MSD sample must also be applied to the parent sample.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 34 of 67

- If the parent sample concentration is >4x the spike concentration, the failing elements of the MS/MSD and parent sample should be flagged with the "QM-4X" qualifier.
- If the spike failure is from what is known or appears to be a non-homogenous matrix the failing element(s) are flagged in the MS/MSD and parent sample with the "QM-06" qualifier.
- If the parent sample concentration is <4x the spike concentration, the MS and/or MSD may be reanalyzed once each to verify a spike recovery failure. If they still do not pass, examine the results of the PS and SRD for possible matrix interference.
- If matrix interference is suspected, the failing elements of the MS and/or MSD sample(s), parent sample and samples of the same matrix, must be flagged with the "QM-05" qualifier. The interpretation of failing spike recoveries for MS/MSD samples can be complicated. For this reason, if the corrective action is unclear, the experienced Task Lead will determine if the data should be qualified or if further sample analysis should be performed (i.e., dilutions of parent, DUP, MS/MSD, PDS and SRD) to help guide qualifying the batch data.

**Note**: Due to the lack of actual sample duplicates for Teflon<sup>®</sup> filter sampling events, the traditional MS/MSD are replaced with Blank Spike Duplicates (BS/BSD). See Section 16.3.1 for information pertaining to BS/BSD.

- As with quartz BSs, it is known that spiked antimony (Sb) recoveries are poor in quartz MS/MSDs. For quartz analyses where Sb is <80% in the MS and/or MSD, Sb in these QC samples and all batch samples should be flagged with the "SL" flag.
- 16.3.4 Post Digestion Spike (PS): The PS is an analyzed aliquot of an extracted sample, preferably same parent sample used for DUP1, that is spiked with the PS standard (See Section 11.9). Preferably, the same sample that is used for the MS/MSD is also spiked for the PS. The spike recovery should be within 74.9% to 125.1% of the true spike value. If the PS fails, the analyst should assess the SRD. If both the PS and SRD fail for the same element it is an indication that matrix interference has occurred and any analytes that have failed should be flagged as possible matrix interference. If the parent sample used for the PS requires dilution, the PS should be prepared by diluting the spiked sample.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 35 of 67

### 16.4 <u>Duplicates</u>

- 16.4.1 Matrix Spike Duplicate (MSD): A spiked laboratory duplicate of a separate strip of the parent (also called source) sample filter used to prepare the matrix spike. The RPD for this duplicate is < 20.1% of the MS sample. This is only applicable to quartz analyses.
- 16.4.2 Blank Spike Duplicate (BSD): A laboratory duplicate of the blank spike prepared using a separate blank Teflon<sup>®</sup> filter. The RPD for this duplicate is < 20.1% of the BS sample. This is only applicable to Teflon<sup>®</sup> analyses.
- 16.4.3 Laboratory Duplicate (DUP1): For Quartz samples this duplicate is prepared with an additional strip of the parent (or source) sample filter. An RPD of < 20.1% is expected for comparison of parent and laboratory duplicate values ≥ 5x MDL; however, sometimes RPDs exceed 20% and may need confirmation before flagging. If one of the two values is less than 5x MDL, substitute 5x MDL for that value when calculating the RPD. Laboratory duplicates that fail the RPD criteria should be qualified with "D-F" on the parent and the DUP1 sample.
- 16.4.4 Collocated Samples (C1, C2): Both filter media types may have collocated samples collected for analysis. Collocated samples are expected to meet the same criteria as laboratory duplicates for both filter types. If one of the two values is less than 5x MDL, substitute 5x the MDL for that value when calculating the RPD. Collocated samples that fail the RPD criteria should be qualified with a "D-F" flag on both samples. See Table 24-3 for frequency and criteria.
- 16.4.5 Analytical Duplicate (or Replicate): A second aliquot of an extracted sample analyzed using the same analytical method. This includes all DUP QC samples for the Teflon method and DUP2 or greater QC samples for the Quartz method. Replicates are typically performed on collocated field samples. One analytical duplicate must be analyzed per 20 samples on an analysis. Preference is given to a collocated pair if available (in which case there should be one DUP for each C1/C2), but any non-blank field sample can be selected. The RPD for analytical duplicates (replicates) shall be < 10.1% for values ≥ 5x MDL. If one of the two values is less than 5x MDL, substitute 5x MDL for that value when calculating the RPD. Analytical replicates are expected to meet criteria for elements that meet the threshold, therefore reanalysis of failures should be considered on case-by-case basis. Replicate analysis that fail should be qualified with an "R-F" flag on both the parent and DUP sample.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 36 of 67

The following guidance should be used when determining if reanalysis is necessary:

### 16.5 Performance Evaluation (PE) Samples

Performance evaluation samples should be obtained as available from independent sources and analyzed as a routine sample. PE samples are prepared and analyzed in the same way as field samples.

### 16.6 Standard Checks

16.6.1 Immediately after the initial calibration, the ICV is analyzed. The measured concentrations should recover within ±10.1%, or 89.9-110.1%, of the actual concentration. If the criteria are not met, reanalyze the standard. If the criteria are still not met, a fresh standard may be prepared and analyzed or repeat the initial calibration and ICV.

This standard is critical for Tier I elements (See section 4.2) so any failures must be reanalyzed. This standard is operational for Tier II elements or non-Tiered elements so failures may not require reanalysis, the CCV and associated samples may be qualified with "LL" or "LK", as appropriate, and "QX".

- 16.6.2 The HSV must be analyzed after the ICB and prior to analysis of samples. The measured concentration should recover within 94.9%–105.1% of the actual concentration. If the HSV fails it may be reanalyzed once. If the HSV still fails, a fresh standard may be prepared and analyzed. If it continues to fail, the instrument must be recalibrated; however, if this is not possible qualify the HSV and correlated field samples that have concentrations above the CCV with the "LL" or "LK" flag, as appropriate, and "QX".
- 16.6.3 Before the analysis of samples and after every 10 samples during a batch analysis, the calibration must be verified using a CCV. Results must be 89.9%–110.1%, of the target value for each analyte to verify that the calibration is valid. If a standard check exceeds the limit, the check standard should be reanalyzed. If the target value exceeds the limit again, a fresh standard may be prepared and analyzed, or the instrument must be recalibrated. Any samples analyzed before or after an invalid CCV must be reanalyzed, minimally for the failing element.

This standard is critical for Tier I elements (See section 4.2) so any failures must be reanalyzed. This standard is operational for Tier II elements or non-Tiered elements so failures may not require reanalysis, the CCV and



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 37 of 67

associated samples may be qualified with "LL" or "LK", as appropriate, and "QX".

### 16.7 <u>Internal Standards</u>

The intensities of all ISTDs must be monitored for every analysis (see Table 24-6 for isotopes). When the intensity of any ISTD fails to register between 60 to 125% of the intensity of that ISTD in the calibration blank, the following procedure is implemented:

- 16.7.1 If the intensities exceed 125% recovery because the internal standard element was determined to be present in the field sample, the sample must be diluted and reanalyzed.
- 16.7.2 If necessary, repeat and increase the dilution until the internal standard intensities fall within the prescribed window, preferably as close to the recovery seen for other unaffected field samples in this analysis.
- 16.7.3 If the intensities are determined to be a result of instrument drift (low or high), stop the analysis, find and correct the problem, recalibrate if needed and reanalyze the affected samples since the last acceptable ISTD recoveries.

