**LABORATORY DATA CONSULTANTS, INC.**
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AECOM
1001 Bishop Street Suite 1600
Honolulu, HI 96813
ATTN: Ms. Alethea Ramos
alethea.ramos@aecom.com

June 26, 2023

SUBJECT: Red Hill Oily Waste Disposal Facility, CTO 18F0176 - Data Validation

Dear Ms. Ramos,

Enclosed are the final validation reports for the fractions listed below. These SDGs were received on October 11, 2022. Attachment 1 is a summary of the samples that were reviewed for each analysis.

**LDC Project #55171_RV1:**

| **SDG #** | **Fraction** |
|---|---|
| 580-118109-1 | Volatiles, Semivolatiles, Polynuclear Aromatic Hydrocarbons, Metals, Wet Chemistry, Gasoline Range Organics, Methane, Polychlorinated Dioxins/Dibenzofurans |
| 580-118118-1 | |

The data validation was performed under Stage 2B & 4 guidelines. The analysis was validated using the following documents, as applicable to each method:

- Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021)
- U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019)
- DoD General Validation Guidelines (November 2019)
- U.S. Department of Defense (DoD) Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS (May 2020)
- U.S. Department of Defense (DoD) Data Validation Guidelines Module 2: Data Validation Procedure for Metals by ICP-OES (May 2020)
- U.S. Department of Defense (DoD) Data Validation Guidelines Module 4: Data Validation Procedure for Organic Analysis by GC (March 2021)
- EPA SW 846, Third Edition, Test Methods for Evaluating Solid Waste, update 1, July 1992; update IIA, August 1993; update II, September 1994; update IIB, January 1995; update III, December 1996; update IIIA, April 1998; IIIB, November 2004; update IV, February 2007; update V, July 2014; update VI, July 2018

Please feel free to contact us if you have any questions.

Sincerely,

Stella Cuenco
scuenco@lab-data.com
Project Manager/Senior Chemist

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292 pages-ADV

Attachment 1

**90/10 2B/4 EDD**

# LDC# 55171 (AECOM - Honolulu, HI / Red Hill Oily Waste, CTO 18F0176)

| LDC | SDG# | DATE REC'D | (3) DATE DUE | VOA (8260D) | | SVOA (8270E) | | PAHs (8270E -SIM) | | (5) Metals (6010D) | | GRO (8260/ LUFT) | | Dioxins (8290A) | | Methane (175) | | Alk. (2320B) | | NO$_3$/ NO$_2$-N (353.2) | | DOC (9060A) | | TOC (9060A) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Matrix: Water/Soil | | | | W | S | W | S | W | S | W | S | W | S | W | S | W | S | W | S | W | S | W | S | W | S | W | S | W | S | W | S | W | S |
| A | 580-118109-1 | 10/11/22 | 11/01/22 | 3 | 0 | 2 | 0 | 2 | 0 | 1 | 0 | 3 | 0 | 2 | 0 | 2 | 0 | 1 | 0 | 1 | 0 | 1 | 0 | 1 | 0 | | | | | | | | |
| B | 580-118118-1 | 10/11/22 | 11/01/22 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 0 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | | | |
| B | 580-118118-1 | 10/11/22 | 11/01/22 | 1 | 0 | 1 | 0 | 1 | 0 | 1 | 0 | 1 | 0 | 1 | 0 | 1 | 0 | 1 | 0 | 1 | 0 | 1 | 0 | 1 | 0 | | | | | | | | |
| Total | TR/SC | | | 5 | 0 | 3 | 0 | 3 | 0 | 2 | 0 | 5 | 0 | 3 | 0 | 4 | 0 | 2 | 0 | 2 | 0 | 2 | 0 | 2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 33 |

Shaded cells indicate Level D validation (all other cells are Level C validation). These sample counts do not include MS/MSD, and DUPs

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**LDC Report#** 55171A1a_RV1

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** May 30, 2023

**Parameters:** Volatiles

**Validation Level:** Stage 2B

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118109-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU147 | 580-118109-1 | Water | 09/19/22 |
| HU148 | 580-118109-2 | Water | 09/19/22 |
| HU146 | 580-118109-3 | Water | 09/19/22 |

# Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS (May 2020). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Volatile Organic Compounds (VOCs) and Tentatively Identified Compounds (TICs) by Environmental Protection Agency (EPA) SW 846 Method 8260D

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

## II. GC/MS Instrument Performance Check

A bromofluorobenzene (BFB) tune was performed at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

For analytes where average relative response factors (RRFs) were utilized, the percent relative standard deviations (%RSD) were less than or equal to 15.0%.

In the case where the laboratory used a calibration curve to evaluate the analytes, all coefficients of determination ($r^2$) were greater than or equal to 0.990.

Average relative response factors (RRF) for all analytes were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0% for all analytes.

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all analytes with the following exceptions:

| Date | Analyte | %D | Associated Samples | Flag | A or P |
|---|---|---|---|---|---|
| 09/27/22 | Vinyl chloride | 21.5 | All samples in SDG 580-118109-1 | UJ (all non-detects) | A |

The percent differences (%D) of the ending continuing calibration verifications (CCVs) were less than or equal to 50.0% for all analytes.

All of the continuing calibration relative response factors (RRF) were within validation criteria.

## V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

| Blank ID | Analysis Date | Analyte | Concentration | Associated Samples |
|---|---|---|---|---|
| MB 580-405161 | 09/27/22 | Hexachlorobutadiene<br>1,3,5-Trichlorobenzene | 0.121 ug/L<br>0.0481 ug/L | All samples in SDG 580-118109-1 |

Sample concentrations were compared to concentrations detected in the laboratory blanks. The sample concentrations were either not detected or were significantly greater (>10X for common contaminants, >5X for other contaminants) than the concentrations found in the associated laboratory blanks.

## VI. Field Blanks

Sample HU146 was identified as a trip blank. No contaminants were found with the following exceptions:

| Blank ID | Collection Date | Analyte | Concentration | Associated Samples |
|---|---|---|---|---|
| HU146 | 09/19/22 | Acetone | 5.4 ug/L | HU147<br>HU148 |

Sample concentrations were compared to concentrations detected in the field blanks. The sample concentrations were either not detected or were significantly greater (>10X for common contaminants, >5X for other contaminants) than the concentrations found in the associated field blanks.

## VII. Surrogates

Surrogates were added to all samples as required by the method. All surrogate recoveries (%R) were within QC limits.

## VIII. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## IX. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

## X. Field Duplicates

Samples HU147 and HU148 were identified as field duplicates. No results were detected in any of the samples.

## XI. Internal Standards

All internal standard areas and retention times were within QC limits.

## XII. Target Analyte and Tentatively Identified Compound Quantitation

All target analyte and tentatively identified compound (TIC) quantitations met validation criteria with the following exceptions:

| Sample | Analyte | Flag | A or P |
|---|---|---|---|
| HU147<br>HU148<br>HU146 | All "unknown" laboratory calibrated analytes reported as TICs | NJ (all detects) | A |

| Sample | Analyte | Finding | Flag | A or P |
|---|---|---|---|---|
| HU147 | p-Isopropyltoluene<br>n-Butylbenzene<br>1,2,3-Trichlorobenzene | All laboratory calibrated analytes reported as tentatively identified compounds (TIC). | J (all detects)<br>J (all detects)<br>J (all detects) | A |
| HU148<br>HU146 | Isopropylbenzene<br>p-Isopropyltoluene<br>n-Butylbenzene<br>1,2,3-Trichlorobenzene | All laboratory calibrated analytes reported as tentatively identified compounds (TIC). | J (all detects)<br>J (all detects)<br>J (all detects)<br>J (all detects) | A |

Raw data were not reviewed for Stage 2B validation.

## XIII. Target Analyte Identification

Raw data were not reviewed for Stage 2B validation.

## XIV. System Performance

Raw data were not reviewed for Stage 2B validation.

## XV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

Due to continuing calibration %D, data were qualified as estimated in three samples.

Due to TICs, data were qualified as presumptive and estimated in three samples.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Volatiles - Data Qualification Summary - SDG 580-118109-1

| Sample | Analyte | Flag | A or P | Reason (Code) |
|---|---|---|---|---|
| HU147<br>HU148<br>HU146 | Vinyl chloride | UJ (all non-detects) | A | Continuing calibration (%D) (c) |
| HU147<br>HU148<br>HU146 | All "unknown" laboratory calibrated analytes reported as TICs | NJ (all detects) | A | Tentatively identified compound quantitation (v) |
| HU147 | p-Isopropyltoluene<br>n-Butylbenzene<br>1,2,3-Trichlorobenzene | J (all detects)<br>J (all detects)<br>J (all detects) | A | Tentatively identified compound quantitation (v) |
| HU148<br>HU146 | Isopropylbenzene<br>p-Isopropyltoluene<br>n-Butylbenzene<br>1,2,3-Trichlorobenzene | J (all detects)<br>J (all detects)<br>J (all detects)<br>J (all detects) | A | Tentatively identified compound quantitation (v) |

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Volatiles - Laboratory Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Volatiles - Field Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

LDC #: 55171A1a

SDG #: 580-118109-1

Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

## Stage 2B

Date: 10/27/22

Page: 1 of 1

Reviewer:

2nd Reviewer:

**METHOD:** GC/MS Volatiles (EPA SW-846 Method 8260D) + TICs

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A A | |
| II. | GC/MS Instrument performance check | A | |
| III. | Initial calibration/ICV | A A | % RSD ≤ 15, r² ICV ≤ 20 |
| IV. | Continuing calibration | SW | CCV ≤ 20/50 |
| V. | Laboratory Blanks | SW | |
| VI. | Field blanks | SW ~~ND~~ | TB = 3 |
| VII. | Surrogate spikes | A | |
| VIII. | Matrix spike/Matrix spike duplicates | N | cs |
| IX. | Laboratory control samples | A | LCS /D |
| X. | Field duplicates | ND | D = 1,2 |
| XI. | Internal standards | A | |
| XII. | Target analyte quantitation | SW | |
| XIII. | Target analyte identification | N | |
| XIV. | System performance | N | |
| XV. | Overall assessment of data | A | |

Note:

| | | | |
|---|---|---|---|
| A = Acceptable | ND = No compounds detected | D = Duplicate | SB=Source blank |
| N = Not provided/applicable | R = Rinsate | TB = Trip blank | OTHER: |
| SW = See worksheet | FB = Field blank | EB = Equipment blank | |

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 2 | HU147 1: MEK | 580-118109-1 | Water | 09/19/22 |
| 2 2 | HU148 1 | 580-118109-2 | Water | 09/19/22 |
| 3 2 | HU146 1 | 580-118109-3 | Water | 09/19/22 |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
| 7 | | | | |
| 8 | | | | |
| 9 | | | | |

Notes:

| | | | | | | |
|---|---|---|---|---|---|---|
| | MB 580-404907 | | | | | |
| | MB 580-405161 | | | | | |
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# TARGET COMPOUND WORKSHEET

**METHOD: VOA**

| | | | | |
|---|---|---|---|---|
| A. Chloromethane | AA. Tetrachloroethene | AAA. 1,3,5-Trimethylbenzene | AAAA. Ethyl tert-butyl ether | A1. 1,3-Butadiene |
| B. Bromomethane | BB. 1,1,2,2-Tetrachloroethane | BBB. 4-Chlorotoluene | BBBB. tert-Amyl methyl ether | B1. Hexane |
| C. Vinyl chloride | CC. Toluene | CCC. tert-Butylbenzene | CCCC. 1-Chlorohexane | C1. Heptane |
| D. Chloroethane | DD. Chlorobenzene | DDD. 1,2,4-Trimethylbenzene | DDDD. Isopropyl alcohol | D1. Propylene |
| E. Methylene chloride | EE. Ethylbenzene | EEE. sec-Butylbenzene | EEEE. Acetonitrile | E1. Freon 11 |
| F. Acetone | FF. Styrene | FFF. 1,3-Dichlorobenzene | FFFF. Acrolein | F1. Freon 12 |
| G. Carbon disulfide | GG. Xylenes, total | GGG. p-Isopropyltoluene | GGGG. Acrylonitrile | G1. Freon 113 |
| H. 1,1-Dichloroethene | HH. Vinyl acetate | HHH. 1,4-Dichlorobenzene | HHHH. 1,4-Dioxane | H1. Freon 114 |
| I. 1,1-Dichloroethane | II. 2-Chloroethylvinyl ether | III. n-Butylbenzene | IIII. Isobutyl alcohol | I1. 2-Nitropropane |
| J. 1,2-Dichloroethene, total | JJ. Dichlorodifluoromethane | JJJ. 1,2-Dichlorobenzene | JJJJ. Methacrylonitrile | J1. Dimethyl disulfide |
| K. Chloroform | KK. Trichlorofluoromethane | KKK. 1,2,4-Trichlorobenzene | KKKK. Propionitrile | K1. 2,3-Dimethyl pentane |
| L. 1,2-Dichloroethane | LL. Methyl-tert-butyl ether | LLL. Hexachlorobutadiene | LLLL. Ethyl ether | L1. 2,4-Dimethyl pentane |
| M. 2-Butanone | MM. 1,2-Dibromo-3-chloropropane | MMM. Naphthalene | MMMM. Benzyl chloride | M1. 3,3-Dimethyl pentane |
| N. 1,1,1-Trichloroethane | NN. Methyl ethyl ketone | NNN. 1,2,3-Trichlorobenzene | NNNN. Iodomethane | N1. 2-Methylpentane |
| O. Carbon tetrachloride | OO. 2,2-Dichloropropane | OOO. 1,3,5-Trichlorobenzene | OOOO.1,1-Difluoroethane | O1. 3-Methylpentane |
| P. Bromodichloromethane | PP. Bromochloromethane | PPP. trans-1,2-Dichloroethene | PPPP. Tetrahydrofuran | P1. 3-Ethylpentane |
| Q. 1,2-Dichloropropane | QQ. 1,1-Dichloropropene | QQQ. cis-1,2-Dichloroethene | QQQQ. Methyl acetate | Q1. 2,2-Dimethylpentane |
| R. cis-1,3-Dichloropropene | RR. Dibromomethane | RRR. m,p-Xylenes | RRRR. Ethyl acetate | R1. 2,2,3- Trimethylbutane |
| S. Trichloroethene | SS. 1,3-Dichloropropane | SSS. o-Xylene | SSSS. Cyclohexane | S1. 2,2,4-Trimethylpentane |
| T. Dibromochloromethane | TT. 1,2-Dibromoethane | TTT. 1,1,2-Trichloro-1,2,2-trifluoroethane | TTTT. Methylcyclohexane | T1. 2-Methylhexane |
| U. 1,1,2-Trichloroethane | UU. 1,1,1,2-Tetrachloroethane | UUU. 1,2-Dichlorotetrafluoroethane | UUUU. Allyl chloride | U1. Nonanal |
| V. Benzene | VV. Isopropylbenzene | VVV. 4-Ethyltoluene | VVVV. Methyl methacrylate | V1. 2-Methylnaphthalene |
| W. trans-1,3-Dichloropropene | WW. Bromobenzene | WWW. Ethanol | WWWW. Ethyl methacrylate | W1. Methanol |
| X. Bromoform | XX. 1,2,3-Trichloropropane | XXX. Di-isopropyl ether | XXXX. cis-1,4-Dichloro-2-butene | X1. 1,2,3-Trimethylbenzene |
| Y. 4-Methyl-2-pentanone | YY. n-Propylbenzene | YYY. tert-Butanol | YYYY. trans-1,4-Dichloro-2-butene | Y1. 2-Propanol |
| Z. 2-Hexanone | ZZ. 2-Chlorotoluene | ZZZ. tert-Butyl alcohol | ZZZZ. Pentachloroethane | Z1. Ethylene Dibromide |

LDC #: 55171A1a

# VALIDATION FINDINGS WORKSHEET

## Continuing Calibration

Page: 1 of ___
Reviewer: FT

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260 D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?
[✓ Y] N N/A Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ?
Y [✓ N] N/A Were all %D and RRFs within the validation criteria of ≤20 %D and ≥0.05 RRF ? (C)

| # | Date | Standard ID | Compound | Finding %D (Limit: <20.0%) | Finding RRF (Limit: >0.05) | Associated Samples | Qualifications |
|---|---|---|---|---|---|---|---|
| | 9/27/22 | CCV 580-405161 | C | 21.5 | | 1→3, | J+/UJ/A ND |
| | 1234 | | | | | MB 580-405161 | |

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LDC #: 55171A1a

# VALIDATION FINDINGS WORKSHEET
## Blanks

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260 B)
Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".
[✓ Y] N N/A Was a method blank associated with every sample in this SDG?
[✓ Y] N N/A Was a method blank analyzed at least once every 12 hours for each matrix and concentration?
[✓ Y] N N/A Was there contamination in the method blanks? If yes, please see the qualifications below.
**Blank analysis date:** 9/27/22
**Conc. units:** ug/L
Associated Samples: 1→3 (ND)

| Compound | Blank ID | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| ▨ | MB 580 | -405161 | | | | | | | | |
| LLL | 0.121 | | | | | | | | | |
| TIC 1,3,5-Trichlorobenzene | 0.0481 | | | | | | | | | |
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**Blank analysis date:**_______
**Conc. units:**_______
Associated Samples:_______

| Compound | Blank ID | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
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All results were qualified using the criteria stated below except those circled.

Note: Common contaminants such as Methylene chloride, Acetone, 2-Butanone, Carbon disulfide and TICs that were detected in samples within ten times the associated method blank concentration were qualified as not detected, "U". Other contaminants within five times the method blank concentration were also qualified as not detected, "U".

LDC #: 55171 A1a

# VALIDATION FINDINGS WORKSHEET

## Field Blanks

Page: 1 of __
Reviewer: FT

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260D)

[✓ Y] N N/A Were field blanks identified in this SDG?

[✓ Y] N N/A Were target compounds detected in the field blanks?

**Blank units:** ug/L **Associated sample units:** ug/L

**Sampling date:** 9/19/22

**Field blank type:** (circle one) Field Blank / Rinsate / Trip Blank / Other: TB Associated Samples: 1, 2 (ND)

| Compound | Blank ID | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 3 | | | | | | | | | |
| F | 5.4 | | | | | | | | | |
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**Blank units:** ____ **Associated sample units:** ____

**Sampling date:** ____

**Field blank type:** (circle one) Field Blank / Rinsate / Trip Blank / Other: ____ Associated Samples: ____

| Compound | Blank ID | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
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CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
Common contaminants such as Methylene chloride, Acetone, 2-Butanone and Carbon disulfide that were detected in samples within ten times the associated field blank concentration were qualified as not detected, "U". Other contaminants within five times the field blank concentration were also qualified as not detected, "U".

LDC #: 55171A1a

# VALIDATION FINDINGS WORKSHEET
## Target Analyte Quantitation

Page: 1 of 1
Reviewer: FT

**METHOD: GCMS VOA 8260** D

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

**Level IV/D Only**

Y N [✓ N/A] Were CRQLs adjusted for sample dilutions, dry weight factors, etc.?

Y N [✓ N/A] Did the reported results for detected target compounds agree within 10.0% of the recalculated results?

(√ )

| # | Associated Samples | Compound Name | Findings | Qualifications |
|---|---|---|---|---|
|  | 1, 2, 3 |  | All unknown reported | NJ /Δ |
|  |  |  | as TIC |  |
|  |  |  |  |  |
|  | 1 | GGG, III, NNN | all calibrated analytes | Jdet /Δ |
|  |  |  | reported as TIC |  |
|  |  |  |  |  |
|  | 2, 3 | VV, GGG, III, NNN | ↓ | Jdet /Δ |
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Comments: See sample calculation verification worksheet for recalculations

**LDC Report#** 55171A2a_RV1

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** June 26, 2023

**Parameters:** Semivolatiles

**Validation Level:** Stage 2B

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118109-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU147 | 580-118109-1 | Water | 09/19/22 |
| HU148 | 580-118109-2 | Water | 09/19/22 |

## Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS (May 2020). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Semivolatile Organic Compounds (SVOCs) and Tentatively Identified Compounds (TICs) by Environmental Protection Agency (EPA) SW 846 Method 8270E

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

## II. GC/MS Instrument Performance Check

A decafluorotriphenylphosphine (DFTPP) tune was performed at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

For analytes where average relative response factors (RRFs) were utilized, the percent relative standard deviations (%RSD) were less than or equal to 15.0%.

In the case where the laboratory used a calibration curve to evaluate the analytes, all coefficients of determination ($r^2$) were greater than or equal to 0.990.

Average relative response factors (RRF) for all analytes were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0% for all analytes with the following exceptions:

| Date | Analyte | %D | Associated Samples | Flag | A or P |
|---|---|---|---|---|---|
| 08/29/22 | Phenol | 23 | All samples in SDG 580-118109-1 | UJ (all non-detects) | A |

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all analytes with the following exceptions:

| Date | Analyte | %D | Associated Samples | Flag | A or P |
|---|---|---|---|---|---|
| 09/24/22 | 2,3,4,6-Tetrachlorophenol<br>2,6-Dinitrotoluene<br>3,3'-Dichlorobenzidine<br>Diethylphthalate<br>Dimethylphthalate<br>Hexachlorobutadiene | 34.0<br>23.0<br>23.0<br>31.1<br>30.6<br>21.4 | All samples in SDG 580-118109-1 | UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects) | A |

The percent differences (%D) of the ending continuing calibration verifications (CCVs) were less than or equal to 50.0% for all analytes.

All of the continuing calibration relative response factors (RRF) were within validation criteria.

## V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

## VI. Field Blanks

No field blanks were identified in this SDG.

## VII. Surrogates

Surrogates were added to all samples as required by the method. All surrogate recoveries (%R) were within QC limits.

## VIII. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## IX. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits.

Relative percent differences (RPD) were within QC limits with the following exceptions:

| LCS ID (Associated Samples) | Analyte | RPD (Limits) | Flag | A or P |
|---|---|---|---|---|
| LCS/LCSD 580-40480 (All samples in SDG 580-118109-1) | Hexachlorobutadiene | 23 (≤20) | NA | - |

## X. Field Duplicates

No field duplicates were identified in this SDG.

## XI. Internal Standards

All internal standard areas and retention times were within QC limits.

## XII. Target Analyte and Tentatively Identified Compounds Quantitation

All tentatively identified compound quantitations met validation criteria with the following exceptions:

| Sample | Analyte | Flag | A or P |
|---|---|---|---|
| All samples in SDG 580-118109-1 | All tentatively identified compounds (TIC) | NJ (all detects) | A |

Raw data were not reviewed for Stage 2B validation.

## XIII. Target Analyte Identification

Raw data were not reviewed for Stage 2B validation.

## XIV. System Performance

Raw data were not reviewed for Stage 2B validation.

## XV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

Due to ICV %D and continuing calibration %D, data were qualified as estimated in two samples.

Due to TICs, data were qualified as presumptive and estimated in two samples.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Semivolatiles - Data Qualification Summary - SDG 580-118109-1

| Sample | Analyte | Flag | A or P | Reason (Code) |
|---|---|---|---|---|
| HU147<br>HU148 | Phenol | UJ (all non-detects) | A | Initial calibration verification (%D) (c) |
| HU147<br>HU148 | 2,3,4,6-Tetrachlorophenol<br>2,6-Dinitrotoluene<br>3,3'-Dichlorobenzidine<br>Diethylphthalate<br>Dimethylphthalate<br>Hexachlorobutadiene | UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects) | A | Continuing calibration (%D) (c) |
| HU147<br>HU148 | All tentatively identified compounds (TIC) | NJ (all detects) | A | Tentatively identified compound quantitation (v) |

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Semivolatiles - Field Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

LDC #: 55171A2a

SDG #: 580-118109-1

Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

## Stage 2B

Date: 10/28/22

Page: 1 of 1

Reviewer:

2nd Reviewer:

**METHOD:** GC/MS Semivolatiles (EPA SW-846 Method 8270E)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A / A | |
| II. | GC/MS Instrument performance check | A | |
| III. | Initial calibration/ICV | A SW | %RSD ≤ 15, r² ICV ≤ 20 |
| IV. | Continuing calibration / ending | SW | CCV ≤ 20/50 |
| V. | Laboratory Blanks | A | |
| VI. | Field blanks | N | |
| VII. | Surrogate spikes | A | |
| VIII. | Matrix spike/Matrix spike duplicates | N | CS |
| IX. | Laboratory control samples | SW | LCS ID |
| X. | Field duplicates | ND | D = 1,2 |
| XI. | Internal standards | A | |
| XII. | Target analyte quantitation | SW | |
| XIII. | Target analyte identification | N | |
| XIV. | System performance | N | |
| XV. | Overall assessment of data | A | |

| Note: | | | | |
|---|---|---|---|---|
| | A = Acceptable | ND = No compounds detected | D = Duplicate | SB=Source blank |
| | N = Not provided/applicable | R = Rinsate | TB = Trip blank | OTHER: |
| | SW = See worksheet | FB = Field blank | EB = Equipment blank | |

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU147 | 580-118109-1 | Water | 09/19/22 |
| 2 | HU148 | 580-118109-2 | Water | 09/19/22 |
| 3 | | | | |
| 4 | | | | |
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Notes:

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|---|---|---|---|---|---|---|---|
| | MB 580-404801 | | | | | | |
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# VALIDATION FINDINGS WORKSHEET

**METHOD:** GC/MS SVOA

| | | | | |
|---|---|---|---|---|
| A. Phenol | CC. Dimethylphthalate | EEE. Bis(2-ethylhexyl)phthalate | GGGG. C30-Hopane | I1. Methyl methanesulfonate |
| B. Bis (2-chloroethyl) ether | DD. Acenaphthylene | FFF. Di-n-octylphthalate | HHHH. 1-Methylphenanthrene | J1. Ethyl methanesulfonate |
| C. 2-Chlorophenol | EE. 2,6-Dinitrotoluene | GGG. Benzo(b)fluoranthene | IIII. 1,4-Dioxane | K1. o,o',o''-Triethylphosphorothioate |
| D. 1,3-Dichlorobenzene | FF. 3-Nitroaniline | HHH. Benzo(k)fluoranthene | JJJJ. Acetophenone | L1. n-Phenylene diamine |
| E. 1,4-Dichlorobenzene | GG. Acenaphthene | III. Benzo(a)pyrene | KKKK. Atrazine | M1. 1,4-Naphthoquinone |
| F. 1,2-Dichlorobenzene | HH. 2,4-Dinitrophenol | JJJ. Indeno(1,2,3-cd)pyrene | LLLL. Benzaldehyde | N1. N-Nitro-o-toluidine |
| G. 2-Methylphenol | II. 4-Nitrophenol | KKK. Dibenz(a,h)anthracene | MMMM. Caprolactam | O1. 1,3,5-Trinitrobenzene |
| H. 2,2'-Oxybis(1-chloropropane) | JJ. Dibenzofuran | LLL. Benzo(g,h,i)perylene | NNNN. 2,6-Dichlorophenol | P1. Pentachlorobenzene |
| I. 4-Methylphenol | KK. 2,4-Dinitrotoluene | MMM. Bis(2-Chloroisopropyl)ether | OOOO. 1,2-Diphenylhydrazine | Q1. 4-Aminobiphenyl |
| J. N-Nitroso-di-n-propylamine | LL. Diethylphthalate | NNN. Aniline | PPPP. 3-Methylphenol | R1. 2-Naphthylamine |
| K. Hexachloroethane | MM. 4-Chlorophenyl-phenyl ether | OOO. N-Nitrosodimethylamine | QQQQ. 3&4-Methylphenol | S1. Triphenylene |
| L. Nitrobenzene | NN. Fluorene | PPP. Benzoic Acid | RRRR. 4-Dimethyldibenzothiophene (4MDT) | T1. Octachlorostyrene |
| M. Isophorone | OO. 4-Nitroaniline | QQQ. Benzyl alcohol | SSSS. 2/3-Dimethyldibenzothiophene (4MDT) | U1. Famphur |
| N. 2-Nitrophenol | PP. 4,6-Dinitro-2-methylphenol | RRR. Pyridine | TTTT. 1-Methyldibenzothiophene (1MDT) | V1. 1,4-phenylenediamine |
| O. 2,4-Dimethylphenol | QQ. N-Nitrosodiphenylamine | SSS. Benzidine | UUUU.. 2,3,4,6-Tetrachlorophenol | W1. Methapyrilene |
| P. Bis(2-chloroethoxy)methane | RR. 4-Bromophenyl-phenylether | TTT. 1-Methylnaphthalene | VVVV. 1,2,4,5-Tetrachlorobenzene | X1. Pentachloroethane |
| Q. 2,4-Dichlorophenol | SS. Hexachlorobenzene | UUU.Benzo(b)thiophene | WWWW.. 2-Picoline | Y1. 3,3'-Dimethylbenzidine |
| R. 1,2,4-Trichlorobenzene | TT. Pentachlorophenol | VVV.Benzonaphthothiophene | XXXX. 3-Methylcholanthrene | Z1. o-Toluidine |
| S. Naphthalene | UU. Phenanthrene | WWW.Benzo(e)pyrene | YYYY. a,a-Dimethylphenethylamine | A2. 1-Naphthylamine |
| T. 4-Chloroaniline | VV. Anthracene | XXX. 2,6-Dimethylnaphthalene | ZZZZ. Hexachloropropene | B2. 4-Aminobiphenyl |
| U. Hexachlorobutadiene | WW. Carbazole | YYY. 2,3,5-Trimethylnaphthalene | A1. N-Nitrosodiethylamine | C2. 4-Nitroquinoline-1-oxide |
| V. 4-Chloro-3-methylphenol | XX. Di-n-butylphthalate | ZZZ. Perylene | B1. N-Nitrosodi-n-butylamine | D2. Hexachloropene |
| W. 2-Methylnaphthalene | YY. Fluoranthene | AAAA. Dibenzothiophene | C1. N-Nitrosomethylethylamine | E2. Bis (2-chloro-1-methylethyl) ether |
| X. Hexachlorocyclopentadiene | ZZ. Pyrene | BBBB. Benzo(a)fluoranthene | D1. N-Nitrosomorpholine | F2. Bifenthrin |
| Y. 2,4,6-Trichlorophenol | AAA. Butylbenzylphthalate | CCCC. Benzo(b)fluorene | E1. N-Nitrosopyrrolidine | G2. Cyfluthrin |
| Z. 2,4,5-Trichlorophenol | BBB. 3,3'-Dichlorobenzidine | DDDD. cis/trans-Decalin | F1. Phenacetin | H2. Cypermethrin |
| AA. 2-Chloronaphthalene | CCC. Benzo(a)anthracene | EEEE. 1,1'-Biphenyl | G1. 2-Acetylaminofluorene | I2. Permethrin (cis/trans) |
| BB. 2-Nitroaniline | DDD. Chrysene | FFFF. Retene | H1. Pronamide | J2. 5-Nitro-o-toluidine |

LDC #:________

# VALIDATION FINDINGS WORKSHEET
## Initial Calibration Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS SVOA (EPA Method 8270 )

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Was an initial calibration verification standard analyzed after each ICAL for each instrument?

