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In reply, please refer to:

March 7, 2025

Rear Admiral Stephen Barnett
Commander, Navy Closure Task Force – Red Hill
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Joint Base Pearl Harbor Hickam, HI 96860-5101
[via email only: stephen.d.barnett.mil@us.navy.mil]

Dear Rear Admiral Barnett:

SUBJECT: DOH Comments on *Quarterly Release Response Report, Red Hill Bulk Fuel Storage Facility*, dated December 10, 2024

The Hawai'i Department of Health (DOH) reviewed the Navy Closure Task Force – Red Hill's (NCTF-RH's) *Quarterly Release Response Report, Red Hill Bulk Fuel Storage Facility*, dated December 10, 2024, hereinafter the "3rdQ 2024 RRR." The report was submitted to comply with release response and closure requirements for the underground storage tank (UST) system that includes the Red Hill Bulk Fuel Storage Facility, in accordance with DOH's May 2022 Emergency Order (EO) and Hawai'i Administrative Rules (HAR) Chapter 11-280.1. The purpose of the Quarterly Release Response Reports is to describe all release response actions taken for that quarter and to provide a plan for future release response actions.

After reviewing the 3rdQ 2024 RRR, the DOH has the following comments. These comments do not require the document to be revised. However, the responses should be incorporated into future Quarterly Release Response Reports.

1. **Section 3.3, PDF page 34** – States, "Once the removal action confirmation sampling results have been evaluated [for the former holding tank and former leach tank site], the Navy will develop site-specific risk-based action levels . . ." Note that site-specific risk-based action levels must be approved by the DOH prior to implementation, in accordance with HAR §11-280.1-65.3(b)(2). Any corrective actions must be conducted in accordance with the EO and HAR Chapter 11-280.1.
2. **Section 3.7.3, PDF page 37** – States the Navy will "begin a 24-month natural source-zone depletion study once Regulatory Agency concurrence of the *Natural Source-Zone Depletion Work Plan* is received (DON 2023b)." The DOH expressed our concerns and expectations regarding this work plan in letters dated April 15, 2024, and July 17, 2024. Based on the

NCTF-RH's August 23, 2024, response, we understood the NCTF-RH intended to move forward regardless of our concerns about the usefulness of this study.

3. **Section 4.4, Table 4-1, PDF page 39** – While the recommended target analytes for middle distillate fuel in Table 4-1 are consistent with the general ones in DOH's guidance document *Evaluation of Environmental Hazards at Sites with Contaminated Soil and Groundwater*, other site-specific analytes have been identified and are routinely analyzed in groundwater (e.g., fuel additives, additional polycyclic aromatic hydrocarbons, etc.). It is unclear whether the NCTF-RH only evaluated the recommended target analytes in Table 4-1 or all analytes against the appropriate DOH environmental action levels (EALs).
4. **Section 6.3.2, PDF page 48** – It is our understanding that a tentatively identified compound (TIC) analysis was conducted on a select number of groundwater samples during this period. This analysis should be discussed (including rationale) in addition to the other analyses mentioned.
5. **Section 8.0, Table 8-1, PDF page 54** – The measurement of criterion for water field duplicates is listed in Table 8-1 as less than or equal to 50 percent. This is not consistent with the listed relative percent difference performance criterion of less than or equal to 30 percent listed in Section 8.3.1, or the recommended replicate precision of 10 to 35 percent provided in Section 10.6.1 of the DOH Hazard Evaluation and Emergency Response Office Technical Guidance Manual. Explain deviations from the guidance.
6. **Section 9.3, PDF page 65** – In Table 9-1, it is unclear if the reported concentrations of total petroleum hydrocarbons (TPH) gasoline range organics, TPH diesel range organics, and TPH oil range organics were added together and then compared to the DOH EAL for TPH-middle distillate fuels, as is specified in the June 12, 2024 DOH guidance document, *Comparison of HIDOH Total Petroleum Hydrocarbons (TPH) Action Levels to Data for Water Samples*. Provide clarification.
7. **Section 9.3, PDF page 67** – While the Table 9-1 footnote states DOH EALs do not represent mandatory cleanup levels, it should be noted that, in accordance with HAR §11-280.1-65.3(b), contaminated soil, groundwater, and surface water must be remediated to either the default Tier 1 Screening Levels in Table 1 of HAR Chapter 11-280.1 (which are based on DOH EALs) or site-specific action levels agreed upon by the DOH. Site-specific action levels have not been presented or agreed upon by DOH.
8. **Section 9.3, PDF page 69** – The project screening criteria in Table 9-2 are based on the DOH Tier 1 EALs for sites where groundwater is a potential drinking water source and the nearest surface water body is greater than 150 meters away. While this criteria may have been appropriate prior to the 2021 release, due to the release(s) in the Adit 3 area and its proximity to Halawa Stream, the use of DOH Tier 1 EALs where the nearest surface water body is less than 150 meters away may be more appropriate.

9. **Section 9.3.1, PDF pages 79-85** – This section (as well as Section 10) states chromatograms were used to determine whether TPH detections came from petroleum or other organic compounds. We also understand a TIC analysis was performed on select samples.

We support the NCTF-RH's efforts to further evaluate TPH detections to determine their chemical composition. However, it is unclear how the chromatograms were used to support the determinations in this report (e.g., what makes them "not characteristic of fuel"), particularly for samples with low-level TPH detections. Provide the DOH a copy of any written procedures outlining how the determination that a detection is or is not petroleum is made.

In addition, based on the enclosed review (*Review of Tentatively Identified Compounds (TICs)*, by NewFields Environmental Forensics Practice, LLC), the TIC analysis does not provide sufficient evidence to determine certain detections of TPH are not from petroleum. To conduct an evaluation that will likely provide sufficient evidence, the NCTF-RH should follow the recommended forensic approach outlined in the Enclosure. This includes collecting and preparing groundwater samples in accordance with U.S. Environmental Protection Agency (EPA) Method 8270E, as well as qualitatively screening target analyte samples for the presence of additional classes of petroleum hydrocarbons using Extracted Ion Current Profiles.

10. **Section 9.3.1, PDF page 79** – States the initial detections of TPH in RHMW21 are "most likely attributable to well construction materials introduced during drilling or due to laboratory artifacts . . ." This determination does not seem to account for evidence of TPH observed during well installation. Additional explanation is needed.
11. **Section 9.3.1.4, PDF page 83** – The data in Table 9-3 is used to justify the NCTF-RH's determination that fuel released from the Red Hill UST system is fully degraded, and therefore, the DOH EAL for fully degraded TPH-middle distillates is applicable. However, Table 9-3 reports a wide range of percentages of polar compounds (10 percent to 100 percent), with 30 of the 41 samples in Table 9-3 indicating a potential degradation of less than 50 percent. While this indicates of some degradation, more evidence is needed to justify using the DOH EAL for fully degraded TPH middle distillates in Table 6-6c of Appendix 1 of the *Evaluation of Environmental Hazards at Sites with Contaminated Soil and Groundwater*, dated July 10, 2024. In addition, while RHWM02 may be the monitoring well with the highest concentrations of TPH detected regularly, the results from all monitoring wells should be used to determine the level of plume degradation.

