



Joint Base Pearl Harbor-Hickam Drinking Water Characterization

Oahu, Hawaii
May 10th, 2024



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Oahu, Hawaii

Submitted to:

Hawaii Department of Health

Safe Drinking Water Branch
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Executive Summary

This report was prepared by NewFields Environmental Forensics Practice, LLC at the request of the Safe Drinking Water Branch of the Hawaii Department of Health (HIDOH). On behalf of the Navy, drinking water samples were collected in 2023 from Joint Base Pearl Harbor-Hickam (JBPHH) as part of the Red Hill Long Term Monitoring Program (LTM) and were analyzed by SGS (Wheat Ridge, CO) for Total Petroleum Hydrocarbons (TPH) using EPA Method 8015D. SGS reported TPH results by carbon range as C₁₀-C₂₄ Diesel Range Organics (DRO) and C₂₄-C₄₀ Oil Range Organics (ORO). A subset of 2023 LTM drinking water samples reported DRO concentrations above the SGS method detection limit (MDL) of 50 micrograms per liter (µg/L) but below the Incident Specific Parameter (ISP) of 266 µg/L. To further investigate the source of DRO detections above the MDL, the HIDOH requested that NewFields perform a forensic chemistry analysis on twelve JBPHH household drinking water samples and three drinking water shaft samples collected from the Waiawa Shaft, Navy Aiea Halawa Shaft, and Red Hill Shaft Adit-3. The household drinking water samples and shaft samples were collected by the HIDOH on February 12th, 13th, and 20th, 2024. The purpose of this report is to evaluate the chemical composition and potential sources of the DRO detections reported by SGS in 2023.

February 2024 household drinking water and shaft samples were analyzed at Alpha Analytical (Mansfield, Massachusetts) by EPA Method 8015D. The 8015D analysis reported low level TPH concentrations below and just above the laboratory reporting limit. NewFields analysis determined that no petroleum hydrocarbons, including petroleum distillates like jet fuel, were detected in any of the household drinking water or shaft samples above the laboratory detection limit. The TPH detections reported in household drinking water and shaft samples are not due to the presence of petroleum but are due to the presence of other non-petroleum chemicals and the limitations of the laboratory methodology. The TPH results for twelve of the fifteen samples were qualified by the laboratory as estimates because they were either detected below the laboratory low-level calibration standard (“J” qualified as estimates) or were determined to contain low-level laboratory contamination (“B” qualified due to laboratory contamination). The remaining three samples contained results that were reported just above the laboratory’s reporting limit.

The twelve household drinking water samples reported TPH concentrations that ranged from 2.0 to 72 µg/L and the three drinking water shaft samples reported TPH concentrations that ranged from 6.0 to 87 µg/L. The TPH results in each sample were carefully screened for petroleum using EPA Method 8260C-PIANO and EPA Method 8270E. These analyses confirmed that no petroleum or petroleum distillates were present in household drinking water or shaft samples above the laboratory detection limit.

The 8270E analysis did determine that household drinking water samples contain low relative levels of non-petroleum related chemicals like brominated organic compounds, and halogenated alcohols. Additionally, household drinking water and shaft samples both contained naturally occurring organic acids. See section 5.0 of this report for a full discussion of laboratory results. The laboratory data can be found in Appendix D.

At the request of HIDOH, additional testing is being performed to further characterize the chemical composition of the non-petroleum related chemicals like brominated organic compounds and halogenated alcohols. The findings of the additional testing will be presented in a supplemental report.

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Acronyms and Abbreviations

CDC	Child Development Center
CFR	Code of Federal Regulation
DPB	Disinfection Byproduct
DPD	N, N-diethyl-p-phenylenediamine
DRO	Diesel Range Organics
DOH	Department of Health
EICP	Electron Ion Current Profile
FID	Flame Ionization Detector
GC	Gas Chromatography
GPS	Geographic Positioning System
GRO	Gasoline Range Organics
HDPE	High Density Polyethylene
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
LLDPE	Linear Low-Density Polyethylene
LTM	Long Term Monitoring
m/z	Mass/Charge
MB	Method Blank
MDL	Method Detection Limit
MS	Mass Spectrometry
NOAA	National Oceanic and Atmospheric Association
NTA	Non-Target Analyte
ORO	Oil Range Organics
PIANO	Paraffin, Isoparaffin, Aromatic, Naphthene, Olefin
SGC	Silica Gel Cleanup
SVOC	Semi-Volatile Organic Compound
TEO	Total Extractable Organics
TIC	Tentatively Identified Compound
TPH	Total Petroleum Hydrocarbons
USEPA	United States Environmental Protection Agency
VOC	Volatile Organic Compounds

1.0 Introduction

This report was prepared by NewFields Environmental Forensics Practice, LLC at the request of the Safe Drinking Water Branch of the Hawaii Department of Health (HIDOH). On behalf of the Navy, drinking water samples were collected in 2023 from Joint Base Pearl Harbor-Hickam (JBPHH) as part of the Red Hill Long Term Monitoring Program (LTM) and were analyzed by SGS (Wheat Ridge, CO) for Total Petroleum Hydrocarbons (TPH) using EPA Method 8015D. SGS reported TPH results by carbon range as C₁₀-C₂₄ Diesel Range Organics (DRO) and C₂₄-C₄₀ Oil Range Organics (ORO). A subset of 2023 LTM drinking water samples reported DRO concentrations above the SGS method detection limit (MDL) of 50 micrograms per liter (µg/L) but below the Incident Specific Parameter (ISP) of 266 µg/L.¹ To further investigate the source of DRO detections above the MDL, the HIDOH requested that NewFields perform a forensic chemistry analysis on JBPHH household drinking water and drinking water shaft samples to evaluate the chemical composition and potential sources of DRO detections. The purpose of this report is to chemically characterize the composition of drinking water samples collected by HIDOH in February of 2024 and to evaluate samples for the presence of petroleum hydrocarbons, or other non-petroleum chemicals. This report also includes a review of EPA Method 8015D and provides an evaluation of the suitability of this method for accurately and precisely measuring low-level TPH and DRO concentrations in aqueous sample matrices. This report presents the findings of this forensic drinking water analysis.