Y N N/A Were all %D within the validation criteria of ≤20 / 30 %D ? (c)

| # | Date | Standard ID | Compound | Finding %D (Limit: ≤ 20 / 30.0%) | Associated Samples | Qualifications |
|---|---|---|---|---|---|---|
| | 8/29/22<br>2133 | ICV 580-402255 | A | 23 | all | J+/UJ/A ND |
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LDC #: 55171A2a

## VALIDATION FINDINGS WORKSHEET

## Continuing Calibration

Page: 1 of 7
Reviewer: FT

**METHOD:** GC/MS BNA (EPA SW 846 Method 8270 E )

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Was a continuing calibration standard analyzed at least once every 12 hours of sample analysis for each instrument?

[✓ Y] N N/A Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ?

Y [✓ N] N/A Were all %D and RRFs within the validation criteria of ≤20%D and ≥0.05 RRF ? (c)

| # | Date | Standard ID | Compound | Finding %D (Limit: ≤20.0%) | Finding RRF (Limit: ≥0.05) | Associated Samples | Qualifications |
|---|---|---|---|---|---|---|---|
| | 9/24/22 | CCV - | UUUU | | 34.0 | All | J+/UJ/A all M |
| | 1509 | 580-404926 | EE | | 23.0 | ↓ | ↓ |
| | | | BBB | | 23.0 | | |
| | | | LL | | 31.1 | | |
| | | | CC | | 30.6 | | |
| | | | U | | 21.4 | | J+/UJ/A |
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LDC #: 55171A2a

# VALIDATION FINDINGS WORKSHEET
## Laboratory Control Samples (LCS)

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS BNA (Method 8270E

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Was a LCS required?

Y [✓ N] N/A Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

(W)

| # | LCS/LCSD ID | Compound | LCS %R (Limits) | LCSD %R (Limits) | RPD (Limits) | Associated Samples | Qualifications |
|---|---|---|---|---|---|---|---|
| | LCSID | U | ( ) | ( ) | 23 (20) | All | Jdets/P ND |
| | 580-404810 | | ( ) | ( ) | ( ) | | |
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LDC #: 55171A2a

# VALIDATION FINDINGS WORKSHEET
## Target Analyte Quantitation

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/GCMS EPA SW 8270 E

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N [✓ N/A] Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?

Y N [✓ N/A] Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?

| # | Date | Sample ID | Compound | Lab RL is higher than QAPP RL | Qualifications |
|---|---|---|---|---|---|
| | | All | all analytes reported as Tentatively Identified Compound (TIC) | | NJ/A |
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Comments: See sample calculation verification worksheet for recalculations

**LDC Report#** 55171A2b

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** November 2, 2022

**Parameters:** Polynuclear Aromatic Hydrocarbons

**Validation Level:** Stage 2B

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118109-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU147 | 580-118109-1 | Water | 09/19/22 |
| HU148 | 580-118109-2 | Water | 09/19/22 |

## Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS (May 2020). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polynuclear Aromatic Hydrocarbons (PAHs) by Environmental Protection Agency (EPA) SW 846 Method 8270E in Selected Ion Monitoring (SIM) mode

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

## II. GC/MS Instrument Performance Check

Instrument performance check was performed at the required frequency.

All ion abundance requirements were met.

## III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

For analytes where average relative response factors (RRFs) were utilized, percent relative standard deviations (%RSD) were less than or equal to 15.0%.

In the case where the laboratory used a calibration curve to evaluate the analytes, all coefficients of determination ($r^2$) were greater than or equal to 0.990.

Average relative response factors (RRF) for all analytes were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0% for all analytes.

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all analytes.

The percent differences (%D) of the ending continuing calibration verifications (CCVs) were less than or equal to 50.0% for all analytes.

All of the continuing calibration relative response factors (RRF) were within validation criteria.

## V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

## VI. Field Blanks

No field blanks were identified in this SDG.

## VII. Surrogates

Surrogates were added to all samples as required by the method. All surrogate recoveries (%R) were within QC limits.

## VIII. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## IX. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

## X. Field Duplicates

Samples HU147 and HU148 were identified as field duplicates. No results were detected in any of the samples.

## XI. Internal Standards

All internal standard areas and retention times were within QC limits.

## XII. Target Analyte Quantitation

Raw data were not reviewed for Stage 2B validation.

## XIII. Target Analyte Identification

Raw data were not reviewed for Stage 2B validation.

## XIV. System Performance

Raw data were not reviewed for Stage 2B validation.

## XV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polynuclear Aromatic Hydrocarbons - Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polynuclear Aromatic Hydrocarbons - Laboratory Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polynuclear Aromatic Hydrocarbons - Field Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

LDC #: 55171A2b

SDG #: 580-118109-1

Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

## Stage 2B

Date: 10/27/22

Page: 1 of 1

Reviewer:

2nd Reviewer:

**METHOD:** GC/MS Polynuclear Aromatic Hydrocarbons (EPA SW-846 Method 8270E-SIM)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A / A | |
| II. | GC/MS Instrument performance check | A | |
| III. | Initial calibration/ICV | A / A | % RSD ≤ 15, r² ICV ≤ 20 |
| IV. | Continuing calibration | A | CCV ≤ 20/50 |
| V. | Laboratory Blanks | A | |
| VI. | Field blanks | N | |
| VII. | Surrogate spikes | A | |
| VIII. | Matrix spike/Matrix spike duplicates | N | cs |
| IX. | Laboratory control samples | A | LCS ID |
| X. | Field duplicates | ND | D = 1,2 |
| XI. | Internal standards | A | |
| XII. | Target analyte quantitation | N | |
| XIII. | Target analyte identification | N | |
| XIV. | System performance | N | |
| XV. | Overall assessment of data | A | |

Note:

| | | | |
|---|---|---|---|
| A = Acceptable | ND = No compounds detected | D = Duplicate | SB=Source blank |
| N = Not provided/applicable | R = Rinsate | TB = Trip blank | OTHER: |
| SW = See worksheet | FB = Field blank | EB = Equipment blank | |

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU147 D | 580-118109-1 | Water | 09/19/22 |
| 2 | HU148 D | 580-118109-2 | Water | 09/19/22 |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
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| 9 | | | | |

Notes:

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | MB: 580-404801 | | | | | | |
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**LDC Report#** 55171A4b

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** December 7, 2022

**Parameters:** Metals

**Validation Level:** Stage 2B

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118109-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU147 | 580-118109-1 | Water | 09/19/22 |

## Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 2: Data Validation Procedure for Metals by ICP-OES (May 2020). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Calcium, Magnesium, Manganese, Potassium, and Sodium by Environmental Protection Agency (EPA) SW 846 Method 6010D

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition.

All technical holding time requirements were met.

## II. Instrument Calibration

Initial and continuing calibrations were performed as required by the method.

The initial calibration verification (ICV) and continuing calibration verification (CCV) standards were within QC limits.

## III. ICP Interference Check Sample Analysis

The frequency of interference check sample (ICS) analysis was met. All criteria were within QC limits.

## IV. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

| Blank ID | Analyte | Maximum Concentration | Associated Samples |
|---|---|---|---|
| ICB/CCB | Calcium<br>Manganese<br>Potassium<br>Sodium | 0.0472 mg/L<br>0.0029 mg/L<br>0.2567 mg/L<br>0.114 mg/L | All samples in SDG 580-118109-1 |

Data qualification by the laboratory blanks was based on the maximum contaminant concentration in the laboratory blanks in the analysis of each analyte. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated laboratory blanks.

## V. Field Blanks

No field blanks were identified in this SDG.

## VI. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## VII. Duplicate Sample Analysis

The laboratory has indicated that there were no duplicate (DUP) analyses specified for the samples in this SDG, and therefore duplicate analyses were not performed for this SDG.

## VIII. Serial Dilution

Serial dilution was not performed for this SDG.

## IX. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

## X. Field Duplicates

No field duplicates were identified in this SDG.

## XI. Target Analyte Quantitation

Raw data were not reviewed for Stage 2B validation.

## XII. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Metals - Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Metals - Laboratory Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Metals - Field Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

LDC #: 55171A4b
SDG #: 580-118109-1
Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET
## Stage 2B

Date: 12/05/22
Page: 1 of 1
Reviewer: NC
2nd Reviewer:

**METHOD:** Metals (EPA SW-846 Method 6010D)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A/A | |
| II. | Instrument Calibration | A | |
| III. | ICP Interference Check Sample (ICS) Analysis | A | |
| IV. | Laboratory Blanks | SW | |
| V. | Field Blanks | N | |
| VI. | Matrix Spike/Matrix Spike Duplicates | N | |
| VII. | Duplicate sample analysis | N | |
| VIII. | Serial Dilution | N | |
| IX. | Laboratory control samples | A | LCS/LCSD |
| X. | Field Duplicates | N | |
| XI. | Target Analyte Quantitation | N | |
| XII. | Overall Assessment of Data | A | |

| Note: | | | | |
|---|---|---|---|---|
| | A = Acceptable | ND = No compounds detected | D = Duplicate | SB=Source blank |
| | N = Not provided/applicable | R = Rinsate | TB = Trip blank | OTHER: |
| | SW = See worksheet | FB = Field blank | EB = Equipment blank | |

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU147 | 580-118109-1 | Water | 09/19/22 |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
| 7 | | | | |
| 8 | | | | |
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| 10 | | | | |
| 11 | | | | |
| 12 | | | | |
| 13 | | | | |
| 14 | | | | |

LDC #: 55171A4b

VALIDATION FINDINGS WORKSHEET

Sample Specific Element Reference

Page 1 of 1

Reviewer:NC

All elements are applicable to each sample as noted below.

| Sample ID | Target Analyte List |
|---|---|
| 1 | Ca, Mg, Mn, K, Na |
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**Analysis Method**

| | |
|---|---|
| ICP | Ca, Mg, Mn, K, Na |

LDC #: 55171A4b

# VALIDATION FINDINGS WORKSHEET

## Laboratory Blank Contamination (PB/ICB/CCB)

Page 1 of 1
Reviewer:NC

METHOD: Trace Metals (EPA SW 846 Methods 6010/6020/7000)
Soil preparation factor applied (if applicable):

Sample Concentration, unless otherwise noted: ug/L

Associated Samples: 1

| | | | | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Analyte** | **PB (mg/L)** | **Maximum ICB/CCB (mg/L)** | **Action Level (ug/L)** | | | | | | | | | |
| Ca | | 0.0472 | 236 | | | | | | | | | |
| Mn | | 0.0029 | 14.5 | | | | | | | | | |
| K | | 0.2567 | 1283.5 | | | | | | | | | |
| Na | | 0.114 | 570 | | | | | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

Comments: The listed analyte concentrtaion is the highest ICB or CCB detected in the analysis. The action level, when applicable, is established at 5X the highest ICB, CCB, or PB concentration.

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** December 7, 2022

**Parameters:** Wet Chemistry

**Validation Level:** Stage 2B

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118109-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU147 | 580-118109-1 | Water | 09/19/22 |
| HU147DUP | 580-118109-1DUP | Water | 09/19/22 |

# Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), and the DoD General Validation Guidelines (November 2019). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following methods:

Alkalinity by Standard Method 2320B
Dissolved Organic Carbon by Environmental Protection Agency (EPA) SW 846 Method 9060A
Nitrate/Nitrite as Nitrogen by EPA Method 353.2
Total Organic Carbon by EPA SW 846 Method 9060A

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition.

All technical holding time requirements were met.

## II. Initial Calibration

All criteria for the initial calibration of each method were met.

## III. Continuing Calibration

Continuing calibration frequency and analysis criteria were met for each method when applicable.

## IV. Laboratory Blanks

Laboratory blanks were analyzed as required by the methods. No contaminants were found in the laboratory blanks.

## V. Field Blanks

No field blanks were identified in this SDG.

## VI. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## VII. Duplicate Sample Analysis

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits.

## VIII. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the methods. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

## IX. Field Duplicates

No field duplicates were identified in this SDG.

## X. Target Analyte Quantitation

Raw data were not reviewed for Stage 2B validation.

## XI. Overall Assessment of Data

The analysis was conducted within all specifications of the methods. No results were rejected or recommended for exclusion in this SDG.

## Red Hill Bulk Storage Facility, CTO 18F0126
## Wet Chemistry - Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

## Red Hill Bulk Storage Facility, CTO 18F0126
## Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

## Red Hill Bulk Storage Facility, CTO 18F0126
## Wet Chemistry - Field Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

LDC #: 55171A6

SDG #: 580-118109-1

Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

## Stage 2B

Date: 12/05/22

Page: 1 of 1

Reviewer: NC

2nd Reviewer:

**METHOD: (Analyte)** Alkalinity (SM2320B), DOC (EPA SW-846 Method 9060A), Nitrate/Nitrite-N (EPA Method 353.2), TOC (EPA SW-846 Method 9060A)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A/A | |
| II | Initial calibration | A | |
| III. | Calibration verification | A | |
| IV | Laboratory Blanks | A | |
| V | Field blanks | N | |
| VI. | Matrix Spike/Matrix Spike Duplicates | N | |
| VII. | Duplicate sample analysis | A | |
| VIII. | Laboratory control samples | A | LCS/LCSD |
| IX. | Field duplicates | N | |
| X. | Target Analyte Quantitation | N | |
| XI. | Overall assessment of data | A | |

Note:
A = Acceptable
N = Not provided/applicable
SW = See worksheet
ND = No compounds detected
R = Rinsate
FB = Field blank
D = Duplicate
TB = Trip blank
EB = Equipment blank
SB=Source blank
OTHER:

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU147 | 580-118109-1 | Water | 09/19/22 |
| 2 | HU147DUP | 580-118109-1DUP | Water | 09/19/22 |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
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LDC #: 55171A6

VALIDATION FINDINGS WORKSHEET

Sample Specific Element Reference

Page 1 of 1

Reviewer: NC

All elements are applicable to each sample as noted below.

| **Sample ID** | **Target Analyte List** |
|---|---|
| 1 | Alkalinity, Bicarbonate Alkalinity as CaCO3, Carbonate Alkalinity as CaCO3, DOC, TOC, Nitrate/Nitrite-N |
| | |
| | |
| | |
| QC | |
| 2 | Alkalinity, Bicarbonate Alkalinity as CaCO3, Carbonate Alkalinity as CaCO3 |
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**LDC Report#** 55171A7

# Laboratory Data Consultants, Inc.
# Data Validation Report

| | |
|---|---|
| **Project/Site Name:** | Red Hill Oily Waste Disposal Facility, CTO 18F0176 |
| **LDC Report Date:** | November 2, 2022 |
| **Parameters:** | Gasoline Range Organics |
| **Validation Level:** | Stage 2B |
| **Laboratory:** | Eurofins, Tacoma, WA |
| **Sample Delivery Group (SDG):** | 580-118109-1 |

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU147 | 580-118109-1 | Water | 09/19/22 |
| HU148 | 580-118109-2 | Water | 09/19/22 |
| HU146 | 580-118109-3 | Water | 09/19/22 |

## Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS (May 2020). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following methods:

Gasoline Range Organics by Environmental Protection Agency (EPA) SW 846 Method 8260 and CADOHS LUFT Method

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

## II. GC/MS Instrument Performance Check

A bromofluorobenzene (BFB) tune was performed at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the methods.

A curve fit, based on the initial calibration, was established for quantitation. The coefficient of determination ($r^2$) was greater than or equal to 0.990.

Average relative response factors (RRF) were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0%.

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0%.

The percent differences (%D) of the ending continuing calibration verifications (CCVs) were less than or equal to 20.0% with the following exceptions:

| Date | Analyte | %D | Associated Samples | Flag | A or P |
|---|---|---|---|---|---|
| 09/27/22 | Gasoline range organics (C6-C12) | 26.8 | All samples in SDG 580-118109-1 | UJ (all non-detects) | A |

All of the continuing calibration relative response factors (RRF) were within validation criteria.

## V. Laboratory Blanks

Laboratory blanks were analyzed as required by the methods. No contaminants were found in the laboratory blanks.

## VI. Field Blanks

Sample HU146 was identified as a trip blank. No contaminants were found.

## VII. Surrogates

Surrogates were added to all samples as required by the methods. All surrogate recoveries (%R) were within QC limits.

## VIII. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## IX. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

## X. Field Duplicates

Samples HU147 and HU148 were identified as field duplicates. No results were detected in any of the samples.

## XI. Internal Standards

All internal standard areas and retention times were within QC limits.

## XII. Target Analyte Quantitation

Raw data were not reviewed for Stage 2B validation.

## XIII. Target Analyte Identification

Raw data were not reviewed for Stage 2B validation.

## XIV. System Performance

Raw data were not reviewed for Stage 2B validation.

## XV. Overall Assessment of Data

The analysis was conducted within all specifications of the methods. No results were rejected or recommended for exclusion in this SDG.

Due to ending CCV %D, data were qualified as estimated in three samples.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Gasoline Range Organics - Data Qualification Summary - SDG 580-118109-1

| Sample | Analyte | Flag | A or P | Reason (Code) |
|---|---|---|---|---|
| HU147<br>HU148<br>HU146 | Gasoline range organics (C6-C12) | UJ (all non-detects) | A | Continuing calibration (ending CCV %D) (c) |

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Gasoline Range Organics - Laboratory Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Gasoline Range Organics - Field Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

LDC #: 55171A7
SDG #: 580-118109-1
Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

## Stage 2B

Date: 10/27/22
Page: 1 of 1
Reviewer:
2nd Reviewer:

**METHOD:** GC/MS Gasoline Range Organics (EPA SW-846 Method 8260/CADOHS LUFT Method)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A/A | |
| II. | GC/MS Instrument performance check | A | |
| III. | Initial calibration/ICV | A/A | r2 ICV ≤ 20 |
| IV. | Continuing calibration | SW | CCV ≤ 20\|20 |
| V. | Laboratory Blanks | A | |
| VI. | Field blanks | ND | TB = 3 |
| VII. | Surrogate spikes | A | |
| VIII. | Matrix spike/Matrix spike duplicates | N | CS |
| IX. | Laboratory control samples | A | LCS ID |
| X. | Field duplicates | ND | D = 1,2 |
| XI. | Internal standards | A | |
| XII. | Target analyte quantitation | N | |
| XIII. | Target analyte identification | N | |
| XIV. | System performance | N | |
| XV. | Overall assessment of data | A | |

| Note: | | | | |
|---|---|---|---|---|
| | A = Acceptable | ND = No compounds detected | D = Duplicate | SB=Source blank |
| | N = Not provided/applicable | R = Rinsate | TB = Trip blank | OTHER: |
| | SW = See worksheet | FB = Field blank | EB = Equipment blank | |

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU147 D | 580-118109-1 | Water | 09/19/22 |
| 2 | HU148 D | 580-118109-2 | Water | 09/19/22 |
| 3 | HU146 TB | 580-118109-3 | Water | 09/19/22 |
| 4 | | | | |
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Notes:

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| | MB 580-405043 | | | | | |
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LDC #: 55171A7

# VALIDATION FINDINGS WORKSHEET

## Continuing Calibration

Page: 1 of 1

Reviewer: FT

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260 / CA DOHS LUFT

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?

[✓ Y] N N/A Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ?

Y [✓ N] N/A Were all %D and RRFs within the validation criteria of ≤20 %D and ≥0.05 RRF ?

(c)

| # | Date | Standard ID | Compound | Finding %D (Limit: ≤20.0%) | Finding RRF (Limit: ≥0.05) | Associated Samples | Qualifications |
|---|---|---|---|---|---|---|---|
| | 9/26/22 | CCV 580-405043 | * | 50.7 | | ~~All~~ | J+/UJ/A ND |
| | 1611 | | | | | MB 580-405043 | |
| | | | | | | | |
| | 9/27/22 | CCV 580-405043 | * | 26.8 | | All -MB | J+/UJ/A ND |
| | 0041 | closing | | | | | |
| | | | * Gasoline Range Organics (C6-C12) | | | | |
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**LDC Report#** 55171A51

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** November 2, 2022

**Parameters:** Methane

**Validation Level:** Stage 2B

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118109-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU147 | 580-118109-1 | Water | 09/19/22 |
| HU146 | 580-118109-3 | Water | 09/19/22 |

## Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 4: Data Validation Procedure for Organic Analysis by GC (March 2021). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Methane by Method RSK-175

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

## II. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0%.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0%.

## III. Continuing Calibration

Continuing calibration was performed at required frequencies.

The percent differences (%D) were less than or equal to 20.0%.

The percent differences (%D) of the ending continuing calibration verifications (CCVs) were less than or equal to 20.0%.

## IV. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

## V. Field Blanks

Sample HU146 was identified as a trip blank. No contaminants were found.

## VI. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## VII. Laboratory Control Samples

Laboratory control samples (LCS) were analyzed as required by the method. Percent recoveries (%R) were within QC limits.

## VIII. Field Duplicates

No field duplicates were identified in this SDG.

## IX. Target Analyte Quantitation

Raw data were not reviewed for Stage 2B validation.

## X. Target Analyte Identification

Raw data were not reviewed for Stage 2B validation.

## XI. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Methane - Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Methane - Laboratory Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Methane - Field Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

LDC #: 55171A51
SDG #: 580-118109-1
Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

Stage 2B

Date: 10/27/22
Page: 1 of 1
Reviewer:
2nd Reviewer:

**METHOD:** GC Methane (Method RSK-175)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A / A | |
| II. | Initial calibration/ICV | A / A | %RSD ≤ 20 ICV ≤ 20 |
| III. | Continuing calibration | A | CCV ≤ 20/20 |
| IV. | Laboratory Blanks | A | |
| V. | Field blanks | ND | TB = 2 |
| VI. | Surrogate spikes | A | |
| VII. | Matrix spike/Matrix spike duplicates | N | cs |
| VIII. | Laboratory control samples | A | lcs |
| IX. | Field duplicates | N | |
| X. | Target analyte quantitation | N | |
| XI. | Target analyte identification | N | |
| XII. | Overall assessment of data | A | |

Note:
A = Acceptable
N = Not provided/applicable
SW = See worksheet
ND = No compounds detected
R = Rinsate
FB = Field blank
D = Duplicate
TB = Trip blank
EB = Equipment blank
SB=Source blank
OTHER:

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU147 | 580-118109-1 | Water | 09/19/22 |
| 2 | HU146 TB | 580-118109-3 | Water | 09/19/22 |
| 3 | | | | |
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Notes:

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| | MB 410-301674 | | | | | | |
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**LDC Report#** 55171A21

# Laboratory Data Consultants, Inc.
# Data Validation Report

| | |
|---|---|
| **Project/Site Name:** | Red Hill Oily Waste Disposal Facility, CTO 18F0176 |
| **LDC Report Date:** | November 2, 2022 |
| **Parameters:** | Polychlorinated Dioxins/Dibenzofurans |
| **Validation Level:** | Stage 2B |
| **Laboratory:** | Eurofins, Tacoma, WA |
| **Sample Delivery Group (SDG):** | 580-118109-1 |

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU147 | 580-118109-1 | Water | 09/19/22 |
| HU148 | 580-118109-2 | Water | 09/19/22 |

## Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), and the DoD General Validation Guidelines (November 2019). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polychlorinated Dioxins/Dibenzofurans by Environmental Protection Agency (EPA) SW 846 Method 8290A

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was resolved with a valley of less than or equal to 25%.

The static resolving power was at least 10,000 (10% valley definition).

## III. Initial Calibration and Initial Calibration Verification

A five point initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all analytes and labeled compounds.

The ion abundance ratios for all PCDDs/PCDFs were within method and validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0% for all analytes and less than or equal to 30.0% for labeled compounds.

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

All of the continuing calibration percent differences (%D) between the initial calibration RRF and the continuing calibration RRF were less than or equal to 20.0% for all analytes and less than or equal to 30.0% for labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within method and validation criteria.

## V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

| Blank ID | Extraction Date | Analyte | Concentration | Associated Samples |
|---|---|---|---|---|
| MB 410-300032 | 09/26/22 | 1,2,3,4,6,7,8-HpCDF | 0.000000365 ug/L | All samples in SDG 580-118109-1 |
| | | 1,2,3,4,7,8-HxCDD | 0.000000318 ug/L | |
| | | 1,2,3,4,7,8-HxCDF | 0.000000326 ug/L | |
| | | 1,2,3,6,7,8-HxCDD | 0.000000163 ug/L | |
| | | 1,2,3,6,7,8-HxCDF | 0.000000277 ug/L | |
| | | 1,2,3,7,8-PeCDF | 0.000000674 ug/L | |
| | | 1,2,3,7,8,9-HxCDD | 0.000000456 ug/L | |
| | | 1,2,3,7,8,9-HxCDF | 0.000000413 ug/L | |
| | | 2,3,4,6,7,8-HxCDF | 0.000000282 ug/L | |
| | | 2,3,4,7,8-PeCDF | 0.000000544 ug/L | |
| | | OCDD | 0.00000101 ug/L | |
| | | OCDF | 0.000000339 ug/L | |
| | | Total HxCDD | 0.000000937 ug/L | |
| | | Total HxCDF | 0.00000130 ug/L | |
| | | Total HpCDF | 0.000000365 ug/L | |
| | | Total PeCDF | 0.00000122 ug/L | |
| | | Total PeCDD/Total PeCDF | 0.00000517 ug/L | |
| | | Total PeCDD | 0.00000195 ug/L | |
| | | Total PeCDF | 0.00000322 ug/L | |

Sample concentrations were compared to concentrations detected in the laboratory blanks. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated laboratory blanks with the following exceptions:

| Sample | Analyte | Reported Concentration | Modified Final Concentration |
|---|---|---|---|
| HU147 | 1,2,3,4,6,7,8-HpCDF | 0.00000036 ug/L | 0.00000036U ug/L |
| | 1,2,3,4,7,8-HxCDD | 0.00000028 ug/L | 0.00000028U ug/L |
| | 1,2,3,6,7,8-HxCDD | 0.00000027 ug/L | 0.00000027U ug/L |
| | 1,2,3,6,7,8-HxCDF | 0.00000025 ug/L | 0.00000025U ug/L |
| | 1,2,3,7,8-PeCDF | 0.00000054 ug/L | 0.00000054U ug/L |
| | 1,2,3,7,8,9-HxCDD | 0.00000013 ug/L | 0.00000013U ug/L |
| | 1,2,3,7,8,9-HxCDF | 0.00000054 ug/L | 0.00000054U ug/L |
| | 2,3,4,6,7,8-HxCDF | 0.00000038 ug/L | 0.00000038U ug/L |
| | 2,3,4,7,8-PeCDF | 0.00000016 ug/L | 0.00000016U ug/L |
| | OCDD | 0.0000015 ug/L | 0.0000015U ug/L |
| | OCDF | 0.00000029 ug/L | 0.00000029U ug/L |
| | Total HxCDD | 0.00000068 ug/L | 0.00000068U ug/L |
| | Total HxCDF | 0.0000012 ug/L | 0.0000012U ug/L |
| | Total HpCDF | 0.00000060 ug/L | 0.00000060U ug/L |
| | Total PeCDF | 0.00000070 ug/L | 0.00000070U ug/L |
| | Total PeCDD/Total PeCDF | 0.0000061 ug/L | 0.0000061U ug/L |
| | Total PeCDD | 0.0000031 ug/L | 0.0000031U ug/L |
| | Total PeCDF | 0.0000030 ug/L | 0.0000030U ug/L |
| HU148 | 1,2,3,7,8-PeCDF | 0.00000025 ug/L | 0.00000025U ug/L |
| | 1,2,3,7,8,9-HxCDD | 0.00000041 ug/L | 0.00000041U ug/L |
| | 1,2,3,7,8,9-HxCDF | 0.00000029 ug/L | 0.00000029U ug/L |
| | 2,3,4,6,7,8-HxCDF | 0.000000027 ug/L | 0.000000027U ug/L |
| | 2,3,4,7,8-PeCDF | 0.00000028 ug/L | 0.00000028U ug/L |
| | OCDD | 0.0000012 ug/L | 0.0000012U ug/L |
| | Total HxCDD | 0.00000041 ug/L | 0.00000041U ug/L |
| | Total HxCDF | 0.00000056 ug/L | 0.00000056U ug/L |
| | Total HpCDF | 0.00000015 ug/L | 0.00000015U ug/L |
| | Total PeCDF | 0.00000053 ug/L | 0.00000053U ug/L |
| | Total PeCDD/Total PeCDF | 0.0000036 ug/L | 0.0000036U ug/L |
| | Total PeCDD | 0.0000024 ug/L | 0.0000024U ug/L |
| | Total PeCDF | 0.0000012 ug/L | 0.0000012U ug/L |

## VI. Field Blanks

No field blanks were identified in this SDG.