### 16.8 Interferences

- 16.8.1 The interference check standards (ICS) are analyzed before the first CCV. They consist of two different standards, the ICSA and ICSAB.
- 16.8.2 The first interference check standard (ICSA) contains known concentrations of the interference analytes of interest with no addition of target elements. Any interferents with known concentrations that are calibrated by the instrument should recover within ±20% of the expected value. All target elements should be less than 3 times the s\*K portion of the MDL (See Appendices I & II as applicable for the determined DQO values for each analysis type). The ICSA is not used for pass/fail determination, but to assess which elements may need correction in the IFB. If an element is greater than 3 x s\*K, that result is subtracted from the IFB before evaluating the precent recovery.
- 16.8.3 The second interference check standard (ICSAB) contains the same concentration of interference analytes in the ICSA as well as a known concentration of reported analytes. The concentration of known analytes should be near the middle of the calibration curve. The values obtained for



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 38 of 67

reported analytes should be within 79.9% to 120.1% of the known concentration of target analytes after ICSA subtraction (if necessary). If the ICSAB fails for any of the target analytes, qualify samples with the "ICS-01" flag and either "LL" or "LK" flag, as appropriate.

#### 16.9 Dilutions

#### 16.9.1 Serial Dilution

The SRD analysis must be performed on one sample per analysis of 20 samples, preferably on the parent sample chosen for the DUP/MS/MSD. This serves as guidance for poor recoveries in the spiked samples that may be due to matrix interference. The same sample matrix used to extract the parent sample must be used as the diluent. After the dilution is applied to the SRD results, the analyte concentration should be within  $\pm 10\%$  RPD of the undiluted sample results if the parent sample analyte concentration is  $\geq$  25x MDL (5x dilution multiplied by  $\geq$  5x MDL). If the SRD, MS/MSD, and PS all fail for the same analyte, a matrix interference must be suspected, and the parent sample and QC data must be flagged for all affected elements. If matrix interference is not suspected, the SRD should be qualified with the "SRD-01" flag.

**Note:** Because the SRD is analytical QC, reanalysis on a different sequence due to prep issues is not helpful, as it does nothing to characterize the original analysis. Care must be taken to prepare the SRD properly; poorly prepared SRDs can only be "corrected" if reprepared and added to the same sequence at the time of discovery.

#### 16.9.2 Sample Dilution

Any samples needing dilution due to high concentration in the sample shall be diluted in an appropriate manner to bring the diluted concentration within the calibration curve and preferably near the mid-point. See Section 16.7 for dilutions required due to high ISTD recoveries. If the parent sample of a DUP/MS/MSD/PS sample needs to be diluted, the dilution should be performed on each of these QC samples (See also Section 16.3.3). If the SRD was performed on the parent sample of a DUP or PS the results from the SRD may be used to report values of that parent sample if the 5 times dilution was appropriate. The pipette serial number used to perform the dilution must be recorded.

**Note:** Dilution also increases the associated MDL by the dilution factor, so care must be taken not to dilute a sample so that the corrected concentration value is less than increased MDL.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 39 of 67

### 16.10 Negative Values

The absolute value of any negative result for blanks and field samples must not exceed the MDL. Reanalysis may remedy such results, or instrument maintenance such as cleaning the sample introduction system may be required.

- a. If the absolute value of any negative sample results exceeds s\*K, the "BR" qualifier must be applied in LIMS.
- b. The absolute value of all instrument blank (CCB) results exceeding s\*K must be flagged with the "QB-04" qualifier in the LIMS. Associated sample data must also be qualified with "LL" or "LK", as appropriate, and the "QB-04" flag.

### 16.11 Initial Demonstration of Capability

Each analyst must demonstrate initial proficiency for analysis by generating analytical data of acceptable accuracy and precision for four LCSs. For demonstration of proficiency, acceptable accuracy and precision is defined as having both RSD ≤20% and percent recovery of 75-125%. This demonstration is repeated whenever new staff receive training or significant changes in extraction procedure or instrumentation are made. These are maintained in the staff training files.

### 16.12 Ongoing Demonstration

To evaluate the continued performance of an analyst annually, it is acceptable to use CCVs, blank spikes, external audit samples, MDLs, or internal blind samples (blind samples at a concentration between LOQ and CCV level). Acceptable percent recovery is within the range of 75-125%. These are evaluated at the beginning of each year.

#### 16.13 Decontaminating/Cleaning Labware

Procedures for proper cleaning and removal of trace metals from labware are found in Section 14.3.1 in SOP ERG-MOR-031. Detergent bath preparation instructions can be found in Section 11.12. Transport/dip baths of DI water ("To" and "From" acid baths) should be labeled with the date they are filled with fresh DI and changed about once every month to minimize contaminants being brought to the acid bath and from being reintroduced to clean labware. This will not only extend the life of the acid bath, but it will help reduce hazardous waste production. The acid bath can be monitored by preparing a 5x dilution that can be analyzed as necessary or whenever contamination issues are suspected.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 40 of 67

#### 17.0 PREVENTION

When possible, minimize the amount of chemicals used in the preparation and analysis of the metals filters to reduce waste.

#### 18.0 DATA REVIEW AND CORRECTIVE ACTION

#### 18.1 Data Review Documentation

Project files, including at a minimum the information required in Section 22, are assembled and maintained by the performing analyst. A review of the data is performed by the Task Lead or designated secondary reviewer using the QC review checklist shown in Figures 24-1 or 24-2 (as applicable) to confirm that quality requirements have been met. Corrections and flags are added to the data consistent with the corrective action required for each review finding. Second level reviewers must complete, initial and date the checklist.

The completed check list is included as part of the data package. Data not meeting SOP requirements are flagged and brought to the attention of the Project Manager for resolution.

### 18.2 Quality Staff Review

A minimum of 10% of the data is reviewed by ERG Quality Staff. Quality staff review checks that all SOP required quality parameters have been met and that data reviewers have completed their review checklists. Additional items may also be reviewed at the discretion of the data reviewer. Quality staff reviews are documented on the review form initiated in Section 18.1 by the Task Lead or data reviewer. Comments or issues with data identified by the Quality Staff reviewer are brought to the attention of the Project Manager for resolution.

Corrective action for metals by ICP/Mass Spectrometry analysis data quality issues are presented in Table 24-3.

If required, a corrective action form is filled out as described in the ERG Laboratory Quality Systems Manual.

#### 19.0 WASTE MANAGEMENT

Hazardous waste disposal is discussed in detail in SOP ERG-MOR-033. Sample digestates are retained for a period of at least 6 months from the sample date. After this time, sample digestates are disposed of by pouring them off in the satellite waste containers located in the laboratory. Used sample vials are disposed of in the laboratory waste bin.



### ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 41 of 67

#### 20.0 MAINTENANCE

The ICP-MS system is maintained under a service contract. The preventative and routine maintenance are performed by the analyst(s) or service engineer. In the event a maintenance issue cannot be addressed by the analyst or Task Lead, a service call is placed with the manufacturer and when appropriate a service engineer will perform any necessary maintenance. All maintenance activities are documented in the instrument maintenance logs.