Rear Admiral Stephen Barnett
March 7, 2025
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If you have any questions regarding this letter, please contact me at KellyAnn.Lee@doh.hawaii.gov or (808) 586-4226.

Sincerely,



KELLY ANN L. LEE
Red Hill Project Coordinator

Enclosure – *Red Hill Long-Term Groundwater Monitoring Program 2025, Review of Tentatively Identified Compounds (TICs)*, dated February 28th, 2025

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Red Hill Long-Term Groundwater
Monitoring Program 2025
Review of Tentatively Identified Compounds (TICs)
Oahu, Hawaii
February 28th, 2025



Red Hill Long-Term Groundwater Monitoring Program 2025

Review of Tentatively Identified Compounds (TICs) Oahu, Hawaii

Submitted to:

Hawaii Department of Health

Solid and Hazardous Waste Branch
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Date:

February 28th, 2025

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Attachments

Attachment A: SGS Laboratory Data Packages

Acronyms and Abbreviations

CFR	Code of Federal Regulation
DRO	Diesel Range Organics
DOH	Department of Health
EAL	Environmental Action Level
EICP	Electron Ion Current Profile
FID	Flame Ionization Detector
GC	Gas Chromatography
GRO	Gasoline Range Organics
ISP	Incident Specific Parameter
JBPHH	Joint Base Pearl Harbor-Hickam
JTF-RH	Joint Task Force – Red Hill
LCS	Laboratory Control Spike
LCSD	Laboratory Control Spike Duplicate
LTM	Long Term Monitoring
m/z	Mass/Charge
MB	Method Blank
MDL	Method Detection Limit
MS	Mass Spectrometry
NTA	Non-Target Analyte
ORO	Oil Range Organics
PAH	Polycyclic Aromatic Hydrocarbon
SVOC	Semi-Volatile Organic Compound
TIC	Tentatively Identified Compound
TPH	Total Petroleum Hydrocarbons
USEPA	United States Environmental Protection Agency

1.0 Introduction

This report was prepared by NewFields Environmental Forensics Practice, LLC at the request of the Solid and Hazardous Waste Branch of the Hawaii Department of Health (HIDOH). In support of the Red Hill Long Term Monitoring Program (LTM), the Navy is conducting groundwater testing for the presence of total petroleum hydrocarbons (TPH) at SGS (Orlando, Florida) using EPA Method 8015D. SGS reports TPH as C₁₀-C₂₄ Diesel Range Organics (TPH-DRO) and C₂₄-C₄₀ Oil Range Organics (TPH-ORO). A subset of LTM groundwater samples reported TPH-DRO concentrations above the laboratory method detection limit (MDL) and in some instances above the Environmental Action Level (EAL) for middle distillate fuels of 127 µg/L. To further characterize the source of TPH-DRO detections, the Navy submitted 23 groundwater samples for tentatively identified compound (TIC) analysis using EPA Method 8270E. TIC analysis characterizes samples for the presence of non-target analytes (NTAs) that are not included in the standard EPA Method 8270E target Analyte list and can be used to identify diagnostic classes of chemicals indicative of petroleum or other non-petroleum sources. However, the Navy's TIC analysis was not specifically focused on identifying petroleum hydrocarbon indicator chemicals, and as a result, does not provide the information necessary to determine if TPH-DRO detections truly contain petroleum. HIDOH requested that NewFields conduct a data quality review of the Navy's TIC analysis, provide an assessment of the usability of the TIC data, and recommend modifications to the Navy's TIC approach that can be used to more definitively evaluate samples for the presence of petroleum. The data quality and usability assessment are focused on determining if the TIC analysis performed by the Navy can be reliably used to determine if petroleum hydrocarbons are present in low-level TPH-DRO and TPH-ORO measurements.

2.0 Laboratory Data Reviewed

The Navy submitted 23 LTM groundwater samples collected between July 15th, 2024, and August 28th, 2024, for TIC analysis by EPA Method 8270E using gas chromatography mass spectrometry (GC/MS). HIDOH requested that NewFields review the TIC analysis performed by SGS on these 23 samples. Table 2-1 provides a summary of the groundwater samples reviewed in this report.

Table 2-1. LTM Groundwater Samples Analyzed for TICs

SGS Report	Laboratory ID	Client Sample ID	Collection Date	Receipt Date	Analysis Date
FC17570	FC17570-1	B24071277-001 (RHMW02)	7/15/2024	7/29/2024	8/2/2024
FC17570	FC17570-3	B24071277-003 (RHMW08)	7/15/2024	7/29/2024	8/2/2024
FC17570	FC17570-4	B24071277-004 (RHMW01R)	7/15/2024	7/29/2024	8/2/2024
FC17570	FC17570-5	B24071277-005 (RHMW01R FD)	7/15/2024	7/29/2024	8/2/2024
FC17570	FC17570-6	B24071406-005 (RHMW03)	7/16/2024	7/29/2024	8/2/2024
FC17570	FC17570-7	B24071406-006 (RHSF-PUMP)	7/16/2024	7/29/2024	8/2/2024
FC17817	FC17817-1	B24071277-001 (RHMW02 SGT)	7/15/2024	8/8/2024	8/12/2024
FC17817	FC17817-3	B24071688-002 (RHP01)	7/18/2024	8/8/2024	8/12/2024
FC17817	FC17817-4	B24071688-003 (RHP02)	7/18/2024	8/8/2024	8/12/2024
FC17817	FC17817-5	B24071879-001 (RHMW21)	7/22/2024	8/8/2024	8/12/2024
FC17817	FC17817-8	B24072012-007 (RHP04B)	7/23/2024	8/8/2024	8/12/2024
FC17817	FC17817-9	B24072275-004 (RHMW18)	7/25/2024	8/8/2024	8/12/2024
FC17817	FC17817-10	B24072301-001 (NMW24)	7/26/2024	8/8/2024	8/12/2024