## VII. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## VIII. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

## IX. Field Duplicates

Samples HU147 and HU148 were identified as field duplicates. No results were detected in any of the samples with the following exceptions:

| | Concentration (ug/L) | | |
|---|---|---|---|
| **Analyte** | **HU147** | **HU148** | **RPD (Limits)** |
| 1,2,3,4,6,7,8-HpCDD | 0.00000082 | 0.00000081 | 1 (≤50) |
| 1,2,3,4,6,7,8-HpCDF | 0.00000036 | 0.0000097U | 186 (≤50) |
| 1,2,3,4,7,8-HxCDD | 0.00000028 | 0.0000097U | 189 (≤50) |
| 1,2,3,4,7,8,9-HpCDF | 0.00000024 | 0.00000015 | 46 (≤50) |
| 1,2,3,6,7,8-HxCDD | 0.00000027 | 0.0000097U | 189 (≤50) |
| 1,2,3,6,7,8-HxCDF | 0.00000025 | 0.0000097U | 190 (≤50) |
| 1,2,3,7,8-PeCDF | 0.00000054 | 0.00000025 | 73 (≤50) |
| 1,2,3,7,8,9-HxCDD | 0.00000013 | 0.00000041 | 104 (≤50) |
| 1,2,3,7,8,9-HxCDF | 0.00000054 | 0.00000029 | 60 (≤50) |
| 2,3,4,6,7,8-HxCDF | 0.00000038 | 0.00000027 | 34 (≤50) |
| 2,3,4,7,8-PeCDF | 0.00000016 | 0.00000028 | 55 (≤50) |
| 2,3,7,8-TCDD | 0.00000014 | 0.0000019U | 173 (≤50) |

| Analyte | Concentration (ug/L) | | RPD (Limits) |
|---|---|---|---|
| | HU147 | HU148 | |
| 2,3,7,8-TCDF | 0.00000016 | 0.0000019U | 169 (≤50) |
| OCDD | 0.0000015 | 0.0000012 | 22 (≤50) |
| OCDF | 0.00000029 | 0.000019U | 194 (≤50) |
| Total HxCDD | 0.00000068 | 0.00000041 | 50 (≤50) |
| Total HxCDF | 0.0000012 | 0.00000056 | 73 (≤50) |
| Total HpCDD | 0.00000082 | 0.00000081 | 1 (≤50) |
| Total HpCDF | 0.00000060 | 0.00000015 | 120 (≤50) |
| Total PeCDF | 0.00000070 | 0.00000053 | 28 (≤50) |
| Total TCDD | 0.00000014 | 0.0000019U | 173 (≤50) |
| Total TCDF | 0.00000016 | 0.0000019U | 169 (≤50) |
| Total PeCDD/Total PeCDF | 0.0000061 | 0.0000036 | 52 (≤50) |
| Total PeCDD | 0.0000031 | 0.0000024 | 25 (≤50) |
| Total PeCDF | 0.0000030 | 0.0000012 | 86 (≤50) |

## X. Labeled Compounds

All percent recoveries (%R) for labeled compounds used to quantitate target analytes were within QC limits.

## XI. Target Analyte Quantitation

All target analyte quantitations met validation criteria with the following exceptions:

| Sample | Analyte | Flag | A or P |
|---|---|---|---|
| All samples in SDG 580-118109-1 | Results flagged "I" by the laboratory as estimated maximum possible concentration (EMPC). | J (all detects) | A |

For sample HU147, a 2nd column confirmation was not performed for 2,3,7,8-TCDF. Result was less than the limit of quantitation (LOQ).

Raw data were not reviewed for Stage 2B validation.

## XII. Target Analyte Identification

Raw data were not reviewed for Stage 2B validation.

## XIII. System Performance

Raw data were not reviewed for Stage 2B validation.

## XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

Due to results reported by the laboratory as EMPC, data were qualified as estimated in two samples.

Due to laboratory blank contamination, data were qualified as not detected in two samples.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polychlorinated Dioxins/Dibenzofurans - Data Qualification Summary - SDG 580-118109-1

| Sample | Analyte | Flag | A or P | Reason (Code) |
|---|---|---|---|---|
| HU147<br>HU148 | Results flagged "I" by the laboratory as estimated maximum possible concentration (EMPC). | J (all detects) | A | Target analyte quantitation (EMPC) (k) |

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polychlorinated Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG 580-118109-1

| Sample | Analyte | Modified Final Concentration | A or P | Code |
|---|---|---|---|---|
| HU147 | 1,2,3,4,6,7,8-HpCDF | 0.00000036U ug/L | A | b |
| | 1,2,3,4,7,8-HxCDD | 0.00000028U ug/L | | |
| | 1,2,3,6,7,8-HxCDD | 0.00000027U ug/L | | |
| | 1,2,3,6,7,8-HxCDF | 0.00000025U ug/L | | |
| | 1,2,3,7,8-PeCDF | 0.00000054U ug/L | | |
| | 1,2,3,7,8,9-HxCDD | 0.00000013U ug/L | | |
| | 1,2,3,7,8,9-HxCDF | 0.00000054U ug/L | | |
| | 2,3,4,6,7,8-HxCDF | 0.00000038U ug/L | | |
| | 2,3,4,7,8-PeCDF | 0.00000016U ug/L | | |
| | OCDD | 0.0000015U ug/L | | |
| | OCDF | 0.00000029U ug/L | | |
| | Total HxCDD | 0.00000068U ug/L | | |
| | Total HxCDF | 0.0000012U ug/L | | |
| | Total HpCDF | 0.00000060U ug/L | | |
| | Total PeCDF | 0.00000070U ug/L | | |
| | Total PeCDD/Total PeCDF | 0.0000061U ug/L | | |
| | Total PeCDD | 0.0000031U ug/L | | |
| | Total PeCDF | 0.0000030U ug/L | | |
| HU148 | 1,2,3,7,8-PeCDF | 0.00000025U ug/L | A | b |
| | 1,2,3,7,8,9-HxCDD | 0.00000041U ug/L | | |
| | 1,2,3,7,8,9-HxCDF | 0.00000029U ug/L | | |
| | 2,3,4,6,7,8-HxCDF | 0.000000027U ug/L | | |
| | 2,3,4,7,8-PeCDF | 0.00000028U ug/L | | |
| | OCDD | 0.0000012U ug/L | | |
| | Total HxCDD | 0.00000041U ug/L | | |
| | Total HxCDF | 0.00000056U ug/L | | |
| | Total HpCDF | 0.00000015U ug/L | | |
| | Total PeCDF | 0.00000053U ug/L | | |
| | Total PeCDD/Total PeCDF | 0.0000036U ug/L | | |
| | Total PeCDD | 0.0000024U ug/L | | |
| | Total PeCDF | 0.0000012U ug/L | | |

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polychlorinated Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG 580-118109-1

No Sample Data Qualified in this SDG

LDC #: 55171A21
SDG #: 580-118109-1
Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

Stage 2B

Date: 10/28/22
Page: 1 of 1
Reviewer: PJ
2nd Reviewer: F

**METHOD:** HRGC/HRMS Polychlorinated Dioxins/Dibenzofurans (EPA SW-846 Method 8290A)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

|  | Validation Area |  | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A /A |  |
| II. | HRGC/HRMS Instrument performance check | A |  |
| III. | Initial calibration/ICV | A /A | % RSD ≤ 20% ICV ≤ 20/30 |
| IV. | Continuing calibration | A | CCV ≤ 20/30 |
| V. | Laboratory Blanks | SW |  |
| VI. | Field blanks | N |  |
| VII. | Matrix spike/Matrix spike duplicates | N | CS |
| VIII. | Laboratory control samples | A | LCS ID |
| IX. | Field duplicates | SW | D = 1,2 |
| X. | Labeled Compounds | A |  |
| XI. | Target analyte quantitation | SW |  |
| XII. | Target analyte identification | N |  |
| XIII. | System performance | N |  |
| XIV. | Overall assessment of data | A |  |

Note:
A = Acceptable
N = Not provided/applicable
SW = See worksheet
ND = No compounds detected
R = Rinsate
FB = Field blank
D = Duplicate
TB = Trip blank
EB = Equipment blank
SB=Source blank
OTHER:

|  | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU147 | 580-118109-1 | Water | 09/19/22 |
| 2 | HU148 | 580-118109-2 | Water | 09/19/22 |
| 3 |  |  |  |  |
| 4 |  |  |  |  |
| 5 |  |  |  |  |
| 6 |  |  |  |  |
| 7 |  |  |  |  |
| 8 |  |  |  |  |
| 9 |  |  |  |  |
| 10 |  |  |  |  |

Notes:

|  |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|
|  | MB 410-300032 |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |

# VALIDATION FINDINGS WORKSHEET

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290A)

| | | | | |
|---|---|---|---|---|
| A. 2,3,7,8-TCDD | F. 1,2,3,4,6,7,8-HpCDD | K. 1,2,3,4,7,8-HxCDF | P. 1,2,3,4,7,8,9-HpCDF | U. Total HpCDD |
| B. 1,2,3,7,8-PeCDD | G. OCDD | L. 1,2,3,6,7,8-HxCDF | Q. OCDF | V. Total TCDF |
| C. 1,2,3,4,7,8-HxCDD | H. 2,3,7,8-TCDF | M. 2,3,4,6,7,8-HxCDF | R. Total TCDD | W. Total PeCDF |
| D. 1,2,3,6,7,8-HxCDD | I. 1,2,3,7,8-PeCDF | N. 1,2,3,7,8,9-HxCDF | S. Total PeCDD | X. Total HxCDF |
| E. 1,2,3,7,8,9-HxCDD | J. 2,3,4,7,8-PeCDF | O. 1,2,3,4,6,7,8-HpCDF | T. Total HxCDD | Y. Total HpCDF |

Notes:________________________________________

LDC #: 55171A21

# VALIDATION FINDINGS WORKSHEET

## Blanks

Page: 1 of 2
Reviewer: FT

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290A)
Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".
Y Were all samples associated with a method blank?
Y Was a method blank performed for each matrix and whenever a sample extraction was performed?
Y Was the method blank contaminated?
**Blank extraction date:** 9/26/22 **Blank analysis date:** **9/27/22**
**Conc. units:** ug/L **Associated samples:** **All (b)**

| Compound | Blank ID | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| ▨ | MB 410-300032 | 5x | | 1 | | 2 | | | | |
| O | 0.000000365 | 0.000001825 | | 0.00000036U | | - | | | | |
| C | 0.000000318 | 0.000001590 | | 0.00000028U | | - | | | | |
| K | 0.000000326 | 0.000001630 | | - | | - | | | | |
| D | 0.000000163 | 0.000000815 | | 0.00000027U | | - | | | | |
| L | 0.000000277 | 0.000001385 | | 0.00000025U | | - | | | | |
| I | 0.000000674 | 0.000003370 | | 0.00000054U | | 0.00000025U | | | | |
| E | 0.000000456 | 0.000002280 | | 0.00000013U | | 0.00000041U | | | | |
| N | 0.000000413 | 0.000002065 | | 0.00000054U | | 0.00000029U | | | | |
| M | 0.000000282 | 0.000001410 | | 0.00000038U | | 0.000000027U | | | | |
| J | 0.000000544 | 0.000002720 | | 0.00000016U | | 0.00000028U | | | | |
| G | 0.00000101 | 0.000005050 | | 0.0000015U | | 0.0000012U | | | | |
| Q | 0.000000339 | 0.000001695 | | 0.00000029U | | - | | | | |
| T | 0.000000937 | 0.000004685 | | 0.00000068U | | 0.00000041U | | | | |

CUIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
All contaminants within five times the method blank concentration were qualified as not detected, "U".

LDC #: 55171A21

# VALIDATION FINDINGS WORKSHEET
## Blanks

Page: 2 of 2
Reviewer: FT

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290A)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y Were all samples associated with a method blank?
Y Was a method blank performed for each matrix and whenever a sample extraction was performed?
Y Was the method blank contaminated?

**Blank extraction date:** 9/26/22 **Blank analysis date:** 9/27/22
**Conc. units:** ug/L **Associated samples:** all (b)

| Compound | Blank ID | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| ▨ | MB 410-300032 | 5x | | 1 | | 2 | | | | |
| X | 0.00000130 | 0.000006500 | | 0.0000012U | | 0.00000056U | | | | |
| Y | 0.000000365 | 0.000001825 | | 0.00000060U | | 0.00000015U | | | | |
| W | 0.00000122 | 0.000006100 | | 0.00000070U | | 0.00000053U | | | | |
| S/W | 0.00000517 | 0.000025850 | | 0.0000061U | | 0.0000036U | | | | |
| S | 0.00000195 | 0.000009750 | | 0.0000031U | | 0.0000024U | | | | |
| W | 0.00000322 | 0.000016100 | | 0.0000030U | | 0.0000012U | | | | |
| | | | | | | | | | | |
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Vdr/Validation Worksheets/Dioxins/8290/MB 410 300032

LDC#:_55171A21

# VALIDATION FINDINGS WORKSHEET
## Field Duplicates

Page:_1__of_1__
Reviewer:__FT____

**METHOD**: EPA SW 846 method 8290A

| Compound | Concentration (ug/L) | | (≤50) |
|---|---|---|---|
| | 1 | 2 | RPD |
| F | 0.00000082 | 0.00000081 | 1 |
| O | 0.00000036 | 0.0000097U | 186 |
| C | 0.00000028 | 0.0000097U | 189 |
| P | 0.00000024 | 0.00000015 | 46 |
| D | 0.00000027 | 0.0000097U | 189 |
| L | 0.00000025 | 0.0000097U | 190 |
| I | 0.00000054 | 0.00000025 | 73 |
| E | 0.00000013 | 0.00000041 | 104 |
| N | 0.00000054 | 0.00000029 | 60 |
| M | 0.00000038 | 0.00000027 | 34 |
| J | 0.00000016 | 0.00000028 | 55 |
| A | 0.00000014 | 0.0000019U | 173 |
| H | 0.00000016 | 0.0000019U | 169 |
| G | 0.0000015 | 0.0000012 | 22 |
| Q | 0.00000029 | 0.000019U | 194 |
| T | 0.00000068 | 0.00000041 | 50 |
| X | 0.0000012 | 0.00000056 | 73 |
| U | 0.00000082 | 0.00000081 | 1 |
| Y | 0.00000060 | 0.00000015 | 120 |
| W | 0.00000070 | 0.00000053 | 28 |
| R | 0.00000014 | 0.0000019U | 173 |
| V | 0.00000016 | 0.0000019U | 169 |
| S/W | 0.0000061 | 0.0000036 | 52 |
| S | 0.0000031 | 0.0000024 | 25 |
| W | 0.0000030 | 0.0000012 | 86 |

LDC #: 55171A2)

# VALIDATION FINDINGS WORKSHEET
## Target Analyte Quantitation

Page: 1 of 1
Reviewer:

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)A

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Were the correct internal standard (IS), quantitation ions and relative response factors (RRF) used to quantitate the compound?
[✓ Y] N N/A Compound quantitation and CRQLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

(K)

| # | Date | Sample ID | Finding | Associated Samples | Qualifications |
|---|---|---|---|---|---|
| | | All | all analyte qualified "I" as (EMPC) estimated maximum possible concentration | | Jdu /A |
| | | 1 | H- NO 2nd column confirmation was performed. Result < LOQ | | text |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

Comments: See sample calculation verification worksheet for recalculations

**LDC Report#** 55171B1a

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** November 2, 2022

**Parameters:** Volatiles

**Validation Level:** Stage 2B & 4

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118118-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU145** | 580-118118-1** | Water | 09/19/22 |
| HU144 | 580-118118-2 | Water | 09/19/22 |

**Indicates sample underwent Stage 4 validation

# Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS (May 2020). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Volatile Organic Compounds (VOCs) and Tentatively Identified Compounds (TICs) by Environmental Protection Agency (EPA) SW 846 Method 8260D

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results. Samples appended with a double asterisk on the cover page were subjected to Stage 4 data validation, which is comprised of the QC summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

## II. GC/MS Instrument Performance Check

A bromofluorobenzene (BFB) tune was performed at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

For analytes where average relative response factors (RRFs) were utilized, the percent relative standard deviations (%RSD) were less than or equal to 15.0%.

In the case where the laboratory used a calibration curve to evaluate the analytes, all coefficients of determination ($r^2$) were greater than or equal to 0.990.

Average relative response factors (RRF) for all analytes were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0% for all analytes.

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all analytes with the following exceptions:

| Date | Analyte | %D | Associated Samples | Flag | A or P |
|---|---|---|---|---|---|
| 09/27/22 | Vinyl chloride | 21.5 | All samples in SDG 580-118118-1 | UJ (all non-detects) | A |

The percent differences (%D) of the ending continuing calibration verifications (CCVs) were less than or equal to 50.0% for all analytes.

All of the continuing calibration relative response factors (RRF) were within validation criteria.

## V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

| Blank ID | Analysis Date | Analyte | Concentration | Associated Samples |
|---|---|---|---|---|
| MB 580-405161 | 09/27/22 | Hexachlorobutadiene<br>1,3,5-Trichlorobenzene | 0.121 ug/L<br>0.0481 ug/L | All samples in SDG 580-118118-1 |

Sample concentrations were compared to concentrations detected in the laboratory blanks. The sample concentrations were either not detected or were significantly greater (>10X for common contaminants, >5X for other contaminants) than the concentrations found in the associated laboratory blanks.

## VI. Field Blanks

Sample HU144 was identified as a trip blank. No contaminants were found.

## VII. Surrogates

Surrogates were added to all samples as required by the method. All surrogate recoveries (%R) were within QC limits.

## VIII. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## IX. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

## X. Field Duplicates

No field duplicates were identified in this SDG.

## XI. Internal Standards

All internal standard areas and retention times were within QC limits.

## XII. Target Analyte and Tentatively Identified Compound Quantitation

All target analyte and tentatively identified compound (TIC) quantitations met validation criteria with the following exceptions:

| Sample | Analyte | Flag | A or P |
|---|---|---|---|
| All samples in SDG 580-118118-1 | All tentatively identified compounds (TIC) | NJ (all detects) | A |

Raw data were not reviewed for Stage 2B validation.

## XIII. Target Analyte Identification

All target analyte identifications met validation criteria for samples which underwent Stage 4 validation. Raw data were not reviewed for Stage 2B validation.

Manual integrations were reviewed and were considered acceptable. The laboratory provided before and after integration printouts.

## XIV. System Performance

The system performance was acceptable for samples which underwent Stage 4 validation. Raw data were not reviewed for Stage 2B validation.

## XV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

Due to continuing calibration %D, data were qualified as estimated in two samples.

Due to TICs, data were qualified as presumptive and estimated in two samples.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Volatiles - Data Qualification Summary - SDG 580-118118-1

| Sample | Analyte | Flag | A or P | Reason (Code) |
|---|---|---|---|---|
| HU145**<br>HU144 | Vinyl chloride | UJ (all non-detects) | A | Continuing calibration (%D) (c) |
| HU145**<br>HU144 | All tentatively identified compounds (TIC) | NJ (all detects) | A | Tentatively identified compound quantitation (v) |

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Volatiles - Laboratory Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Volatiles - Field Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

LDC #: 55171B1a
SDG #: 580-118118-1
Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET
Stage 2B/4

Date: 10/31/22
Page: 1 of 1
Reviewer:
2nd Reviewer:

**METHOD:** GC/MS Volatiles (EPA SW-846 Method 8260D) + TICs

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A A | |
| II. | GC/MS Instrument performance check | A | |
| III. | Initial calibration/ICV | A, A | % RSD ≤ 15, r² ICV ≤ 20 |
| IV. | Continuing calibration | SW | CCV ≤ 20/50 |
| V. | Laboratory Blanks | SW | |
| VI. | Field blanks | ND | TB = 2 |
| VII. | Surrogate spikes | A | |
| VIII. | Matrix spike/Matrix spike duplicates | N | CS |
| IX. | Laboratory control samples | A | LCS/D |
| X. | Field duplicates | N | |
| XI. | Internal standards | A | |
| XII. | Target analyte quantitation | SW | Not reviewed for Stage 2B validation. |
| XIII. | Target analyte identification | A | Not reviewed for Stage 2B validation. MI |
| XIV. | System performance | A | Not reviewed for Stage 2B validation. |
| XV. | Overall assessment of data | A | |

Note:

| | | | |
|---|---|---|---|
| A = Acceptable | ND = No compounds detected | D = Duplicate | SB=Source blank |
| N = Not provided/applicable | R = Rinsate | TB = Trip blank | OTHER: |
| SW = See worksheet | FB = Field blank | EB = Equipment blank | |

** Indicates sample underwent Stage 4 validation

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 12+ | HU145** 1 = MEF | 580-118118-1** | Water | 09/19/22 |
| 2 2 | HU144 1 > TB | 580-118118-2 | Water | 09/19/22 |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
| 7 | | | | |
| 8 | | | | |
| 9 | | | | |

Notes:

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | MB 580-404 | 907 | | | | | |
| 2 | MB 580- 4051 | 61 | | | | | |
| | | | | | | | |
| | | | | | | | |

LDC #: 55171B1a

## VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: FT

**Method:** Volatiles (EPA SW 846 Method 8260D)

| Validation Area | Yes | No | NA | Findings/Comments |
|---|---|---|---|---|
| ***I. Technical holding times*** | | | | |
| Were all technical holding times met? | ✓ | | | |
| Was cooler temperature criteria met? | ✓ | | | |
| ***II. GC/MS Instrument performance check*** | | | | |
| Were the BFB performance results reviewed and found to be within the specified criteria? | ✓ | | | |
| Were all samples analyzed within the 12 hour clock criteria? | ✓ | | | |
| ***IIIa. Initial calibration*** | | | | |
| Did the laboratory perform a 5 point calibration prior to sample analysis? | ✓ | | | |
| Were all percent relative standard deviations (%RSD) $\leq$ 15% and relative response factors (RRF) within method criteria? | ✓ | | | |
| Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of $\geq$ 0.990? | ✓ | | | |
| ***IIIb. Initial Calibration Verification*** | | | | |
| Was an initial calibration verification standard analyzed after each initial calibration for each instrument? | ✓ | | | |
| Were all percent differences (%D) $\leq$ 20%? | ✓ | | | |
| ***IV. Continuing calibration*** | | | | |
| Was a continuing calibration standard analyzed at least once every 12 hours for each instrument? | ✓ | | | |
| Were all percent differences (%D) $\leq$ 20% and relative response factors (RRF) within method criteria? Were all percent differences (%D) $\leq$ 50% in the ending CCV? | | ✓ | | |
| ***V. Laboratory Blanks*** | | | | |
| Was a laboratory blank associated with every sample in this SDG? | ✓ | | | |
| Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration? | ✓ | | | |
| Was there contamination in the laboratory blanks? | | ✓ | | |
| ***VI. Field blanks*** | | | | |
| Were field blanks were identified in this SDG? | ✓ | | | |
| Were target analytes detected in the field blanks? | | ✓ | | |
| ***VII. Surrogate spikes*** | | | | |
| Were all surrogate percent recovery (%R) within QC limits? | ✓ | | | |
| If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R outside of criteria? | | | ✓ | |
| ***VIII. Matrix spike/Matrix spike duplicates*** | | | | |
| Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG? | | | ✓ | |
| Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits? | | | ✓ | |

LDC #: 55171B1a

## VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: FT

| | | | | |
|---|---|---|---|---|
| | | | | |
| ***IX. Laboratory control samples*** | | | | |
| Was an LCS analyzed per analytical batch? | ✓ | | | |
| Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits? | ✓ | | | |
| ***X. Field duplicates*** | | | | |
| Were field duplicate pairs identified in this SDG? | | ✓ | | |
| Were target analytes detected in the field duplicates? | | | ✓ | |
| ***XI. Internal standards*** | | | | |
| Were internal standard area counts within -50% to +100% of the associated calibration standard? | ✓ | | | |
| Were retention times within ± 30 seconds of the associated calibration standard? | ✓ | | | |
| ***XII. Target analyte quantitation*** | | | | |
| Did the laboratory LOQs/RLs meet the QAPP LOQs/RLs? | ✓ | | | |
| Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the target analyte? | ✓ | | | |
| Were target analyte quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation? | ✓ | | | |
| ***XIII. Target analyte identification*** | | | | |
| Were relative retention times (RRT's) within ± 0.06 RRT units of the standard? | ✓ | | | |
| Did analyte spectra meet specified EPA "Functional Guidelines" criteria? | ✓ | | | |
| Were chromatogram peaks verified and accounted for? | ✓ | | | |
| Were manual integrations reviewed and found acceptable? | ✓ | | | |
| Did the laboratory provide before and after integration printouts? | ✓ | | | |
| ***XIV. System performance*** | | | | |
| System performance was found to be acceptable. | ✓ | | | |
| ***XV. Overall assessment of data*** | | | | |
| Overall assessment of data was found to be acceptable. | ✓ | | | |

# TARGET COMPOUND WORKSHEET

**METHOD: VOA**

| | | | | |
|---|---|---|---|---|
| A. Chloromethane | AA. Tetrachloroethene | AAA. 1,3,5-Trimethylbenzene | AAAA. Ethyl tert-butyl ether | A1. 1,3-Butadiene |
| B. Bromomethane | BB. 1,1,2,2-Tetrachloroethane | BBB. 4-Chlorotoluene | BBBB. tert-Amyl methyl ether | B1. Hexane |
| C. Vinyl choride | CC. Toluene | CCC. tert-Butylbenzene | CCCC. 1-Chlorohexane | C1. Heptane |
| D. Chloroethane | DD. Chlorobenzene | DDD. 1,2,4-Trimethylbenzene | DDDD. Isopropyl alcohol | D1. Propylene |
| E. Methylene chloride | EE. Ethylbenzene | EEE. sec-Butylbenzene | EEEE. Acetonitrile | E1. Freon 11 |
| F. Acetone | FF. Styrene | FFF. 1,3-Dichlorobenzene | FFFF. Acrolein | F1. Freon 12 |
| G. Carbon disulfide | GG. Xylenes, total | GGG. p-Isopropyltoluene | GGGG. Acrylonitrile | G1. Freon 113 |
| H. 1,1-Dichloroethene | HH. Vinyl acetate | HHH. 1,4-Dichlorobenzene | HHHH. 1,4-Dioxane | H1. Freon 114 |
| I. 1,1-Dichloroethane | II. 2-Chloroethylvinyl ether | III. n-Butylbenzene | IIII. Isobutyl alcohol | I1. 2-Nitropropane |
| J. 1,2-Dichloroethene, total | JJ. Dichlorodifluoromethane | JJJ. 1,2-Dichlorobenzene | JJJJ. Methacrylonitrile | J1. Dimethyl disulfide |
| K. Chloroform | KK. Trichlorofluoromethane | KKK. 1,2,4-Trichlorobenzene | KKKK. Propionitrile | K1. 2,3-Dimethyl pentane |
| L. 1,2-Dichloroethane | LL. Methyl-tert-butyl ether | LLL. Hexachlorobutadiene | LLLL. Ethyl ether | L1. 2,4-Dimethyl pentane |
| M. 2-Butanone | MM. 1,2-Dibromo-3-chloropropane | MMM. Naphthalene | MMMM. Benzyl chloride | M1. 3,3-Dimethyl pentane |
| N. 1,1,1-Trichloroethane | NN. Methyl ethyl ketone | NNN. 1,2,3-Trichlorobenzene | NNNN. Iodomethane | N1. 2-Methylpentane |
| O. Carbon tetrachloride | OO. 2,2-Dichloropropane | OOO. 1,3,5-Trichlorobenzene | OOOO.1,1-Difluoroethane | O1. 3-Methylpentane |
| P. Bromodichloromethane | PP. Bromochloromethane | PPP. trans-1,2-Dichloroethene | PPPP. Tetrahydrofuran | P1. 3-Ethylpentane |
| Q. 1,2-Dichloropropane | QQ. 1,1-Dichloropropene | QQQ. cis-1,2-Dichloroethene | QQQQ. Methyl acetate | Q1. 2,2-Dimethylpentane |
| R. cis-1,3-Dichloropropene | RR. Dibromomethane | RRR. m,p-Xylenes | RRRR. Ethyl acetate | R1. 2,2,3- Trimethylbutane |
| S. Trichloroethene | SS. 1,3-Dichloropropane | SSS. o-Xylene | SSSS. Cyclohexane | S1. 2,2,4-Trimethylpentane |
| T. Dibromochloromethane | TT. 1,2-Dibromoethane | TTT. 1,1,2-Trichloro-1,2,2-trifluoroethane | TTTT. Methyl cyclohexane | T1. 2-Methylhexane |
| U. 1,1,2-Trichloroethane | UU. 1,1,1,2-Tetrachloroethane | UUU. 1,2-Dichlorotetrafluoroethane | UUUU. Allyl chloride | U1. Nonanal |
| V. Benzene | VV. Isopropylbenzene | VVV. 4-Ethyltoluene | VVVV. Methyl methacrylate | V1. 2-Methylnaphthalene |
| W. trans-1,3-Dichloropropene | WW. Bromobenzene | WWW. Ethanol | WWWW. Ethyl methacrylate | W1. Methanol |
| X. Bromoform | XX. 1,2,3-Trichloropropane | XXX. Di-isopropyl ether | XXXX. cis-1,4-Dichloro-2-butene | X1. 1,2,3-Trimethylbenzene |
| Y. 4-Methyl-2-pentanone | YY. n-Propylbenzene | YYY. tert-Butanol | YYYY. trans-1,4-Dichloro-2-butene | Y1. 2-Propanol |
| Z. 2-Hexanone | ZZ. 2-Chlorotoluene | ZZZ. tert-Butyl alcohol | ZZZZ. Pentachloroethane | Z1. |

LDC #: 55171B1a

# VALIDATION FINDINGS WORKSHEET
## Continuing Calibration

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260 D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?
[✓ Y] N N/A Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ?
Y [✓ N] N/A Were all %D and RRFs within the validation criteria of ≤20 %D and ≥0.05 RRF ?

| # | Date | Standard ID | Compound | Finding %D (Limit: <20.0%) | Finding RRF (Limit: >0.05) | Associated Samples | Qualifications |
|---|---|---|---|---|---|---|---|
| | 9/27/22 | CCV 580-405161 | C | 21.5 | | All, | J+/UJ/A ND |
| | 1234 | | | | | MB 580-405161 | |
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CONCAL.wpd

LDC #: 55171B/a

# VALIDATION FINDINGS WORKSHEET

## Blanks

Page: 1 of 1

Reviewer: FT

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260 )

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Was a method blank associated with every sample in this SDG?