- 20.1 The following maintenance procedures need to be addressed daily.
  - 20.1.1 Check sample waste container level.
  - 20.1.2 Inspect liquid argon cylinder supply and its pressure to the instrument.
  - 20.1.3 Inspect chiller coolant level and connections for possible leaks.
  - 20.1.4 Inspect torch and aerosol injector tubes.
  - 20.1.5 Inspect nebulizer for clogs.
  - 20.1.6 Inspect sample capillary tubing to be sure it is clean and in good condition.
  - 20.1.7 Check peristaltic pump tubing before operation.
  - 20.1.8 At the end of each analysis, flush system for 5 minutes with the plasma on with rinse blank.
  - 20.1.9 Inspect vacuum pump oil level and replace as needed.
  - 20.1.10 Inspect sample and skimmer cones for excessive salt build-up.
- 20.2 The following maintenance procedures should be addressed as needed (common things to check if instrument performance indicates maintenance is needed).
  - 20.2.1 Clean torch components.
  - 20.2.2 Inspect and clean the RF coil.
  - 20.2.3 Inspect nebulizer spray pattern.
  - 20.2.4 Check nebulizer components.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 42 of 67

- 20.2.5 Check spray chamber drain fitting for leaks.
- 20.2.6 Check that pump rollers are clean.
- 20.2.7 Clean skimmer and sampling cones (See Section 20.4) and inspect orifices for damage or corrosion. Replace cones as needed.
- 20.2.8 Inspect autosampler rinse pump rollers and clean or replace as necessary.

### 20.3 <u>Cleaning Glass Sample Introduction Components</u>

- 20.3.1 Place torch and injector into a 2% Citranox detergent and sonicate for 15 minutes. If they have stubborn deposits, they can then be placed into an acid bath containing 5% HNO<sub>3</sub> and 2% HCl and sonicated for another 15 minutes. The spray chamber should be initially cleaned in a 2% Contrad NF detergent solution and sonicated for 15 minutes, as this restores the wetting surface of the glass best.
- 20.3.2 Rinse the components thoroughly first with hot tap water to remove detergents and then triple rinse with ultrapure water. Alternatively, the components can be sonicated in ultrapure water for another 15 minutes after the hot tap water rinse.
- 20.3.3 Allow components to completely dry in the Class 100 clean hood. If not immediately used, store in metal-free sealed plastic bags.

#### 20.4 Cleaning and Conditioning of Sample and Skimmer Cones

- 20.4.1 Remove cones from interface. Remove cone insert from skimmer cone.
- 20.4.2 Place cones and skimmer insert into 2% Citranox and sonicate until salt buildup is gone (15 minutes is usually enough). Rinse thoroughly with warm or hot tap water.
- 20.4.3 Place cones and skimmer insert into ultrapure water in an ultrasonic bath for 5 minutes.
- 20.4.4 Rinse thoroughly with ultrapure water.
- 20.4.5 Place in Class 100 hood or leave to air dry on a dust free cloth (e.g., Technicloth®) or Kimwipes® may be used. For cones that are still dirty after the procedure described in 20.4 and believed to be usable, refer to the Spectron Cone Cleaning Guide for additional procedures. Cones that are no



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 43 of 67

longer usable may be returned to the manufacturer for recycling/credit and platinum cones can be sent in for refurbishing.

20.4.6 Condition the newly cleaned cones by aspirating the cone conditioning solution in Table 24-11 for approximately 45 minutes, followed by 15 minutes of the Quartz IFA standard in Appendix I. Rinse the sample introduction system by aspirating rinse blank until background levels are acceptable.

#### 21.0 SHORTHAND PROCEDURE

The flow chart shown in Figure 24-4 shows the procedural steps and sequence for analysis of inorganic samples.

#### 22.0 DOCUMENTATION AND DOCUMENT CONTROL

- 22.1 All information concerning standard preparation, instrument conditions, etc., must be documented in the appropriate binders (i.e., Daily Performance Reports, Standards Log etc.) and/or electronically in either the LIMS or the local instrument computer.
- 22.2 All calculations and the type of method for determining concentration must be recorded in the analyst's notebook. Any unusual problems or conditions must also be noted.
- 22.3 Record all maintenance performed on each instrument in their respective maintenance logbook.
- 22.4 Reviewer must sign instrument maintenance notebook on a regular basis.
- 22.5 Any hard copies of instrument data should be filed chronologically. Electronic copies of instrument data are maintained on the Y: drive in the "ICP-MS Data" folder.
- 22.6 It is imperative the project documentation be updated following each analysis. Analysts will copy all raw instrument (i.e., Qtegra Lab Books and exported Excel xml files for transfer into the LIMS) and QC files (performance reports and lab book PDFs, etc.) to a designated corporate network shared drive at the completion of each analysis sequence or batch for backup. Primary data reviewers will use the data on the shared network drive for their data review process. The completed data packages ready for upload into ERG's LIMS will be retained on the network drive as the backup for this data.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 44 of 67

22.7 All processed data are archived in the LIMS and on the shared network drive and stored for at least five years in the laboratory. An archive copy of a data package is retained for at least five years in the laboratory data storage. The data backup should include enough information to manually generate the numbers used for reporting.

#### 22.8 Reporting

- 22.8.1 Sample results are uploaded into the LIMS in ng/L as analyzed. Any dilutions performed must be accounted for in the instrument software. The internal standard recoveries must be included with the result calculation. Final results are typically reported in ng/m³ to three significant figures although some clients receive results in  $\mu g/filter$ . If required by the sampling organization, results can also be reported in  $\mu g/m³$  by multiplying the ng/m³ results by 1,000.
- 22.8.2 Sample results should not be corrected based on analyte results from the laboratory blanks, field, trip, or filter lot blanks provided by sampling agencies. Due to uncontrollable and variable inherent background of trace metals present in blank quartz filter strips used to prepare method blanks (BLK2) and blank spikes (BS1), sometimes failing recoveries in BS1 are unavoidable for target metals, such as chromium and lead. Sometimes nontarget metals such as aluminum may also cause BS1 percent recovery failures. When this occurs, these failing elements are flagged with "BS-02" to indicate the noted background present in blank strips used for the preparation of these batch QC samples. Original data may not be manipulated; however, it should be noted on the review form which metals are greater than the MDL.
- 22.8.3 If a dilution was necessary, the diluted value will be reported to the sampling agency with a qualifier, "D".
- 22.8.4 Data should meet all specifications as presented in Table 24-3. If data does not meet specifications, corrective reporting actions listed must be followed (flag or invalidate data).

#### 23.0 REFERENCES

Code of Federal Regulations – 40 CFR Part 50, Appendix G – Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air – January 1, 2011.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 45 of 67

Code of Federal Regulations – 40 CFR Part 136, Appendix B – Definition and Procedure for the Determination of the Method Detection Limit – Revision 1.11.

Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP/MS). Compendium Method IO-3.5, *In:* Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air. Center for Environmental Research Information, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH, 45268, June 1999.

EPA Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry. Revision 5.4, EMMC Version, 1994.

ERG's Corporate Health and Safety Manual.

ERG's Laboratory Quality Systems Manual.

ERG-MOR-031. Standard Operating Procedure for Cleaning Glassware and Syringes for Organic Analysis. Revision Number 6, Effective: October 20, 2017.

ERG-MOR-033. Standard Operating Procedure for Hazardous Waste. Revision Number 5, Effective: February 8, 2016.

ERG-MOR-045. Standard Operating Procedure for Sample Receipt at the ERG Chemical Laboratory. Revision Number 9, Effective: March 6, 2017.

ERG-MOR-084. Standard Operating Procedure for the Preparation and Extraction of High Volume Quartz and Glass Fiber Filters for Metals by ICP-MS using Methods IO 3.1, 3.5 and FEM Method EQL-0512-201. Revision Number 14, Effective: January 16, 2017.

ERG-MOR-085. Standard Operating Procedure for the Extraction of 47 mm Filters for Metals by ICP-MS using Methods IO 3.1, 3.5 and FEM Method EQL-0512-202. Revision Number 13, Effective: January 16, 2017.