1.1 Limitations of Method 8015D in Low-Level DRO Analysis

It is important to note early in this report that EPA Method 8015D analysis is both a conventional laboratory technique used for testing water samples for petroleum hydrocarbons and that Method 8015D was not designed to identify specific chemicals or classes of chemicals in drinking water samples. The utility and limitations of Method 8015D are discussed in greater detail below.

EPA Method 8015D uses Gas Chromatography with a Flame Ionization Detector (GC/FID) and provides a bulk measurement of organic matter detected within a defined carbon range (e.g. gasoline range organics-GRO, diesel range organics-DRO, oil range organics-ORO).² The organic matter measured by EPA Method 8015D can include but is not limited to the presence of petroleum hydrocarbons. GC/FID analysis alone cannot identify or differentiate the specific types of chemicals reported within a bulk TPH measurement. At higher relative concentrations it may be clear that a drinking water sample contains petroleum hydrocarbons, but at low levels confirmation analysis is needed to identify, or tentatively identify the specific chemical constituents. When analyzing samples with very low levels of organic matter, it is important to characterize the specific classes of chemicals present using a method like GC Mass Spectrometry (GC/MS). GC/MS analysis can be used to determine if low-level organic matter in

¹ Red Hill Interagency Task Force.2024. Lines of Evidence Evaluation of TPH Detects Observed During Long-Term Monitoring (LTM).

² USEPA.2003. Validated Test Method 8015D: Nonhalogenated Organics Using GC/FID.
[Validated Test Method 8015D: Nonhalogenated Organics Using GC/FID](#)

a sample is truly composed of petroleum hydrocarbons or arises from other non-petroleum related organic matter like non-petroleum chemicals or naturally occurring biogenic materials like plant waxes or organic acids. Additionally, water samples prepared for EPA Method 8015D analysis by separatory funnel (liquid-liquid extraction) using dichloromethane (DCM, EPA Method 3510C) do not generally undergo any sample cleanup procedures, such as silica-gel or activated alumina, to remove non-petroleum organic matter. Sample extraction Method 3510C states that, "This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures." The use of EPA Method 3510C, without silica gel or activated alumina cleanup, is a more conservative analytical approach to measuring TPH because it includes a broad profile of chemical constituents that may include various non-polar and polar petroleum hydrocarbons and other non-petroleum related organic matter.

Depending on site conditions, this can result in sample extracts that contain multiple classes of chemicals potentially including petroleum hydrocarbons, non-petroleum chemicals, and other naturally occurring biogenic materials and their polar degradation products such as organic acids.³ One such specific site condition is the environment in the JBPHH water distribution system, where the chemistry of the drinking water and contaminants can be affected by the addition of chlorine and the formation of chlorinated disinfection products.

EPA Method 8015D uses the analyte names TPH and DRO to define the carbon ranges in which organic chemicals are present, but the method is not able to definitively determine if low-level petroleum hydrocarbons are present or if TPH and DRO concentrations are due to the presence of non-petroleum chemicals or other naturally occurring materials. A more accurate description of low-level TPH, DRO and ORO results is Total Extractable Organics (TEO). TEO more broadly accounts for the potential presence of both petroleum hydrocarbons, and other non-petroleum chemicals. To be clear, carefully evaluating low-level TPH and DRO detections is critically important to protecting human and ecological health, and some low-level TPH and DRO results may not contain petroleum hydrocarbons and are more accurately described as TEO.⁴ A critical step in evaluating low-level TPH, DRO, ORO or TEO data is to perform a confirmatory analysis by a more highly resolved method like GC-Mass Spectrometry (GC/MS) which can potentially identify the classes of chemicals and determine if petroleum hydrocarbons or other types of chemicals are present. The forensic characterization described in this report recognizes the limitations of EPA Method 8015D and utilizes supplemental forensic methods to

³ USEPA. 2016. Method 3510C: Separatory Funnel Liquid-Liquid Extraction, part of Test Methods for Evaluating Solid Waste, Physical/Chemical Methods

<https://www.epa.gov/sites/default/files/2015-12/documents/3510c.pdf>

⁴ This is a known limitation of EPA Method 8015D, for example during the Mississippi Canyon 252 (Deepwater Horizon) Natural Resource Damage Assessment, the National Oceanic and Atmospheric Association (NOAA) reported TPH C₉-C₄₄ as "Total Extractable Material (TEM)" to account for the presence of both petroleum and non-petroleum related chemicals.

<https://pub-data.diver.orr.noaa.gov/field-files/MC%20252%20Analytical%20QAP%20V4%200%20FINAL.pdf>

further evaluate drinking water samples for the presence of petroleum hydrocarbons, and other non-petroleum chemicals.

1.2 Limitations of this Study

The intent of this investigative study is to use advanced forensic techniques to enhance the quality of detections and confirm the potential presence of petroleum hydrocarbons in a drinking water matrix. The number of samples in this investigation is limited and as such the results are specific to the location tested and not representative of all buildings or residences in the JBP HH Public Water System (PWS) HI0000360 or the consecutive Aliamanu Military Reservation (AMR) HI0000337 PWS.