[✓ Y] N N/A Was a method blank analyzed at least once every 12 hours for each matrix and concentration?

[✓ Y] N N/A Was there contamination in the method blanks? If yes, please see the qualifications below.

**Blank analysis date:** 9/27/22

**Conc. units:** ug/L

Associated Samples: All (ND)

| Compound | Blank ID | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| ▨ | MB 580-40516) | | | | | | | | | |
| LLL | 0.12) | | | | | | | | | |
| TIC 1,3,5- | 0.048) | | | | | | | | | |
| Trichlorobenzene | | | | | | | | | | |
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**Blank analysis date:**

**Conc. units:**

Associated Samples:

| Compound | Blank ID | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| ▨ | | | | | | | | | | |
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All results were qualified using the criteria stated below except those circled.

Note: Common contaminants such as Methylene chloride, Acetone, 2-Butanone, Carbon disulfide and TICs that were detected in samples within ten times the associated method blank concentration were qualified as not detected, "U". Other contaminants within five times the method blank concentration were also qualified as not detected, "U".

LDC #: 55171B1a

# VALIDATION FINDINGS WORKSHEET

## Target Analyte Quantitation and Tentatively Identified Commpound

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?

[✓ Y] N N/A Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?

| # | Sample ID | Analytes | Finding | Qualifications |
|---|---|---|---|---|
| | all | all unkown and analytes reported as Tentatively Identified Compounds (TIC) | | NJ/A (v) |
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Comments: See sample calculation verification worksheet for recalculations

LDC #: 55171B1a

# VALIDATION FINDINGS WORKSHEET
## Initial Calibration Calculation Verification

Page: _1___ of __1__
Reviewer: ____FT___

METHOD: GCMS 8260D

The calibration factors (RRFF), average RRFF, and relative standard deviation (%RSD) were recalculated for compounds identified below using the following calculations:

RRF = (Ax)(Cis)/(Ais)(Cx)
average RRF = sum of the RRFs/number of standards
%RSD = 100 * (S/X)

Where:

Ax = Area of compound
Cx = Concentration of compound
S = Standard deviation of the RRFs
X = Mean of the RRFs
Ais = Area of associated internal standard
Cis = Concentration of internal Standard

| # | Standard ID | Calibration Date | Compound | Reported (RRF 5ug/L std) | Recalculated (RRF 5ug/Lstd) | Reported AverageRRF (Initial) | Recalculated Average RRF (Initial) | Reported %RSD | Recalculated %RSD |
|---|---|---|---|---|---|---|---|---|---|
|  | ICAL | 9/18/2022 | K | 0.4505 | 0.4505 | 0.4477 | 0.4477 | 6.7 | 6.7 |
|  | TACO48 |  | CC | 1.7177 | 1.7177 | 1.7010 | 1.7010 | 7.8 | 7.8 |
|  |  |  | JJJ | 1.3211 | 1.3211 | 1.2712 | 1.2712 | 6.2 | 6.2 |

LDC #: 55171B1a

# VALIDATION FINDINGS WORKSHEET
## Continuing Calibration Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260D)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the analytes identified below using the following calculation:

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
RRF = $(A_x)(C_{is})/(A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
RRF = continuing calibration RRF
$A_x$ = Area of compound, $A_{is}$ = Area of associated internal standard
$C_x$ = Concentration of compound, $C_{is}$ = Concentration of internal standard

| # | Standard ID | Calibration Date | Compound (Reference internal Standard) | Average RRF (initial) | Reported RRF (CC) | Recalculated RRF (CC) | Reported %D | Recalculated %D |
|---|---|---|---|---|---|---|---|---|
| 1 | CCV<br>TAC048 | 9/27/22<br>1234 | K (1st internal standard) | 0.4477 | 0.4814 | 0.4814 | 7.5 | 7.5 |
| | | | CC (2nd internal standard) | 1.7010 | 1.802 | 1.802 | 6.0 | 6.0 |
| | | | JJJ (3rd internal standard) | 1.2712 | 1.363 | 1.363 | 7.2 | 7.2 |
| | | | (4th internal standard) | | | | | |
| 2 | CCV<br>TAC 113 | 9/23/22<br>1735 | MEK (1st internal standard) | 0.0183 | 0.0181 | 0.0181 | 1.5 | 1.5 |
| | | | (2nd internal standard) | | | | | |
| | | | (3rd internal standard) | | | | | |
| | | | (4th internal standard) | | | | | |
| 3 | | | (1st internal standard) | | | | | |
| | | | (2nd internal standard) | | | | | |
| | | | (3rd internal standard) | | | | | |
| | | | (4th internal standard) | | | | | |
| 4 | | | (1st internal standard) | | | | | |
| | | | (2nd internal standard) | | | | | |
| | | | (3rd internal standard) | | | | | |
| | | | (4th internal standard) | | | | | |

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 55171B1a

# VALIDATION FINDINGS WORKSHEET

## Surrogate Results Verification

Page: 1 of 1
Reviewer: FT
2nd reviewer:

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260~~B~~) D

The percent recoveries (%R) of surrogates were recalculated for the analytes identified below using the following calculation:

% Recovery: SF/SS * 100

Where: SF = Surrogate Found
SS = Surrogate Spiked

**Sample ID:** #1

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Dibromofluoromethane | 10.0 | 11.3 | 113 | 113 | 0 |
| 1,2-Dichloroethane-d4 | ↓ | 10.4 | 104 | 104 | ↓ |
| Toluene-d8 | ↓ | 10.1 | 101 | 101 | ↓ |
| Bromofluorobenzene | ↓ | 9.02 | 90 | 90 | ↓ |

**Sample ID:**

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Dibromofluoromethane | | | | | |
| 1,2-Dichloroethane-d4 | | | | | |
| Toluene-d8 | | | | | |
| Bromofluorobenzene | | | | | |

**Sample ID:**

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Dibromofluoromethane | | | | | |
| 1,2-Dichloroethane-d4 | | | | | |
| Toluene-d8 | | | | | |
| Bromofluorobenzene | | | | | |

**Sample ID:**

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Dibromofluoromethane | | | | | |
| 1,2-Dichloroethane-d4 | | | | | |
| Toluene-d8 | | | | | |
| Bromofluorobenzene | | | | | |

**Sample ID:**

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Dibromofluoromethane | | | | | |
| 1,2-Dichloroethane-d4 | | | | | |
| Toluene-d8 | | | | | |
| Bromofluorobenzene | | | | | |

LDC #: 5517 1B/a

# VALIDATION FINDINGS WORKSHEET
## Laboratory Control Sample Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS VOA (EPA Method 8260 B)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratoy control sample and laboratory control sample duplicate (if applicable) were recalculated for the analytes identified below using the following calculation:

% Recovery = 100 * SSC/SA    Where: SSC = Spiked sample concentration
SA = Spike added

RPD = I LCSC - LCSDC I * 2/(LCSC + LCSDC)    LCSC = Laboraotry control sample concentration  LCSDC = Laboratory control sample duplicate concentration

LCS ID: lcs 1D 580-405161

| Compound | Spike Added (ug/L) | | Spiked Sample Concentration (ug/L) | | LCS Percent Recovery | | LCSD Percent Recovery | | LCS/LCSD RPD | |
|---|---|---|---|---|---|---|---|---|---|---|
| | LCS | LCSD | LCS | LCSD | Reported | Recalc. | Reported | Recalc. | Reported | Recalculated |
| 1,1-Dichloroethene | 5.0 | 5.0 | 5.28 | 5.00 | 106 | 106 | 100 | 100 | 5 | 5 |
| Trichloroethene | | | 4.26 | 4.34 | 85 | 85 | 87 | 87 | 2 | 2 |
| Benzene | | | 5.54 | 5.20 | 111 | 111 | 104 | 104 | 6 | 6 |
| Toluene | | | 5.14 | 5.28 | 103 | 103 | 106 | 106 | 3 | 3 |
| Chlorobenzene | | | 5.29 | 5.37 | 106 | 106 | 107 | 107 | 1 | 1 |

Comments: Refer to Laboratory Control Sample findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 55171B/a

# VALIDATION FINDINGS WORKSHEET

## Sample Calculation Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260 B)

[✓ Y] N N/A Were all reported results recalculated and verified for all level IV samples?

[✓ Y] N N/A Were all recalculated results for detected target analytes agree within 10.0% of the reported results?

Concentration = $\dfrac{(A_x)(I_s)(DF)}{(A_{is})(RRF)(V_o)(\%S)}$

| | | |
|---|---|---|
| $A_x$ | = | Area of the characteristic ion (EICP) for the compound to be measured |
| $A_{is}$ | = | Area of the characteristic ion (EICP) for the specific internal standard |
| $I_s$ | = | Amount of internal standard added in nanograms (ng) |
| RRF | = | Relative response factor of the calibration standard. |
| $V_o$ | = | Volume or weight of sample pruged in milliliters (ml) or grams (g). |
| Df | = | Dilution factor. |
| %S | = | Percent solids, applicable to soils and solid matrices only. |

Example:

Sample I.D. #1 , R :

Conc. = $\dfrac{\frac{80}{435509}(10)+0.057}{0.5793}$

$= 0.10156$

| # | Sample ID | Compound | Reported Concentration (ug/L) | Calculated Concentration (ug/L) | Qualification |
|---|---|---|---|---|---|
| | #1 | R | 0.10 | 0.10156 | |
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**LDC Report#** 55171B2a_RV1

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** June 26, 2023

**Parameters:** Semivolatiles

**Validation Level:** Stage 4

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118118-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU145 | 580-118118-1 | Water | 09/19/22 |

# Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS (May 2020). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Semivolatile Organic Compounds (SVOCs) and Tentatively Identified Compounds (TICs) by Environmental Protection Agency (EPA) SW 846 Method 8270E

All sample results were subjected to Stage 4 data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met with the following exceptions:

| Sample | Analyte | Total Days From Sample Collection Until Extraction | Required Holding Time (in Days) From Sample Collection Until Extraction | Flag | A or P |
|---|---|---|---|---|---|
| HU145 | 2,4,5-Trichlorophenol<br>2,4-Dichlorophenol<br>2-Chlorophenol | 10 | 7 | UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects) | A |

## II. GC/MS Instrument Performance Check

A decafluorotriphenylphosphine (DFTPP) tune was performed at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

For analytes where average relative response factors (RRFs) were utilized, the percent relative standard deviations (%RSD) were less than or equal to 15.0%.

In the case where the laboratory used a calibration curve to evaluate the analytes, all coefficients of determination ($r^2$) were greater than or equal to 0.990.

Average relative response factors (RRF) for all analytes were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0% for all analytes with the following exceptions:

| Date | Analyte | %D | Associated Samples | Flag | A or P |
|---|---|---|---|---|---|
| 08/29/22 | Phenol | 23 | All samples in SDG 580-118118-1 | UJ (all non-detects) | A |

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all analytes with the following exceptions:

| Date | Analyte | %D | Associated Samples | Flag | A or P |
|---|---|---|---|---|---|
| 09/27/22 | 1,2,4-Trichlorobenzene<br>Dimethylphthalate<br>Pentachlorophenol<br>Hexachlorobenzene | 20.8<br>24.4<br>25.8<br>21.0 | All samples in SDG 580-118118-1 | UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects) | A |

The percent differences (%D) of the ending continuing calibration verifications (CCVs) were less than or equal to 50.0% for all analytes.

All of the continuing calibration relative response factors (RRF) were within validation criteria.

## V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

## VI. Field Blanks

No field blanks were identified in this SDG.

## VII. Surrogates

Surrogates were added to all samples as required by the method. Surrogate recoveries (%R) were not within QC limits. Using professional judgment, no data were qualified when one base or one acid surrogate %R was outside the QC limits and the %R was greater than or equal to 10%.

## VIII. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## IX. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits.

Relative percent differences (RPD) were within QC limits with the following exceptions:

| LCS ID (Associated Samples) | Analyte | RPD (Limits) | Flag | A or P |
|---|---|---|---|---|
| LCS/LCSD 580-404969 (All samples in SDG 580-118118-1) | 2,3,4,6-Tetrachlorophenol<br>2,4,6-Trichlorophenol<br>2,4-Dinitrophenol<br>Hexachlorobutadiene<br>Pentachlorophenol<br>Phenol | 48 (≤20)<br>35 (≤20)<br>68 (≤20)<br>24 (≤20)<br>61 (≤20)<br>24 (≤20) | NA | - |

## X. Field Duplicates

No field duplicates were identified in this SDG.

## XI. Internal Standards

All internal standard areas and retention times were within QC limits.

## XII. Target Analyte and Tentatively Identified Compounds Quantitation

All tentatively identified compound quantitations met validation criteria with the following exceptions:

| Sample | Analyte | Flag | A or P |
|---|---|---|---|
| All samples in SDG 580-118118-1 | All tentatively identified compounds (TIC) | NJ (all detects) | A |

## XIII. Target Analyte Identification

All target analyte identifications met validation criteria.

Manual integrations were reviewed and were considered acceptable. The laboratory provided before and after integration printouts.

## XIV. System Performance

The system performance was acceptable.

## XV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

Due to technical holding time, ICV %D, and continuing calibration %D, data were qualified as estimated in one sample.

Due to TICs, data were qualified as presumptive and estimated in one sample.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Semivolatiles - Data Qualification Summary - SDG 580-118118-1

| Sample | Analyte | Flag | A or P | Reason (Code) |
|---|---|---|---|---|
| HU145 | 2,4,5-Trichlorophenol<br>2,4-Dichlorophenol<br>2-Chlorophenol | UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects) | A | Technical holding times (h) |
| HU145 | Phenol | UJ (all non-detects) | A | Initial calibration verification (%D) (c) |
| HU145 | 1,2,4-Trichlorobenzene<br>Dimethylphthalate<br>Pentachlorophenol<br>Hexachlorobenzene | UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects)<br>UJ (all non-detects) | A | Continuing calibration (%D) (c) |
| HU145 | All tentatively identified compounds (TIC) | NJ (all detects) | A | Tentatively identified compound quantitation (v) |

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Semivolatiles - Field Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

LDC #: 55171B2a

SDG #: 580-118118-1

Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

## Stage 4

Date: 11/1/22

Page: 1 of 1

Reviewer:

2nd Reviewer:

**METHOD:** GC/MS Semivolatiles (EPA SW-846 Method 8270E)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A / SW | |
| II. | GC/MS Instrument performance check | A | |
| III. | Initial calibration/ICV | A SW | % RSD ≤ 15, r² ICV ≤ 20 |
| IV. | Continuing calibration ending | SW | CCV ≤ 20/50 |
| V. | Laboratory Blanks | A | |
| VI. | Field blanks | N | |
| VII. | Surrogate spikes | SW | |
| VIII. | Matrix spike/Matrix spike duplicates | N | cs |
| IX. | Laboratory control samples | SW | LCS ID |
| X. | Field duplicates | N | |
| XI. | Internal standards | A | |
| XII. | Target analyte quantitation | SW | |
| XIII. | Target analyte identification | A | MI |
| XIV. | System performance | A | |
| XV. | Overall assessment of data | A | |

Note:
A = Acceptable
N = Not provided/applicable
SW = See worksheet
ND = No compounds detected
R = Rinsate
FB = Field blank
D = Duplicate
TB = Trip blank
EB = Equipment blank
SB=Source blank
OTHER:

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU145 RE = Z, Q, C (2) | 580-118118-1 | Water | 09/19/22 |
| 2 | | | | |
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Notes:

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | MB 580-404969 | | | | | | |
| 2 | MB 580-405394 | | | | | | |
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| | | | | | | | |

LDC #: 55171B2a

## VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: FT

**Method:** Semivolatiles (EPA SW 846 Method 8270E)

| Validation Area | Yes | No | NA | Findings/Comments |
|---|---|---|---|---|
| ***I. Technical holding times*** | | | | |
| Were all technical holding times met? | ~~✓~~ | ✓ | | |
| Was cooler temperature criteria met? | ✓ | | | |
| ***II. GC/MS Instrument performance check*** | | | | |
| Were the DFTPP performance results reviewed and found to be within the specified criteria? | ✓ | | | |
| Were all samples analyzed within the 12 hour clock criteria? | ✓ | | | |
| ***IIIa. Initial calibration*** | | | | |
| Did the laboratory perform a 5 point calibration prior to sample analysis? | ✓ | | | |
| Were all percent relative standard deviations (%RSD) ≤ 15% and relative response factors (RRF) within method criteria? | ✓ | | | |
| Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990? | ✓ | | | |
| ***IIIb. Initial Calibration Verification*** | | | | |
| Was an initial calibration verification standard analyzed after each initial calibration for each instrument? | ✓ | | | |
| Were all percent differences (%D) ≤ 20%? | ~~✓~~ | ✓ | | |
| ***IV. Continuing calibration*** | | | | |
| Was a continuing calibration standard analyzed at least once every 12 hours for each instrument? | ✓ | | | |
| Were all percent differences (%D) ≤ 20% and relative response factors (RRF) within method criteria? Were all percent differences (%D) ≤ 50% for closing calibration verification? | | ✓ | | |
| ***V. Laboratory Blanks*** | | | | |
| Was a laboratory blank associated with every sample in this SDG? | ✓ | | | |
| Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration? | ✓ | | | |
| Was there contamination in the laboratory blanks? If yes, please see the blanks validation findings worksheet. | | ✓ | | |
| ***VI. Field blanks*** | | | | |
| Were field blanks were identified in this SDG? | | ✓ | | |
| Were target analytes detected in the field blanks? | | | ✓ | |
| ***VII. Surrogate spikes*** | | | | |
| Were all surrogate percent recovery (%R) within QC limits? | | ✓ | | |
| If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R? | | ✓ | | |
| If any percent recoveries (%R) was less than 10%, was a reanalysis performed to confirm %R ? | | | ✓ | |
| ***VIII. Matrix spike/Matrix spike duplicates*** | | | | |
| Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG? | | | ✓ | |

LDC #: 55171B2a

## VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: FT

| Validation Area | Yes | No | NA | Findings/Comments |
|---|---|---|---|---|
| Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits? | | | ✓ | |
| ***IX. Laboratory control samples*** | | | | |
| Was an LCS analyzed per extraction batch? | ✓ | | | |
| Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits? | | ✓ | | |
| ***X. Field duplicates*** | | | | |
| Were field duplicate pairs identified in this SDG? | | ✓ | | |
| Were target analytes detected in the field duplicates? | | | ✓ | |
| ***XI. Internal standards*** | | | | |
| Were internal standard area counts within -50% to +100% of the associated calibration standard? | ✓ | | | |
| Were retention times within + 30 seconds of the associated calibration standard? | ✓ | | | |
| ***XII. Target analyte quantitation*** | | | | |
| Did the laboratory LOQs/RLs meet the QAPP LOQs/RLs? | ✓ | | | |
| Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the target analyte? | ✓ | | | |
| Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation? | ✓ | | | |
| ***XIII. Target analyte identification*** | | | | |
| Were relative retention times (RRT's) within + 0.06 RRT units of the standard? | ✓ | | | |
| Did compound spectra meet specified EPA "Functional Guidelines" criteria? | ✓ | | | |
| Were chromatogram peaks verified and accounted for? | ✓ | | | |
| Were manual integrations reviewed and found acceptable? | ✓ | | | |
| Did the laboratory provide before and after integration printouts? | ✓ | | | |
| ***XIV. System performance*** | | | | |
| System performance was found to be acceptable. | ✓ | | | |
| ***XV. Overall assessment of data*** | | | | |
| Overall assessment of data was found to be acceptable. | ✓ | | | |

# VALIDATION FINDINGS WORKSHEET

**METHOD:** GC/MS SVOA

| | | | | |
|---|---|---|---|---|
| A. Phenol | CC. Dimethylphthalate | EEE. Bis(2-ethylhexyl)phthalate | GGGG. C30-Hopane | I1. Methyl methanesulfonate |
| B. Bis (2-chloroethyl) ether | DD. Acenaphthylene | FFF. Di-n-octylphthalate | HHHH. 1-Methylphenanthrene | J1. Ethyl methanesulfonate |
| C. 2-Chlorophenol ✓ DCB | EE. 2,6-Dinitrotoluene | GGG. Benzo(b)fluoranthene | IIII. 1,4-Dioxane | K1. o,o',o''-Triethylphosphorothioate |
| D. 1,3-Dichlorobenzene | FF. 3-Nitroaniline | HHH. Benzo(k)fluoranthene | JJJJ. Acetophenone | L1. n-Phenylene diamine |
| E. 1,4-Dichlorobenzene | GG. Acenaphthene | III. Benzo(a)pyrene | KKKK. Atrazine | M1. 1,4-Naphthoquinone |
| F. 1,2-Dichlorobenzene | HH. 2,4-Dinitrophenol | JJJ. Indeno(1,2,3-cd)pyrene | LLLL. Benzaldehyde | N1. N-Nitro-o-toluidine |
| G. 2-Methylphenol | II. 4-Nitrophenol | KKK. Dibenz(a,h)anthracene | MMMM. Caprolactam | O1. 1,3,5-Trinitrobenzene |
| H. 2,2'-Oxybis(1-chloropropane) | JJ. Dibenzofuran | LLL. Benzo(g,h,i)perylene | NNNN. 2,6-Dichlorophenol | P1. Pentachlorobenzene |
| I. 4-Methylphenol | KK. 2,4-Dinitrotoluene | MMM. Bis(2-Chloroisopropyl)ether | OOOO. 1,2-Diphenylhydrazine | Q1. 4-Aminobiphenyl |
| J. N-Nitroso-di-n-propylamine | LL. Diethylphthalate | NNN. Aniline | PPPP. 3-Methylphenol | R1. 2-Naphthylamine |
| K. Hexachloroethane | MM. 4-Chlorophenyl-phenyl ether | OOO. N-Nitrosodimethylamine | QQQQ. 3&4-Methylphenol | S1. Triphenylene |
| L. Nitrobenzene | NN. Fluorene | PPP. Benzoic Acid | RRRR. 4-Dimethyldibenzothiophene (4MDT) | T1. Octachlorostyrene |
| M. Isophorone | OO. 4-Nitroaniline | QQQ. Benzyl alcohol | SSSS. 2/3-Dimethyldibenzothiophene (4MDT) | U1. Famphur |
| N. 2-Nitrophenol | PP. 4,6-Dinitro-2-methylphenol | RRR. Pyridine | TTTT. 1-Methyldibenzothiophene (1MDT) | V1. 1,4-phenylenediamine |
| O. 2,4-Dimethylphenol | QQ. N-Nitrosodiphenylamine | SSS. Benzidine | UUUU.. 2,3,4,6-Tetrachlorophenol | W1. Methapyrilene |
| P. Bis(2-chloroethoxy)methane | RR. 4-Bromophenyl-phenylether | TTT. 1-Methylnaphthalene | VVVV. 1,2,4,5-Tetrachlorobenzene | X1. Pentachloroethane |
| Q. 2,4-Dichlorophenol ✓ NPT | SS. Hexachlorobenzene | UUU.Benzo(b)thiophene | WWWW.. 2-Picoline | Y1. 3,3'-Dimethylbenzidine |
| R. 1,2,4-Trichlorobenzene | TT. Pentachlorophenol | VVV.Benzonaphthothiophene | XXXX. 3-Methylcholanthrene | Z1. o-Toluidine |
| S. Naphthalene | UU. Phenanthrene | WWW.Benzo(e)pyrene | YYYY. a,a-Dimethylphenethylamine | A2. 1-Naphthylamine |
| T. 4-Chloroaniline | VV. Anthracene | XXX. 2,6-Dimethylnaphthalene | ZZZZ. Hexachloropropene | B2. 4-Aminobiphenyl |
| U. Hexachlorobutadiene | WW. Carbazole | YYY. 2,3,5-Trimethylnaphthalene | A1. N-Nitrosodiethylamine | C2. 4-Nitroquinoline-1-oxide |
| V. 4-Chloro-3-methylphenol | XX. Di-n-butylphthalate | ZZZ. Perylene | B1. N-Nitrosodi-n-butylamine | D2. Hexachloropene |
| W. 2-Methylnaphthalene | YY. Fluoranthene | AAAA. Dibenzothiophene | C1. N-Nitrosomethylethylamine | E2. Bis (2-chloro-1-methylethyl) ether |
| X. Hexachlorocyclopentadiene | ZZ. Pyrene | BBBB. Benzo(a)fluoranthene | D1. N-Nitrosomorpholine | F2. Bifenthrin |
| Y. 2,4,6-Trichlorophenol | AAA. Butylbenzylphthalate | CCCC. Benzo(b)fluorene | E1. N-Nitrosopyrrolidine | G2. Cyfluthrin |
| Z. 2,4,5-Trichlorophenol ✓ AVT | BBB. 3,3'-Dichlorobenzidine | DDDD. cis/trans-Decalin | F1. Phenacetin | H2. Cypermethrin |
| AA. 2-Chloronaphthalene | CCC. Benzo(a)anthracene | EEEE. 1,1'-Biphenyl | G1. 2-Acetylaminofluorene | I2. Permethrin (cis/trans) |
| BB. 2-Nitroaniline | DDD. Chrysene | FFFF. Retene | H1. Pronamide | J2. 5-Nitro-o-toluidine |

LDC #: 55171B2a

## VALIDATION FINDINGS WORKSHEET
## Technical Holding Times

Page: 1 of 1
Reviewer: ft

All circled dates have exceeded the technical holding times.
[✓ Y] N N/A Were all cooler temperatures within validation criteria?

**METHOD** : GC/MA BNA SW846 Method 8270 (h)

| Sample ID | Matrix | Preserved | Sampling Date | Extraction date | Analysis date | Total # of Days | Qualifier |
|---|---|---|---|---|---|---|---|
| 1 | w | | 9/19/22 | 9/29/22 | 9/29/22 | 10 | J-/UJ/A |
| | | | | | | | ND |
| | | | | | qual | Z, Q, | C |
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**TECHNICAL HOLDING TIME CRITERIA**

| | |
|---|---|
| Water: | Extracted within 7 days, analyzed within 40 days. |
| Soil: | Extracted within 14 days, analyzed within 40 days. |

LDC #: 55171B2a

# VALIDATION FINDINGS WORKSHEET

## Initial Calibration Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS BNA (EPA SW 846 Method 8270 E)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Was an initial calibration verification standard analyzed after each ICAL for each instrument?