Federal Advisory Committee on Detection and Quantitation Approaches and Uses In Clean Water Act Programs. Appendix D: DQ FAC Single Laboratory Procedure v2.4, 8/30/2007.

Selection, Preparation and Extraction of Filter Material. Compendium Method IO-3.1, *In:* Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air. Center for Environmental Research Information, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH, 45268, June 1999.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 46 of 67

Spectron Cone Cleaning Guide. By Lawrence Neufeld, Spectron, Inc. Website: https://spectronus.com/assets/product\_documents/Cone\_Cleaning\_Final\_909.pdf

Standard Operating Procedure (SOP) For The Trace Elemental Analysis of Ambient Air Particulate Samples Using Inductively Coupled Plasma - Mass Spectrometry (ICP-MS). California Environmental Protection Agency, Air Resources Board, (SOP MLD 061), Rev. No. 1, January 1, 2007.

Technical Assistance Document for the National Air Toxics Trends Stations Program, Revision 4, July 2022.

Thermo Scientific iCAP Q / iCAP RQ ICP-MS Software Manual, Qtegra ISDS Software Version 2.8 SR1, Revision B, June 2017.

Thermo Scientific iCAP Q Operating Manual, Revision C - 1288090.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 47 of 67

### 24.0 TABLES, DIAGRAMS, FLOWCHARTS, VALIDATION DATA

### Figure 24-1. Quality Control Review Form for Quartz Fiber Filters

Sequence ID: Cal Curve (Method):		Instrument:			Batch: Date:	
10% Review Sample ID	s:	Analyst: Reviewer:			Date:	
Parameter	Acceptance Criteria	Analyst Check (Initials and Date)	Task Lead/Data (Initials and Date)	10% QA Review (Initials and Date)	Optl Addl QA Review (Initials and Date)	Comments
		Instrument QC		·		
Daily Performance Report STD Mode	See instrument generated PDF or Table 24-7 in SOP 095 for performance report requirements					
Daily Performance Report KED Mode	See instrument generated PDF or Table 24-7 in SOP 095 for performance report requirements					
Initial Calibration Standards (IC)	≥0.995 correlation coefficient & RSD ≤10%. RSE for CAL2 within 30% of nominal; CAL5 RSE within 10%. non-Tier 1 QX and LJ					
Initial Calibration Verification (ICV)	Recovery 89.9-110.1%. non-Tier 1 QX and LJ					
Initial Calibration Blank (ICB)	Absolute value must be < s*K					
High Calibration Verification (HCV)	Recovery 94.9-105.1% non-Tier 1 QX and LJ					
Interference Check Standard (ISCA/IFA)	None – any values which exceed 3 times s*K will be used to correct the IFB result					
Interference Check Standard (ICSAB/IFB)	Recovery 79.9-120.1% of nominal after IFA correction (if IFA > 3x s*K) ICS-01 and LK or LL					
Continuing Calibration Verification (CCV)	Recovery 89.9-110.1% non-Tier 1 QX and LJ					

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# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 48 of 67

#### SOP ERG-MOR-095 Quality Control Review Form Metals Analysis -- 8x10" Quartz Filters 2023-2

Parameter	Parameter Acceptance Criteria  Continuing Calibration Absolute value must be < s*K QB-04		Parameter Acceptance Criteria (Initials Date	Analyst Check (Initials and Date)	Task Lead/Data (Initials and Date)	10% QA Review (Initials and Date)	Optl Addl QA Review (Initials and Date)	Comments
Continuing Calibration Blanks (CCB)								
Internal Standard Response	Recovery must be between 60 and 125% of the measured intensity of the calibration blank QX and LJ							
Calibration Range Check	Reporting elements for unknown/field samples within range of highest calibration standard (or LDR if one is in use). <b>D</b> or E							
		Extraction QC						
Review Batch Details/ Check Sample Volume	Check COC or filter envelope against Bench Sheet to make sure sample volumes are correct. Review Bench sheet/Extraction sheet							
Laboratory Reagent Blank (LRB/BLK1)	Absolute value must be < MDL. <b>B</b>							
Method Blank (MB/BLK2)	Absolute value must be < MDL. QB-01							
Laboratory Control Sample (LCS/BS)	Recovery 79.9-120.1% GC-BS							
Reagent Blank Spike (RBS)	Recovery 79.9-120.1% LJ							
Collocated Samples (C1/C2)	<20.1% RPD when concentration of either sample is ≥ 5x the MDL (see Section 16.4.4 and 16.4.3 for details). D-F							
Duplicate (DUP1) (Laboratory Duplicate)	$<$ 20.1% RPD for samples values $\ge$ 5x MDL (see Section 16.4.3 for details). <b>D-F</b>							

This review check sheet must be completed by primary data reviewer/TL/QA. Potential qualifiers in **Bold**. Specific details on use of qualifiers in SOP.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 49 of 67

#### SOP ERG-MOR-095 Quality Control Review Form Metals Analysis -- 8x10" Quartz Filters 2023-2

Parameter	Acceptance Criteria	Analyst Check (Initials and Date)	Task Lead/Data (Initials and Date)	10% QA Review (Initials and Date)	Optl Addl QA Review (Initials and Date)	Comments
Replicate Analyses (Analytical Duplicate)	<10.1% RPD when concentration of the parent sample is ≥ 5x the MDL (see Section 16.4.5 for details). R-F rep, col, and sequence					
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	79.9-120.1% when the parent sample concentration is less than 4 times the spike concentration; Sb recovery should be 74.9-125.1% QM-#					
Serial Dilution (SRD)	Recovery 89.9 to 110.1% of undiluted sample if the parent sample concentration is > than 25x the MDL. SRD-01					
Post Digestion Spike (PDS)	Recovery 74.9%-125.1% <b>PS-01</b>					
	—total	porting Requirem	ents			
Field Blank	<mdl (in="" <b="" m³)="" ng="">FB-01</mdl>					
Manual Check of Calculations	Hand calculate an equation - a unit conversion equation, a sample concentration equation, a dilution factor calculation, etc. – to verify calculation					
Check LIMS Qualifiers	Check the LIMS data flags (see above flagging and hold time, O-04 or O-05)					
Negative Sample Values	Negative sample values must be less than the absolute value of the MDL.					
MDL	Check to ensure the MDLs in LIMS are current					

This review check sheet must be completed by primary data reviewer/TL/QA. Potential qualifiers in **Bold**. Specific details on use of qualifiers in SOP.



# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 50 of 67

### Figure 24-2. Quality Control Review Form for Teflon Filters

#### SOP ERG-MOR-095 Quality Control Review Form Metals Analysis -- 47mm Filters 2023-2 Sequence ID: Instrument: Batch: Cal Curve (Method): Date: Analyst: 10% Review Sample IDs: Reviewer: Date: Analyst Check Optl Addl QA Lead/Data Review Review (Initials and Date) Parameter Acceptance Criteria (Initials and Comments (Initials and (Initials and Date) Date) Date) Instrument OC Daily Performance Report STD Mode See instrument generated PDF or Table 24-7 in SOP 095 for performance report requirements See instrument generated PDF or Table 24-7 in SOP 095 for performance report Daily Performance Report KED Mode requirements ≥0.995 correlation coefficient & RSD ≤10%. RSE for CAL2 within 30% of nominal; CAL5 Initial Calibration Standards (IC) RSE within 10%. non-Tier 1 QX and LJ Initial Calibration Verification Recovery 89.9-110.1%. non-Tier 1 QX and Absolute value must be < s\*K Initial Calibration Blank (ICB) High Calibration Verification (HCV) Recovery 94.9-105.1%. non-Tier 1 QX and LJ Interference Check Standard None - any values which exceed 3 times (ISCA/IFA) s\*K will be used to correct the IFB result Interference Check Standard (ICSAB/IFB) Recovery 79.9-120.1% of nominal after IFA correction (if IFA > 3x s\*K) ICS-01 and LK or LL Recovery 89.9-110.1% non-Tier 1 QX Continuing Calibration Verification (CCV) and LJ

This review check sheet must be completed by primary data reviewer/TL/QA. Potential qualifiers in Bold. Specific details on use of qualifiers in SOP.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 51 of 67

#### SOP ERG-MOR-095 Quality Control Review Form Metals Analysis -- 47mm Filters 2023-2

Parameter	Acceptance Criteria	Analyst Check (Initials and Date)	Task Lead/Data (Initials and Date)	10% QA Review (Initials and Date)	Optl Addl QA Review (Initials and Date)	Comments
Continuing Calibration Blanks (CCB)	Absolute value must be < s*K QB-04					
Internal Standard Response	nal Standard Response Recovery must be between 60 and 125% of the measured intensity of the calibration blank OX and LJ					
Calibration Range Check  Reporting elements for unknown/field samples within range of highest calibration standard (or LDR if one is in use). <b>D or E</b>						
		Extraction QC				
Review Batch Details/ Check Sample Volume	Check COC or filter envelope against Bench Sheet to make sure sample volumes are correct. Review Bench sheet/Extraction sheet					
Laboratory Reagent Blank (LRB/BLK1)	Absolute value must be < MDL. B					
Method Blank (MB/BLK2)	Absolute value must be < MDL. QB-01					
Laboratory Control Sample (LCS/BS/BSD)	Recovery 79.9-120.1% and < 20.1% RPD. GC-BS					
Reagent Blank Spike (RBS)	Recovery 79.9-120.1% LJ					
Collocated Samples (C1/C2)	<20.1% RPD when concentration of either sample is ≥ 5x the MDL (see Sections 16.4.4 and 16.4.3 for details). D-F					
Replicate Analyses (Analytical Duplicate)	<10.1% RPD when concentration of the parent sample is ≥ 5x the MDL(see Section 16.4.5 for details). R-F rep, col, and sequence					

This review check sheet must be completed by primary data reviewer/TL/QA. Potential qualifiers in Bold. Specific details on use of qualifiers in SOP.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 52 of 67

#### SOP ERG-MOR-095 Quality Control Review Form Metals Analysis -- 47mm Filters 2023-2

Parameter	Acceptance Criteria	Analyst Check (Initials and Date)	Task Lead/Data (Initials and Date)	10% QA Review (Initials and Date)	Optl Addl QA Review (Initials and Date)	Comments
Serial Dilution (SRD)	±10% RPD of undiluted sample if the parent sample concentration is > than 25x the MDL. SRD-01					
Post Digestion Spike (PDS)	Recovery 74.9%-125.1% <b>PS-01</b>					
	Re	porting Requirem	ents			
Field Blank	< MDL (in ng/m³) FB-01					
Manual Check of Calculations	Hand calculate an equation - a unit conversion equation, a sample concentration equation, a dilution factor calculation, etc. – to verify calculation					
Check LIMS Qualifiers	Check the LIMS data flags (see above flagging and hold time, O-04 or O-05)					
Negative Sample Values	Negative sample values must be less than the absolute value of the MDL.					
MDL	Check to ensure the MDLs in LIMS are current					

This review check sheet must be completed by primary data reviewer/TL/QA. Potential qualifiers in Bold. Specific details on use of qualifiers in SOP.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

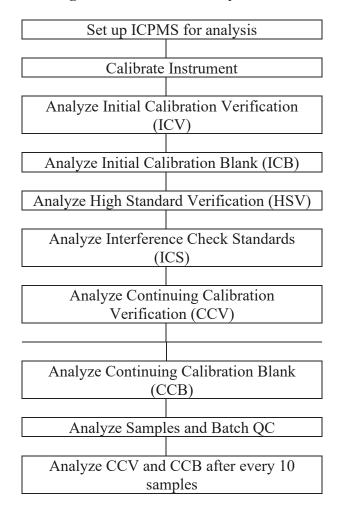
Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 53 of 67

Figure 24-3. Flow Diagram for ICP-MS Analysis for PM10 or TSP Filters





# ERG PROPRIETARY INFORMATION Standard Operating Procedure

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 54 of 67

Table 24-1. 2023 Method Detection Limits (MDLs) for Metals Hi-Vol Quartz Filters

	MDL	MDL
	By FAC	by FAC
Element	(ng/L)	$(ng/m^3)$ §
Aluminum *	116,000	32.1
Antimony	159	0.0441
Arsenic	34.5	0.00955
Barium *	3,430	0.948
Beryllium	12.00	0.0033
Cadmium	393.0	0.109
Calcium *	1,060,000	292
Chromium†	7,350	2.03
Cobalt	56.3	0.0156
Copper *	10,900	3
Iron *	87,600	24.2
Lead	999	0.276
Magnesium *	348,000	96.4
Manganese	4,290	1.19
Molybdenum *	769	0.213
Nickel	2,900	0.801
Phosphorus*	4,540,000	1250
Potassium*	137,000	38
Rubidium *	66.3	0.0183
Selenium	39.9	0.011
Sodium*	7,220,000	2000
Strontium *	2,360	0.652
Thallium *	1.82	0.000503
Thorium *	10.9	0.003
Uranium *	61.4	0.017
Vanadium*	178	0.0492
Zinc *	353,000	97.7

<sup>\*</sup> Non-target elements for EPAs UATMP/NATTS program.

<sup>†</sup> Total Chromium.

<sup>§</sup> Units of ng/m³ assumes 1627 m³ sampling volume.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 55 of 67

Table 24-2. 2023 Method Detection Limits (MDLs) for Metals 47 mm Teflon® Filters

	MDL	MDL
	by FAC	by FAC
Element	(ng/L)	(ng/m <sup>3</sup> )§
Aluminum *	9330	19.4
Antimony	39.5	0.0822
Arsenic	11.5	0.024
Barium *	215	0.446
Beryllium	3.4	0.00707
Cadmium	3.65	0.00759
Calcium *	103000	214
Chromium†	3090	6.43
Cobalt	9.3	0.0193
Copper *	309	0.644
Iron *	7180	14.9
Lead	31	0.0645
Magnesium *	10100	21
Manganese	244	0.508
Molybdenum *	23.3	0.0485
Nickel	141	0.292
Phosphorus*	3840	7.98
Potassium*	25800	53.7
Rubidium *	24.3	0.0506
Selenium	20.4	0.0424
Sodium*	18500	38.4
Strontium *	79.5	0.165
Thallium *	0.44	0.000915
Thorium *	1.09	0.00227
Uranium *	0.707	0.00147
Vanadium*	35.3	0.0734
Zinc *	27400	57.1

<sup>\*</sup> Non-target elements for EPAs UATMP/NATTS program.

<sup>†</sup> Total Chromium.