2.0 Sample Collection

The HODOH collected 12 drinking water samples from JBP HH households and 3 shaft samples from the Waiawa Shaft, Navy Aiea Halawa Shaft, and Red Hill Shaft Adit-3 on February 12th, 13th, and 20th, 2024. Samples were collected according to HODOH standard operating procedures for sample collection. Table 2-1 includes a summary of the sample collection dates, sample locations and free chlorine concentrations (mg/L) measured in the field by HODOH. The free chlorine concentration is a measure of the chlorine content added during the drinking water disinfection process. The chlorine concentrations measured in household drinking water samples are consistent with state and federal safety guidelines.

Table 2-1. Sample Collection Information

Field ID	Laboratory ID	Collection Date	Address	Cl ₂ (mg/L)	Received Cl ₂ Disinfection (Y/N)
021224-002-01	L2408333-01	2/12/2024	5792A Erne Avenue	0.79	Yes
021224-002-02	L2408333-02	2/12/2024	1824 Madrona Place	0.71	Yes
021224-002-03	L2408333-03	2/12/2024	6363 Papaya Lane	0.55	Yes
021224-002-04	L2408333-04	2/12/2024	6653 Plumeria Loop	0.63	Yes
021224-002-05	L2408333-05	2/12/2024	419 Valley View Loop	0.66	Yes
021224-002-06	L2408333-06	2/12/2024	269 Etccl Court	0.21	Yes
021224-009-01	L2408333-07	2/12/2024	Bldg 570, Ford Island CDC 319 Lexington Blvd	0.59	Yes
021224-009-02	L2408333-08	2/12/2024	Bldg 4655, Catlin School Age Center 4655 Catlin Dr	0.61	Yes

Field ID	Laboratory ID	Collection Date	Address	Cl ₂ (mg/L)	Received Cl ₂ Disinfection (Y/N)
021224-009-03	L2408333-09	2/12/2024	15 Julian Ave	0.69	Yes
021224-009-04	L2408333-10	2/12/2024	546 Ohana Nui Circle	0.76	Yes
021324-009-01	L2408333-11	2/13/2024	Waiawa Shaft	0.02	No
021324-009-02	L2408333-12	2/13/2024	Navy Aiea Halawa Shaft	NA	No
021324-009-03	L2408333-13	2/13/2024	Red Hill Shaft - Adit 3	0.04	No
021324-009-04	L2408333-14	2/13/2024	417 Valley View Loop	0.58	Yes
022024-009-01	L2408333-16	2/20/2024	731 Ohana Nui Circle	0.67	Yes

2.1 Sampling Location Selection and History

The selection of residential and child development center (CDC) sample locations was based on five residence sampled under the October 2023 Premise Plumbing Investigation as well as seven locations that reported elevated TPH (GRO, DRO and RRO range) detections during LTM periods 5 and 6. In this study, a drinking water shaft sample was collected from the Waiawa shaft because it is the sole source of drinking water to JBPHH PWS HI0000360 and the consecutive AMR PWS HI0000337 system. Samples were also collected from the Navy Aiea Halawa Shaft, and Red Hill Shaft to further chemically characterize these drinking water resources. The majority of residential TPH results, reported during LTM periods 5 and 6, were detected between the method detection limit (MDL) and the reporting limit (RL) 50-80 ug/L. Table 2-1-1 summarizes the historic residential/CDC sampling results for TPH collected and tested by the US Navy for the locations included in the study.

Table 2-1-1. LTM Residential Sample Locations and Previous Sample Results

Location	Date	TPH Result (Total GRO, DRO, ORO) µg/L
5792A Erne Avenue	7/19/2023	96.5
	2/12/2024	ND
	2/12/2024	ND
1824 Madrona Place	9/26/2023	139
	12/28/2023	ND
	2/12/2024	ND
	2/12/2024	ND

Table 2-1-1. LTM Residential Sample Locations and Previous Sample Results (continued)

Location	Date	TPH Result (Total GRO, DRO, ORO) µg/L
6363 Papaya Lane	9/13/2023	240.3
	9/13/2023	58.5
	2/12/2024	ND
	2/12/2024	ND
6653 Plumeria Loop	9/14/2023	93.7
	9/14/2023	58.3
	2/12/2024	ND
	2/12/2024	ND
419 Valley View Loop	1/29/2024	ND
	2/12/2024	ND
	2/12/2024	74
269 Etccl Court	10/20/2023	71.2
	2/12/2024	ND
	2/12/2024	ND
Bldg 570, Ford Island CDC 319 Lexington Blvd	3/17/2022	ND
	3/17/2022	58.1
	4/12/2022	ND
	4/12/2022	ND
	5/6/2022	ND
	5/6/2022	ND
	8/24/2022	57.2
	8/24/2022	51.1
	2/14/2023	70.6
	2/14/2023	76.9
	9/22/2023	87.6
	9/22/2023	73
	1/18/2024	ND
	1/18/2024	ND
	2/12/2024	ND
	2/12/2024	ND
2/27/2024	68.2	
2/27/2024	71.2	

Table 2-1-1. LTM Residential Sample Locations and Previous Sample Results (continued)

Location	Date	TPH Result (Total GRO, DRO, ORO) µg/L
Bldg 4655, Catlin School Age Center 4655 Catlin Dr	4/1/2022	ND
	4/1/2022	ND
	5/9/2022	ND
	5/9/2022	ND
	5/9/2022	ND
	5/31/2022	ND
	5/31/2022	59.3
	9/6/2022	ND
	9/6/2022	ND
	2/22/2023	60.3
	2/22/2023	ND
	8/28/2023	82.6
	8/28/2023	56.8
	1/11/2024	158.5
	1/11/2024	99.9
2/12/2024	ND	
2/12/2024	ND	
2/26/2024	ND	
2/26/2024	ND	
15 Julian Ave	3/29/2022	ND
	10/20/2023	50.2
	2/12/2024	ND
	2/12/2024	ND
546 Ohana Nui Circle	10/20/2023	ND
	2/12/2024	ND
	2/12/2024	ND
417 Valley View Loop	9/14/2023	84.3
	9/14/2023	199.2
	2/13/2024	ND
	2/13/2024	ND
731 Ohana Nui Circle	10/20/2023	56
	10/25/2023	65.9
	2/12/2024	ND
	2/12/2024	ND