Table 2-1. LTM Groundwater Samples Analyzed for TICs - continued

SGS Report Number	SGS Laboratory ID	Client Sample ID	Collection Date	Receipt Date	Analysis Date
FC18391	FC18391-1	RHMW01R-WGN01LF-2408A	8/5/2024	8/28/2024	8/30/2024
FC18391	FC18391-2	RHMW02-WGN01LF-2408A	8/5/2024	8/28/2024	8/30/2024
FC18391	FC18391-3	RHMW03-WGN01LF-2408A	8/6/2024	8/28/2024	8/30/2024
FC18391	FC18391-4	RHMW21-WGN01LF-2408A	8/5/2024	8/28/2024	8/30/2024
FC18391	FC18391-5	RHP08-WGFD01LF-2408A	8/6/2024	8/28/2024	8/30/2024
FC18994	FC18994-2	RHMW02-WGN01LF-2408B	8/22/2024	9/21/2024	10/4/2024
FC18994	FC18994-3	RHMW06-WGN01LF-2408B	8/22/2024	9/21/2024	10/4/2024
FC18994	FC18994-4	RHMW21-WGN01LF-2408B	8/22/2024	9/21/2024	10/4/2024
FC18994	FC18994-5	RHMW03-WGN01LF-2408B	8/20/2024	9/21/2024	10/4/2024
FC18994	FC18994-6	NMW25-WGN01LF-2408B	8/28/2024	9/21/2024	10/4/2024

3.0 Analytical Methods

The LTM groundwater samples listed above (Table 2-1) were first analyzed by Navy for TPH-DRO and TPH-ORO using EPA Method 8015D. The LTM samples were not initially prepared for EPA Method 8270E analysis, and as a result were not extracted using EPA Method 8270E extraction surrogates (EPA Method 8270E, 7.8, 2018). Additionally, a subset of the 23 LTM groundwater samples were analyzed outside of the analytical holding time for sample extracts. These data quality issues and the potential effects on data usability are described in greater detail below.

3.1 EPA Method 8270E Target Analyte Analysis

EPA Method 8270E is used to determine the concentration of semi-volatile organic compounds (SVOCs) in a variety of environmental matrices including groundwater. SGS reported 72 SVOC target analytes that include polycyclic aromatic hydrocarbons (PAHs), phenols, phthalates, chlorine, and nitrogen containing compounds, organic acids, and alcohols (see Attachment A: SGS Laboratory Data Packages for a full list of target analytes).

In addition to target analytes, GC/MS analysis also provides a gas chromatogram that can be used qualitatively to observe the presence of organic contaminants like petroleum hydrocarbon residues. The gas chromatogram is the instrumental output from the GC/MS analysis that depicts the distribution of SVOC compounds that compose a sample. The gas chromatogram is read from left to right. SVOC compounds in a gas chromatogram appear in order of decreasing volatility; the most volatile compounds appear earliest in the chromatogram, with compounds of lower volatility appearing sequentially relative to their increasing boiling points.¹ The discrete peaks that appear in the chromatogram represent individual compounds, the heights of which are proportional to their relative concentrations in the sample. When petroleum is present a chromatogram may contain an unresolved complex mixture (UCM) that is composed of a

¹ The elution order (e.g., retention time) of peaks on the chromatogram is a function of volatility with the most volatile compounds eluting early in the GC run (e.g., n-C₁₀), and the less volatile hydrocarbons eluting later in the GC run (e.g., n-C₃₀).

complex mixture of aromatic and aliphatic hydrocarbons and if weathered, their degradation components. Hydrocarbon source types, and specifically the UCM present in a chromatogram are commonly characterized by the boiling range or carbon range of the petroleum material (e.g., GRO, DRO, ORO). It is this fundamental gas chromatographic feature – the GC “fingerprint” – that allows the environmental chemist to identify the presence of petroleum and to potentially distinguish one hydrocarbon product from another (i.e., petroleum including crude oil, light and middle distillate fuels, MGP derived pyrogenic tar products, naturally occurring hydrocarbons, etc.).² This type of GC fingerprint is commonly reviewed using GC/FID chromatograms, but in the absence of GC/FID chromatograms, GC/MS total ion chromatograms can provide a similar type of qualitative information. Only GC/MS total ion chromatograms were available for the 23 LTM groundwater samples reviewed in this report.

3.2 EPA Method 8270E Tentatively Identified Compound (TIC) Analysis

TICs are a useful tool that can be used to characterize environmental samples for contaminants not included in conventional target analyte lists. EPA Method 8270E includes guidance for performing TIC analysis, at a minimum, laboratories performing TIC analysis should follow EPA Method 8270E’s stated TIC procedure (EPA 2018, 11.6.2). TICs are not measured as target analytes but instead are tentatively identified by performing secondary analysis of mass spectral data. If TICs are detected, mass spectral peaks are compared to the National Institute of Standards and Technology (NIST, 2005) electron ionization mass spectral library of suspect TICs.³ The most accurate procedure to confirm the presence of a chemical in the environment is to perform a target analyte analysis using a discrete analytical standard that exactly matches the identity of the target analyte of interest. However, there are thousands of potential analytes of interest, and analytical standards are not available for the majority of these potential chemicals. When analytical standards are not available, TIC analysis is used to evaluate the tentative identity of chemicals.^{4,5,6}

SGS operated the EPA Method 8270E analysis in GC/MS full-scan mode to screen for C₉ to C₄₄₊ compounds with masses ranging from 35 mass/charge (m/z) to 500 m/z. Additionally, SGS applied a filtering parameter to only evaluate TIC peaks with a minimum of 500 area counts. This means that any suspect TIC peak with < 500 area counts were not considered by SGS in their TIC analysis. This could potentially censor low level suspected TICs, including low level

² Douglas, G.D., Emsbo-Mattingly, S.D., Stout, S.A., Uhler, A.D., and McCarthy, K.J. (2015) Hydrocarbon fingerprinting methods. In: Introduction to Environmental Forensics, 3rd Ed., B. Murphy and R. Morrison, Eds., Academic Press, NY, pp. 201-310.

³ NIST. 2005. NIST, EPA, NIH Electron Ionization Mass Spectrometry (EI-MS) Library.

⁴ Sobus, J.R., Wambaugh, J.F., Isaacs, K.K. *et al.* Integrating tools for non-targeted analysis research and chemical safety evaluations at the US EPA. *J Expo Sci Environ Epidemiol* **28**, 411–426 (2018). <https://doi.org/10.1038/s41370-017-0012-y>

⁵ USEPA Region III. 2006. Tentatively Identified Compounds What are they and why are they important?

<https://19january2017snapshot.epa.gov/sites/production/files/2015-06/documents/tics.pdf>

⁶ USEPA. 1997. Technical Assessment of the Current Tentatively Identified Compound (TIC) Protocol. EPA/600/R-97/011.

hydrocarbons TICs indicative of petroleum (e.g., alkylated PAHs), from being characterized and tentatively identified.