<sup>§</sup> Units of ng/m³ assumes 24.04 m³ sampling volume.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 56 of 67

## Table 24-3. Summary of Quality Control Procedures for Metals Analysis

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Daily Performance Check (DPR) STD Mode	Before each analysis	See Table 24-5	Repeat analysis of DPR     Re-optimize instrument tuning parameters     Reprepare DPR standard     Perform instrument maintenance
Daily Performance Check (DPR) KED Mode	Before each analysis	See Table 24-5	Repeat analysis of DPR     Re-optimize instrument tuning parameters     Reprepare DPR standard     Perform instrument maintenance
Initial Calibration Standards (IC)	At least 5 non-zero calibration points and a blank before each analysis	Coefficient of determination ( $R^2$ ) $\geq 0.995$ & RSD < 10.1%.	Repeat analysis of calibration standards     Reprepare calibration standards and reanalyze     Reprepare 1 failing elements with QX and LJ and void Tier 1 failing elements in ICAL and sample data
Initial Calibration Verification (ICV)	Immediately after calibration	Recovery 89.9-110.1%	Repeat analysis of ICV     Reprepare ICV standard     Recalibrate and reanalyze     Flag non-Tier 1 failing elements with QX and LJ and void Tier 1 failing elements in ICV, ICAL and sample data
Initial Calibration Blank (ICB)	Immediately after ICV	Absolute value must be < s*K portion of the MDL	Reanalyze ICB     Locate and resolve contamination problems before continuing     Recalibrate when appropriate
High standard verification (HSV or HCV)	After ICB and before ICS	Recovery from 94.9-105.1%	Repeat analysis of HSV     Reprepare HSV     Non-Tier I elements may be flagged instead of requiring reanalysis
Interference Check Standard (ICSA/IFA)	Following the HSV	Within determined DQO criteria (See Section 16.8.2)	None
Interference Check Standard (ICSAB/IFB)	Following the ICSA	Recovery 79.9-120.1% of true value when accounting for IFA correction (if IFA > 3x s*K)	Repeat analysis of ICSAB     Reprepare ICSAB and analyze     Flag failing elements with ICS-01 and LK or LL
Continuing Calibration Verification (CCV)	Analyze before samples, after every 10 samples, and at the end of each run	Recovery 89.9-110.1%	1) Reanalyze CCV 2) Reprepare CCV 3) Recalibrate and reanalyze samples since last acceptable CCV 4) Flag non-Tier 1 failing elements with QX and LJ and void Tier 1 failing elements in CCV and sample data
Continuing Calibration Blanks (CCB)	Analyzed after each CCV	Absolute value must be < s*K portion of the MDL	1) Reanalyze CCB 2) Reanalyze samples since last acceptable CCB 3) Flag any samples associated with failing CCBs with QB-04



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 57 of 67

## Table 24-3 (Cont'd) Summary of Quality Control Procedures for Metals Analysis

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Laboratory Reagent Blank (LRB/BLK1)	1 per batch of ≤ 20 samples	Absolute value must be < MDL	Reanalyze for verification     Reanalyze for verification     Reanalyze for verification     Reanalyze for all batch QC and samples must be flagged B     When enough sample filter remains, a reextraction and analysis of the batch should be considered
Method Blank (MB/BLK2)	1 per batch of $\leq$ 20 samples	Absolute value < MDL	Reanalyze for verification     Flag the failing elements in the samples in the batch QB-01
Laboratory Control Sample (RBS/BS/BSD)	1 BS per batch of ≤ 20 Quartz filters, a minimum of 1 per batch 1 BS/BSD per batch of ≤ 20 samples	Recovery 79.9-120.1%.  Sb recovery 74.9 to 125.1%.  BS/BSD RPD < 20.1%.	1) Reanalyze 2) Flag data GC-BS 3) Re-prepare sample batch if recovery for most elements fail criteria (quartz). 4) Sb failures must be flagged on BS and all samples, "SL"
Duplicate (DUP1) (Laboratory Duplicate)	1 per batch of ≤ 20 samples	< 20.1% RPD for quartz sample, <10.1% RPD for Teflon samples, and duplicate values ≥ 5x MDL (see Section 16.4.3 for details)	1) Check for matrix interference 2) Repeat duplicate analysis if necessary 3) Flag data D-F (see Section 16.4.3 for procedure)
Replicate Analysis (Analytical Duplicate)	On a minimum one sample per batch, ensuring 6 per site per year	< 10.1% RPD for sample and duplicate values ≥ 5x MDL (see Section 16.4.5 for details)	1) Repeat replicate analysis if necessary 2) Flag rep, parent, and sequence data R-F (see Section 16.4.5 for procedure)
Collocated Samples (C1/C2)	10% of samples annually (for sites that conduct collocated sampling)	< 20.1% RPD for sample and collocate values ≥ 5x MDL (see Section 16.4.4 and 16.4.3 for details)	1) Repeat C1 and/or C2 analyses if necessary. 2) Flag C1 and C2 data if necessary D-F (see Section 16.4.3 for procedure)
Matrix Spike (MS) and Matrix Spike Duplicate (MSD) for 8x10" Quartz filters only	1 per batch of ≤ 20 samples	Quartz Recovery 79.9- 120.1%when the parent sample concentration is less than 4 times the spike concentration. Sb recovery 74.9 to 125.1%.	1) Flag MS and parent sample if recovery for only one or two elements fail criteria, or when a matrix interference is confirmed by SRD and/or PS results with appropriate MS-## 2) Reanalyze
		Not applicable to Teflon method	3) Reprepare sample batch if recovery for most elements fail criteria or contamination is evident.  4) Sb failures must be flagged on MS/MSD and all samples, "SL"
MS/MSD RPD for 8 x 10" Quartz filters only	1 per batch of ≤ 20 samples	RPD < 20.1% for elements ≥ 5 x MDL in at least one of the duplicates Not applicable to Teflon method	Check for 4x spike concentration and non-homogenous matrix, flag as necessary with appropriate MS-##     Reanalyze for verification
Post Digestion Spike (PS)	1 per batch of ≤ 20 samples	Recovery 74.9%-125.1%	Flag failed elements for parent sample and PS PS-01     Reprepare PS if preparation issue is suspected reason for failure.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 58 of 67

### Table 24-3 (Cont'd) Summary of Quality Control Procedures for Metals Analysis

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Serial Dilution (SRD)	1 per batch of ≤ 20 samples	Recovery 89.9 to 110.1% of undiluted sample if the element concentration is ≥ 25x MDL	Re-prepare dilution if preparation issue is suspected reason for failure.     Flag failed analytes in SRD and Parent SRD-01
Field Blank	As received	< MDL in ng/m <sup>3</sup>	1) Flag failed elements in FB FB-01. Samples from last to next acceptable FB will be flagged in AQS.
Internal Standards (ISTD)	Every Calibration, QC and Field Sample	Recovery 60-125% of the measured intensity of the calibration blank	1) If drift suspected, stop analysis and determine cause, recalibrate if necessary 2) Reanalyze sample 3) If recovery > 125% due to inherent ISTD, dilute sample and reanalyze 4) Flag failed elements in sequence QX and LJ

### **Table 24-4. Optimization Procedures**

Procedure	When to Perform
Mass Calibration	Before each analysis
Detector Cross Calibration	Before each analysis
Nebulizer Gas Optimization	As necessary
Instrument Performance Check	Before each analysis
Detector Optimization:	As necessary
Pulse Stage Voltage	
Analog Stage Voltage	



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 59 of 67

Table 24-5. Instrument Mass Calibration & Performance Report Specifications iCAP-Q and RQ Criteria