Y [✓ N] N/A Were all %D within the validation criteria of ≤ 20% 30 % D ? c

| # | Date | Standard ID | Compound | Finding %D (Limit: ≤20.0% 30%) | Associated Samples | Qualifications |
|---|---|---|---|---|---|---|
|  | 8/29/22<br>12/33 | ICV 580-402255 | A | 23 | All | J+/UJ/A ND |
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LDC #: 55171B2a

# VALIDATION FINDINGS WORKSHEET
## Continuing Calibration

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS SVOA(EPA Method 8270 E)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Was a continuing calibration standard analyzed at least once every 12 hours of sample analysis for each instrument?

Y N N/A Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ?

Y [✓ N] N/A Were all %D and RRFs within the validation criteria of ≤20 %D and ≥0.05 RRF ?

(c)

| # | Date | Standard ID | Compound | Finding %D (Limit: <20.0%) | Finding RRF (Limit: >0.05) | Associated Samples | Qualifications |
|---|---|---|---|---|---|---|---|
| | 9/27/22 | CCV 5BG-405079 | R | 20.8 | | All | J+ /UJ/A all ND |
| | 1023 | | CC | 24.4 | | ↓ | ↓ |
| | | | TT | 25.8 | | ↓ | J+ /UJ/A |
| | | | SS | 21.0 | | All | J+ /UJ/A |
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LDC #: 55171B2a

# VALIDATION FINDINGS WORKSHEET

## Surrogate Recovery

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS BNA (EPA SW 846 Method 8270 E)

Please see qualification below for all questions answered "N". Not applicable questions are identified as "N/A".

Y [✓ N] N/A Were percent recoveries (%R) for surrogates within QC limits?

Y [✓ N] N/A If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R? (S)

Y [✓ N] [✓ N/A] If any %R was less than 10 percent, was a reanalysis performed to confirm %R?

| # | Sample ID | Surrogate | %R (Limits) | Qualifications |
|---|---|---|---|---|
| | 1 | TPH | 162 (50-134) | no qual |
| | | | ( ) | |
| | | | ( ) | |
| | MB 580-404969 | TBP | 12 (43-140) | J-/X/P all Acids |
| | | ~~PHL-d5~~ 2FP | 2 (19-119) | ↓ ↓ |
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(NBZ) = Nitrobenzene - d5
(FBP) = 2-Fluorobiphenyl
(TPH) = Terphenyl - d14
(2FP) = 2-Fluorophenol
(TBP) = 2,4,6 -Tribromophenol
(2CP) = 2-Chlorophenol - d4

LDC #: 55171B2a

# VALIDATION FINDINGS WORKSHEET
## Laboratory Control Samples (LCS)

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS BNA (Method ) 8270E

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Was a LCS required?

Y [✓ N] N/A Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits? (w)

| # | LCS/LCSD ID | Compound | LCS %R (Limits) | LCSD %R (Limits) | RPD (Limits) | Associated Samples | Qualifications |
|---|---|---|---|---|---|---|---|
| | lcsD 580- | ~~Z~~ | ~~34 (53-123)~~ | ( ) | ( ) | All | |
| | 404969 | F? Q | 26 (47-121) | ( ) | ( ) | ↓ | |
| | | ~~C~~ | ~~26 (38-117)~~ | ( ) | ( ) | | |
| | | UUUU | ( ) | ( ) | 48 (20) | | Jdet/P all ND |
| | | Y | ( ) | ( ) | 35 ( ↓ ) | | ↓ |
| | | HH | ( ) | ( ) | 68 ( ) | | |
| | | U | ( ) | ( ) | 24 ( ) | | |
| | | TT | ( ) | ( ) | 61 ( ) | | |
| | | Δ | ( ) | ( ) | 24 ( ) | | |
| | | | ( ) | ( ) | ( ) | | |
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LDC #: 55171B2a

# VALIDATION FINDINGS WORKSHEET

## Target Analyte Quantitation and Tentatively Identified Commpound

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS SVOA (EPA SW 846 Method 8270E)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

[✓ Y] N N/A Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?

[✓ Y] N N/A Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?

| # | Sample ID | Analytes | Finding | Qualifications |
|---|---|---|---|---|
| | all | all analytes reported as Tentatively Identified Compounds | | NJ/A (v) |
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Comments: See sample calculation verification worksheet for recalculations

LDC #: 55171B2a

# VALIDATION FINDINGS WORKSHEET

## Initial Calibration Calculation Verification

Page: 1 of 1
Reviewer: FT

METHOD: GCMS 8270E

The calibration factors (RRFF), average RRFF, and relative standard deviation (%RSD) were recalculated for compounds identified below using the following calculations:

RRF = (Ax)(Cis)/(Ais)(Cx)
average RRF = sum of the RRFs/number of standards
%RSD = 100 * (S/X)

Where:

Ax = Area of compound
Cx = Concentration of compound
S = Standard deviation of the RRFs
X = Mean of the RRFs
Ais = Area of associated internal standard
Cis = Concentration of internal Standard

| # | Standard ID | Calibration Date | Compound | Reported (RRF 500 std) | Recalculated (RRF500 std) | Reported AverageRRF (Initial) | Recalculated Average RRF (Initial) | Reported %RSD | Recalculated %RSD |
|---|---|---|---|---|---|---|---|---|---|
|  | ICAL | 8/29/2022 | A | 1.5842 | 1.5842 | 1.5785 | 1.5785 | 13.5 | 13.5 |
|  | TACO51 |  | U | 0.2759 | 0.2759 | 0.2869 | 0.2869 | 14.0 | 14.0 |
|  |  |  | LL | 1.3644 | 1.3644 | 1.2628 | 1.2628 | 10.6 | 10.6 |
|  |  |  | SS | 0.2259 | 0.2259 | 0.2501 | 0.2501 | 8.7 | 8.7 |
|  |  |  | BBB | 0.4468 | 0.4468 | 0.4217 | 0.4217 | 7.6 | 7.6 |
|  |  |  |  |  |  |  |  |  |  |

082922 TACO51

LDC #: 55171B2a

## VALIDATION FINDINGS WORKSHEET
## Continuing Calibration Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS BNA (EPA SW 846 Method 8270E)
The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the target analytes identified below using the following calculation:

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
RRF = $(A_x)(C_{is})/(A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
$A_x$ = Area of target analyte
$C_x$ = Concentration of target analyte
RRF = continuing calibration RRF
$A_{is}$ = Area of associated internal standard
$C_{is}$ = Concentration of internal standard

| # | Standard ID | Calibration Date | Target Analyte (Internal Standard) | Average RRF (Initial) | Reported RRF (CC) | Recalculated RRF (CC) | Reported %D | Recalculated %D |
|---|---|---|---|---|---|---|---|---|
| 1 | ccV | 9/27/22<br>1023 | A (1st IS) | 1.5785 | 1.762 | 1.762 | 11.6 | 11.6 |
| | | | U (2nd IS) | 0.2869 | 0.3433 | 0.3433 | 19.7 | 19.7 |
| | | | LL (3rd IS) | 1.2628 | 1.475 | 1.475 | 16.8 | 16.8 |
| | | | SS (4th IS) | 0.2501 | 0.2896 | 0.2896 | 15.8 | 15.8 |
| | | | BBB (5th IS) | 0.4217 | 0.4899 | 0.4899 | 16.2 | 16.2 |
| | | | (6th IS) | | | | | |
| 2 | ~~ccV~~ | ~~9/29/22~~ | ~~R~~ FT (1st IS) | ~~0.3665~~ | ~~0.4129~~ | ~~0.4129~~ | ~~12.7~~ | ~~12.7~~ |
| | | | (2nd IS) | | | | | |
| | | | (3rd IS) | | | | | |
| | | | (4th IS) | | | | | |
| | | | (5th IS) | | | | | |
| | | | (6th IS) | | | | | |
| 3 | ccV | 9/29/22<br>177 | C (1st IS) | 1.279 | 1.283 | 1.283 | 0.4 | 0.4 |
| | | | Δ (L) (2nd IS) | 1000 | 975 | 975 | 2.5 | 2.5 |
| | | | Z (L) (3rd IS) | 1000 | 886 | 886 | 11.4 | 11.4 |
| | | | (4th IS) | | | | | |
| | | | (5th IS) | | | | | |
| | | | (6th IS) | | | | | |

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 55171B2a

# VALIDATION FINDINGS WORKSHEET
## Surrogate Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS Semivolatiles (EPA SW 846 Method 8270 E)

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS * 100

Where: SF = Surrogate Found
SS = Surrogate Spiked

**Sample ID:** #1

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Nitrobenzene-d5 | 1000.0 | 911.4 | 91 | 91 | 0 |
| 2-Fluorobiphenyl | | 795.9 | 80 | 80 | |
| Terphenyl-d14 | | 1071.7 | 107 | 107 | |
| Phenol-d5 | | 268.6 | 27 | 27 | |
| 2-Fluorophenol | | 585.1 | 59 | 59 | |
| 2,4,6-Tribromophenol | ↓ | 768.9 | 77 | 77 | ↓ |

**Sample ID:**

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Nitrobenzene-d5 | | | | | |
| 2-Fluorobiphenyl | | | | | |
| Terphenyl-d14 | | | | | |
| Phenol-d5 | | | | | |
| 2-Fluorophenol | | | | | |
| 2,4,6-Tribromophenol | | | | | |

LDC #: 55171B2a

# VALIDATION FINDINGS WORKSHEET

## Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS BNA (EPA SW 846 Method 8270)E

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the target analytes identified below using the following calculation:

$$SSC = \frac{(Ax)(C_{IS})(Fv)(Df)}{(A_{is})(RRF)(Vs\ or\ Ws)(\%S/100)}$$

%Recovery = (SSC/SA)*100

RPD =(({SSCLCS - SSCLCSD} * 2) / (SSCLCS + SSCLCSD))*100

Where:
$A_x$= Area of the target analyte
$A_{is}$= Area for the specific internal standard
$C_{is}$ = Concentration of internal standard
Fv =Final volume of extract
Df= Dilution factor
RRF= Average relative response factor of the target analyte
Ws= Initial weight of the sample
%S= Percent Solid
SSC = Spiked sample concentration
LCS = Laboratory control sample
LCSD = Laboratory control sample duplicate
Vs= Initial volume of the sample

LCS/LCSD samples: lcsID 580-404969

| Compound | Spike Added (ug/L) | | Spike Concentration (ug/L) | | LCS Percent Recovery | | LCSD Percent Recovery | | LCS/LCSD RPD | |
|---|---|---|---|---|---|---|---|---|---|---|
| | LCS | LCSD | LCS | LCSD | Reported | Recalc | Reported | Recalc | Reported | Recalculated |
| Phenol | 2.0 | 2.0 | 0.659 | 0.840 | 33 | 33 | 42 | 42 | 24 | 24 |
| N-Nitroso-di-n-propylamine | NA | | | | | | | | | |
| 4-Chloro-3-methylphenol | | | | | | | | | | |
| Acenaphthene | ↓ | | | | | | | | | |
| Pentachlorophenol | 4.0 | 4.0 | 1.74 | 3.28 | 43 | 43 | 82 | 82 | 61 | 61 |
| Pyrene | ND | | | | | | | | | |
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LDC #: 55171B2a

# VALIDATION FINDINGS WORKSHEET

## Sample Calculation Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS BNA (EPA SW 846 Method 8270E)

The concentration of the sample was calculated for the target analyte identified below using the following calculation:

$$\text{Concentration} = \frac{(A_x)(I_s)(V_t)(DF)(2.0)}{(A_{is})(RRF)(V_o)(V_i)(\%S)}$$

$A_x$ = Area of the characteristic ion (EICP) for the target analyte to be measured
$A_{is}$ = Area of the characteristic ion (EICP) for the specific internal standard
$I_s$ = Amount of internal standard added in nanograms (ng)
$V_o$ = Volume or weight of sample extract in milliliters (ml) or grams (g).
$V_i$ = Volume of extract injected in microliters (ul)
$V_t$ = Volume of the concentrated extract in microliters (ul)
Df = Dilution Factor.
%S = Percent solids, applicable to soil and solid matrices only.
2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. LCS 580-404969 Δ

$$\text{Conc.} = \frac{(28303)(100.0)\quad(2)}{(5444)(1.5785)(1000)}$$

= 0.6587 ug/L

| # | Sample ID | Target Analyte | Reported Concentration (ug/L) | Calculated Concentration (ug/L) | Qualification |
|---|---|---|---|---|---|
| | LCS | Δ | 0.659 | 0.6587 | |
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**LDC Report#** 55171B2b

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** November 2, 2022

**Parameters:** Polynuclear Aromatic Hydrocarbons

**Validation Level:** Stage 4

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118118-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU145 | 580-118118-1 | Water | 09/19/22 |

## Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS (May 2020). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polynuclear Aromatic Hydrocarbons (PAHs) by Environmental Protection Agency (EPA) SW 846 Method 8270E in Selected Ion Monitoring (SIM) mode

All sample results were subjected to Stage 4 data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

## II. GC/MS Instrument Performance Check

Instrument performance check was performed at the required frequency.

All ion abundance requirements were met.

## III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

For analytes where average relative response factors (RRFs) were utilized, percent relative standard deviations (%RSD) were less than or equal to 15.0%.

In the case where the laboratory used a calibration curve to evaluate the analytes, all coefficients of determination ($r^2$) were greater than or equal to 0.990.

Average relative response factors (RRF) for all analytes were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0% for all analytes.

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all analytes.

The percent differences (%D) of the ending continuing calibration verifications (CCVs) were less than or equal to 50.0% for all analytes.

All of the continuing calibration relative response factors (RRF) were within validation criteria.

## V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

## VI. Field Blanks

No field blanks were identified in this SDG.

## VII. Surrogates

Surrogates were added to all samples as required by the method. All surrogate recoveries (%R) were within QC limits.

## VIII. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## IX. Laboratory Control Samples

Laboratory control samples (LCS) were analyzed as required by the method. Percent recoveries (%R) were within QC limits.

## X. Field Duplicates

No field duplicates were identified in this SDG.

## XI. Internal Standards

All internal standard areas and retention times were within QC limits.

## XII. Target Analyte Quantitation

All target analyte quantitations met validation criteria.

## XIII. Target Analyte Identification

All target analyte identifications met validation criteria.

## XIV. System Performance

The system performance was acceptable.

## XV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polynuclear Aromatic Hydrocarbons - Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polynuclear Aromatic Hydrocarbons - Laboratory Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polynuclear Aromatic Hydrocarbons - Field Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

LDC #: 55171B2b
SDG #: 580-118118-1
Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET
## Stage 4

Date: 10/31/22
Page: 1 of 1
Reviewer:
2nd Reviewer:

**METHOD:** GC/MS Polynuclear Aromatic Hydrocarbons (EPA SW-846 Method 8270E-SIM)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A / A | |
| II. | GC/MS Instrument performance check | A | |
| III. | Initial calibration/ICV | A / A | % RSD ≤ 15, r² ICV ≤ 20 |
| IV. | Continuing calibration | A | CCV ≤ 20/50 |
| V. | Laboratory Blanks | A | |
| VI. | Field blanks | N | |
| VII. | Surrogate spikes | A | |
| VIII. | Matrix spike/Matrix spike duplicates | N | cs |
| IX. | Laboratory control samples | A | lcs |
| X. | Field duplicates | N | |
| XI. | Internal standards | A | |
| XII. | Target analyte quantitation | A | |
| XIII. | Target analyte identification | A | M I |
| XIV. | System performance | A | |
| XV. | Overall assessment of data | A | |

| Note: | | | | |
|---|---|---|---|---|
| | A = Acceptable | ND = No compounds detected | D = Duplicate | SB=Source blank |
| | N = Not provided/applicable | R = Rinsate | TB = Trip blank | OTHER: |
| | SW = See worksheet | FB = Field blank | EB = Equipment blank | |

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU145 | 580-118118-1 | Water | 09/19/22 |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
| 7 | | | | |
| 8 | | | | |
| 9 | | | | |

Notes:

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | MB 580-404969 | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

LDC #: 55171B2b

## VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: FT

**Method:** Semivolatiles (EPA SW 846 Method 8270 E) SIM

| Validation Area | Yes | No | NA | Findings/Comments |
|---|---|---|---|---|
| ***I. Technical holding times*** | | | | |
| Were all technical holding times met? | ✓ | | | |
| Was cooler temperature criteria met? | ✓ | | | |
| ***II. GC/MS Instrument performance check*** | | | | |
| Were the DFTPP performance results reviewed and found to be within the specified criteria? | ✓ | | | |
| Were all samples analyzed within the 12 hour clock criteria? | ✓ | | | |
| ***IIIa. Initial calibration*** | | | | |
| Did the laboratory perform a 5 point calibration prior to sample analysis? | ✓ | | | |
| Were all percent relative standard deviations (%RSD) ≤ 15% and relative response factors (RRF) within method criteria? | ✓ | | | |
| Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990? | ✓ | | | |
| ***IIIb. Initial Calibration Verification*** | | | | |
| Was an initial calibration verification standard analyzed after each initial calibration for each instrument? | ✓ | | | |
| Were all percent differences (%D) ≤ 20%? | ✓ | | | |
| ***IV. Continuing calibration*** | | | | |
| Was a continuing calibration standard analyzed at least once every 12 hours for each instrument? | ✓ | | | |
| Were all percent differences (%D) ≤ 20% and relative response factors (RRF) within method criteria? Were all percent differences (%D) ≤ 50% for closing calibration verification? | ✓ | | | |
| ***V. Laboratory Blanks*** | | | | |
| Was a laboratory blank associated with every sample in this SDG? | ✓ | | | |
| Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration? | ✓ | | | |
| Was there contamination in the laboratory blanks? If yes, please see the blanks validation findings worksheet. | | ✓ | | |
| ***VI. Field blanks*** | | | | |
| Were field blanks were identified in this SDG? | | ✓ | | |
| Were target analytes detected in the field blanks? | | | ✓ | |
| ***VII. Surrogate spikes*** | | | | |
| Were all surrogate percent recovery (%R) within QC limits? | ✓ | | | |
| If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R? | | | ✓ | |
| If any percent recoveries (%R) was less than 10%, was a reanalysis performed to confirm %R ? | | | ✓ | |
| ***VIII. Matrix spike/Matrix spike duplicates*** | | | | |
| Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG? | | | ✓ | |

LDC #: 55711B2b

## VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: FT

| Validation Area | Yes | No | NA | Findings/Comments |
|---|---|---|---|---|
| Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits? | | | ✓ | |
| ***IX. Laboratory control samples*** | | | | |
| Was an LCS analyzed per extraction batch? | ✓ | | | |
| Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits? | ✓ | | | |
| ***X. Field duplicates*** | | | | |
| Were field duplicate pairs identified in this SDG? | | ✓ | | |
| Were target analytes detected in the field duplicates? | | | ✓ | |
| ***XI. Internal standards*** | | | | |
| Were internal standard area counts within -50% to +100% of the associated calibration standard? | ✓ | | | |
| Were retention times within ± 30 seconds of the associated calibration standard? | ✓ | | | |
| ***XII. Target analyte quantitation*** | | | | |
| Did the laboratory LOQs/RLs meet the QAPP LOQs/RLs? | ✓ | | | |
| Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the target analyte? | ✓ | | | |
| Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation? | ✓ | | | |
| ***XIII. Target analyte identification*** | | | | |
| Were relative retention times (RRT's) within ± 0.06 RRT units of the standard? | ✓ | | | |
| Did compound spectra meet specified EPA "Functional Guidelines" criteria? | ✓ | | | |
| Were chromatogram peaks verified and accounted for? | ✓ | | | |
| Were manual integrations reviewed and found acceptable? | ✓ | | | |
| Did the laboratory provide before and after integration printouts? | ✓ | | | |
| ***XIV. System performance*** | | | | |
| System performance was found to be acceptable. | ✓ | | | |
| ***XV. Overall assessment of data*** | | | | |
| Overall assessment of data was found to be acceptable. | ✓ | | | |

# VALIDATION FINDINGS WORKSHEET

**METHOD:** GC/MS SVOA

| | | | | |
|---|---|---|---|---|
| A. Phenol | CC. Dimethylphthalate | EEE. Bis(2-ethylhexyl)phthalate | GGGG. C30-Hopane | I1. Methyl methanesulfonate |
| B. Bis (2-chloroethyl) ether | DD. Acenaphthylene | FFF. Di-n-octylphthalate | HHHH. 1-Methylphenanthrene | J1. Ethyl methanesulfonate |
| C. 2-Chlorophenol | EE. 2,6-Dinitrotoluene | GGG. Benzo(b)fluoranthene | IIII. 1,4-Dioxane | K1. o,o',o"-Triethylphosphorothioate |
| D. 1,3-Dichlorobenzene | FF. 3-Nitroaniline | HHH. Benzo(k)fluoranthene | JJJJ. Acetophenone | L1. n-Phenylene diamine |
| E. 1,4-Dichlorobenzene | GG. Acenaphthene | III. Benzo(a)pyrene | KKKK. Atrazine | M1. 1,4-Naphthoquinone |
| F. 1,2-Dichlorobenzene | HH. 2,4-Dinitrophenol | JJJ. Indeno(1,2,3-cd)pyrene | LLLL. Benzaldehyde | N1. N-Nitro-o-toluidine |
| G. 2-Methylphenol | II. 4-Nitrophenol | KKK. Dibenz(a,h)anthracene | MMMM. Caprolactam | O1. 1,3,5-Trinitrobenzene |
| H. 2,2'-Oxybis(1-chloropropane) | JJ. Dibenzofuran | LLL. Benzo(g,h,i)perylene | NNNN. 2,6-Dichlorophenol | P1. Pentachlorobenzene |
| I. 4-Methylphenol | KK. 2,4-Dinitrotoluene | MMM. Bis(2-Chloroisopropyl)ether | OOOO. 1,2-Diphenylhydrazine | Q1. 4-Aminobiphenyl |
| J. N-Nitroso-di-n-propylamine | LL. Diethylphthalate | NNN. Aniline | PPPP. 3-Methylphenol | R1. 2-Naphthylamine |
| K. Hexachloroethane | MM. 4-Chlorophenyl-phenyl ether | OOO. N-Nitrosodimethylamine | QQQQ. 3&4-Methylphenol | S1. Triphenylene |
| L. Nitrobenzene | NN. Fluorene | PPP. Benzoic Acid | RRRR. 4-Dimethyldibenzothiophene (4MDT) | T1. Octachlorostyrene |
| M. Isophorone | OO. 4-Nitroaniline | QQQ. Benzyl alcohol | SSSS. 2/3-Dimethyldibenzothiophene (4MDT) | U1. Famphur |
| N. 2-Nitrophenol | PP. 4,6-Dinitro-2-methylphenol | RRR. Pyridine | TTTT. 1-Methyldibenzothiophene (1MDT) | V1. 1,4-phenylenediamine |
| O. 2,4-Dimethylphenol | QQ. N-Nitrosodiphenylamine | SSS. Benzidine | UUUU.. 2,3,4,6-Tetrachlorophenol | W1. Methapyrilene |
| P. Bis(2-chloroethoxy)methane | RR. 4-Bromophenyl-phenylether | TTT. 1-Methylnaphthalene | VVVV. 1,2,4,5-Tetrachlorobenzene | X1. Pentachloroethane |
| Q. 2,4-Dichlorophenol | SS. Hexachlorobenzene | UUU.Benzo(b)thiophene | WWWW.. 2-Picoline | Y1. 3,3'-Dimethylbenzidine |
| R. 1,2,4-Trichlorobenzene | TT. Pentachlorophenol | VVV.Benzonaphthothiophene | XXXX. 3-Methylcholanthrene | Z1. o-Toluidine |
| S. Naphthalene | UU. Phenanthrene | WWW.Benzo(e)pyrene | YYYY. a,a-Dimethylphenethylamine | A2. 1-Naphthylamine |
| T. 4-Chloroaniline | VV. Anthracene | XXX. 2,6-Dimethylnaphthalene | ZZZZ. Hexachloropropene | B2. 4-Aminobiphenyl |
| U. Hexachlorobutadiene | WW. Carbazole | YYY. 2,3,5-Trimethylnaphthalene | A1. N-Nitrosodiethylamine | C2. 4-Nitroquinoline-1-oxide |
| V. 4-Chloro-3-methylphenol | XX. Di-n-butylphthalate | ZZZ. Perylene | B1. N-Nitrosodi-n-butylamine | D2. Hexachloropene |
| W. 2-Methylnaphthalene | YY. Fluoranthene | AAAA. Dibenzothiophene | C1. N-Nitrosomethylethylamine | E2. Bis (2-chloro-1-methylethyl) ether |
| X. Hexachlorocyclopentadiene | ZZ. Pyrene | BBBB. Benzo(a)fluoranthene | D1. N-Nitrosomorpholine | F2. Bifenthrin |
| Y. 2,4,6-Trichlorophenol | AAA. Butylbenzylphthalate | CCCC. Benzo(b)fluorene | E1. N-Nitrosopyrrolidine | G2. Cyfluthrin |
| Z. 2,4,5-Trichlorophenol | BBB. 3,3'-Dichlorobenzidine | DDDD. cis/trans-Decalin | F1. Phenacetin | H2. Cypermethrin |
| AA. 2-Chloronaphthalene | CCC. Benzo(a)anthracene | EEEE. 1,1'-Biphenyl | G1. 2-Acetylaminofluorene | I2. Permethrin (cis/trans) |
| BB. 2-Nitroaniline | DDD. Chrysene | FFFF. Retene | H1. Pronamide | J2. 5-Nitro-o-toluidine |

LDC #: 55171B2b

# VALIDATION FINDINGS WORKSHEET
## Initial Calibration Calculation Verification

Page: 1 of 1
Reviewer: FT

METHOD: GCMS 8270D SIM

The calibration factors (RRFF), average RRFF, and relative standard deviation (%RSD) were recalculated for compounds identified below using the following calculations:

RRF = (Ax)(Cis)/(Ais)(Cx)
average RRF = sum of the RRFs/number of standards
%RSD = 100 * (S/X)

Where:
Ax = Area of compound
Cx = Concentration of compound
S = Standard deviation of the RRFs
X = Mean of the RRFs
Ais = Area of associated internal standard
Cis = Concentration of internal Standard

| # | Standard ID | Calibration Date | Compound | Reported (RRF 500ug/Lstd) | Recalculated (RRF 500ug/L std) | Reported AverageRRF (Initial) | Recalculated Average RRF (Initial) | Reported %RSD | Recalculated %RSD |
|---|---|---|---|---|---|---|---|---|---|
|  | ICAL | 3/24/2022 | S | 1.0387 | 1.0387 | 1.0388 | 1.0388 | 6.0 | 6.0 |
|  |  |  | GG | 1.3013 | 1.3013 | 1.2744 | 1.2744 | 3.0 | 3.0 |
|  | SEA101 |  | UU | 1.2092 | 1.2092 | 1.1719 | 1.1719 | 6.2 | 6.2 |
|  |  |  | DDD | see curve |  |  |  |  |  |
|  |  |  | III | 1.1663 | 1.1663 | 1.0795 | 1.0795 | 10.9 | 10.9 |

032422 SEA101

**LDC #: 55171B2b** **Validation Findings Worksheet** **Page:__1__ of __1__**

## Initial Calibration Calculation Verification

**FT**

Method: 8270E SIM

| Calibration Date | Instrument/Column | Compound | Standard | (Y) Response | (X) Conc. | (X^2) Conc. |
|---|---|---|---|---|---|---|
| 3/24/2022 | SEA101 | DDD | 1 | 0.016 | 0.01 | 0.0001 |
| | | | 2 | 0.034 | 0.02 | 0.0004 |
| | | | 3 | 0.068 | 0.05 | 0.0025 |
| | | | 4 | 0.151 | 0.1 | 0.01 |
| | | | 5 | 0.311 | 0.2 | 0.04 |
| | | | 6 | 0.750 | 0.5 | 0.25 |
| | | | 7 | 1.533 | 1 | 1 |
| | | | 8 | 2.995 | 2 | 4 |
| | | | 9 | 6.952 | 5 | 25 |
| | | | 10 | 13.807 | 10 | 100 |
| | | | 11 | 27.760 | 20 | 400 |
| | | | 12 | 65.375 | 50 | 2500 |
| | | | 13 | 118.050 | 100 | 10000 |

| **Regression Output** | **Calculated** | | ***Reported*** | |
|---|---|---|---|---|
| Constant | c | 0.0037 | c | 0.2105 |
| Std Err of Y Est | | | | |
| R Squared | | 0.9999906 | | 0.9970000 |
| Degrees of Freedom | | | | |
| | a | b | a | b |
| X Coefficient(s) | 1.43267E+00 | -2.5210E-03 | 1.47230E+00 | -3.1000E-05 |
| Std Err of Coef. | | | | |
| | | | | |
| Correlation Coefficient | | 0.999995 | | |
| Coefficient of Determination (r^2) | | 0.999991 | | |