Additionally, LTM monitoring samples tested from the Waiawa shaft reported detections of Volatile Organic Compounds (VOCs) and Semi-Volatile Organic Compounds (SVOCs). Table 2-1-2 summarizes past detections of VOCs and SVOCs reported from the Waiawa shaft. It should be noted that Dieldrin and 1,2-dibromo-3-chloropropane were not detected in recent monitoring activities and will be monitored in the 2023-2025 compliance sampling period. During the Navy’s LTM program Bis(2-ethylhexyl)phthalate was detected in 7 of 9 samples taken between May 2022 to January 2024. All detections were below the Maximum Contaminant Level (MCL) of 6 µg/L (ppb).

Table 2-1-2. Historical Detection of VOC's and SVOCs at Waiawa Shaft

Compound	Location	Date	Result µg/L
Dieldrin	Waiawa Shaft Chlorinator	4/26/2017	0.01
1,2-dibromo-3-chloropropane	Waiawa Shaft Chlorinator	4/12/2012	0.014
Bis(2-ethylhexyl)phthalate*	Waiawa Shaft Chlorinator	5/6/2022	0.52
		7/14/2022	0.55
		7/13/2023	0.61
		1/12/2024	0.67
		1/30/2024	1.1
		1/30/2024	0.95
		1/12/2024	0.76

* This result from Navy LTM was detected after the water advisory for JBPHH and AMR was lifted.

3.0 Analytical Methods

The samples collected by HODOH in February 2024 (Table 2-1) were analyzed using the following methods specifically developed for the forensic characterization of petroleum hydrocarbons and the evaluation of non-petroleum chemicals.⁵ All samples were shipped to Alpha Analytical, Inc. in Mansfield, Massachusetts (Alpha) for forensic testing. The Alpha analytical data package can be found in Appendix D. The forensic testing performed on household drinking water and shaft samples included the following laboratory analyses.

3.1 Modified EPA Method 8015D

Samples were analyzed using a modified version of EPA Method 8015D to determine the TPH concentration (C₉-C₄₄) and simultaneously provide a detailed fingerprint of hydrocarbons present in each sample. This analysis was used to screen samples for the presence of petroleum, and if present to characterize the general boiling ranges and types of petroleum present. Following solvent extraction by EPA Method 3510C with dichloromethane (DCM) sample extracts were analyzed by GC/FID using a high-resolution extended acquisition method (> 70-minutes) to fully resolve the presence of any potential complex hydrocarbon mixtures. The concentration of TPH in drinking water samples is reported in µg/L. To monitor the sensitivity, accuracy, and precision of TPH data, each laboratory quality control batch was prepared with a method blank (MB), lab control sample (LCS), and lab control sample duplicate (LCSD). The laboratory reporting limit for TPH ranges from 31 to 33 µg/L depending on the volume of sample collected.

⁵ 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.

3.2 Modified EPA Method 8260C-PIANO

Samples were also analyzed using a modified version of EPA Method 8260C to determine the concentrations of 88 volatile hydrocarbon target analytes by purge-and-trap using GC/MS operated in full scan mode. The target analytes reported by this method are specifically focused on characterizing C₅ – C₁₃ range petroleum distillates and are grouped into five hydrocarbon compound classes: paraffins, isoparaffins, aromatics, naphthenes, and olefins (PIANO). In addition to PIANO target analytes, various oxygenated compounds commonly found in oxygenated and reformulated gasolines and other petroleum distillates were also included in the analysis such as tert-butyl alcohol (TBA), methyl-tert-butyl ether (MTBE), di-isopropyl ether (DIPE), ethyl-tert-butyl ether (ETBE), and tert-amyl-methyl ether (TAME). If petroleum is present in samples, these results provide a basis to characterize and distinguish different types of light petroleum products like gasoline, kerosene, and jet fuels. The concentrations of target compounds in drinking water and shaft samples are reported in µg/L. To monitor the sensitivity, accuracy, and precision of PIANO data the laboratory prepared a MB, LCS, and LCSD. A full list of PIANO target analytes can be found in Table 3-2-1 (Attached).

In addition to analyzing drinking water samples for the target analytes listed in Table 3-2-1, samples were also qualitatively screened for the presence of additional classes of petroleum hydrocarbons by reviewing Extracted Ion Current Profiles (EICPs). EICPs allow for a detailed review of each sample and provide data that is used to assess the presence of both target and non-target petroleum hydrocarbon analytes.⁶ This is a comprehensive way to evaluate the presence of petroleum hydrocarbons in drinking water samples. Table 3-2-2 includes an inventory of the EICPs and diagnostic compound classes that were reviewed for the presence of C₅ – C₁₃ petroleum hydrocarbons and other non-petroleum chemicals. The reporting limit for PIANO analysis varies by target analyte and ranges from 2.0 to 5.0 µg/L.

Table 3-2-2. C₅-C₁₃ VOC-PIANO EICPs

Compound Class	EICPs (m/z)
Saturated Hydrocarbons	43, 57, 85
Alkylated Benzenes	78, 92, 106, 120, 134, 148
Alkylated Naphthalenes	128, 142, 156

⁶ Emsbo-Mattingly, S.D. and E.R. Litman. 2016. "Polycyclic Aromatic Hydrocarbon Homolog and Isomer Fingerprinting." 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.