When evaluating the identity of a TIC, it is important to carefully evaluate the quality of the spectral match between field samples and the NIST reference library. The measure of the similarity between field sample mass spectrum and the NIST reference library spectrum is called the quality score. The quality score is reported by the NIST library and assigns a percent (%) similarity between the sample and library mass spectra. It is important to note that the tentative identification of a TIC is not considered to be verified until the suspect compound can be confirmed using a certified analytical standard. SGS does not appear to have applied a quality match screening criteria for the reported TICs. This means that TICs with both high quality scores, and exceptionally low quality scores were reported. The mass spectrum for TICs with low quality match scores needs to be carefully reviewed and should be used with caution (EPA, 1997).

Additionally, the SGS TIC analysis did not report the full chemical name for all reported TICs. If the TIC name exceeded a set number of characters the chemical name was truncated and not fully reported. Truncated TIC chemical names are difficult to evaluate for quality and reliability because the full chemical name of the TIC cannot be verified.

3.3 SGS EPA Method 8270E Quality Control Deviations

None of the 23 LTM groundwater samples analyzed by SGS for EPA Method 8270E TICs were prepared with extractions surrogates. Extraction surrogates are required by EPA Method 8270E (EPA Method 8270E, 7.8, 2018) and by the Department of Defense (DOD) Quality Systems Manual 6.0 (QSM 6.0) Module 4: Quality Systems for Chemical Testing (DOD, QSM 7.2.3.C, 2023). Extraction surrogates reflect the chemistry of the target analytes being tested and allow the laboratory and data user to evaluate if the samples were adequately extracted (surrogate percent recovery = extraction efficiency). The absence of surrogates in the 23 LTM groundwater samples fails the stated SGS and DOD QSM surrogate recovery acceptance criteria (Attachment A: SGS Data Packages; DOD QSM, Appendix C, Table C26).

According to EPA's National Functional Guidelines for Organics Analysis, "If surrogate standards were not added to the samples and blanks or the concentrations of surrogates in the samples and blanks are not as specified, use professional judgment to qualify detects and non-detects. Examine the data package narrative and standards and sample preparation logs included in the data package or notify the designated project management personnel who may arrange for the laboratory to repeat the analyses as specified and/or to provide any missing information. **In the event that a reanalysis cannot be performed, qualify the data as unusable (R)**" (EPA, pg. 152, 2020). Under standard EPA data validation procedures, the data for the 23 LTM groundwater samples would be qualified as unusable and rejected.

Additionally, the custody and extraction records found in the SGS data packages do not provide a traceable accounting of the true sample collection and extraction dates of the samples. For example, SGS Report FC17570 reports the collection date for samples FC17570-1 to FC17570-

5 as July 15th, 2024. However, a note in the SGS case narrative states that, “Sample extracted April 03-2024 13:12 extract received from Energy Labs. Sample received outside the holding time. Insufficient extract for reanalysis.” This case narrative note indicates the sample was extracted at “Energy Labs” (not SGS) over 3-months prior to the sample being analyzed at SGS by EPA Method 8270E. This exceeds the EPA National Functional Guidelines extract analytical holding time of 40-days (EPA, pg. 53, 2020). Based on the SGS data package documentation, it is unclear when the 23 LTM groundwater samples were collected and/or extracted (SGS Lab Packages: FC17570, FC17817, FC18391, FC18994). It does appear that SGS is listing the EPA Method 8015D extraction date (performed at Energy Labs and possibly at SGS for some samples), as the sample collection date for the EPA Method 8270E TIC analysis (Tabel 2-1). For a subset of samples, it also appears that the samples were analyzed outside of analytical holding time, although due to the available documentation this is not verifiable for all reported samples. If samples were analyzed outside of holding time, this would also require that the data be additionally qualified as estimated and potentially unusable.

The absence of extraction surrogates and exceeding analytical holding times significantly impairs the usability of this data. If this data were being used for strict regulatory purposes the data would need to be qualified as unusable and rejected for use in decision making. For the purposes of this TIC analysis, the data should be treated with caution. See section 6.0 of this report for recommendations on developing a sampling and analysis plan for the evaluation of petroleum in groundwater samples that satisfies the requirements of the DOD’s QSM 6.0 and the EPA’s National Functional Guidelines for EPA Method 8270E analysis.

4.0 EPA Method 8270E Results

The LTM groundwater sample data was reviewed using a tiered forensic approach. The three tiers included, 1) a qualitative review of the EPA Method 8270E GC/MS total ion chromatogram for the presence of petroleum, 2) a review of the EPA Method 8270E target analyte data for the presence of petroleum hydrocarbon indicator chemicals like PAHs, and 3) a review of the EPA Method 8270E TIC analysis for the presence of petroleum and non-petroleum related non-target analytes. The findings of this analysis are presented below.

4.1 Tier I: Qualitative Review of EPA Method 8270E Total Ion Chromatograms

The GC/MS total ion chromatogram for each sample was qualitatively reviewed for the presence of an unresolved complex mixture (UCM) that is indicative of petroleum. Six of the twenty-one LTM groundwater samples reviewed contained a UCM or other chromatographic feature, like gasoline range peaks, which are potentially indicative of petroleum. The petroleum residues observed in these samples are heavily weathered, and do not appear to be the result of a recent petroleum release. Table 4-1 provides a summary of the samples and the approximate carbon range of the potential petroleum hydrocarbon material. Qualitatively reviewing GC/MS chromatograms is not a definitive method for identifying petroleum, especially at low levels. Samples qualitatively identified as containing petroleum hydrocarbons should undergo further forensic testing to more fully characterize the classes and composition of hydrocarbons present.

Sample B24071277-005 (RHMW01R) and associated field duplicate B24071277-005 (RHMW01R FD) both contain low-level DRO residues. Field duplicate B24071277-005 (RHMW01R FD) also contains a heavier ORO residue that is not present in sample B24071277-005 (RHMW01R). The source of the ORO material is unknown, it is possible this could be due to sample heterogeneity, or it could be carryover from a contaminated sample analyzed prior to sample B24071277-005 (RHMW01R FD).

Samples not listed in Table 4-1 to not contain chromatographic features that indicate the presence of petroleum.