LDC #: 55171B2b

# VALIDATION FINDINGS WORKSHEET
## Continuing Calibration Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS BNA (EPA SW 846 Method 8270 E)
The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the target analytes identified below using the following calculation:

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
RRF = $(A_x)(C_{is})/(A_{is})(C_x)$

Where:
ave. RRF = initial calibration average RRF
RRF = continuing calibration RRF
$A_x$ = Area of target analyte
$A_{is}$ = Area of associated internal standard
$C_x$ = Concentration of target analyte
$C_{is}$ = Concentration of internal standard

| # | Standard ID | Calibration Date | Target Analyte (Internal Standard) | | Average RRF (Initial) | Reported | Recalculated | Reported | Recalculated |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | RRF (CC) | RRF (CC) | %D | %D |
| 1 | ccv | 9/27/22<br>1027 | S | (1st IS) | 1.0388 | 0.9148 | 0.9148 | 11.9 | 11.9 |
| | | | GG | (2nd IS) | 1.2744 | 1.193 | 1.193 | 6.4 | 6.4 |
| | | | uu | (3rd IS) | 1.1719 | 0.998 | 0.998 | 14.8 | 14.8 |
| | | | DDD (Q) | (4th IS) | 500 | 464 | 464 | 7.3 | 7.3 |
| | | | III | (5th IS) | 1.0795 | 1.034 | 1.034 | 4.2 | 4.2 |
| | | | | (6th IS) | | | | | |
| 2 | | | | (1st IS) | | | | | |
| | | | | (2nd IS) | | | | | |
| | | | | (3rd IS) | | | | | |
| | | | | (4th IS) | | | | | |
| | | | | (5th IS) | | | | | |
| | | | | (6th IS) | | | | | |
| 3 | | | | (1st IS) | | | | | |
| | | | | (2nd IS) | | | | | |
| | | | | (3rd IS) | | | | | |
| | | | | (4th IS) | | | | | |
| | | | | (5th IS) | | | | | |
| | | | | (6th IS) | | | | | |

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 55171B2b

# VALIDATION FINDINGS WORKSHEET

## Surrogate Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS Semivolatiles (EPA SW 846 Method 8270 E)

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS * 100

Where: SF = Surrogate Found
SS = Surrogate Spiked

**Sample ID:** #1

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| ~~Nitrobenzene-d5~~ W - d10 | 1000 | 689.1 | 69 | 69 | 0 |
| 2-Fluorobiphenyl YY - d10 | ↓ | 695.9 | 101 | 101 | ↓ |
| Terphenyl-d14 TPH - d14 | ↓ | 1146.1 | 115 | 115 | ↓ |
| Phenol-d5 | | | | | |
| 2-Fluorophenol | | | | | |
| ~~2,4,6-Tribromophenol~~ | | | | | |

**Sample ID:**

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Nitrobenzene-d5 | | | | | |
| 2-Fluorobiphenyl | | | | | |
| Terphenyl-d14 | | | | | |
| Phenol-d5 | | | | | |
| 2-Fluorophenol | | | | | |
| 2,4,6-Tribromophenol | | | | | |

LDC #: 55171B2b

# VALIDATION FINDINGS WORKSHEET

## Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS BNA (EPA SW 846 Method 8270)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the target analytes identified below using the following calculation:

$$SSC = \frac{(A_X)(C_{IS})(Fv)(Df)}{(A_{IS})(RRF)(Vs \text{ or } Ws)(\%S/100)}$$

%Recovery = (SSC/SA)*100

RPD =((({SSCLCS - SSCLCSD} * 2) / (SSCLCS + SSCLCSD))*100

Where:
$A_X$= Area of the target analyte
$A_{IS}$= Area for the specific internal standard
$C_{IS}$ = Concentration of internal standard
Fv =Final volume of extract
Df= Dilution factor
RRF= Average relative response factor of the target analyte
Ws= Initial weight of the sample
%S= Percent Solid
SSC = Spiked sample concentration
LCS = Laboratory control sample
LCSD = Laboratory control sample duplicate
Vs= Initial volume of the sample

LCS/LCSD samples: LCS 580-404969

| Compound | Spike Added (ug/L) | | Spike Concentration (ug/L) | | LCS Percent Recovery | | LCSD Percent Recovery | | LCS/LCSD RPD | |
|---|---|---|---|---|---|---|---|---|---|---|
| ▨ | LCS | LCSD | LCS | LCSD | Reported | Recalc | Reported | Recalc | Reported | Recalculated |
| Phenol | | | | | | | | | | |
| N-Nitroso-di-n-propylamine | | | | | | | | | | |
| 4-Chloro-3-methylphenol | | | | | | | | | | |
| Acenaphthene | 2.0 | NA | 1.67 | NA | 83 | 83 | | | | |
| Pentachlorophenol | | | | | | | | | | |
| Pyrene | 2.0 | NA | 1.87 | NA | 94 | 94 | NA | | | |
| | | | | | | | | | | |
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LDC #: 55171B2b

# VALIDATION FINDINGS WORKSHEET
## Sample Calculation Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS BNA (EPA SW 846 Method 8270E)

The concentration of the sample was calculated for the target analyte identified below using the following calculation:

$$\text{Concentration} = \frac{(A_x)(I_s)(V_t)(DF)(2.0)}{(A_{is})(RRF)(V_o)(V_i)(\%S)}$$

$A_x$ = Area of the characteristic ion (EICP) for the target analyte to be measured

$A_{is}$ = Area of the characteristic ion (EICP) for the specific internal standard

$I_s$ = Amount of internal standard added in nanograms (ng)

$V_o$ = Volume or weight of sample extract in milliliters (ml) or grams (g).

$V_i$ = Volume of extract injected in microliters (ul)

$V_t$ = Volume of the concentrated extract in microliters (ul)

Df = Dilution Factor.

%S = Percent solids, applicable to soil and solid matrices only.

2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. LCS 580-404969 GG

$$\text{Conc.} = \frac{(261946)(100.0)(2)}{(24632)(1.2744)(1000)}$$

$$= 1.67 \text{ ug/L}$$

| # | Sample ID | Target Analyte | Reported Concentration ( ug/L ) | Calculated Concentration ( ug/L ) | Qualification |
|---|---|---|---|---|---|
| | LCS | GG | 1.67 | 1.67 | |
| | | | | | |
| | | | | | |
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**LDC Report#** 55171B4b

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** December 7, 2022

**Parameters:** Metals

**Validation Level:** Stage 4

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118118-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU145 | 580-118118-1 | Water | 09/19/22 |

## Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 2: Data Validation Procedure for Metals by ICP-OES (May 2020). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Calcium, Magnesium, Manganese, Potassium, and Sodium by Environmental Protection Agency (EPA) SW 846 Method 6010D

All sample results were subjected to Stage 4 data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition.

All technical holding time requirements were met.

## II. Instrument Calibration

Initial and continuing calibrations were performed as required by the method.

The initial calibration verification (ICV) and continuing calibration verification (CCV) standards were within QC limits.

## III. ICP Interference Check Sample Analysis

The frequency of interference check sample (ICS) analysis was met. All criteria were within QC limits.

## IV. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

| Blank ID | Analyte | Maximum Concentration | Associated Samples |
|---|---|---|---|
| ICB/CCB | Calcium<br>Magnesium<br>Manganese<br>Potassium | 0.0919 mg/L<br>0.0636 mg/L<br>0.0023 mg/L<br>0.186 mg/L | All samples in SDG 580-118118-1 |

Data qualification by the laboratory blanks was based on the maximum contaminant concentration in the laboratory blanks in the analysis of each analyte. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated laboratory blanks.

## V. Field Blanks

No field blanks were identified in this SDG.

## VI. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## VII. Duplicate Sample Analysis

The laboratory has indicated that there were no duplicate (DUP) analyses specified for the samples in this SDG, and therefore duplicate analyses were not performed for this SDG.

## VIII. Serial Dilution

Serial dilution was not performed for this SDG.

## IX. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

## X. Field Duplicates

No field duplicates were identified in this SDG.

## XI. Target Analyte Quantitation

All target analyte quantitation met validation criteria.

## XII. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

## Red Hill Bulk Storage Facility, CTO 18F0126
## Metals - Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Bulk Storage Facility, CTO 18F0126
## Metals - Laboratory Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Bulk Storage Facility, CTO 18F0126
## Metals - Field Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

LDC #: 55171B4b
SDG #: 580-118118-1
Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

## Stage 4

Date: 12/05/22
Page: 1 of 1
Reviewer: NC
2nd Reviewer:

**METHOD:** Metals (EPA SW-846 Method 6010D)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A/A | |
| II. | Instrument Calibration | A | |
| III. | ICP Interference Check Sample (ICS) Analysis | A | |
| IV. | Laboratory Blanks | SW | |
| V. | Field Blanks | N | |
| VI. | Matrix Spike/Matrix Spike Duplicates | N | |
| VII. | Duplicate sample analysis | N | |
| VIII. | Serial Dilution | N | |
| IX. | Laboratory control samples | A | LCS/LCSD |
| X. | Field Duplicates | N | |
| XI. | Target Analyte Quantitation | A | |
| XII. | Overall Assessment of Data | A | |

Note:
A = Acceptable
N = Not provided/applicable
SW = See worksheet
ND = No compounds detected
R = Rinsate
FB = Field blank
D = Duplicate
TB = Trip blank
EB = Equipment blank
SB=Source blank
OTHER:

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU145 | 580-118118-1 | Water | 09/19/22 |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
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| 7 | | | | |
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| 10 | | | | |
| 11 | | | | |
| 12 | | | | |
| 13 | | | | |
| 14 | | | | |

LDC #: 55171B4b

VALIDATION FINDINGS CHECKLIST

Reviewer:NC

| | | | | |
|---|---|---|---|---|
| METHOD: Trace Metals (EPA SW 846 Methods 6010/6020/7000) | | | | |
| **Validation Area** | **Yes** | **No** | **NA** | **Comments** |
| **I. Technical holding times** | | | | |
| Were all technical holding times met? | Yes | | | |
| Were all water samples preserved to a pH of <2. | Yes | | | |
| **II. ICP-MS Tune** | | | | |
| Were mass resolutions within 0.1 amu for all isotopes in the tuning solution? | | | NA | |
| Were %RSDs of isoptoes in the tuning solution ≤5%? | | | NA | |
| **III. Calibration** | | | | |
| Were all instruments calibrated daily? | Yes | | | |
| Were the proper standards used? | Yes | | | |
| Were all initial and continuing calibration verifications within the 90-110% (80-120% for mercury) QC limits? | Yes | | | |
| Were the low level standard checks within 70-130%? | Yes | | | |
| Were all initial calibration correlation coefficients within limits as specifed by the method? | Yes | | | |
| **IV. Blanks** | | | | |
| Was a method blank associated with every sample in this SDG? | Yes | | | |
| Was there contamination in the method blanks? | | No | | |
| Was there contamination in the initial and continuing calibration blanks? | Yes | | | |
| **V. Interference Check Sample** | | | | |
| Were the interference check samples performed daily? | Yes | | | |
| Were the AB solution recoveries within 80-120%? | Yes | | | |
| **VI. Matrix Spike/Matrix Spike Duplicates/Laboratory Duplicates** | | | | |
| Were MS/MSD recoveries within the QC limits? (If the sample concentration exceeded the spike concentration by a factor of 4, no action was taken.) | | | NA | |
| Were the MS/MSD or laboratory duplicate relative percent differences (RPDs) within the QC limits? | | | NA | |
| **VII. Laboratory Control Samples** | | | | |
| SDG? | Yes | | | |

| | | | | |
|---|---|---|---|---|
| Were the LCS recoveries and RPDs (if applicable) within QC limits? | Yes | | | |
| METHOD: Trace Metals (EPA SW 846 Methods 6010/6020/7000) | | | | |
| **Validation Area** | **Yes** | **No** | **NA** | **Comments** |
| **VIII. Internal Standards** | | | | |
| Were all percent recoveries within the 30-120% (60-125% for EPA Method 200.8) QC limits? | | | NA | |
| If the recoveries were outside the limits, was a reanalysis performed? | | | NA | |
| **IX. Serial Dilution** | | | | |
| Were all percent differences <10%? | | | NA | |
| Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data. | | | NA | |
| **X. Target Analyte Quantitation** | | | | |
| Were all reporting limits adjusted to reflect sample dilutions? | Yes | | | |
| Were all soil samples dry weight corrected? | | | NA | |
| **XI. Overall Assessment of Data** | | | | |
| Was the overall assessment of the data found to be acceptable? | Yes | | | |
| **XII. Field Duplicates** | | | | |
| Were field duplicates identifed in this SDG? | | No | | |
| Were target analytes detected in the field duplicates? | | | NA | |
| **XIII. Field Blanks** | | | | |
| Were field blanks identified in this SDG? | | No | | |
| Were target analytes detected in the field blanks? | | | NA | |

LDC #: 55171B4b

VALIDATION FINDINGS WORKSHEET

Sample Specific Element Reference

Page 1 of 1

Reviewer:NC

All elements are applicable to each sample as noted below.

| Sample ID | Target Analyte List |
|---|---|
| 1 | Ca, Mg, Mn, K, Na |
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**Analysis Method**

| | |
|---|---|
| ICP | Ca, Mg, Mn, K, Na |

LDC #: 55171B4b

# VALIDATION FINDINGS WORKSHEET

## Laboratory Blank Contamination (PB/ICB/CCB)

Page 1 of 1
Reviewer:NC

METHOD: Trace Metals (EPA SW 846 Methods 6010/6020/7000)
Soil preparation factor applied (if applicable):

Sample Concentration, unless otherwise noted: mg/L

Associated Samples: 1

| | | | | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Analyte** | **PB (mg/L)** | **Maximum ICB/CCB (mg/L)** | **Action Level (ug/L)** | | | | | | | | | |
| Ca | | 0.0919 | 459.5 | | | | | | | | | |
| Mg | | 0.0636 | 318 | | | | | | | | | |
| Mn | | 0.0023 | 11.5 | | | | | | | | | |
| K | | 0.186 | 930 | | | | | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

Comments: The listed analyte concentrtaion is the highest ICB or CCB detected in the analysis. The action level, when applicable, is established at 5X the highest ICB, CCB, or PB concentration.

# VALIDATION FINDINGS CHECKLIST

## Calibration Calculation Verification

Reviewer:NC

METHOD: Trace Metals (EPA SW 846 Methods 6010/6020/7000)

An intial calibration verification (ICV), continuing calibration verification (CCV), low level calibration check (LLCC), and interference check sample (ICSAB) percent recovery (%R) was recalculated for each type of analysis using the following formula:

%R = (Found/True) x 100

Found = concentration of each analyte measured in the analysis
True = concentration of each analyte in the source

| Standard ID | Type of Analysis | Element | Found (mg/L) | True (mg/L) | Recalculated %R | Reported %R | Acceptable (Y/N) |
|---|---|---|---|---|---|---|---|
| ICV 580-406044/9 | ICP | Ca | 38.98 | 40 | 97.45 | 97 | Y |
| CCV 580-406044/26 | ICP | Na | 100.9 | 100 | 100.9 | 101 | Y |
| ICVL 580-406044/11 | ICP | Mn | 0.0206 | 0.02 | 103 | 103 | Y |
| ICSAB 580-406044/13 | ICP | Mg | 507 | 500 | 101.4 | 101 | Y |

# VALIDATION FINDINGS CHECKLIST

## Quality Control Sample Recalculations

METHOD: Trace Metals (EPA SW 846 Methods 6010/6020/7000)

Percent recoveries (%R) for the laboratory control sample (LCS), matrix spike (MS), and post digestion spike (PDS) were recalculated using the following formula:

%R = (Found/True) x 100

Found = concentration of each analyte measured in the analysis. For the MS calculation, Found = SSR (Spiked Sample Result) - SR (Sample Result)

True = concentration of each analyte in the source

The sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

RPD = (Absolute value(S-D)x 200) / (S+D)

S = Original sample concentration

D = Duplicate sample concentration

The serial dilution percent difference (%D) was recalculated using the following formula.

%D = (Absolute value (I - SDR)) x 100 / (I)

I = Initial sample result

SDR = Serial dilution result (with a 5x dilution applied)

| Sample ID | Type of Analysis | Element | Found/S/I | True/D/SDR | Recalculated %R/RPD/%D | Reported %R/RPD/%D | Acceptable (Y/N) |
|---|---|---|---|---|---|---|---|
| LCS 580-405900/23-A | LCS | Mn | 1024 | 1000 | 102.4 | 102 | Y |

## VALIDATION FINDINGS CHECKLIST
## Sample Calculation Verification

METHOD: Trace Metals (EPA SW 846 Methods 6010/6020/7000)
Analytes were recalculated and verified using the following equation:
Concentration = (Result from raw data x Final volume x Dilution factor) / (Initial volume)

| Sample ID | Analyte | Raw Data (ug/L) | Dilution | Initial Weight/ Volume (mL) | Final Volume (mL) | Reported Result (ug/L) | Recalculated Result (ug/L) | Acceptable (Y/N) |
|---|---|---|---|---|---|---|---|---|
| 1 | K | 3515 | 1 | 50 | 50 | 3500 | 3515 | Y |

**LDC Report#** 55171B6

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** December 7, 2022

**Parameters:** Wet Chemistry

**Validation Level:** Stage 4

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118118-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU145 | 580-118118-1 | Water | 09/19/22 |

## Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), and the DoD General Validation Guidelines (November 2019). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Alkalinity by Standard Method 2320B
Dissolved Organic Carbon by Environmental Protection Agency (EPA) SW 846 Method 9060A
Nitrate/Nitrite as Nitrogen by EPA Method 353.2
Total Organic Carbon by EPA SW 846 Method 9060A

All sample results were subjected to Stage 4 data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

J+ (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation.

J- (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation.

J (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate.

U (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s).

UJ (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate.

X (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended.

NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition.

All technical holding time requirements were met.

## II. Initial Calibration

All criteria for the initial calibration of each method were met.

## III. Continuing Calibration

Continuing calibration frequency and analysis criteria were met for each method when applicable.

## IV. Laboratory Blanks

Laboratory blanks were analyzed as required by the methods. No contaminants were found in the laboratory blanks.

## V. Field Blanks

No field blanks were identified in this SDG.

## VI. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## VII. Duplicate Sample Analysis

The laboratory has indicated that there were no duplicate (DUP) analyses specified for the samples in this SDG, and therefore duplicate analyses were not performed for this SDG.

## VIII. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the methods. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

## IX. Field Duplicates

No field duplicates were identified in this SDG.

## X. Target Analyte Quantitation

All target analyte quantitations were acceptable.

## XI. Overall Assessment of Data

The analysis was conducted within all specifications of the methods. No results were rejected or recommended for exclusion in this SDG.

## Red Hill Bulk Storage Facility, CTO 18F0126
## Wet Chemistry - Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Bulk Storage Facility, CTO 18F0126
## Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Bulk Storage Facility, CTO 18F0126
## Wet Chemistry - Field Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

LDC #: 55171B6
SDG #: 580-118118-1
Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

## Stage 4

Date: 12/05/22
Page: 1 of 1
Reviewer: NC
2nd Reviewer:

**METHOD: (Analyte)** Alkalinity (SM2320B), DOC (EPA SW-846 Method 9060A), Nitrate/Nitrite-N (EPA Method 353.2), TOC (EPA SW-846 Method 9060A)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A/A | |
| II | Initial calibration | A | |
| III. | Calibration verification | A | |
| IV | Laboratory Blanks | A | |
| V | Field blanks | N | |
| VI. | Matrix Spike/Matrix Spike Duplicates | N | |
| VII. | Duplicate sample analysis | N | |
| VIII. | Laboratory control samples | A | LCS/LCSD |
| IX. | Field duplicates | N | |
| X. | Target Analyte Quantitation | A | |
| XI. | Overall assessment of data | A | |

| | | | | |
|---|---|---|---|---|
| Note: | A = Acceptable | ND = No compounds detected | D = Duplicate | SB=Source blank |
| | N = Not provided/applicable | R = Rinsate | TB = Trip blank | OTHER: |
| | SW = See worksheet | FB = Field blank | EB = Equipment blank | |

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU145 | 580-118118-1 | Water | 09/19/22 |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
| 7 | | | | |
| 8 | | | | |
| 9 | | | | |
| 10 | | | | |
| 11 | | | | |
| 12 | | | | |
| 13 | | | | |

Reviewer: NC

| METHOD: Inorganics | | | | |
|---|---|---|---|---|
| **Validation Area** | **Yes** | **No** | **NA** | **Comments** |
| **I. Technical holding times** | | | | |
| Were all technical holding times met? | Yes | | | |
| **II. Calibration** | | | | |
| Were all instruments calibrated at the required frequency? | Yes | | | |
| Were the proper number of standards used? | Yes | | | |
| Were all initial and continuing calibration verifications within the QC limits? | Yes | | | |
| Were all initial calibration correlation coefficients within limits as specifed by the method? | Yes | | | |
| Were balance checks performed as required? | | | NA | |
| **III. Blanks** | | | | |
| Was a method blank associated with every sample in this SDG? | Yes | | | |
| Was there contamination in the method blanks? | | No | | |
| Was there contamination in the initial and continuing calibration blanks? | | No | | |
| **IV. Matrix Spike/Matrix Spike Duplicates/Laboratory Duplicates** | | | | |
| Were MS/MSD recoveries within the QC limits? (If the sample concentration exceeded the spike concentration by a factor of 4, no action was taken.) | | | NA | |
| Were the MS/MSD or laboratory duplicate relative percent differences (RPDs) within the QC limits? | | | NA | |
| **V. Laboratory Control Samples** | | | | |
| Was a LCS analyzed for each batch in the SDG? | Yes | | | |
| Were the LCS recoveries and RPDs (if applicable) within QC limits? | Yes | | | |
| **X. Sample Result Verification** | | | | |
| Were all reporting limits adjusted to reflect sample dilutions? | Yes | | | |
| Were all soil samples dry weight correcteo | | | NA | |
| **XI. Overall Assessment of Data** | | | | |
| Was the overall assessment of the data found to be acceptable? | Yes | | | |
| **XII. Field Duplicates** | | | | |
| Were field duplicates identifed in this SDG? | | No | | |
| Were target analytes detected in the field duplicates? | | | NA | |
| **XIII. Field Blanks** | | | | |
| Were field blanks identified in this SDG? | | No | | |
| Were target analytes detected in the field blanks? | | | NA | |

LDC #: 55171B6

# VALIDATION FINDINGS WORKSHEET

## Sample Specific Element Reference

Page 1 of 1

Reviewer: NC

All elements are applicable to each sample as noted below.

| **Sample ID** | **Target Analyte List** |
|---|---|
| 1 | Alkalinity, Bicarbonate Alkalinity as CaCO3, Carbonate Alkalinity as CaCO3, DOC, TOC, Nitrate /Nitrite as N |
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LDC #: 55171B6

# VALIDATION FINDINGS CHECKLIST

## Initial and Continuing Calibration Calculation Verification

Page 1 of 1
Reviewer: NC

METHOD: Inorganics

The correlation coefficient (r) for the calibration of Nitrate Nitrite as N were recalculated.
Calibration date:
An initial or continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:
%R = (Found/True) x 100
Found = concentration of each analyte measured in the analysis of the ICV or CCV solution
True = concentration of each analyte in the ICV or CCV source

| Type of Analysis | Analyte | Standard | Concentration (mg/L) | Area | Recalculated r or $r^2$ | Reported r or $r^2$ | Acceptable (Y/N) |
|---|---|---|---|---|---|---|---|
| Initial Calibration | Nitrate Nitrite as N | s1 | 4 | 792795.2 | 0.999928 | 0.99992801 | Y |
| | | s2 | 3 | 593734.8 | | | |
| | | s3 | 1 | 202233.7 | | | |
| | | s4 | 0.5 | 106606.1 | | | |
| | | s5 | 0.2 | 42425.2 | | | |
| | | s6 | 0.1 | 20513.4 | | | |
| | | s7 | 0 | 138.800 | | | |
| | | s8 | | | | | |
| | | s9 | | | | | |
| | | s10 | | | | | |
| | | s11 | | | | | |
| | | s12 | | | | | |

| Type of Analysis | Analyte | Found (mg/L) | True (mg/L) | | Recalculated r or $r^2$ | Reported r or $r^2$ | Acceptable (Y/N) |
|---|---|---|---|---|---|---|---|
| ICV 580-405526/1 | Alkalinity | 103.233333 | 100 | | 103.233333 | 103 | Y |
| CCV 280-588735/15 | TOC | 24.85 | 25 | | 99.4 | 99 | Y |
| CCV 280-588744/15 | DOC | 25.254 | 25 | | 101.016 | 101 | Y |

LDC #: 55171B6

# VALIDATION FINDINGS CHECKLIST

## Quality Control Sample Recalculations

Page 1 of 1
Reviewer: NC

METHOD: Inorganics

Percent recoveries (%R) for the laboratory control sample (LCS) and matrix spike (MS) were recalcuated using the following formula:
%R = (Found/True) x 100
Found = concentration of each analyte measured in the analysis. For the MS calculation, Found = SSR (Spiked Sample Result) - SR (Sample Result)
True = concentration of each analyte in the source
The sample and duplicate relative percent difference (RPD) was recalculated using the following formula:
RPD = (Absolute value(S-D)x 200) / (S+D)
S = Original sample concentration
D = Duplicate sample concentration

| Sample ID | Type of Analysis | Element | Found/S | True/D | Recalculated %R/RPD | Reported %R/RPD | Acceptable (Y/N) |
|---|---|---|---|---|---|---|---|
| LCS 580-405526/2 | LCS | Alkalinity | 92625 | 100000 | 92.625 | 93 | Y |
| LCS 280-588735/3 | LCS | TOC | 24230 | 25000 | 96.92 | 97 | Y |
| LCS 280-588744/14 | LCS | DOC | 25278 | 25000 | 101.112 | 101 | Y |
| LCS 410-299872/176 | LCS | Nitrate Nitrite as N | 2490 | 2500 | 99.6 | 100 | Y |

LDC #: 55171B6

# VALIDATION FINDINGS CHECKLIST

## Sample Calculation Verification

Page 1 of 1
Reviewer: NC

METHOD: Inorganics

Analytes were recalculated and verified using the following equation:

Concentration = (Result from raw data x Final volume x Dilution factor) / (Percent solids (if applicable) x Initial weight or volume)

| Sample ID | Analyte | Raw Data (ug/L) | Dilution | Initial Volume (mL) | Final Volume (mL) | Reported Result (ug/L) | Recalculated Result (ug/L) | Acceptable (Y/N) |
|---|---|---|---|---|---|---|---|---|
| 1 | Alkalinity | 101491.6667 | 1 | 30 | 30 | 100000 | 101491.6667 | Y |
| 1 | TOC | -66.45 | 1 | 20 | 20 | 800U | -66.45 | Y |
| 1 | DOC | 1018.785 | 1 | 20 | 20 | 1000 | 1018.785 | Y |
| 1 | Nitrate Nitrite as N | 346 | 1 | 1 | 1 | 350 | 346 | Y |

**LDC Report#** 55171B7

# Laboratory Data Consultants, Inc.
# Data Validation Report

| | |
|---|---|
| **Project/Site Name:** | Red Hill Oily Waste Disposal Facility, CTO 18F0176 |
| **LDC Report Date:** | November 2, 2022 |
| **Parameters:** | Gasoline Range Organics |
| **Validation Level:** | Stage 2B & 4 |
| **Laboratory:** | Eurofins, Tacoma, WA |
| **Sample Delivery Group (SDG):** | 580-118118-1 |

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU145** | 580-118118-1** | Water | 09/19/22 |
| HU144 | 580-118118-2 | Water | 09/19/22 |

**Indicates sample underwent Stage 4 validation

## Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS (May 2020). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following methods:

Gasoline Range Organics by Environmental Protection Agency (EPA) SW 846 Method 8260 and CADOHS LUFT Method

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results. Samples appended with a double asterisk on the cover page were subjected to Stage 4 data validation, which is comprised of the QC summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

## II. GC/MS Instrument Performance Check

A bromofluorobenzene (BFB) tune was performed at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the methods.

A curve fit, based on the initial calibration, was established for quantitation. The coefficient of determination ($r^2$) was greater than or equal to 0.990.

Average relative response factors (RRF) were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0%.

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0%.

The percent differences (%D) of the ending continuing calibration verifications (CCVs) were less than or equal to 20.0%.

All of the continuing calibration relative response factors (RRF) were within validation criteria.

## V. Laboratory Blanks

Laboratory blanks were analyzed as required by the methods. No contaminants were found in the laboratory blanks.

## VI. Field Blanks

Sample HU144 was identified as a trip blank. No contaminants were found.

## VII. Surrogates

Surrogates were added to all samples as required by the methods. All surrogate recoveries (%R) were within QC limits.

## VIII. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## IX. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

## X. Field Duplicates

No field duplicates were identified in this SDG.

## XI. Internal Standards

All internal standard areas and retention times were within QC limits.