3.3 Modified EPA Method 8270E Non-Target Analyte (NTA) and Tentatively Identified Compound (TIC) Analysis

If TPH results were reported above the detection limit, a modified version of EPA Method 8270E was used to further characterize the results by performing EICP screening for petroleum hydrocarbons and to identify the presence of other non-target analytes (NTAs) such as non-petroleum chemicals. EPA Method 8270E was operated in full-scan mode to screen for C₉ – C₄₄₊ compounds with masses ranging from 35 m/z to 450 m/z.

Similar to the PIANO EICP analysis, a series of diagnostic EICPs were also reviewed to carefully examine each sample for the presence of C₉ – C₄₄₊ petroleum hydrocarbons. Table 3-3-1 includes an inventory of the EICPs and diagnostic compound classes that were reviewed for the presence of C₉ – C₄₄₊ petroleum hydrocarbons and other non-petroleum chemicals.

Table 3-3-1. C₉-C₄₄₊ SVOC 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

In addition to EICP screening, samples were also screened for the presence of non-petroleum NTA peaks. NTAs are not measured as target analytes by standard regulatory compliance methods like EPA Method 8270E and instead are identified by carefully examining chromatograms for peaks detected above the laboratory detection limit, which are not 1) target analytes, 2) laboratory internal standards, or 3) present in the laboratory method blank. Peaks that meet these criteria are identified as NTAs and are subjected to further analysis by comparing NTA mass spectrum to the National Institute of Standards and Technology (NIST) mass spectral library to tentatively identify the compounds (TICs).⁷ 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

⁷ NIST. 2005. NIST, EPA, NIH Electron Ionization Mass Spectrometry (EI-MS) Library.

analytical standards are not available for the majority of these potential chemicals. When analytical standards are not available, NTA analysis is used to tentatively identify compounds.^{8,9} When evaluating the identity of a TIC, it is important to carefully evaluate the quality of the spectral match between field sample and 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 spectrum. In this preliminary analysis, only NTAs with quality scores >50% were assigned TICs. In some cases, it is not possible to definitively identify the chemical of interest, but it may be possible to determine the class of compound (e.g. organic acid, or organic bromate).¹⁰ The laboratory reporting limit for the EPA Method 8270E EICP and NTA analysis is 1.0 to 5.0 µg/L depending on the compound class.

3.4 Free Chlorine by USEPA DPD Method

HIDOH performed a field analysis for free chlorine (Cl₂) in all drinking water samples collected. To perform this analysis HIDOH used the USEPA N,N-diethyl-phenylenediamine (DPD) method which was conducted using the Hach DR300 Colorimeter field instrument. The linear range of the Hach DR300 field instrument ranges from 0.02 to 2.0 mg/L of free chlorine. The free chlorine concentrations measured in each sample are reported in Table 2-1.

4.0 Evaluation of Forensic Chemistry Data

In this report the interpretation of forensic chemistry data relies on established hydrocarbon “fingerprinting” laboratory methods, namely a forensic modification of EPA Method 8015D for Total Petroleum Hydrocarbons (TPH) using GC/FID, a modification of EPA Method 8260C designed to measure volatile petroleum hydrocarbons such as PIANO-VOCs using GC/MS, and a modification of EPA Method 8270E for semi-volatile Organics by GC/MS used to screen EICPs for petroleum hydrocarbons and evaluate samples for NTAs and if present, to identify TICs. These forensic methods were used in a tiered fashion to systematically screen each sample for the presence of petroleum hydrocarbons and other non-petroleum chemicals, and to chemically characterize any materials that are detected.

The tiered forensic approach was conducted according to the following framework:

4.1 Tier I: Modified EPA Method 8015D High Resolution GC/FID Fingerprint

- In Tier I analysis, samples are screened for petroleum hydrocarbons using a modified version of EPA Method 8015D. The forensic modification of method

⁸ 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>

⁹ 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>

¹⁰ USEPA.1997. Technical Assessment of the Current Tentatively Identified Compound (TIC) Protocol. EPA/600/R-97/011.

8015D uses high resolution GC/FID fingerprints that provide greater separation between carbon ranges and allows for a more accurate assessment of potential source materials.

- EPA Method 8015D analysis helps answer the question: Are C₉-C₄₄₊ extractable organics present above the laboratory detection limit?
- If there are C₉-C₄₄₊ extractable organics present above the laboratory detection limit, samples are then submitted for Tier II analysis using a more highly resolved forensic analysis that can identify specific chemicals or classes of chemicals.

4.2 Tier II: Confirmation Testing by GC/MS for C₅-C₁₃ and C₉-C₄₄₊ Hydrocarbons and Other Purgeable or Extractable Organics

- Tier I samples containing TPH results above the laboratory detection limit are then analyzed by EPA Method 8260D-PIANO for C₅-C₁₃ petroleum hydrocarbons and other purgeable organics and by Method 8270E for C₉-C₄₄₊ petroleum hydrocarbons and other extractable organics.
- Tier II confirmation analysis by GC/MS is used to perform target analyte, EICP, and NTA analysis to identify purgeable, or extractable chemical constituents present in low level TPH measurements. Tier II GC/MS confirmation analysis is used to determine if TPH measurements are truly petroleum hydrocarbons or contain other non-petroleum constituents.

4.3 Tier III: Petroleum Characterization

- If petroleum hydrocarbons are detected during Tier I or Tier II analysis, samples are then analyzed by a modified version of EPA Method 8270E for parent and Alkylated PAHs. In Tier III analysis, Alkylated PAH and PIANO-VOC data are used quantitatively to chemically characterize petroleum hydrocarbon residues, determine and differentiate source type, and evaluate the degree of environmental weathering.