Danamatan	Peak	Sensitiv	ity/Criteria*	DCD
Parameter	Width	iCAP-Q	iCAP-RQ	RSD
		Standard Mod	e	
Bkg4.5	NA	< 1.0 cps	< 1.0 cps	N/A
7Li	0.65-0.85	> 50,000 cps	> 55,000 cps	< 2% RSD
24Mg	0.65-0.85	> 500,000 cps	> 500,000 cps	< 2% RSD
25Mg	0.65-0.85	> 70,000 cps	> 80,000 cps	< 2% RSD
26Mg	0.65-0.85	> 80,000 cps	> 100,000 cps	< 2% RSD
59Co	0.65-0.85	> 100,000 cps	> 100,000 cps	< 2% RSD
115In	0.65-0.85	> 220,000 cps	> 240,000 cps	< 2% RSD
206Pb	0.65-0.85	> 70,000 cps	> 80,000 cps	< 2% RSD
207Pb	0.65-0.85	> 60,000 cps	> 70,000 cps	< 2% RSD
208Pb	0.65-0.85	> 100,000 cps	> 160,000 cps	< 2% RSD
238U	0.65-0.85	> 300,000 cps	> 330,000 cps	< 2% RSD
130Ce16O/130Ce	NA	< 0.02	< 0.02	N/A
137Ba++/137Ba+	NA	< 0.03	< 0.03	N/A
Bkg220.7	NA	< 2.0 cps	< 2.0 cps	N/A
Analyzer Pressure	NA	< 10 <sup>-6</sup> mbar	< 10 <sup>-6</sup> mbar	NA
		KED Mode		
Bkg4.5	NA	< 0.5 cps	< 0.5 cps	N/A
24Mg	0.65-0.85	> 3,000 cps	> 10,000 cps	< 5% RSD
25Mg	0.65-0.85	> 500 cps	> 2,000 cps	< 5% RSD
26Mg	0.65-0.85	> 600 cps	> 3,000 cps	< 5% RSD
59Co	0.65 - 0.85	> 30,000 cps	> 30,000 cps	< 2% RSD
115In	0.65-0.85	> 30,000 cps	> 35,000 cps	< 2% RSD
206Pb	0.65-0.85	> 60,000 cps	> 100,000 cps	< 2% RSD
207Pb	0.65-0.85	> 50,000 cps	> 90,000 cps	< 2% RSD
208Pb	0.65-0.85	> 80,000 cps	> 200,000 cps	< 2% RSD
238U	0.65-0.85	> 80,000 cps	> 85,000 cps	< 2% RSD
130Ce16O/130Ce	NA	< 0.01	< 0.01	N/A
59Co/35Cl16O	NA	> 18.0	> 18.0	N/A
Bkg220.7	NA	< 2.0 cps	< 2.0 cps	N/A

<sup>\*</sup>cps – Counts per second

<sup>† –</sup> There are no vacuum requirements for KED mode.



# **ERG PROPRIETARY INFORMATION Standard Operating Procedure**

Procedure Number: ERG-MOR-095

Revision Number: 6

Revision Date: August 31, 2023

Page: 60 of 67

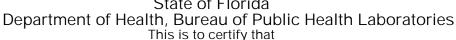
Table 24-6. Analytical Isotopes for Quantitation and Monitoring of Reported Elements

Element	Quantitation	Monitored/Confirmation
	Isotope(s)	Isotope(s)
Aluminum	27	NA
Antimony	121	123
Arsenic	75	NA
Beryllium	9	NA
Barium	137	135
Bismuth (ISTD)	209	NA
Cadmium	111	106, 108, 114
Calcium	43	NA
Chromium	52	53
Cobalt	59	NA
Copper	63	65
Iron	57	54
Germanium (ISTD)	73	NA
Holmium (ISTD)	165	NA
Indium (ISTD)	115	NA
Lead	206, 207, 208	NA
Lithium (ISTD)	6	NA
Magnesium	25	23
Manganese	55	NA
Molybdenum	98	92, 94, 95, 97
Nickel	60	62
Rubidium	85	NA
Scandium (ISTD)	45	NA
Selenium	78	77, 82
Strontium	88	NA
Terbium (ISTD)	159	NA
Thallium	205	203
Thorium	232	NA
Uranium	238	NA
Vanadium	51	NA
Yttrium (ISTD)	89	NA
Zinc	66	67, 68

NA = Not applicable/none.









E87673

EASTERN RESEARCH GROUP, INC. 601 KEYSTONE PARK DRIVE SUITE 700 MORRISVILLE, NC 27560

has complied with Florida Administrative Code 64E-1, for the examination of environmental samples in the following categories

AIR AND EMISSIONS - EXTRACTABLE ORGANICS, AIR AND EMISSIONS - GENERAL CHEMISTRY, AIR AND EMISSIONS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Public Health Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2023 Expiration Date: June 30, 2024

GREAT STATE STATE OF THE STATE

Susanne Crowe, MHA
Interim Chief Bureau of Public Health Laboratories
DH Form 1697, 7/04

NON-TRANSFERABLE E87673-25-07/01/2023 Supersedes all previously issued certificates





# Laboratory Scope of Accreditation

Page 1

of 3

Attachment to Certificate #: E87673-25, expiration date June 30, 2024. This listing of accredited analytes should be used only when associated with a valid certificate.

EPA Lab Code: State Laboratory ID: **E87673** NC00124 (919) 468-7924

E87673 Eastern Research Group, Inc. **601 Keystone Park Drive Suite 700** Morrisville, NC 27560

Matrix:	Air and Emissions				
Analyte#	Analyte	Method/Tech	Method Code	Category	Effective Date
5160	1,1,1-Trichloroethane	EPA TO-15	10248803	Volatile Organics	10/1/2001
5110	1,1,2,2-Tetrachloroethane	EPA TO-15	10248803	Volatile Organics	10/1/2001
5165	1,1,2-Trichloroethane	EPA TO-15	10248803	Volatile Organics	10/1/2001
4630	1,1-Dichloroethane	EPA TO-15	10248803	Volatile Organics	10/1/2001
4640	1,1-Dichloroethylene	EPA TO-15	10248803	Volatile Organics	10/1/2001
5155	1,2,4-Trichlorobenzene	EPA TO-15	10248803	Volatile Organics	10/1/2001
5210	1,2,4-Trimethylbenzene	EPA TO-15	10248803	Volatile Organics	11/13/2020
4585	1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA TO-15	10248803	Volatile Organics	11/13/2020
4610	1,2-Dichlorobenzene	EPA TO-15	10248803	Volatile Organics	10/1/2001
4635	1,2-Dichloroethane	EPA TO-15	10248803	Volatile Organics	10/1/2001
4655	1,2-Dichloropropane	EPA TO-15	10248803	Volatile Organics	11/13/2020
5215	1,3,5-Trimethylbenzene	EPA TO-15	10248803	Volatile Organics	11/13/2020
9318	1,3-Butadiene	EPA TO-15	10248803	Volatile Organics	11/13/2020
4615	1,3-Dichlorobenzene	EPA TO-15	10248803	Volatile Organics	10/1/2001
4620	1,4-Dichlorobenzene	EPA TO-15	10248803	Volatile Organics	10/1/2001
4410	2-Butanone (Methyl ethyl ketone, MEK)	EPA TO-11A	10311805	Extractable Organics	11/13/2020
4995	4-Methyl-2-pentanone (MIBK)	EPA TO-15	10248803	Volatile Organics	10/1/2001
5500	Acenaphthene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
5505	Acenaphthylene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
4300	Acetaldehyde	EPA TO-11A	10311805	Extractable Organics	10/1/2001
4315	Acetone	EPA TO-11A	10311805	Extractable Organics	10/1/2001
4320	Acetonitrile	EPA TO-15	10248803	Volatile Organics	10/1/2001
4325	Acrolein (Propenal)	EPA TO-15	10248803	Volatile Organics	11/13/2020
4340	Acrylonitrile	EPA TO-15	10248803	Volatile Organics	10/1/2001
5555	Anthracene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
1005	Antimony	EPA IO-3.5/ICP-MS	10246603	General Chemistry	1/25/2007
1010	Arsenic	EPA IO-3.5/ICP-MS	10246603	General Chemistry	1/25/2007
5570	Benzaldehyde	EPA TO-11A	10311805	Extractable Organics	10/1/2001
4375	Benzene	EPA TO-15	10248803	Volatile Organics	10/1/2001
5575	Benzo(a)anthracene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
5580	Benzo(a)pyrene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
5585	Benzo(b)fluoranthene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
5605	Benzo(e)pyrene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
5590	Benzo(g,h,i)perylene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
5600	Benzo(k)fluoranthene	EPA TO-13A	10248405	Extractable Organics	1/25/2007