## XII. Target Analyte Quantitation

All target analyte quantitations met validation criteria for samples which underwent Stage 4 validation. Raw data were not reviewed for Stage 2B validation.

## XIII. Target Analyte Identification

All target analyte identifications met validation criteria for samples which underwent Stage 4 validation. Raw data were not reviewed for Stage 2B validation.

## XIV. System Performance

The system performance was acceptable for samples which underwent Stage 4 validation. Raw data were not reviewed for Stage 2B validation.

## XV. Overall Assessment of Data

The analysis was conducted within all specifications of the methods. No results were rejected or recommended for exclusion in this SDG.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Gasoline Range Organics - Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Gasoline Range Organics - Laboratory Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Gasoline Range Organics - Field Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

LDC #: 55171B7

SDG #: 580-118118-1

Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

## Stage 2B/4

Date: 10/31/22

Page: 1 of 1

Reviewer:

2nd Reviewer:

**METHOD:** GC/MS Gasoline Range Organics (EPA SW-846 Method 8260/CADOHS LUFT Method)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A/A | |
| II. | GC/MS Instrument performance check | A | |
| III. | Initial calibration/ICV | A/A | $r^2$ ICV ≤ 20 |
| IV. | Continuing calibration pending | A | CCV ≤ 20/20 |
| V. | Laboratory Blanks | A | |
| VI. | Field blanks | ND | TB = 2 |
| VII. | Surrogate spikes | A | |
| VIII. | Matrix spike/Matrix spike duplicates | N | cs |
| IX. | Laboratory control samples | A | LCS/D |
| X. | Field duplicates | N | |
| XI. | Internal standards | A | |
| XII. | Target analyte quantitation | A | Not reviewed for Stage 2B validation. |
| XIII. | Target analyte identification | A | Not reviewed for Stage 2B validation. |
| XIV. | System performance | A | Not reviewed for Stage 2B validation. |
| XV. | Overall assessment of data | A | |

Note:

| | | | |
|---|---|---|---|
| A = Acceptable | ND = No compounds detected | D = Duplicate | SB=Source blank |
| N = Not provided/applicable | R = Rinsate | TB = Trip blank | OTHER: |
| SW = See worksheet | FB = Field blank | EB = Equipment blank | |

** Indicates sample underwent Stage 4 validation

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU145** | 580-118118-1** | Water | 09/19/22 |
| 2 | HU144 TB | 580-118118-2 | Water | 09/19/22 |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
| 7 | | | | |
| 8 | | | | |
| 9 | | | | |

Notes:

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | MB 580-405701 | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

LDC #: 55171B7

# VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: FT

**Method:** Volatiles (EPA SW 846 Method 8260 / CA DoHS LUFT)

| Validation Area | Yes | No | NA | Findings/Comments |
|---|---|---|---|---|
| ***I. Technical holding times*** | | | | |
| Were all technical holding times met? | ✓ | | | |
| Was cooler temperature criteria met? | ✓ | | | |
| ***II. GC/MS Instrument performance check*** | | | | |
| Were the BFB performance results reviewed and found to be within the specified criteria? | ✓ | | | |
| Were all samples analyzed within the 12 hour clock criteria? | ✓ | | | |
| ***IIIa. Initial calibration*** | | | | |
| Did the laboratory perform a 5 point calibration prior to sample analysis? | ✓ | | | |
| Were all percent relative standard deviations (%RSD) ≤ 15% and relative response factors (RRF) within method criteria? | | | ✓ | |
| Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990? | ✓ | | | |
| ***IIIb. Initial Calibration Verification*** | | | | |
| Was an initial calibration verification standard analyzed after each initial calibration for each instrument? | ✓ | | | |
| Were all percent differences (%D) ≤ 20%? | ✓ | | | |
| ***IV. Continuing calibration*** | | | | |
| Was a continuing calibration standard analyzed at least once every 12 hours for each instrument? | ✓ | | | |
| Were all percent differences (%D) ≤ 20% and relative response factors (RRF) within method criteria? Were all percent differences (%D) ≤ ~~50%~~ 20 in the ending CCV? | ✓ | | | |
| ***V. Laboratory Blanks*** | | | | |
| Was a laboratory blank associated with every sample in this SDG? | ✓ | | | |
| Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration? | ✓ | | | |
| Was there contamination in the laboratory blanks? | | ✓ | | |
| ***VI. Field blanks*** | | | | |
| Were field blanks were identified in this SDG? | ✓ | | | |
| Were target analytes detected in the field blanks? | | ✓ | | |
| ***VII. Surrogate spikes*** | | | | |
| Were all surrogate percent recovery (%R) within QC limits? | ✓ | | | |
| If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R outside of criteria? | | | ✓ | |
| ***VIII. Matrix spike/Matrix spike duplicates*** | | | | |
| Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG? | | | ✓ | |
| Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits? | | | ✓ | |

LDC #: 55171B7

## VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: FT

| | | | | |
|---|---|---|---|---|
| | | | | |
| ***IX. Laboratory control samples*** | | | | |
| Was an LCS analyzed per analytical batch? | ✓ | | | |
| Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits? | ✓ | | | |
| ***X. Field duplicates*** | | | | |
| Were field duplicate pairs identified in this SDG? | | ✓ | | |
| Were target analytes detected in the field duplicates? | | | ✓ | |
| ***XI. Internal standards*** | | | | |
| Were internal standard area counts within -50% to +100% of the associated calibration standard? | ✓ | | | |
| Were retention times within ± 30 seconds of the associated calibration standard? | ✓ | | | |
| ***XII. Target analyte quantitation*** | | | | |
| Did the laboratory LOQs/RLs meet the QAPP LOQs/RLs? | ✓ | | | |
| Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the target analyte? | ✓ | | | |
| Were target analyte quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation? | ✓ | | | |
| ***XIII. Target analyte identification*** | | | | |
| Were relative retention times (RRT's) within ± 0.06 RRT units of the standard? | ✓ | | | |
| Did analyte spectra meet specified EPA "Functional Guidelines" criteria? | ✓ | | | |
| Were chromatogram peaks verified and accounted for? | ✓ | | | |
| Were manual integrations reviewed and found acceptable? | ✓ | | | |
| Did the laboratory provide before and after integration printouts? | | | ✓ | |
| ***XIV. System performance*** | | | | |
| System performance was found to be acceptable. | ✓ | | | |
| ***XV. Overall assessment of data*** | | | | |
| Overall assessment of data was found to be acceptable. | ✓ | | | |

# TARGET COMPOUND WORKSHEET

## METHOD: VOA

| | | | | |
|---|---|---|---|---|
| A. Chloromethane | AA. Tetrachloroethene | AAA. 1,3,5-Trimethylbenzene | AAAA. Ethyl tert-butyl ether | A1. 1,3-Butadiene |
| B. Bromomethane | BB. 1,1,2,2-Tetrachloroethane | BBB. 4-Chlorotoluene | BBBB. tert-Amyl methyl ether | B1. Hexane |
| C. Vinyl choride | CC. Toluene | CCC. tert-Butylbenzene | CCCC. 1-Chlorohexane | C1. Heptane |
| D. Chloroethane | DD. Chlorobenzene | DDD. 1,2,4-Trimethylbenzene | DDDD. Isopropyl alcohol | D1. Propylene |
| E. Methylene chloride | EE. Ethylbenzene | EEE. sec-Butylbenzene | EEEE. Acetonitrile | E1. Freon 11 |
| F. Acetone | FF. Styrene | FFF. 1,3-Dichlorobenzene | FFFF. Acrolein | F1. Freon 12 |
| G. Carbon disulfide | GG. Xylenes, total | GGG. p-Isopropyltoluene | GGGG. Acrylonitrile | G1. Freon 113 |
| H. 1,1-Dichloroethene | HH. Vinyl acetate | HHH. 1,4-Dichlorobenzene | HHHH. 1,4-Dioxane | H1. Freon 114 |
| I. 1,1-Dichloroethane | II. 2-Chloroethylvinyl ether | III. n-Butylbenzene | IIII. Isobutyl alcohol | I1. 2-Nitropropane |
| J. 1,2-Dichloroethene, total | JJ. Dichlorodifluoromethane | JJJ. 1,2-Dichlorobenzene | JJJJ. Methacrylonitrile | J1. Dimethyl disulfide |
| K. Chloroform | KK. Trichlorofluoromethane | KKK. 1,2,4-Trichlorobenzene | KKKK. Propionitrile | K1. 2,3-Dimethyl pentane |
| L. 1,2-Dichloroethane | LL. Methyl-tert-butyl ether | LLL. Hexachlorobutadiene | LLLL. Ethyl ether | L1. 2,4-Dimethyl pentane |
| M. 2-Butanone | MM. 1,2-Dibromo-3-chloropropane | MMM. Naphthalene | MMMM. Benzyl chloride | M1. 3,3-Dimethyl pentane |
| N. 1,1,1-Trichloroethane | NN. Methyl ethyl ketone | NNN. 1,2,3-Trichlorobenzene | NNNN. Iodomethane | N1. 2-Methylpentane |
| O. Carbon tetrachloride | OO. 2,2-Dichloropropane | OOO. 1,3,5-Trichlorobenzene | OOOO.1,1-Difluoroethane | O1. 3-Methylpentane |
| P. Bromodichloromethane | PP. Bromochloromethane | PPP. trans-1,2-Dichloroethene | PPPP. Tetrahydrofuran | P1. 3-Ethylpentane |
| Q. 1,2-Dichloropropane | QQ. 1,1-Dichloropropene | QQQ. cis-1,2-Dichloroethene | QQQQ. Methyl acetate | Q1. 2,2-Dimethylpentane |
| R. cis-1,3-Dichloropropene | RR. Dibromomethane | RRR. m,p-Xylenes | RRRR. Ethyl acetate | R1. 2,2,3- Trimethylbutane |
| S. Trichloroethene | SS. 1,3-Dichloropropane | SSS. o-Xylene | SSSS. Cyclohexane | S1. 2,2,4-Trimethylpentane |
| T. Dibromochloromethane | TT. 1,2-Dibromoethane | TTT. 1,1,2-Trichloro-1,2,2-trifluoroethane | TTTT. Methyl cyclohexane | T1. 2-Methylhexane |
| U. 1,1,2-Trichloroethane | UU. 1,1,1,2-Tetrachloroethane | UUU. 1,2-Dichlorotetrafluoroethane | UUUU. Allyl chloride | U1. Nonanal |
| V. Benzene | VV. Isopropylbenzene | VVV. 4-Ethyltoluene | VVVV. Methyl methacrylate | V1. 2-Methylnaphthalene |
| W. trans-1,3-Dichloropropene | WW. Bromobenzene | WWW. Ethanol | WWWW. Ethyl methacrylate | W1. Methanol |
| X. Bromoform | XX. 1,2,3-Trichloropropane | XXX. Di-isopropyl ether | XXXX. cis-1,4-Dichloro-2-butene | X1. 1,2,3-Trimethylbenzene |
| Y. 4-Methyl-2-pentanone | YY. n-Propylbenzene | YYY. tert-Butanol | YYYY. trans-1,4-Dichloro-2-butene | Y1. 2-Propanol |
| Z. 2-Hexanone | ZZ. 2-Chlorotoluene | ZZZ. tert-Butyl alcohol | ZZZZ. Pentachloroethane | Z1. |

**LDC#: 55171B7**

# VALIDATION FINDINGS WORKSHEET
## Initial Calibration Calculation Verification

**Page:__1____of__1___**
**Reviewer:____FT_____**

**Method: GRO C6-C12**

| Calibration Date | System | Compound | Standard | (Y) Response | (X) Concentration |
|---|---|---|---|---|---|
| 9/22/2022 | SEA102 | GRO (C6-C12) | 1 | 14.1175 | 5 |
| | | | 2 | 21.508 | 10 |
| | | | 3 | 46.095 | 25 |
| | | | 4 | 88.545 | 50 |
| | | | 5 | 188.51 | 100 |
| | | | 6 | 847.65 | 500 |
| | | | | 1635.3 | 1000 |
| | | | 7 | 2222.7 | 1500 |
| | | | 8 | 3585.66 | 2600 |

**Regression Output** ***Reported***

| | | |
|---|---|---|
| Constant | *59.665151* | *60.640000* |
| Std Err of Y Est | | |
| R Squared | *0.994955* | *0.992000* |
| Degrees of Freedom | | |
| | | |
| X Coefficient(s) | *1.401226* | *1.599700* |
| Std Err of Coef. | | |
| | | |
| Correlation Coefficient | *0.997474* | |
| Coefficient of Determination (r^2) | *0.994955* | *0.992000* |

092122 SEA102 C6 C12 Linear

LDC #: 55171B7

# VALIDATION FINDINGS WORKSHEET
## Continuing Calibration Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260/LAFT

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the analytes identified below using the following calculation:

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
RRF = $(A_x)(C_{is})/(A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
RRF = continuing calibration RRF
$A_x$ = Area of compound, $A_{is}$ = Area of associated internal standard
$C_x$ = Concentration of compound, $C_{is}$ = Concentration of internal standard

| # | Standard ID | Calibration Date | Compound (Reference internal Standard) | Average RRF (initial) | Reported RRF (CC) | Recalculated RRF (CC) | Reported %D | Recalculated %D |
|---|---|---|---|---|---|---|---|---|
| 1 | ccV<br>SEA102 | 10/1/22<br>1451 | GRO C6-C12 (1st internal standard) | 1.00 | 1.18 | 1.18 | 18 | 18 |
| | | | (2nd internal standard) | | | | | |
| | | | (3rd internal standard) | | | | | |
| | | | (4th internal standard) | | | | | |
| 2 | | | (1st internal standard) | | | | | |
| | | | (2nd internal standard) | | | | | |
| | | | (3rd internal standard) | | | | | |
| | | | (4th internal standard) | | | | | |
| 3 | | | (1st internal standard) | | | | | |
| | | | (2nd internal standard) | | | | | |
| | | | (3rd internal standard) | | | | | |
| | | | (4th internal standard) | | | | | |
| 4 | | | (1st internal standard) | | | | | |
| | | | (2nd internal standard) | | | | | |
| | | | (3rd internal standard) | | | | | |
| | | | (4th internal standard) | | | | | |

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 55171B7

# VALIDATION FINDINGS WORKSHEET
## Surrogate Results Verification

Page: 1 of 1
Reviewer: FT
2nd reviewer:

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260B)

The percent recoveries (%R) of surrogates were recalculated for the analytes identified below using the following calculation:

% Recovery: SF/SS * 100

Where: SF = Surrogate Found
SS = Surrogate Spiked

**Sample ID:** #1

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Dibromofluoromethane | | | | | |
| 1,2-Dichloroethane-d4 | | | | | |
| Toluene-d8 | | | | | |
| Bromofluorobenzene | 10.0 | 11.3 | 113 | 113 | 0 |

**Sample ID:**

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Dibromofluoromethane | | | | | |
| 1,2-Dichloroethane-d4 | | | | | |
| Toluene-d8 | | | | | |
| Bromofluorobenzene | | | | | |

**Sample ID:**

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Dibromofluoromethane | | | | | |
| 1,2-Dichloroethane-d4 | | | | | |
| Toluene-d8 | | | | | |
| Bromofluorobenzene | | | | | |

**Sample ID:**

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Dibromofluoromethane | | | | | |
| 1,2-Dichloroethane-d4 | | | | | |
| Toluene-d8 | | | | | |
| Bromofluorobenzene | | | | | |

**Sample ID:**

| | Surrogate Spiked | Surrogate Found | Percent Recovery Reported | Percent Recovery Recalculated | Percent Difference |
|---|---|---|---|---|---|
| Dibromofluoromethane | | | | | |
| 1,2-Dichloroethane-d4 | | | | | |
| Toluene-d8 | | | | | |
| Bromofluorobenzene | | | | | |

LDC #: 55171B7

# VALIDATION FINDINGS WORKSHEET
## Laboratory Control Sample Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS VOA (EPA Method 8260/LUFT

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratoy control sample and laboratory control sample duplicate (if applicable) were recalculated for the analytes identified below using the following calculation:

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration
SA = Spike added

RPD = I LCSC - LCSDC I * 2/(LCSC + LCSDC) LCSC = Laboraotry control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS ID: LCS ID 580-405701

| Compound | Spike Added (ug/L) | | Spiked Sample Concentration (ug/L) | | LCS Percent Recovery | | LCSD Percent Recovery | | LCS/LCSD RPD | |
|---|---|---|---|---|---|---|---|---|---|---|
| ▨ | LCS | LCSD | LCS | LCSD | Reported | Recalc. | Reported | Recalc. | Reported | Recalculated |
| GRO C6-C12<br>~~1,1-Dichloroethene~~ | 1000 | 1000 | 1000 | 941 | 100 | 100 | 94 | 94 | 6 | 6 |
| ~~Trichloroethene~~ | | | | | | | | | | |
| ~~Benzene~~ | | | | | | | | | | |
| ~~Toluene~~ | | | | | | | | | | |
| ~~Chlorobenzene~~ | | | | | | | | | | |

Comments: Refer to Laboratory Control Sample findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 55171B7

# VALIDATION FINDINGS WORKSHEET
## Sample Calculation Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS VOA (EPA SW 846 Method 8260 LUFT

[✓ Y] N N/A Were all reported results recalculated and verified for all level IV samples?

[✓ Y] N N/A Were all recalculated results for detected target analytes agree within 10.0% of the reported results?

Concentration = $\frac{(A_x)(I_s)(DF)}{(A_{is})(RRF)(V_o)(\%S)}$

$A_x$ = Area of the characteristic ion (EICP) for the compound to be measured

$A_{is}$ = Area of the characteristic ion (EICP) for the specific internal standard

$I_s$ = Amount of internal standard added in nanograms (ng)

RRF = Relative response factor of the calibration standard.

$V_o$ = Volume or weight of sample pruged in milliliters (ml) or grams (g).

Df = Dilution factor.

%S = Percent solids, applicable to soils and solid matrices only.

Example:

Sample I.D. lcs 580-405701 GRO C6-C12

$$\text{Conc.} = \frac{\left(\frac{61602557}{370789}(10) - 60.640\right)}{(1.5997)}$$

1000.66 ug/L

| # | Sample ID | Compound | Reported Concentration (ug/L) | Calculated Concentration (ug/L) | Qualification |
|---|---|---|---|---|---|
| | lcs | GRO C6-C12 | 1000 | 1000.66 | |
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**LDC Report#** 55171B51

# Laboratory Data Consultants, Inc.
# Data Validation Report

**Project/Site Name:** Red Hill Oily Waste Disposal Facility, CTO 18F0176

**LDC Report Date:** November 2, 2022

**Parameters:** Methane

**Validation Level:** Stage 2B & 4

**Laboratory:** Eurofins, Tacoma, WA

**Sample Delivery Group (SDG):** 580-118118-1

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU145** | 580-118118-1** | Water | 09/19/22 |
| HU144 | 580-118118-2 | Water | 09/19/22 |

**Indicates sample underwent Stage 4 validation

# Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), the DoD General Validation Guidelines (November 2019), and the U.S. Department of Defense (DoD) Data Validation Guidelines Module 4: Data Validation Procedure for Organic Analysis by GC (March 2021). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Methane by Method RSK-175

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results. Samples appended with a double asterisk on the cover page were subjected to Stage 4 data validation, which is comprised of the QC summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

## II. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0%.

Retention time windows were established as required by the method.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0%.

## III. Continuing Calibration

Continuing calibration was performed at required frequencies.

The percent differences (%D) were less than or equal to 20.0%.

The percent differences (%D) of the ending continuing calibration verifications (CCVs) were less than or equal to 20.0%.

Retention times of all analytes in the calibration standards were within the established retention time windows.

## IV. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

## V. Field Blanks

Sample HU144 was identified as a trip blank. No contaminants were found.

## VI. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## VII. Laboratory Control Samples

Laboratory control samples (LCS) were analyzed as required by the method. Percent recoveries (%R) were within QC limits.

## VIII. Field Duplicates

No field duplicates were identified in this SDG.

## IX. Target Analyte Quantitation

All target analyte quantitations met validation criteria for samples which underwent Stage 4 validation. Raw data were not reviewed for Stage 2B validation.

## X. Target Analyte Identification

All target analyte identifications met validation criteria for samples which underwent Stage 4 validation. Raw data were not reviewed for Stage 2B validation.

Manual integrations were reviewed and were considered acceptable. The laboratory provided before and after integration printouts.

## XI. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Methane - Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Methane - Laboratory Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Methane - Field Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

LDC #: 55171B51 **VALIDATION COMPLETENESS WORKSHEET** Date: 10/31/22
SDG #: 580-118118-1 Stage 2B/4 Page: 1 of 1
Laboratory: Eurofins, Tacoma, WA
Reviewer:
2nd Reviewer:

**METHOD:** GC Methane (Method RSK-175)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A / A | |
| II. | Initial calibration/ICV | A A | % RSD / ICV ≤ 20 |
| III. | Continuing calibration ending | A | ccv ≤ 20/20 |
| IV. | Laboratory Blanks | A | |
| V. | Field blanks | ND | TB = ~~ND~~ 2 |
| VI. | Surrogate spikes | A | |
| VII. | Matrix spike/Matrix spike duplicates | N | CS |
| VIII. | Laboratory control samples | A | LCS |
| IX. | Field duplicates | N | |
| X. | Target analyte quantitation | A | Not reviewed for Stage 2B validation. MI |
| XI. | Target analyte identification | A | Not reviewed for Stage 2B validation. |
| XII | Overall assessment of data | A | |

| Note: | | | | |
|---|---|---|---|---|
| | A = Acceptable | ND = No compounds detected | D = Duplicate | SB=Source blank |
| | N = Not provided/applicable | R = Rinsate | TB = Trip blank | OTHER: |
| | SW = See worksheet | FB = Field blank | EB = Equipment blank | |

** Indicates sample underwent Stage 4 validation

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU145** | 580-118118-1** | Water | 09/19/22 |
| 2 | HU144 TB | 580-118118-2 | Water | 09/19/22 |
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Notes:

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|---|---|---|---|---|---|---|---|
| | MB 410-301674 | | | | | | |
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LDC #: 55171Bs)

## VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: FT

**Method:** ✓ GC __ HPLC

| Validation Area | Yes | No | NA | Findings/Comments |
|---|---|---|---|---|
| ***I. Technical holding times*** | | | | |
| Were all technical holding times met? | ✓ | | | |
| Was cooler temperature criteria met? | ✓ | | | |
| ***IIa. Initial calibration*** | | | | |
| Did the laboratory perform a 5 point calibration prior to sample analysis? | ✓ | | | |
| Were all percent relative standard deviations (%RSD) < 20%? | ✓ | | | |
| Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990? | | | ✓ | |
| Were the RT windows properly established? | ✓ | | | |
| ***IIb. Initial calibration verification*** | | | | |
| Was an initial calibration verification standard analyzed after each initial calibration for each instrument? | ✓ | | | |
| Were all percent differences (%D) ≤ 20%? | ✓ | | | |
| ***III. Continuing calibration*** | | | | |
| Was a continuing calibration analyzed daily? | ✓ | | | |
| Were all percent differences (%D) ≤ 20%? | ✓ | | | |
| Were all the retention times within the acceptance windows? | ✓ | | | |
| ***IV. Laboratory Blanks*** | | | | |
| Was a laboratory blank associated with every sample in this SDG? | ✓ | | | |
| Was a laboratory blank analyzed for each matrix and concentration? | ✓ | | | |
| Was there contamination in the laboratory blanks? | | ✓ | | |
| ***V. Field Blanks*** | | | | |
| Were field blanks identified in this SDG? | ✓ | | | |
| Were target analytes detected in the field blanks? | | ✓ | | |
| ***VI. Surrogate spikes*** | | | | |
| Were all surrogate percent recovery (%R) within the QC limits? | ✓ | | | |
| If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R? | | | ✓ | |
| If any %R was less than 10 percent, was a reanalysis performed to confirm %R? | | | ✓ | |
| ***VII. Matrix spike/Matrix spike duplicates*** | | | | |
| Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG? | | | ✓ | |
| Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits? | | | ✓ | |
| ***VIII. Laboratory control samples*** | | | | |
| Was an LCS analyzed per analytical or extraction batch? | ✓ | | | |
| Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits? | ✓ | | | |

LDC #: 55171B5 )

## VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: FT

| Validation Area | Yes | No | NA | Findings/Comments |
|---|---|---|---|---|
| ***IX. Field duplicates*** | | | | |
| Were field duplicate pairs identified in this SDG? | | ✓ | | |
| Were target analytes detected in the field duplicates? | | | ✓ | |
| ***X. Target analyte quantitation*** | | | | |
| Did the laboratory LOQs/RLs meet the QAPP LOQs/RLs? | ✓ | | | |
| Were analyte quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation? | ✓ | | | |
| ***XI. Target analyte identification*** | | | | |
| Were the retention times of reported detects within the RT windows? | ✓ | | | |
| Were manual integrations reviewed and found acceptable? | ✓ | | | |
| Did the laboratory provide before and after integration printouts? | ✓ | | | |
| ***XIII. Overall assessment of data*** | | | | |
| Overall assessment of data was found to be acceptable. | ✓ | | | |

LDC #: 55171B51

# VALIDATION FINDINGS WORKSHEET
## Initial Calibration Calculation Verification

Page: 1 of 1
Reviewer: FT
2nd Reviewer: ________

**METHOD:** [✓ GC] HPLC________

The calibration factors (CF) and relative standard deviation (%RSD) were recalculated using the following calculations:

CF = A/C
Average CF = sum of the CF/number of standards
%RSD = 100 * (S/X)

Where: A = Area of compound
C = Concentration of compound
S = Standard deviation of calibration factors
X = Mean of calibration factors

| | | | | Reported | Recalculated | Reported | Recalculated | Reported | Recalculated |
|---|---|---|---|---|---|---|---|---|---|
| # | Standard ID | Calibration Date | Compound | CF ( 59.8 std) | CF ( 59.8 std) | CF (initial) | CF (intial) | %RSD | %RSD |
| 1 | ICAL<br>HP Plot | 7/19/22 | Methane | 1756426 | 1756426 | 1668929.44 | 1668929.44 | 4.3 | 4.3 |
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Comments: Refer to Initial Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 55171B51

# VALIDATION FINDINGS WORKSHEET
## Continuing Calibration Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC ✓ HPLC ____

The percent difference (%D) of the initial calibration average Calibration Factors (CF) and the continuing calibration CF were recalculated for the target analytes identified below using the following calculation:

% Difference = 100 * (ave. CF -CF)/ave.CF

Where: ave. CF = initial calibration average CF
CF = continuing calibration CF
A = Area of target analyte
C = Concentration of target analyte

| # | Standard ID | Calibration Date | Target Analyte | Average CF(Ical)/ CCV Conc. | Reported CF/ Conc. CCV | Recalculated CF/ Conc. CCV | Reported %D | Recalculated %D |
|---|---|---|---|---|---|---|---|---|
| 1 | ccv | 9/30/22<br>0827 | Methane | 1668929 | 1786201 | 1786201 | 7.0 | 7.0 |
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Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 5517/B5/

# VALIDATION FINDINGS WORKSHEET

## Surrogate Results Verification

Page: 1 of 1

Reviewer: FT

**METHOD:** [✓ GC] __ HPLC

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS * 100

Where: SF = Surrogate Found
SS = Surrogate Spiked

**Sample ID:** #1

| Surrogate | Column/Detector | Surrogate Spiked | Surrogate Found | Percent Recovery | Percent Recovery | Percent Difference |
|---|---|---|---|---|---|---|
| | | | | Reported | Recalculated | |
| Propene | | 20 | 19.8 | 99 | 99 | 0 |
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**Sample ID:**

| Surrogate | Column/Detector | Surrogate Spiked | Surrogate Found | Percent Recovery | Percent Recovery | Percent Difference |
|---|---|---|---|---|---|---|
| | | | | Reported | Recalculated | |
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| | Surrogate Compound | | Surrogate Compound | | Surrogate Compound | | Surrogate Compound | | Surrogate Compound |
|---|---|---|---|---|---|---|---|---|---|
| A | Chlorobenzene (CBZ) | G | Octacosane | M | Benzo(e)Pyrene | S | 1-Chloro-3-Nitrobenzene | Y | Tetrachloro-m- xylene |
| B | 4-Bromofluorobenzene (BFB) | H | Ortho-Terphenyl | N | Terphenyl-D14 | T | 3,4-Dinitrotoluene | Z | 2-Bromonaphthalene |
| C` | a,a,a-Trifluorotoluene | I | Fluorobenzene (FBZ) | O | Decachlorobiphenyl (DCB) | U | Tripentyltin | AA | Chloro-octadecane |
| D | Bromochlorobenene | J | n-Triacontane | P | 1-methylnaphthalene | V | Tri-n-propyltin | BB | 2,4-Dichlorophenylacetic acid |
| E | 1,4-Dichlorobutane | K | Hexacosane | Q | Dichlorophenyl Acetic Acid (DCAA) | W | Tributyl Phosphate | CC | 2,5-Dibromotoluene |
| F | 1,4-Difluorobenzene (DFB) | L | Bromobenzene | R | 4-Nitrophenol | X | Triphenyl Phosphate | | |