5.0 Results

Twelve household drinking water and three drinking water shaft samples were analyzed for the presence of petroleum hydrocarbons and screened for the presence of NTAs. As described in detail above, household drinking water and shaft samples were analyzed by EPA Method 8015D, EPA 8260C-PIANO, EPA Method 8270E-SVOC-TIC.

The following section presents a summary of results according to the tiered forensic framework.

5.1 Tier I: Review of EPA 8015D TPH Results and Chromatograms

The GC/FID chromatograms for household drinking water and shaft samples were carefully reviewed for the presence of petroleum. **The GC/FID chromatograms do not indicate the**

presence of petroleum, or petroleum distillates (including jet fuel), in any samples.
 Appendix A provides a summary of GC/FID chromatograms reviewed in the Tier I analysis.

Table 5-1 provides a summary of TPH results, laboratory reporting limits and laboratory qualifiers. The household drinking water samples have TPH concentrations ranging from 2.0 µg/L to 71 µg/L. While the shaft water samples report TPH concentrations ranging from 6.0 to 86 µg/L. These low-level TPH results are reported near, and below the reporting limit (< RL are “J” flagged as estimates), and several results are “B” flagged due to contamination in the laboratory method blank.

Table 5-1 EPA Method 8015D Results

Client ID	Lab ID	Units	Reporting Limit	TPH Result	Lab Qualifier
021224-002-01	L2408333-01	µg/L	32	72	
021224-002-02	L2408333-02	µg/L	32	27	JB
021224-002-03	L2408333-03	µg/L	32	42	B
021224-002-04	L2408333-04	µg/L	32	24	JB
021224-002-05	L2408333-05	µg/L	32	15	JB
021224-002-06	L2408333-06	µg/L	32	19	JB
021224-009-01	L2408333-07	µg/L	32	57	B
021224-009-02	L2408333-08	µg/L	32	54	B
021224-009-03	L2408333-09	µg/L	32	55	B
021224-009-04	L2408333-10	µg/L	32	61	B
021324-009-01	L2408333-11	µg/L	32	6.0	J
021324-009-02	L2408333-12	µg/L	32	87	
021324-009-03	L2408333-13	µg/L	32	19	J
021324-009-04	L2408333-14	µg/L	32	61	
022024-009-01	L2408333-16	µg/L	32	2.0	J

5.1.1 Data Quality and Data Usability Review of TPH Results

There are three factors that need to be carefully considered when evaluating the quality and usability of the EPA Method 8015D TPH results presented in this report, 1) the laboratory's data quality objectives (blank contamination, reporting limits, and laboratory data qualifiers), 2) method artifacts that introduce bias into TPH results, and 3) the presence of non-petroleum NTAs in the C₉-C₄₄₊ TPH carbon range.

- 1) This report presents low-level TPH results for fifteen samples. Five TPH sample results are "B" flagged by the laboratory due to laboratory blank contamination. This means the results in the drinking water samples are not meaningfully different than the TPH result reported from the method blank. Four TPH results are "JB" flagged, meaning the samples contained low-level laboratory contamination, but the TPH results are below the reporting limit and are flagged as estimates. Three samples reported TPH results that are "J" flagged as estimated values for being below the reporting limit, and three TPH samples results are reported above the reporting limit with no qualifier.
- 2) In low-level samples, EPA Method 8015D is particularly vulnerable to high bias in the C₂₈ to C₄₄₊ carbon range. The Interstate Technology and Regulatory Council's TPH guidance reports that, "The analysis [of TPH] is limited to C₃₅ because most labs have a difficult time getting to C₄₄ without discrimination and significant column bleed."¹¹ Column bleed refers to an analytical phenomenon where the gas chromatography column sheds part of the polymer stationary phase during analysis and subsequently, positively responds in the range > C₂₈ mimicking the presence of TPH hydrocarbon mass. If not carefully accounted for, column bleed will introduce a positive bias into TPH results. This phenomenon presents a particular challenge in low-level drinking water analysis.
- 3) As discussed above in section 1.2, EPA Method 8015D results can potentially contain both petroleum and non-petroleum chemicals. The household drinking water and shaft samples all contain non-petroleum NTAs. These NTAs introduce a high bias into the TPH results. For example, there is an NTA present in all the household drinking water samples, within the C₉-C₄₄₊ TPH carbon range at an ~ retention time of 5.15 minutes). By comparing the TPH chromatograms to the 8270E mass spectrum, the peak was identified as a non-petroleum compound. However, this peak is not present in the shaft samples. After subtracting this non-petroleum NTA peak from the household drinking water samples, six of the tap water samples have non-detect TPH levels, two samples have reduced concentrations below the reporting limit of 32 µg/L, while the four remaining household drinking water samples remain below the reporting limit and are "B" flagged for blank contamination.

These three factors provide important context regarding the reliability of low-level TPH results. Of the fifteen samples analyzed, twelve of the samples are either "B" flagged for blank

¹¹ ITRC.2018.TPH Risk Evaluation at Petroleum-Contaminated Sites. Section 4: TPH Fundamentals. Washington D.D.: ITRC, TPH Risk Evaluation Team.

contamination or are reported below the reporting limit and “J” flagged as estimates. This indicates that these twelve TPH sample results are not meaningfully different than the laboratory method blank and are not reliable TPH detections. It is also important to remember that EPA Method 8015D has an inherent high bias in the C₂₈₊ carbon range due to column bleed that likely introduces variability into low-level results. Finally, it is important to consider that all fifteen-household drinking water and shaft samples contain non-petroleum NTAs that introduce low-level contributions into the final TPH results. The TPH concentrations presented in this report contain contributions from laboratory artifacts and non-petroleum NTA chemicals. If these TPH results were corrected for laboratory contamination and the presence of non-petroleum NTAs, the TPH results would be non-detect for all fifteen drinking water and shaft samples.