Table 4-1. Summary of LTM Groundwater Chromatograms Containing Petroleum

SGS Laboratory ID	Client Sample ID	Collection Date	Tentative Petroleum Carbon Range ⁷	Degree of Weathering
FC17570-1	B24071277-001 (RHMW02)	7/15/2024	DRO	Heavily Weathered
FC17570-4	B24071277-004 (RHMW01R)	7/15/2024	DRO	Heavily Weathered
FC17570-5	B24071277-005 (RHMW01R FD)	7/15/2024	DRO/ORO	Heavily Weathered
FC17817-1	B24071277-001 (RHMW02 SGT)	7/15/2024	Low Level UCM	NA
FC18391-2	RHMW02-WGN01LF-2408A	8/5/2024	GRO/DRO	Heavily Weathered
FC18994-2	RHMW02-WGN01LF-2408B	8/22/2024	DRO	Heavily Weathered
FC18994-5	RHMW03-WGN01LF-2408B	8/20/2024	GRO Peaks	Heavily Weathered

4.2 Tier II: Review of EPA Method 8270E Target Analytes

The LTM groundwater samples analyzed by EPA Method 8270E reported non-detects for over 99% of the SVOC target analytes evaluated. Four LTM samples reported estimated detections of either PAHs, phthalates, or benzoic acid. Sample RHMW02 reported estimated results (“J” qualified) for naphthalene, and 1-methylnaphthalene, while sample RHMW02 SGT reported an estimated result for naphthalene. The presence of naphthalene and 1-methylnaphthalene can be indicative of the presence of petroleum or other anthropogenic sources of hydrocarbons arising from industrial activities.

Sample RHP02 reported an estimated concentration of bis(2-Ethylhexyl)phthalate and sample RHMW21 reported an estimated concentration of benzoic acid. Neither of these low-level detections are generally associated with the presence of petroleum hydrocarbons. Due to the absence of extraction surrogates, non-detects may be unreliable, and need to be treated with caution.

⁷ Diesel Range Organics (DRO), Gasoline Range Organics (GRO), and Oil Range Organics (ORO) carbon ranges are estimated using EPA Method 8270E internal quantitation standards.

Table 4-2. Summary of EPA Method 8270E Target Analyte Detections

SGS Laboratory ID	Client Sample ID	Collection Date	Naphthalene (µg/L)	1-Methyl-naphthalene (µg/L)	bis(2-Ethylhexyl) phthalate (µg/L)	Benzoic Acid (µg/L)
FC17570-1	B24071277-001 (RHMW02)	7/15/2024	2.4 J	0.81 J	< DL	< DL
FC17817-1	B24071277-001 (RHMW02 SGT)	7/15/2024	1.3 J	< DL	< DL	< DL
FC17817-4	B24071688-003 (RHP02)	7/18/2024	< DL	< DL	2.0 J	< DL
FC17817-5	B24071879-001 (RHMW21)	7/22/2024	< DL	< DL	< DL	33.9 J

4.3 Tier III: Review of Tentatively Identified Compounds

The SGS TIC results were evaluated for the presence of non-target petroleum hydrocarbon indicator compounds like alkylated benzenes, and alkylated PAHs. When reviewing samples for TICs, it is important to review the TICs detected in laboratory method blanks and equipment blanks to ensure that field sample TIC detections are true *in situ* chemicals and not artifacts from laboratory processing or sample collection. The most common TICs detected in the SGS TIC analysis were not from site impacts but instead were from laboratory standards (e.g., o-terphenyl), and background laboratory or field contamination (e.g., amylene hydride, cyclic siloxanes, and methyl furans).

However, there were also TICs identified in select samples that could be potentially indicative of *in situ* conditions. These TICs were characterized as hydrocarbons of multiple classes, and organic acids. A summary of the TICs identified in relevant samples is provided below. Samples not listed below did not contain TICs that were diagnostic of petroleum or other types of uncharacterized site contamination.

B24071277-001 (RHMW02)

Sample B24071277-001 (RHMW02) contains a mixture of carboxylic acids including methylated benzoic acids, hexa-, and octadecenoic acids, and cyclic hydrocarbons like cyclohexanes and fused bicyclic hydrocarbons like indane. These TICs could be indicative of degraded petroleum.

B24071406-005 (RHMW03)

Sample B24071406-005 (RHMW03) contains a limited number of hydrocarbons, and oxygenated hydrocarbon TICs that could be indicative of degraded petroleum.

Additionally, sample B24071406-005 (RHMW03) also contains a series of siloxanes that are known to be common laboratory and sample collection artifacts and are not likely to be associated with true site contamination.

RHMW03-WGN01LF-2408B

Sample RHMW03-WGN01LF-2408B contains a limited number of cyclic, branched alkene, and alkane hydrocarbons TICs that could be indicative of degraded petroleum.

A summary of SGS reported TICs can be found in Table 4.3. Table 4.3 lists the TIC with the highest NIST mass spectral library quality score for each TIC peak. TICs reported by SGS that were detected in lab or equipment blanks or are known lab standards are highlighted in grey. Attachment A: SGS Data Packages provides a full listing of all TICs reported by SGS.

5.0 Discussion of EPA Method 8270E Data

The TIC analysis of 23 LTM groundwater sample did not provide data of sufficient quality or chemical specificity to determine if TPH-DRO or TPH-ORO measurements truly contained petroleum. The TIC data has significant quality control issues that limit the reliability and usability of these results. The approach used by SGS, to analyze groundwater samples prepared for EPA Method 8015D analysis for TICs using EPA Method 8270E without adequate quality controls (e.g., 8270E extraction surrogates) is not advisable. Additionally, the sample custody records, including the date of sample collection, and sample extraction (and the laboratory performing the extraction) are not verifiable based on the documentation in the SGS data packages. At least a subset of samples appears to have been analyzed outside of the analytical holding time for extracts. According to the EPA's National Functional Guidelines these data are unusable and should be qualified as rejected ("R").

The Tier I review of the GC/MS chromatograms observed petroleum in six samples (Table 4-1) of these samples, the presence of petroleum in RHMW02 and RHMW02 SGT was further supported by naphthalene and/or 1-methylnaphthalene target analyte detections and the presence of select hydrocarbon TICs that could be associated with petroleum. Sample RHMW03-WGN01LF-2408B also contained TICs that could be associated with the presence of petroleum.

The SGS framework used to perform the TIC analysis was not specifically focused on diagnostic hydrocarbons TICs known to be indicative of petroleum. Due to the limitations of the TIC analysis, and the stated data quality issues, this data is not reliable for use in evaluating the presence of petroleum residues in LTM groundwater samples. A recommended approach is provided below in Section 6.

6.0 Recommended Forensic Approach for Characterizing Petroleum Hydrocarbons using Extracted Ion Current Profiles

If low-level TPH-DRO or TPH-ORO results are reported above the laboratory detection limit, and below the EAL for middle distillate fuels of 127 µg/L, or if non-petroleum chemicals are suspected of interfering with TPH measurements, a forensic approach can be used to determine if petroleum hydrocarbons or other non-petroleum chemicals are truly present in the TPH measurement. Samples should be collected, and prepared following all requirements found in EPA Method 8270E, and the EPA's National Functional Guidelines for Organics Analysis. This could include collecting enough sample volume to analyze samples for both TPH by EPA

Method 8015D and SVOCs by EPA Method 8270E. As a matter of good lab practice, samples not properly prepared for EPA Method 8270E analysis should not be analyzed using this method.