LDC #: 55171B51

# VALIDATION FINDINGS WORKSHEET

## Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** [✓ **GC**] __**HPLC**

The percent recoveries (%R) and relative percent differences (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the target analytes identified below using the following calculation:

%Recovery = 100 * (SSC/SA)
RPD =((({SSCLCS - SSCLCSD} * 2) / (SSCLCS + SSCLCSD))*100

Where SSC = Spiked sample concentration
LCS = Laboratory Control Sample
SA = Spike added
LCSD = Laboratory Control Sample duplicate

**LCS/LCSD** samples: LCS 410-301674

| Compound | Spike Added (ug/L) | | Spike Sample Concentration (ug/L) | | LCS Percent Recovery | | LCSD Percent Recovery | | LCS/LCSD RPD | |
|---|---|---|---|---|---|---|---|---|---|---|
| | LCS | LCSD | LCS | LCSD | Reported | Recalc. | Reported | Recalc. | Reported | Recalc. |
| Methane | 59.8 | NA | 66.7 | NA | 112 | 112 | | | | |
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Comments:

LDC #: 5517/B5/

# VALIDATION FINDINGS WORKSHEET
## Sample Calculation Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** [✓ GC] __ HPLC

The concentration of the sample was calculated for the target analyte identified below using the following calculation:

$$\text{Concentration} = \frac{(A)(Fv)(Df)}{(RF)(Vs \text{ or } Ws)(\%S/100)}$$

A= Area or height of the target analyte to be measured
Fv= Final Volume of extract
Df= Dilution Factor
RF= Average response factor of the target analyte In the initial calibration
Vs= Initial volume of the sample
Ws= Initial weight of the sample
%S= Percent Solid

Example:

Sample ID. LCS 410 - 301674 Methane

$$\text{Concentration} = \frac{(111234316)}{(1668929)} =$$

= 66.65 ug/L

| # | Sample ID | Target analyte | Reported Concentrations ( ug/L ) | Recalculated Results Concentrations ( ug/L ) | Qualifications |
|---|---|---|---|---|---|
| | LCS | Methane | ~~P2~~ F7 | 66.65 | |
| | | | 66.7 | | |
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Comments:

**LDC Report#** 55171B21

# Laboratory Data Consultants, Inc.
# Data Validation Report

| | |
|---|---|
| **Project/Site Name:** | Red Hill Oily Waste Disposal Facility, CTO 18F0176 |
| **LDC Report Date:** | November 2, 2022 |
| **Parameters:** | Polychlorinated Dioxins/Dibenzofurans |
| **Validation Level:** | Stage 4 |
| **Laboratory:** | Eurofins, Tacoma, WA |
| **Sample Delivery Group (SDG):** | 580-118118-1 |

| Sample Identification | Laboratory Sample Identification | Matrix | Collection Date |
|---|---|---|---|
| HU145 | 580-118118-1 | Water | 09/19/22 |

## Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Site Assessment Work Plan, Red Hill Oily Waste Disposal Facility, Pearl Harbor HI FISC Site 22, Joint Base Pearl Harbor-Hickam, Oahu, Hawaii (February 2021), the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.3 (2019), and the DoD General Validation Guidelines (November 2019). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polychlorinated Dioxins/Dibenzofurans by Environmental Protection Agency (EPA) SW 846 Method 8290A

All sample results were subjected to Stage 4 data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

| | |
|---|---|
| J+ | (Estimated, High Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying high bias, due to non-conformances discovered during data validation. |
| J- | (Estimated, Low Bias): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated, displaying low bias, due to non-conformances discovered during data validation. |
| J | (Estimated, Bias Indeterminate): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation. Bias is indeterminate. |
| U | (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected due to the presence of contaminants detected in the associated blank(s). |
| UJ | (Non-detected estimated): The analyte was not detected and the associated numerical value is approximate. |
| X | (Exclusion of data recommended): The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Exclusion of the data is recommended. |
| NA | (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data. |

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

## Qualification Code Reference

| | |
|---|---|
| a | ICP Serial Dilution %D was not within control limits. |
| b | Presumed contamination from preparation (method blank). |
| c | Calibration %RSD, r, $r^2$, %D or %R was noncompliant. |
| d | The analysis with this flag should not be used because another more technically sound analysis is available. |
| e | MS/MSD or Duplicate RPD was high. |
| f | Presumed contamination from FB or ER. |
| g | ICP ICS results were unsatisfactory. |
| h | Holding times were exceeded. |
| i | Internal standard performance was unsatisfactory. |
| k | Estimated Maximum Possible Concentration (HRGC/HRMS only) |
| l | LCS/LCSD %R was not within control limits. |
| m | Result exceeded the calibration range. |
| o | Cooler temperature or temperature blank was noncompliant and/or sample custody problems. |
| p | RPD between two columns was high (GC only). |
| q | MS/MSD recovery was not within control limits. |
| s | Surrogate recovery was not within control limits. |
| t | Presumed contamination from trip blank. |
| v | Unusual problems found with the data not defined elsewhere. Description of the problem can be found in the validation report. |
| w | LCS/LCSD RPD was high. |
| y | Chemical recovery was not within control limits (Radiochemistry only). |

## I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was resolved with a valley of less than or equal to 25%.

The static resolving power was at least 10,000 (10% valley definition).

## III. Initial Calibration and Initial Calibration Verification

A five point initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all analytes and labeled compounds.

The ion abundance ratios for all PCDDs/PCDFs were within method and validation criteria.

The minimum S/N ratio was greater than or equal to 2.5 for each analyte and greater than or equal to 10 for each labeled compound.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 20.0% for all analytes and less than or equal to 30.0% for labeled compounds.

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

All of the continuing calibration percent differences (%D) between the initial calibration RRF and the continuing calibration RRF were less than or equal to 20.0% for all analytes and less than or equal to 30.0% for labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within method and validation criteria.

The minimum S/N ratio was greater than or equal to 10 for each analyte and labeled compound.

## V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

| Blank ID | Extraction Date | Analyte | Concentration | Associated Samples |
|---|---|---|---|---|
| MB 410-300032 | 09/26/22 | 1,2,3,4,6,7,8-HpCDF | 0.000000365 ug/L | All samples in SDG 580-118118-1 |
| | | 1,2,3,4,7,8-HxCDD | 0.000000318 ug/L | |
| | | 1,2,3,4,7,8-HxCDF | 0.000000326 ug/L | |
| | | 1,2,3,6,7,8-HxCDD | 0.000000163 ug/L | |
| | | 1,2,3,6,7,8-HxCDF | 0.000000277 ug/L | |
| | | 1,2,3,7,8-PeCDF | 0.000000674 ug/L | |
| | | 1,2,3,7,8,9-HxCDD | 0.000000456 ug/L | |
| | | 1,2,3,7,8,9-HxCDF | 0.000000413 ug/L | |
| | | 2,3,4,6,7,8-HxCDF | 0.000000282 ug/L | |
| | | 2,3,4,7,8-PeCDF | 0.000000544 ug/L | |
| | | OCDD | 0.00000101 ug/L | |
| | | OCDF | 0.000000339 ug/L | |
| | | Total HxCDD | 0.000000937 ug/L | |
| | | Total HxCDF | 0.00000130 ug/L | |
| | | Total HpCDF | 0.000000365 ug/L | |
| | | Total PeCDF | 0.00000122 ug/L | |
| | | Total PeCDD/Total PeCDF | 0.00000517 ug/L | |
| | | Total PeCDD | 0.00000195 ug/L | |
| | | Total PeCDF | 0.00000322 ug/L | |

Sample concentrations were compared to concentrations detected in the laboratory blanks. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated laboratory blanks with the following exceptions:

| Sample | Analyte | Reported Concentration | Modified Final Concentration |
|---|---|---|---|
| HU145 | 1,2,3,4,7,8-HxCDD | 0.00000012 ug/L | 0.00000012U ug/L |
| | 2,3,4,7,8-PeCDF | 0.00000032 ug/L | 0.00000032U ug/L |
| | OCDD | 0.00000061 ug/L | 0.00000061U ug/L |
| | OCDF | 0.00000042 ug/L | 0.00000042U ug/L |
| | Total HxCDD | 0.00000012 ug/L | 0.00000012U ug/L |
| | Total HpCDF | 0.00000029 ug/L | 0.00000029U ug/L |
| | Total PeCDF | 0.00000032 ug/L | 0.00000032U ug/L |
| | Total PeCDD/Total PeCDF | 0.0000017 ug/L | 0.0000017U ug/L |
| | Total PeCDD | 0.00000073 ug/L | 0.00000073U ug/L |
| | Total PeCDF | 0.0000010 ug/L | 0.0000010U ug/L |

## VI. Field Blanks

No field blanks were identified in this SDG.

## VII. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

## VIII. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

## IX. Field Duplicates

No field duplicates were identified in this SDG.

## X. Labeled Compounds

All percent recoveries (%R) for labeled compounds used to quantitate target analytes were within QC limits.

## XI. Target Analyte Quantitation

All target analyte quantitations met validation criteria with the following exceptions:

| Sample | Analyte | Flag | A or P |
|---|---|---|---|
| All samples in SDG 580-118118-1 | Results flagged "I" by the laboratory as estimated maximum possible concentration (EMPC). | J (all detects) | A |

## XII. Target Analyte Identification

All target analyte identifications met validation criteria.

## XIII. System Performance

The system performance was acceptable.

## XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected or recommended for exclusion in this SDG.

Due to results reported by the laboratory as EMPC, data were qualified as estimated in one sample.

Due to laboratory blank contamination, data were qualified as not detected in one sample.

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polychlorinated Dioxins/Dibenzofurans - Data Qualification Summary - SDG 580-118118-1

| Sample | Analyte | Flag | A or P | Reason (Code) |
|---|---|---|---|---|
| HU145 | Results flagged "I" by the laboratory as estimated maximum possible concentration (EMPC). | J (all detects) | A | Target analyte quantitation (EMPC) (k) |

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polychlorinated Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG 580-118118-1

| Sample | Analyte | Modified Final Concentration | A or P | Code |
|---|---|---|---|---|
| HU145 | 1,2,3,4,7,8-HxCDD | 0.00000012U ug/L | A | b |
| | 2,3,4,7,8-PeCDF | 0.00000032U ug/L | | |
| | OCDD | 0.00000061U ug/L | | |
| | OCDF | 0.00000042U ug/L | | |
| | Total HxCDD | 0.00000012U ug/L | | |
| | Total HpCDF | 0.00000029U ug/L | | |
| | Total PeCDF | 0.00000032U ug/L | | |
| | Total PeCDD/Total PeCDF | 0.0000017U ug/L | | |
| | Total PeCDD | 0.00000073U ug/L | | |
| | Total PeCDF | 0.0000010U ug/L | | |

## Red Hill Oily Waste Disposal Facility, CTO 18F0176
## Polychlorinated Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG 580-118118-1

No Sample Data Qualified in this SDG

LDC #: 55171B21

SDG #: 580-118118-1

Laboratory: Eurofins, Tacoma, WA

# VALIDATION COMPLETENESS WORKSHEET

Stage 4

Date: 11/01/22

Page: 1 of 1

Reviewer:

2nd Reviewer:

**METHOD:** HRGC/HRMS Polychlorinated Dioxins/Dibenzofurans (EPA SW-846 Method 8290A)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

| | Validation Area | | Comments |
|---|---|---|---|
| I. | Sample receipt/Technical holding times | A/A | |
| II. | HRGC/HRMS Instrument performance check | A | |
| III. | Initial calibration/ICV | A/A | % RSD ≤ 20 ICV ≤ 20/30 |
| IV. | Continuing calibration | A | CCV ≤ 20/30 |
| V. | Laboratory Blanks | SW | |
| VI. | Field blanks | N | |
| VII. | Matrix spike/Matrix spike duplicates | N | cs |
| VIII. | Laboratory control samples | A | LCS ID |
| IX. | Field duplicates | N | |
| X. | Labeled Compounds | A | |
| XI. | Target analyte quantitation | SW | |
| XII. | Target analyte identification | A | |
| XIII. | System performance | A | |
| XIV. | Overall assessment of data | A | |

Note:

| | | | |
|---|---|---|---|
| A = Acceptable | ND = No compounds detected | D = Duplicate | SB=Source blank |
| N = Not provided/applicable | R = Rinsate | TB = Trip blank | OTHER: |
| SW = See worksheet | FB = Field blank | EB = Equipment blank | |

| | Client ID | Lab ID | Matrix | Date |
|---|---|---|---|---|
| 1 | HU145 | 580-118118-1 | Water | 09/19/22 |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
| 7 | | | | |
| 8 | | | | |
| 9 | | | | |
| 10 | | | | |

Notes:

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | MB410 300032 | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

LDC #: 55171B21

## VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: FT
2nd Reviewer:

**Method:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290A)

| Validation Area | Yes | No | NA | Findings/Comments |
|---|---|---|---|---|
| ***I. Technical holding times*** | | | | |
| All technical holding times were met. | ✓ | | | |
| Cooler temperature criteria was met. | ✓ | | | |
| ***II. GC/MS Instrument performance check*** | | | | |
| Was PFK exact mass 380.9760 verified? | ✓ | | | |
| Were the retention time windows established for all homologues? | ✓ | | | |
| Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers < 25% ? | ✓ | | | |
| Is the static resolving power at least 10,000 (10% valley definition)? | ✓ | | | |
| Was the mass resolution adequately check with PFK? | ✓ | | | |
| Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified? | ✓ | | | |
| ***IIIa. Initial calibration*** | | | | |
| Was the initial calibration performed at 5 concentration levels? | ✓ | | | |
| Were all percent relative standard deviations (%RSD) ≤ 20% for all analytes and labeled compounds ? | ✓ | | | |
| Did all calibration standards meet the Ion Abundance Ratio criteria? | ✓ | | | |
| Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10? | ✓ | | | |
| ***IIIb. Initial Calibration Verification*** | | | | |
| Was an initial calibration verification standard analyzed after each initial calibration for each instrument? | ✓ | | | |
| Were all percent differences (%D) ≤ 20% for unlabeled compounds and ≤30% for labeled compounds ? | ✓ | | | |
| ***IV. Continuing calibration*** | | | | |
| Was a contiuning calibration performed at the beginning and end of each 12 hour period? | ✓ | | | |
| Were all percent differences (%D) ≤ 20% for unlabeled compounds and ≤ 30% for labeled compounds ? | ✓ | | | |
| Did all routine calibration standards meet the Ion Abundance Ratio criteria? | ✓ | | | |
| Was the signal to noise ratio for each target compound and for each recovery and internal standard ≥ 10? | ✓ | | | |
| ***V. Laboratory Blanks*** | | | | |
| Was a method blank associated with every sample in this SDG? | ✓ | | | |
| Was a method blank performed for each matrix and whenever a sample extraction was performed? | ✓ | | | |
| Was there contamination in the method blanks? | ✓ | | | |
| ***VI. Field blanks*** | | | | |
| Field blanks were identified in this SDG. | | ✓ | | |
| Target compounds were detected in the field blanks. | | | ✓ | |

LDC #: 55171B2

## VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: FT
2nd Reviewer:

| | | | | |
|---|---|---|---|---|
| ***VII. Matrix spike/Matrix spike duplicates*** | | | | |
| Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG? | | | ✓ | |
| Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits? | | | ✓ | |
| ***VIII. Laboratory control samples*** | | | | |
| Was an LCS analyzed per extraction batch? | ✓ | | | |
| Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits? | ✓ | | | |
| ***IX. Field duplicates*** | | | | |
| Field duplicate pairs were identified in this SDG. | | ✓ | | |
| Target compounds were detected in the field duplicates. | | | ✓ | |
| ***X. Labeled Compoubds*** | | | | |
| Were internal standard recoveries within the 40-135% criteria? | ✓ | | | |
| Was the minimum S/N ratio of all internal standard peaks ≥ 10? | ✓ | | | |
| ***XI. Compound quantitation*** | | | | |
| Did the laboratory LOQs/RLs meet the QAPP LOQs/RLs? | ✓ | | | |
| Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound? | ✓ | | | |
| Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation? | ✓ | | | |
| ***XII. Target compound identification*** | | | | |
| For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard? | ✓ | | | |
| For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration? | ✓ | | | |
| For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution? | ✓ | | | |
| Did compound spectra contain all characteristic ions listed in the table attached? | ✓ | | | |
| Was the Ion Abundance Ratio for the two quantitation ions within criteria? | ✓ | | | |
| Was the signal to noise ratio for each target compound and labeled standard ≥ 2.5? | ✓ | | | |
| Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)? | ✓ | | | |
| For PCDF identification, was any signal (S/N ≥ 2.5, at ± seconds RT) detected in the corresponding PCDPE channel? | ✓ | | | |
| Was an acceptable lock mass recorded and monitored? | ✓ | | | |
| ***XIII. System performance*** | | | | |
| System performance was found to be acceptable. | ✓ | | | |
| ***XIV. Overall assessment of data*** | | | | |
| Overall assessment of data was found to be acceptable. | ✓ | | | |

# VALIDATION FINDINGS WORKSHEET

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

| | | | | |
|---|---|---|---|---|
| A. 2,3,7,8-TCDD | F. 1,2,3,4,6,7,8-HpCDD | K. 1,2,3,4,7,8-HxCDF | P. 1,2,3,4,7,8,9-HpCDF | U. Total HpCDD |
| B. 1,2,3,7,8-PeCDD | G. OCDD | L. 1,2,3,6,7,8-HxCDF | Q. OCDF | V. Total TCDF |
| C. 1,2,3,4,7,8-HxCDD | H. 2,3,7,8-TCDF | M. 2,3,4,6,7,8-HxCDF | R. Total TCDD | W. Total PeCDF |
| D. 1,2,3,6,7,8-HxCDD | I. 1,2,3,7,8-PeCDF | N. 1,2,3,7,8,9-HxCDF | S. Total PeCDD | X. Total HxCDF |
| E. 1,2,3,7,8,9-HxCDD | J. 2,3,4,7,8-PeCDF | O. 1,2,3,4,6,7,8-HpCDF | T. Total HxCDD | Y. Total HpCDF |

Notes:______________________________________________________

LDC #: 55171B21

# VALIDATION FINDINGS WORKSHEET
## Blanks

Reviewer: FT

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290A)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y Were all samples associated with a method blank?

Y Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y Was the method blank contaminated?

**Blank extraction date:** 9/26/22 **Blank analysis date:** **9/27/22**

**Conc. units:** ug/L **Associated samples:** **All (b)**

| Compound | Blank ID | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| ▨ | MB 410-300032 | 5x | | 1 | | | | | | |
| O | 0.000000365 | 0.000001825 | | - | | | | | | |
| C | 0.000000318 | 0.000001590 | | 0.00000012U | | | | | | |
| K | 0.000000326 | 0.000001630 | | - | | | | | | |
| D | 0.000000163 | 0.000000815 | | - | | | | | | |
| L | 0.000000277 | 0.000001385 | | - | | | | | | |
| I | 0.000000674 | 0.000003370 | | - | | | | | | |
| E | 0.000000456 | 0.000002280 | | - | | | | | | |
| N | 0.000000413 | 0.000002065 | | - | | | | | | |
| M | 0.000000282 | 0.000001410 | | - | | | | | | |
| J | 0.000000544 | 0.000002720 | | 0.00000032U | | | | | | |
| G | 0.00000101 | 0.000005050 | | 0.00000061U | | | | | | |
| Q | 0.000000339 | 0.000001695 | | 0.00000042U | | | | | | |
| T | 0.000000937 | 0.000004685 | | 0.00000012U | | | | | | |

CUIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
All Ycontaminants within five times the method blank concentration were qualified as not detected, "U".

vdr/validation worksheet/dioxins/8290/MB 410 300032 55171B21

LDC #: **55171B21**

# VALIDATION FINDINGS WORKSHEET
## Blanks

Page: 2 of 2
Reviewer: FT

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290A)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y Were all samples associated with a method blank?
Y Was a method blank performed for each matrix and whenever a sample extraction was performed?
Y Was the method blank contaminated?

**Blank extraction date:** 9/26/22 **Blank analysis date:** 9/27/22
**Conc. units:** ug/L **Associated samples:** all (b)

| Compound | Blank ID | Sample Identification | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | MB 410-300032 | 5x | | 1 | | | | | | |
| X | 0.00000130 | 0.000006500 | | - | | | | | | |
| Y | 0.000000365 | 0.000001825 | | 0.00000029U | | | | | | |
| W | 0.00000122 | 0.000006100 | | 0.00000032U | | | | | | |
| S/W | 0.00000517 | 0.000025850 | | 0.0000017U | | | | | | |
| S | 0.00000195 | 0.000009750 | | 0.00000073U | | | | | | |
| W | 0.00000322 | 0.000016100 | | 0.0000010U | | | | | | |
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| | | - | | | | | | | | |

LDC #: 55171B21

# VALIDATION FINDINGS WORKSHEET
## Target Analyte Quantitation

Page: 1 of 1
Reviewer:

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)A

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Were the correct internal standard (IS), quantitation ions and relative response factors (RRF) used to quantitate the compound?
Y N N/A Compound quantitation and CRQLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

| # | Date | Sample ID | Finding | Associated Samples | Qualifications |
|---|---|---|---|---|---|
| | | All | All results qualified | | J dets/A (K) |
| | | | "I" as EMPC | | |
| | | | | | |
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Comments: See sample calculation verification worksheet for recalculations

LDC #: 55171B21

# VALIDATION FINDINGS WORKSHEET
## Initial Calibration Calculation Verification

Page: 1 of 1
Reviewer: FT

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290A)

The Relative Response Factor (RRF), average RRF, and percent relative standard deviation (%RSD) were recalculated for the compounds identified below using the following calculations:

RRF = $(A_x)(C_{is})/(A_{is})(C_x)$
average RRF = sum of the RRFs/number of standards
%RSD = 100 * (S/X)

$A_x$ = Area of Compound
$C_x$ = Concentration of compound,
S= Standard deviation of the RRFs,

$A_{is}$ = Area of associated internal standard
$C_{is}$ = Concentration of internal standard
X = Mean of the RRFs

| # | Standard ID | Calibration Date | Compound (IS) | Reported RRF (10/50/100 std) | Recalculated RRF (10/50/100 std) | Reported Average RRF (Initial) | Recalculated Average RRF (Initial) | Reported %RSD | Recalculated %RSD |
|---|---|---|---|---|---|---|---|---|---|
| 1 | ICAL DF18471 | 1/6/2022 | 2,3,7,8-TCDF (13C-2,3,7,8-TCDD) | 1.5760 | 1.5760 | 1.1309 | 1.1309 | 15.1 | 15.1 |
| | | | 2,3,7,8-TCDD (13C-2,3,7,8-TCDF) | 1.0589 | 1.0589 | 1.1359 | 1.1359 | 16.7 | 16.7 |
| | | | 1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD) | 1.0168 | 1.0168 | 1.0526 | 1.0526 | 5.1 | 5.1 |
| | | | 1,2,3,4,6,7,8-HpCDD (13C-1,2,3,4,6,7,8,-HpCDD) | 1.0509 | 1.0509 | 1.0671 | 1.0671 | 8.3 | 8.3 |
| | | | OCDF (13C-OCDF) | 0.9190 | 0.9190 | 0.9320 | 0.9320 | 4.0 | 4.0 |

LDC #: 55171B21

# VALIDATION FINDINGS WORKSHEET
## Continuing Calibration Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290A)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
RRF = $(A_x)(C_{is})/(A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
RRF = continuing calibration RRF
$A_x$ = Area of compound, $A_{is}$ = Area of associated internal standard
$C_x$ = Concentration of compound, $C_{is}$ = Concentration of internal standard

| # | Standard ID | Calibration Date | Compound (Reference Internal Standard) | Average RRF (initial) | Reported | Recalculated | Reported | Recalculated |
|---|---|---|---|---|---|---|---|---|
| | | | | | RRF (CC) | RRF (CC) | %D | %D |
| 1 | ccv | 9/27/22<br>1002 | 2,3,7,8-TCDF ($^{13}$C-2,3,7,8-TCDF) | 1.1309 | 1.149 | 1.149 | 1.6 | 1.6 |
| | | | 2,3,7,8-TCDD ($^{13}$C-2,3,7,8-TCDD) | 1.1359 | 1.232 | 1.232 | 8.4 | 8.4 |
| | | | 1,2,3,6,7,8-HxCDD ($^{13}$C-1,2,3,6,7,8-HxCDD) | 1.0526 | 1.155 | 1.155 | 9.7 | 9.7 |
| | | | 1,2,3,4,6,7,8-HpCDD ($^{13}$C-1,2,4,6,7,8,-HpCDD) | 1.0671 | 1.093 | 1.093 | 2.4 | 2.4 |
| | | | OCDF ($^{13}$C-OCDD) | 0.9320 | 0.9814 | 0.9814 | 5.3 | 5.3 |
| 2 | | | 2,3,7,8-TCDF ($^{13}$C-2,3,7,8-TCDF) | | | | | |
| | | | 2,3,7,8-TCDD ($^{13}$C-2,3,7,8-TCDD) | | | | | |
| | | | 1,2,3,6,7,8-HxCDD ($^{13}$C-1,2,3,6,7,8-HxCDD) | | | | | |
| | | | 1,2,3,4,6,7,8-HpCDD ($^{13}$C-1,2,4,6,7,8,-HpCDD) | | | | | |
| | | | OCDF ($^{13}$C-OCDD) | | | | | |
| 3 | | | 2,3,7,8-TCDF ($^{13}$C-2,3,7,8-TCDF) | | | | | |
| | | | 2,3,7,8-TCDD ($^{13}$C-2,3,7,8-TCDD) | | | | | |
| | | | 1,2,3,6,7,8-HxCDD ($^{13}$C-1,2,3,6,7,8-HxCDD) | | | | | |
| | | | 1,2,3,4,6,7,8-HpCDD ($^{13}$C-1,2,4,6,7,8,-HpCDD) | | | | | |
| | | | OCDF ($^{13}$C-OCDD) | | | | | |

Comments: Refer to Routine Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 55171B21

# VALIDATION FINDINGS WORKSHEET

## Laboratory Control Sample Results Verification

Page: 1 of 1
Reviewer: FT

**METHOD:** GC/MS Dioxins/Dibenzofurans (EPA SW 846 Method 8290A)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratoy control sample and laboratory control sample duplicate (if applicable) were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration
SA = Spike added

RPD = I LCS - LCSD I * 2/(LCS + LCSD) LCS = Laboraotry control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: lcs/D 410 - 300032

| Compound | Spike Added ( ug/L ) | | Spiked Sample Concentration ( ug/L ) | | LCS Percent Recovery | | LCSD Percent Recovery | | LCS/LCSD RPD | |
|---|---|---|---|---|---|---|---|---|---|---|
| | LCS | LCSD | LCS | LCSD | Reported | Recalc | Reported | Recalc | Reported | Recalc |
| 2,3,7,8-TCDD | 0.000200 | 0.000200 | 0.000209 | 0.000211 | 104 | 104 | 106 | 506 | 1 | 1 |
| 1,2,3,7,8-PeCDD | 0.00100 | 0.00100 | 0.00111 | 0.00110 | 111 | 111 | 110 | 110 | 0 | 0 |
| 1,2,3,4,7,8-HxCDD | 0.00100 | 0.00100 | 0.00106 | 0.00111 | 106 | 106 | 111 | 111 | 5 | 5 |
| 1,2,3,4,7,8,9-HpCDF | 0.00100 | 0.00100 | 0.00105 | 0.00105 | 105 | 105 | 105 | 105 | 0 | 0 |
| OCDF | 0.00200 | 0.00200 | 0.00219 | 0.00213 | 109 | 109 | 106 | 106 | 3 | 3 |
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Comments: Refer to Laboratory Control Sample findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 55171B21

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Reviewer: B

# VALIDATION FINDINGS WORKSHEET

## Sample Calculation Verification

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290A)

[✓ Y] N N/A Were all reported results recalculated and verified for all level IV samples?
[✓ Y] N N/A Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

Concentration = $\frac{(A_x)(I_s)(DF)}{(A_{is})(RRF)(V_o)(\%S)}$

| | | |
|---|---|---|
| $A_x$ | = | Area of the characteristic ion (EICP) for the compound to be measured |
| $A_{is}$ | = | Area of the characteristic ion (EICP) for the specific internal standard |
| $I_s$ | = | Amount of internal standard added in nanograms (ng) |
| $V_o$ | = | Volume or weight of sample extract in milliliters (ml) or grams (g). |
| RRF | = | Relative Response Factor (average) from the initial calibration |
| Df | = | Dilution Factor. |
| %S | = | Percent solids, applicable to soil and solid matrices only. |

Example:

Sample I.D. #1, OCDF:

Conc. = $\frac{(146)(200)(20)}{(1419676)(0.9320)(1048.5)(1000)}$

= 0.00000042096 ug/L

| # | Sample ID | Compound | Reported Concentration (ug/L) | Calculated Concentration (ug/L) | Qualification |
|---|---|---|---|---|---|
| | #1 | OCDF | 0.00000042 | 0.00000042096 | |
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