5.2 Tier II: Review of 8260D-PIANO-VOC and 8270E EICPs and NTAs

The Tier II forensic analysis is focused on determining the chemical composition of the low-level TPH results reported for household drinking water and shaft samples. The samples were further analyzed for C₅-C₁₃ PIANO VOCs and C₉-C₄₄₊ SVOCs. Tier II analysis is discussed in greater detail below.

5.2.1 C₅ – C₁₃ EPA Method 8260C PIANO Target Analyte Results and EICP Review

The PIANO-VOC data does not indicate the presence of petroleum or petroleum distillates (like jet fuel) in any household drinking water or shaft samples. No PIANO VOC target analytes were detected in 11 of the 12-household drinking water or in any of the 3 shaft samples. One household drinking water sample (021224-002-03) reported estimated detections of naphthalene, 2-methylnaphthalene and 1-methylnaphthalene below the laboratory reporting limit (“J” flagged as estimates). The detection of naphthalene in this sample was also “B” flagged for laboratory contamination due to the presence of naphthalene in the laboratory method blank. The detection of these naphthalene and methylnaphthalene analytes are likely due to laboratory contamination and not due to the presence of low-level petroleum in sample 021224-002-03.

In addition to reviewing PIANO target analytes, the EICPs listed in section 3-2-2 (Saturated Hydrocarbons, Alkylated Benzenes and Alkylated Naphthalenes) were carefully reviewed for the presence of petroleum hydrocarbons. These EICPs do not contain alkylated benzene or alkylated PAH isomer patterns indicative of the presence of petroleum hydrocarbons. Chromatographic peaks present in these profiles were also observed in the laboratory method blank EICPs and are internal standards or lab artifacts.¹² Appendix B provides the PIANO EICPs that were reviewed.

¹² 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.

The absence of petroleum hydrocarbons in the PIANO-VOC target analyte evaluation and EICP analysis further confirm that the household drinking water and shaft samples do not contain petroleum, or petroleum distillates (like jet fuel) above the laboratory detection limit in the C₅-C₁₃ carbon range.

5.2.2 C₉ – C₄₄₊ EPA Method 8270E EICP, Target Analyte, NTA and TIC Analysis

The 8270E C₉-C₄₄₊ data do not indicate the presence of petroleum or petroleum distillates (like jet fuel) in any household drinking water or shaft samples. The 8270E data was first analyzed for the presence of petroleum hydrocarbons using the EICPs presented in Table 3-3-1, followed by the evaluation and tentative identification of other non-target analytes present in the water samples. The 8270E GC/MS Chromatograms and EICPs are provided in Appendix C.

5.2.2.1 C₉ – C₄₄₊ 8270E EICP Review for Petroleum Hydrocarbons

First, the EICPs listed in Table 3-3-1 (Saturated Hydrocarbons, Alkylcyclohexanes, Alkylated Benzenes, Alkylated Naphthalenes, Alkylated Fluorenes, Alkylated Phenanthrene/Anthracenes) were carefully reviewed for the presence of petroleum hydrocarbons. These EICPs do not contain alkylated benzene or alkylated PAH isomer patterns indicative of the presence of petroleum hydrocarbons.¹³ Chromatographic peaks present in these profiles were also observed in the laboratory method blank EICPs and are internal standards or lab artifacts. The EICPs confirm that none of the peaks in the 8270E SVOC chromatograms are from petroleum hydrocarbons (Appendix C).

5.2.2.2 C₉ – C₄₄₊ 8270E Target Analyte Review

EPA Method 8270E target analytes were qualitatively reviewed in the GC/MS chromatograms (Appendix C). There are low-level target compounds identified in the water samples. All household drinking water and shaft samples contain phthalates including diethylphthalate, di-n-butylphthalate, butylbenzylphthalate, and bis(2-ethylhexyl)phthalate. Phthalates are known sampling and laboratory artifacts, and it is likely these compounds are associated with field or laboratory activities and not site conditions.

All drinking water samples have a chromatographic peak with a retention time of approximately 4.88 minutes. This peak is identified as bis(2-chloroethyl)ether in sample 021224-002-01. However, this chromatographic peak is not identified in the other drinking water samples because the ratio of primary and secondary ion scans used to identify this peak did not fall within the appropriate confirmation range. The lack of ion confirmation is likely due to the low-level nature of this peak and the loss of mass spectral resolution at lower concentrations.

Sample 021324-009-02 contains the target analyte, Caprolactam and sample 022024-009-01

¹³ 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.

contains the target analyte, 1,2-Dichlorobenzene; however, this peak has a small relative abundance.

5.2.2.3 C₉ – C₄₄₊ 8270E NTA and TIC Review

Household drinking water and shaft samples were screened for the presence of NTAs by EPA Method 8270E. This analysis determined that the household drinking water samples contained low relative levels of tentatively identified brominated organic compounds, and halogenated alcohols. The household drinking water and shaft samples also contain naturally occurring organic acids. These NTAs do not arise from petroleum sources and are not associated with petroleum distillates like jet fuel. At the direction of HDOH, further testing is being conducted to thoroughly characterize the origin and chemical composition of these tentatively identified compounds. The results of this additional testing will be presented in a supplemental report.

5.3 Tier III: Forensic Alkylated PAH Analysis

Tier I and Tier II analysis clearly confirm there is no petroleum present in these samples above the EPA Method 8015D, EPA Method 8260C PIANO-VOC and EPA Method 8270E detection limits. Due to the absence of petroleum in these samples there was no utility in further analyzing drinking water samples by petroleum specific forensic methods. This evaluation only required Tier I and Tier II analysis.