Clients and Customers are urged to verify the laboratory's current certification status with Certification Type **NELAP** the Environmental Laboratory Certification Program. **Issue Date: 7/1/2023** Expiration Date: 6/30/2024





### Laboratory Scope of Accreditation

Page 2

Certification Type

Expiration Date: 6/30/2024

**NELAP** 

of 3

Attachment to Certificate #: E87673-25, expiration date June 30, 2024. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87673 EPA Lab Code: NC00124 (919) 468-7924

E87673 Eastern Research Group, Inc. 601 Keystone Park Drive Suite 700 Morrisville, NC 27560

Matrix:	Air and Emissions				
Analyte#	Analyte	Method/Tech	Method Code	Category	Effective Date
1020	Beryllium	EPA IO-3.5/ICP-MS	10246603	General Chemistry	1/25/2007
4400	Bromoform	EPA TO-15	10248803	Volatile Organics	11/13/2020
4430	Butyraldehyde	EPA TO-11A	10311805	Extractable Organics	11/13/2020
1030	Cadmium	EPA IO-3.5/ICP-MS	10246603	General Chemistry	1/25/2007
4450	Carbon disulfide	EPA TO-15	10248803	Volatile Organics	11/13/2020
4455	Carbon tetrachloride	EPA TO-15	10248803	Volatile Organics	10/1/2001
4475	Chlorobenzene	EPA TO-15	10248803	Volatile Organics	10/1/2001
4485	Chloroethane	EPA TO-15	10248803	Volatile Organics	10/1/2001
4505	Chloroform	EPA TO-15	10248803	Volatile Organics	10/1/2001
4525	Chloroprene	EPA TO-15	10248803	Volatile Organics	10/1/2001
1040	Chromium	EPA IO-3.5/ICP-MS	10246603	General Chemistry	1/25/2007
1045	Chromium VI	ERG-MOR-063, Rev. 4	60039045	General Chemistry	9/22/2009
5855	Chrysene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
4645	cis-1,2-Dichloroethylene	EPA TO-15	10248803	Volatile Organics	10/1/2001
4680	cis-1,3-Dichloropropene	EPA TO-15	10248803	Volatile Organics	10/1/2001
1050	Cobalt	EPA IO-3.5/ICP-MS	10246603	General Chemistry	1/25/2007
5856	Coronene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
4545	Crotonaldehyde	EPA TO-11A	10311805	Extractable Organics	10/1/2001
5895	Dibenz(a,h)anthracene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
4575	Dibromochloromethane	EPA TO-15	10248803	Volatile Organics	11/13/2020
4625	Dichlorodifluoromethane	EPA TO-15	10248803	Volatile Organics	11/13/2020
4760	Ethyl acrylate	EPA TO-15	10248803	Volatile Organics	11/13/2020
4765	Ethylbenzene	EPA TO-15	10248803	Volatile Organics	10/1/2001
4795	Ethylene oxide	EPA TO-15	10248803	Volatile Organics	11/13/2020
6265	Fluoranthene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
6270	Fluorene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
4815	Formaldehyde	EPA TO-11A	10311805	Extractable Organics	10/1/2001
4835	Hexachlorobutadiene	EPA TO-15	10248803	Volatile Organics	11/13/2020
3825	Hexanaldehyde	EPA TO-11A	10311805	Extractable Organics	11/13/2020
5315	Indeno(1,2,3-cd)pyrene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
1075	Lead	EPA IO-3.5/ICP-MS	10246603	General Chemistry	1/25/2007
1090	Manganese	EPA IO-3.5/ICP-MS	10246603	General Chemistry	1/25/2007
1095	Mercury	EPA IO-3.5/ICP-MS	10246603	General Chemistry	1/25/2007
4950	Methyl bromide (Bromomethane)	EPA TO-15	10248803	Volatile Organics	10/1/2001
4960	Methyl chloride (Chloromethane)	EPA TO-15	10248803	Volatile Organics	10/1/2001





# Laboratory Scope of Accreditation

Page 3

of 3

Attachment to Certificate #: E87673-25, expiration date June 30, 2024. This listing of accredited analytes should be used only when associated with a valid certificate.

EPA Lab Code: State Laboratory ID: **E87673** NC00124 (919) 468-7924

E87673 Eastern Research Group, Inc. **601 Keystone Park Drive Suite 700** Morrisville, NC 27560

Matrix:	Air and Emissions				
Analyte#	Analyte	Method/Tech	Method Code	Category	Effective Date
4990	Methyl methacrylate	EPA TO-15	10248803	Volatile Organics	10/1/2001
5000	Methyl tert-butyl ether (MTBE)	EPA TO-15	10248803	Volatile Organics	10/1/2001
4975	Methylene chloride	EPA TO-15	10248803	Volatile Organics	10/1/2001
5005	Naphthalene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
1105	Nickel	EPA IO-3.5/ICP-MS	10246603	General Chemistry	1/25/2007
5250	o-Xylene	EPA TO-15	10248803	Volatile Organics	11/13/2020
6608	Perylene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
6615	Phenanthrene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
3965	Propionaldehyde (Propanal)	EPA TO-11A	10311805	Extractable Organics	10/1/2001
4836	Propylene (Propene)	EPA TO-15	10248803	Volatile Organics	11/13/2020
6665	Pyrene	EPA TO-13A	10248405	Extractable Organics	1/25/2007
1140	Selenium	EPA IO-3.5/ICP-MS	10246603	General Chemistry	1/25/2007
5100	Styrene	EPA TO-15	10248803	Volatile Organics	10/1/2001
5115	Tetrachloroethylene (Perchloroethylene)	EPA TO-15	10248803	Volatile Organics	11/13/2020
5140	Toluene	EPA TO-15	10248803	Volatile Organics	10/1/2001
4700	trans-1,2-Dichloroethylene	EPA TO-15	10248803	Volatile Organics	11/13/2020
4685	trans-1,3-Dichloropropene	EPA TO-15	10248803	Volatile Organics	10/1/2001
5170	Trichloroethene (Trichloroethylene)	EPA TO-15	10248803	Volatile Organics	10/1/2001
5175	Trichlorofluoromethane	EPA TO-15	10248803	Volatile Organics	11/13/2020
4040	Valeraldehyde (Pentanal)	EPA TO-11A	10311805	Extractable Organics	10/1/2001
5235	Vinyl chloride	EPA TO-15	10248803	Volatile Organics	10/1/2001
5260	Xylene (total)	EPA TO-15	10248803	Volatile Organics	10/1/2001