In addition to analyzing groundwater samples for EPA Method 8270E target analytes samples should also be qualitatively screened for the presence of additional classes of petroleum hydrocarbons using Extracted Ion Current Profiles (EICPs). EICPs allow for a detailed review of each sample's GC/MS data that is used to assess the presence of both target and non-target petroleum hydrocarbon analytes. This is a comprehensive way to evaluate the presence of hundreds of non-target petroleum hydrocarbons in groundwater samples. EPA Method 8270E should be operated in full-scan mode to screen for $C_9 - C_{44+}$ compounds with masses ranging from 35 m/z to 450 m/z. Table 6-1 includes an inventory of the EICPs and diagnostic compound classes that are recommended when reviewing sample data for the presence of $C_9 - C_{44+}$ petroleum hydrocarbons and other non-petroleum chemicals.⁸ Screening groundwater samples for petroleum hydrocarbons using EICPs provides a diagnostic technique specifically focused on detecting petroleum. The classes of petroleum hydrocarbons screened for using EICPs occur in homologous series (e.g., C1-, C2-, C3- and C4-alkylated naphthalenes) that have known patterns of alkylated PAH isomers. The presence of these isomers, and the recognition of known petroleum patterns provide a reliable metric to perform low-level petroleum screening. The laboratory reporting limit for the EPA Method 8270E EICP and TIC analysis is 1.0 to 5.0 µg/L depending on the compound class. Samples that contain petroleum should be submitted for full forensic characterization to determine the specific source(s) of contamination and a more thorough evaluation of the degree of weathering.

Table 6-1. C_9 - C_{44+} Aliphatic and Alkylated PAH EICPs

Compound Class	EICPs (m/z)
Saturated Hydrocarbons	85, 83
Alkylated Benzenes	78, 92, 106, 120, 134, 148
Alkylated Naphthalenes	128, 142, 156, 170, 184
Alkylated Fluorenes	166, 180, 194, 208
Alkylated Phenanthrene/Anthracenes	178, 192, 206, 220, 234

⁸ Emsbo-Mattingly, S.D. and E.R. Litman. 2016. "Polycyclic Aromatic Hydrocarbon Homolog and Isomer Fingerprinting." Figures 5.6 to 5.9. In: Standard Handbook of Oil Spill Environmental Forensics: Fingerprinting and Source Identification, 2nd Ed., S.A. Stout and Z. Wang, Eds., Elsevier Publishing Co., Boston, MA. ISBN 9780128038321.

7.0 Summary of Conclusions

The review of the SGS TIC data presented in this report resulted in the following conclusions:

- 1) The SGS EPA Method 8270E target analyte and TIC data reported in Data Packages FC17570, FC17817, FC18391 have significant data quality issues and should be used cautiously if not qualified as unusable.
 - a. LTM groundwater samples were not prepared with appropriate EPA Method 8270E extraction surrogates. The efficiency of the extraction process has not been demonstrated, and target analyte and TIC non-detects are not reliable.
 - b. The custody records for these samples are not verifiable based on the SGS Data Packages. At least samples in FC17570 appear to have been extracted at a non-SGS laboratory and analyzed by SGS for TICs over 3 months after sample extraction.
- 2) Based on a qualitative review of the EPA Method 8270E GC/MS total ion chromatograms, 6 of the 23 LTM groundwater samples appear to contain weathered petroleum.
- 3) Naphthalene was detected as a target analyte in samples B24071277-001 (RHMW02) and B24071277-001 (RHMW02 SGT), and 1-methylnaphthalene was detected in sample B24071277-001 (RHMW02).
- 4) The majority of reported TICs in the 23 LTM groundwater samples are 1) laboratory analytical standards or 2) are associated with artifacts from laboratory processing or field sample collection.
- 5) Three LTM groundwater samples identified as containing petroleum in Tier I contained TICs potentially arising from petroleum hydrocarbons (RHMW02, RHMW02 SGT and RHMW03-WGN01LF-2408B).
- 6) Three LTM ground water samples identified as containing petroleum in Tier I did not contain TICs indicative of petroleum or contained TICs of equivocal spectral match quality (B24071277-004 RHMW01R, RHMW02-WGN01LF-2408A, and RHMW02-WGN01LF-2408B).
- 7) The TIC analysis was not of sufficient data quality or chemical specificity to be used reliably in characterizing low-level TPH-DRO or TPH-ORO measurements for the presence of petroleum.
- 8) Using EICP screening, focused on homologous series of diagnostic petroleum hydrocarbons (e.g., alkylated PAHs) is a reliable approach for characterizing low level TPH-DRO and TPH-ORO measurements for the presence of petroleum.