6.0 Conclusions

The forensic analysis presented in this report resulted in the following conclusions:

- 1) No household drinking water or shaft samples contain petroleum, or petroleum distillates (including jet fuel) above the laboratory detection limit.
- 2) All household drinking water samples contained tentatively identified brominated organic compounds and halogenated alcohols.
- 3) Samples tested from the Waiawa Shaft, Navy Aiea Halawa Shaft, and Red Hill shaft did not contain the tentatively identified brominated organic or halogenated alcohol compounds.
- 4) Household drinking water and Waiawa Shaft, Navy Aiea Halawa Shaft, and Red Hill Shaft samples contained naturally occurring biogenic compounds like organic acids.
- 5) At the direction of HDOH, additional testing is being conducted to characterize the origin and chemical composition of the tentatively identified brominated organic and halogenated alcohol compounds. The findings of this analysis will be presented in a supplemental report.

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Table 3-2-1: EPA Method 8260C-PIANO-VOC Target Analytes

Class	Abbrev	Analytes	Class	Abbrev	Analytes
I	IP	ISOPENTANE	P	C8	OCTANE
O	1P	1-PENTENE	ADD	12DBE	1,2-DIBROMOETHANE
O	2M1B	2-METHYL-1-BUTENE	A	EB	ETHYLBENZENE
P	C5	PENTANE	S	2ETHIO	2-ETHYLTHIOPHENE
O	T2P	TRANS-2-PENTENE	A	MPX	P/M-XYLENE
O	C2P	CIS-2-PENTENE	O	1N	1-NONENE
OX	TBA	TERTIARY BUTANOL	SHC	C9	NONANE (C9)
N	CYP	CYCLOPENTANE	A	STY	STYRENE
I	23DMB	2,3-DIMETHYLBUTANE	A	OX	O-XYLENE
I	2MP	2-METHYLPENTANE	A	IPB	ISOPROPYLBENZENE
OX	MTBE	METHYL TERT BUTYL ETHER	A	PROPB	N-PROPYLBENZENE
I	3MP	3-METHYLPENTANE	A	1M3EB	1-METHYL-3-ETHYLBENZENE
O	1HEX	1-HEXENE	A	4ET	1-METHYL-4-ETHYLBENZENE
P	C6	N-HEXANE	A	135TMB	1,3,5-TRIMETHYLBENZENE
OX	DIPE	ISOPROPYL ETHER	O	1D	1-DECENE
OX	ETBE	ETHYL-TERT-BUTYL-ETHER	A	1M2EB	1-METHYL-2-ETHYLBENZENE
I	22DMP	2,2-DIMETHYLPENTANE	P	C10	DECANE (C10)
N	MCYP	METHYLCYCLOPENTANE	A	124TMB	1,2,4-TRIMETHYLBENZENE
I	24DMP	2,4-DIMETHYLPENTANE	A	SECBUT	SEC-BUTYLBENZENE
ADD	12DCA	1,2-DICHLOROETHANE	A	1M3IPB	1-METHYL-3-ISOPROPYLBENZENE
N	CH	CYCLOHEXANE	A	1M4IPB	1-METHYL-4-ISOPROPYLBENZENE
I	2MH	2-METHYLHEXANE	A	1M2IPB	1-METHYL-2-ISOPROPYLBENZENE
A	B	BENZENE	A	INDA	INDANE
I	23DMP	2,3-DIMETHYLPENTANE	A	1M3PB	1-METHYL-3-N-PROPYLBENZENE
S	THIO	THIOPHENE	A	1M4PB	1-METHYL-4-N-PROPYLBENZENE
I	3MH	3-METHYLHEXANE	A	BUTB	N-BUTYLBENZENE
O	TAME	TERTIARY-AMYL METHYL ETHER 1-HEPTENE/1,2-DMCP (TRANS)	A	12DM4EB	1,2-DIMETHYL-4-ETHYLBENZENE
I	ISO	ISOCTANE	A	12DEB	1,2-DIETHYLBENZENE
P	C7	HEPTANE	A	1M2PB	1-METHYL-2-N-PROPYLBENZENE
N	MCYH	METHYLCYCLOHEXANE	A	14DM2EB	1,4-DIMETHYL-2-ETHYLBENZENE
I	25DMH	2,5-DIMETHYLHEXANE	P	C11	UNDECANE
I	24DMH	2,4-DIMETHYLHEXANE	A	13DM4EB	1,3-DIMETHYL-4-ETHYLBENZENE
I	223TMP	2,2,3-TRIMETHYLPENTANE	A	13DM5EB	1,3-DIMETHYL-5-ETHYLBENZENE
I	234TMP	2,3,4-TRIMETHYLPENTANE	A	13DM2EB	1,3-DIMETHYL-2-ETHYLBENZENE
I	233TMP	2,3,3-TRIMETHYLPENTANE	A	12DM3EB	1,2-DIMETHYL-3-ETHYLBENZENE
I	23DMH	2,3-DIMETHYLHEXANE	A	1245TMP	1,2,4,5-TETRAMETHYLBENZENE
I	2MHEP	2-METHYLHEPTANE	A	PENTB	N-PENTYLBENZENE
I	3MHEP	3-METHYLHEPTANE	P	C12	DODECANE (C12)
I	3EH	3-ETHYLHEXANE	A	N0	NAPHTHALENE
A	T	TOLUENE	S	BT0	BENZOTHIOPHENE
S	2MTHIO	2-METHYLTHIOPHENE	ADD	MMT	MMT
S	3MTHIO	3-METHYLTHIOPHENE	SHC	C13	TRIDECANE
O	1O	1-OCTENE	A	2MN	2-METHYLNAPHTHALENE
			A	1MN	1-METHYLNAPHTHALENE