8.0 References

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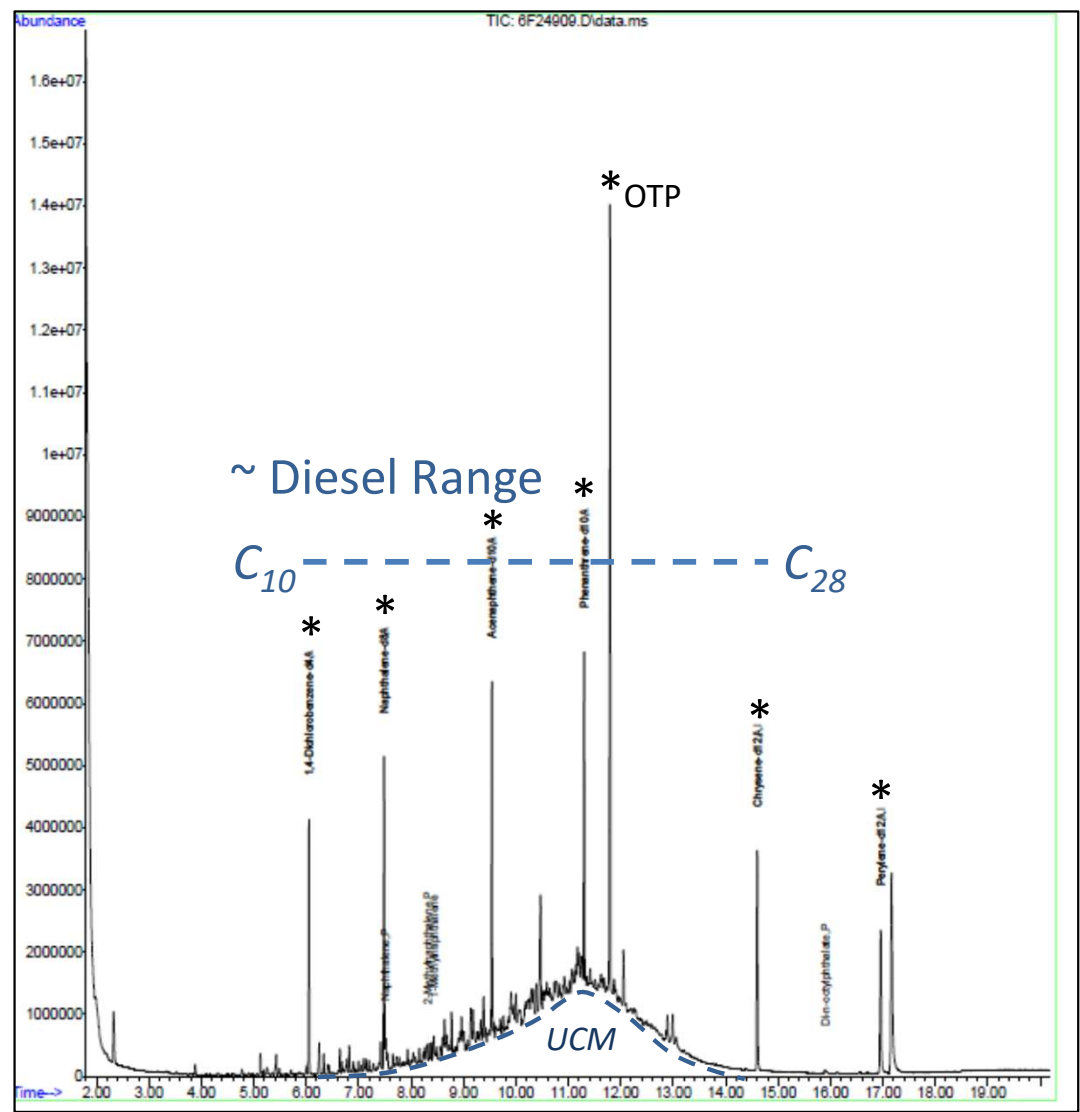
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Figure 1. Example 8270E GC/MS Total Ion Chromatogram



Lab Report	Lab ID	Sample ID
FC17570	FC17570-1	B24071277-001 (RHMW02)

* Lab Standards



Table 4.3a. Tentatively Identified Compound (TIC) Summary – SGS Report FC17570

Lab ID	Sample ID	TIC Name	MW	Chemical Formula	CAS#	Q Score	Detected in Method Blank	Lab Standard
FC17570-1	B24071277-001 (RHMW02)	Amylene Hydrate	88	C5H12O	000075-85-4	74	Y	
		Cyclohexane, 1-ethyl-2-methyl-	126	C9H18	004923-77-7	64		
		Indane	118	C9H10	000496-11-7	93		
		1,1-Dimethyl-4-methylenecyclohexane	124	C9H16	1000210-04-7	53		
		Benzoic acid, 2,4,6-trimethyl-	164	C10H12O2	000480-63-7	76		
		Benzoic acid, 2,4,5-trimethyl-	164	C10H12O2	000528-90-5	55		
		1-Naphthalenecarboxylic acid, 5	176	C11H12O2	004242-18-6	94		
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
		n-Hexadecanoic acid	256	C16H32O2	000057-10-3	98		
		9-Octadecenoic acid	282	C18H34O2	000112-79-8	99		
		Octadecanoic acid	284	C18H36O2	000057-11-4	98		
FC17570-3	B24071277-003 (RHMW08)	Amylene Hydrate	88	C5H12O	000075-85-4	74	Y	
		2-Butenal,3-methyl-	84	C5H8O	000107-86-8	53	Y	
		2-Hexene, 3,4,4-trimethyl-	126	C9H18	053941-19-8	72		
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
FC17570-4	B24071277-004 (RHMW01R)	Amylene Hydrate	88	C5H12O	000075-85-4	74	Y	
		2-Butenal,3-methyl-	84	C5H8O	000107-86-8	53	Y	
		Cyclohexane, 1-ethyl-2-methyl-	126	C9H18	004923-77-7	72		
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
FC17570-5	B24071277-005 (RHMW01R FD)	Amylene Hydrate	88	C5H12O	000075-85-4	74	Y	
		2-Butenal,3-methyl-	84	C5H8O	000107-86-8	53	Y	
		Cyclohexane, 1-methyl-2-propyl	140	C10H20	004291-79-6	72		
FC17570-6	B24071406-005 (RHMW03)	Amylene Hydrate	88	C5H12O	000075-85-4	74	Y	
		3-Penten-2-one, 4-methyl-	98	C6H10O	000141-79-7	91		
		Cyclotrisiloxane, hexamethyl-	222	C6H18O3Si3	000541-05-9	91		
		2-Butene, 1-bromo-3-methyl-	148	C5H9Br	000870-63-3	64		
		Cyclopentane, 1,2,3,4,5-pentamet	140	C10H20	1000152-79-7	58		
		Cyclotetrasiloxane, octamethyl-	296	C8H24O4Si4	000556-67-2	91		
		Ethylidenecyclobutane	82	C6H10	001528-21-8	58		
		Cyclopentasiloxane, decamethyl	370	C10H30O5Si5	000541-02-6	87		
		Cyclotetrasiloxane, octamethyl-	296	C8H24O4Si4	000556-67-2	58		
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
FC17570-7	B24071406-006 (RHSF-PUMP)	Amylene Hydrate	88	C5H12O	000075-85-4	74	Y	
		Cyclotrisiloxane, hexamethyl-	222	C6H18O3Si3	000541-05-9	86		
		2-Butenal,3-methyl-	84	C5H8O	000107-86-8	53	Y	
		Cyclohexane, (1,2,2-trimethylbut	182	C13H26	061142-21-0	64		
		o-Terphenyl	230	C18H14	000084-15-1	98		Y



 TIC identified in laboratory method blank or equipment blank and is likely a lab artifact or standard (e.g., o-terphenyl) or an artifact of field collection.



Table 4.3b. Tentatively Identified Compound (TIC) Summary – SGS Report FC17817

Lab ID	Sample ID	TIC Name	MW	Chemical Formula	CAS#	Q Score	Detected in Method Blank	Lab Standard
FC17817-1	B24071277-001 (RHMW02 SGT)	o-Terphenyl	230	C ₁₈ H ₁₄	000084-15-1	98		Y
FC17817-3	B24071688-002 (RHP01)	o-Terphenyl	230	C ₁₈ H ₁₄	000084-15-1	98		Y
FC17817-4	B24071688-003 (RHP02)	o-Terphenyl	230	C ₁₈ H ₁₄	000084-15-1	98		Y
FC17817-5	B24071879-001 (RHMW21)	Butanoic acid	88	C ₄ H ₈ O ₂	000107-92-6	91		
		o-Terphenyl	230	C ₁₈ H ₁₄	000084-15-1	98		Y
		9,12,15-Octadecatrien-1-ol	264	C ₁₈ H ₃₂ O	000506-44-5	97		
FC17817-8	B24072012-007 (RHP04B)	1-Propene, 1,2,3-trichloro-	144	C ₃ H ₃ Cl ₃	013116-57-9	64	Y	
		o-Terphenyl	230	C ₁₈ H ₁₄	000084-15-1	98		Y
FC17817-9	B24072275-004 (RHMW18)	Butane, 2-chloro-2-methyl-	106	C ₅ H ₁₁ Cl	000594-36-5	50		
		1-Propene, 1,2,3-trichloro-	144	C ₃ H ₃ Cl ₃	000096-19-5	68	Y	
		o-Terphenyl	230	C ₁₈ H ₁₄	000084-15-1	98		Y
FC17817-10	B24072301-001 (NMW24)	Cyclotrisiloxane, hexamethyl-	222	C ₆ H ₁₈ O ₃ Si ₃	000541-05-9	91	Y	
		1-Propene, 1,1,2-trichloro-	144	C ₃ H ₃ Cl ₃	021400-25-9	83	Y	
		o-Terphenyl	230	C ₁₈ H ₁₄	000084-15-1	98		Y

 TIC identified in laboratory method blank or equipment blank and is likely a lab artifact or standard (e.g., o-terphenyl) or an artifact of field collection.


MW: Molecular Weight

Q Score: Quality Match Score between the field sample TIC mass spectrum and the NIST library mass spectrum.



Table 4.3c. Tentatively Identified Compound (TIC) Summary – SGS Report FC18391

Lab ID	Sample ID	TIC Name	MW	Chemical Formula	CAS#	Q Score	Detected in Method Blank	Lab Standard
FC18391-1	RHMW01R-WGN01LF-2408A	Amylene Hydrate	88	C5H12O	000075-85-4	74	Y	
		Cyclohexane, (1,2,2-trimethylbut	182	C13H26	061142-21-0	72		
		2-Pentene, 2,3,4-trimethyl-	112	C8H16	000565-77-5	64		
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
FC18391-2	RHMW02-WGN01LF-2408A	Amylene Hydrate	88	C5H12O	000075-85-4	74	Y	
		Propanoic acid, 2-methyl-, 3-met	158	C9H18O2	002050-01-3	72	Y	
		2-Hexene, 3,4,4-trimethyl-	126	C9H18	053941-19-8	72	Y	
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
FC18391-3	RHMW03-WGN01LF-2408A	Amylene Hydrate	88	C5H12O	000075-85-4	74	Y	
		3,4-Diethyl hexane	142	C10H22	019398-77-7	72		
		Sulfurous acid, cyclohexylmethyl	304	C16H32O3S	1000309-21-8	72		
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
FC18391-4	RHMW21-WGN01LF-2408A	Amylene Hydrate	88	C5H12O	000075-85-4	74	Y	
		Propanoic acid, 2-methyl-, 3-met	158	C9H18O2	002050-01-3	72	Y	
		Cyclohexane, 1-methyl-2-propyl-	140	C10H20	004291-79-6	64		
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
FC18391-5	RHP08-WGFD01LF-2408A	3,4-Diethyl hexane	142	C10H22	019398-77-7	72		
		Sulfurous acid, cyclohexylmethyl	304	C16H32O3S	1000309-21-8	72		
		o-Terphenyl	230	C18H14	000084-15-1	98		Y

 TIC identified in laboratory method blank or equipment blank and is likely a lab artifact or standard (e.g., o-terphenyl) or an artifact of field collection.


MW: Molecular Weight

Q Score: Quality Match Score between the field sample TIC mass spectrum and the NIST library mass spectrum.



Table 4.3d. Tentatively Identified Compound (TIC) Summary – SGS Report FC18894

Lab ID	Sample ID	TIC Name	MW	Chemical Formula	CAS#	Q Score	Detected in Method Blank	Lab Standard
FC18994-2	RHMW02-WGN01LF-2408B	Furan, 2,5-dimethyl-	96	C6H8O	000625-86-5	94	Y	
		2-(2-Ethyl-1,3-dimethyl-cyclopentyl)-	182	C12H22O	1000186-82-4	50		
		Tricyclo[4.3.1.1 ^{3,8}]undecane, 1	180	C12H20O	021898-95-3	43		
		1-Naphthalenecarboxylic acid, 5	176	C11H12O2	004242-18-6	94		
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
		Triphenylphosphine oxide	278	C18H15OP	000791-28-6	92	Y	
FC18994-3	RHMW06-WGN01LF-2408B	Furan, 2,5-dimethyl-	96	C6H8O	000625-86-5	94	Y	
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
		Formylmethylenetriphenylphosphorane	304	C20H17OP	028900-91-6	90	Y	
FC18994-4	RHMW21-WGN01LF-2408B	Furan, 2,5-dimethyl-	96	C6H8O	000625-86-5	94	Y	
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
		Formylmethylenetriphenylphosphorane	304	C20H17OP	028900-91-6	90	Y	
FC18994-5	RHMW03-WGN01LF-2408B	Furan, 2,5-dimethyl-	96	C6H8O	000625-86-5	94	Y	
		Hexane, 3-methyl-4-methylene-	112	C8H16	003404-67-9	43		
		2-Butenal, 3-methyl-	84	C5H8O	000107-86-8	58	Y	
		1-Butanol, 3-methyl-, carbonate	202	C11H22O3	002050-95-5	78		
		Cyclohexane, 1,1-dimethyl-	112	C8H16	000590-66-9	64		
		Pentene, 2,3,4-trimethyl-	112	C8H16	000565-77-5	72		
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
		Acetic acid, (triphenylphosphorane)	334	C21H19O2P	002605-67-6	91	Y	
FC18994-6	NMW25-WGN01LF-2408B	Furan, 2,5-dimethyl-	96	C6H8O	000625-86-5	94	Y	
		o-Terphenyl	230	C18H14	000084-15-1	98		Y
		Formylmethylenetriphenylphosphorane	304	C20H17OP	028900-91-6	90	Y	

 TIC identified in laboratory method blank or equipment blank and is likely a lab artifact or standard (e.g., o-terphenyl) or an artifact of field collection.

MW: Molecular Weight

Q Score: Quality Match Score between the field sample TIC mass spectrum and the NIST library mass spectrum.