DATA VALIDATION REPORT
Wilcox Oil Company Superfund Site RI/FS
April 2017 Sampling Event

Site Name:	Wilcox Oil Company Superfund Site Bristow, Creek County, Oklahoma
Laboratory:	Eurofins Lancaster Laboratories Environmental, Lancaster Pennsylvania
QA Reviewer:	Kim Wallace-Wymore EA Engineering, Science, and Technology, Inc. (EA), PBC
Sample Delivery Group Nos.:	WLC06, WLC07, WLC08, WLC10, WLC11, WLC12
Sample Identification:	See Table 1
Matrix:	Soil and Water
QC Criteria Reviewed:	Section 2.0
Laboratory Report Date:	June 2017

1.0 INTRODUCTION

Soil samples were collected during April 2017 in support of the Wilcox Oil Company Superfund Site Remedial Investigation/ Feasibility Study (RI/FS). The samples were shipped via overnight courier to Eurofins Lancaster Laboratories Environmental, Lancaster, Pennsylvania for analysis. Samples were analyzed in accordance with USEPA SW-846 *Test Methods for Evaluating Solid Waste* (EPA 1986) and Texas Natural Resource Conservation Commission *Total Petroleum Hydrocarbons TNRCC Method 1005* (TNRCC 2001). The respective analytical parameters and testing methods are provided below.

- Polychlorinated biphenyls (PCBs) – Method SW8082A
- Dioxins/Furans – Method SW8290A
- Total petroleum hydrocarbons (TPH) – TNRCC Method 1005
- Reactivity (cyanide and sulfide) – SW846 Chapter 7.3
- Corrosivity (pH) – Method SW9045D
- Ignitability – Method SW-846 Chapter 7
- Toxicity characteristic leaching procedure (TCLP) volatile organic compounds (VOCs) – Method SW1311/8260CTCLP semivolatile organic compounds (SVOCs) – Method SW1311/8270D
- TCLP Resource Conservation and Recovery Act (RCRA) metals – Method

SW1311/6010C
- TCLP mercury – Method SW1311/7470A
- VOCs – Method SW8020B
- SVOCs – Method SW8270D
- RCRA metals – Method SW6010C
- Mercury – Method SW7471B

The data was validated in accordance with requirements specified in the following documents:

- *Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, EPA 540-R-014-002 (EPA 2014*)
- *Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review, EPA 540-R-013-01 (EPA 2014)*
- *Sampling and Analysis Plan (SAP) for Remedial Investigation/Feasibility Study Wilcox Oil Company Superfund Site, Bristow, Creek County, Oklahoma (EA 2016)*

Section 2.0 of this validation report identifies the criteria reviewed for each analytical method as appropriate based on EPA guidelines. Section 3.0 contains the definitions of the qualifiers to be applied to the data results based on the validation process. Section 4.0 provides an assessment of the overall data quality, and Section 5 provides the references to the guidelines and documents used in performing the review of the data set.

Table 1─Sample Cross-Reference provides a list of the field sample identification (ID), laboratory sample delivery group (SDG) and sample ID, sample matrix, sample collection date, and analysis parameters. The results of these analyses are discussed in Section 4.0, Data Assessment.

2.0 DATA VALIDATION CRITERIA

The criteria listed below were evaluated as part of the validation process, as applicable to the analytical method.

- Deliverables
- Chain of custody and sample receipt
- Holding times
- Calibration (initial and continuing)
- Ion ratio (dioxin/furan)
- Blanks (trip blanks, method blanks and calibration blanks)
- Laboratory control sample (LCS)
- Matrix spike (MS), matrix spike duplicate (MSD), and/or laboratory duplicate
- Surrogate recoveries
- Serial Dilution
- Labeled compound recoveries
- Field quality control (QC) samples
- Sample quantitation and reported detection limits
- Overall assessment of data

TABLE 1
SAMPLE CROSS-REFERENCE TABLE

Field Sample ID	Laboratory Sample Delivery Group	Sample ID	Matrix	Date Collected	Analysis Parameters
ETF-WC-01	WLC06	WLC06-01	Soil	25-Apr-17	PCBs Method SW8082A Dioxins/Furans Method SW8290A Petroleum Hydrocarbons TX 1005 RCI SW-846 Chapter 7
ETF-WC-01	WLC06	WLC06-02	Soil	25-Apr-17	TCLP SVOCs Method SW1311/8270D TCLP Metals Method SW1311/6010C TCLP Mercury Method SW1311/7470A
ETF-WC-01	WLC06	WLC06-03	Soil	25-Apr-17	TCLP VOCs Method SW1311/8260C
WPA-WC-02	WLC07	WLC07-01	Soil	25-Apr-17	PCBs Method SW8082A Dioxins/Furans Method SW8290A Petroleum Hydrocarbons TX 1005 RCI SW-846 Chapter 7
WPA-WC-02	WLC07	WLC07-02	Soil	25-Apr-17	TCLP SVOCs Method SW1311/8270D TCLP Metals Method SW1311/6010C TCLP Mercury Method SW1311/7470A
WPA-WC-02	WLC07	WLC07-03	Soil	25-Apr-17	TCLP VOCs Method SW1311/8260C
WPA-WC-01	WLC08	WLC08-01	Soil	25-Apr-17	PCBs Method SW8082A Dioxins/Furans Method SW8290A Petroleum Hydrocarbons TX 1005 RCI SW-846 Chapter 7
WPA-WC-01	WLC08	WLC08-02	Soil	25-Apr-17	TCLP SVOCs Method SW1311/8270D TCLP Metals Method SW1311/6010C TCLP Mercury Method SW1311/7470A

Field Sample ID	Laboratory Sample Delivery Group	Sample ID	Matrix	Date Collected	Analysis Parameters
WPA-WC-01	WLC08	WLC08-03	Soil	25-Apr-17	TCLP VOCs Method SW1311/8260C
ETF-WC-01	WLC10	WLC10-01	Soil	25-Apr-17	VOCs Method SW8260B SVOCs Method SW8270D Metals SW6010C Mercury SW7471B
WPA-WC-02	WLC11	WLC11-01	Soil	25-Apr-17	VOCs Method SW8260B SVOCs Method SW8270D Metals SW6010C Mercury SW7471B
WPA-WC-01	WLC12	WLC12-01	Soil	25-Apr-17	VOCs Method SW8260B SVOCs Method SW8270D Metals SW6010C Mercury SW7471B

NOTE:
PCB – Polychlorinated biphenyls
RCI – Reactivity, corrosivity, ignitibility
SVOC – Semivolatile organic compound
TCLP – Toxicity characteristic leaching procedure
VOC – Volatile organic compound

3.0 GLOSSARY OF DATA QUALIFIERS

The following definitions provide a brief explanation for the data qualifiers that may be applied to the data during the data review process. The definitions are consistent with EPA guidance (2014).

No Qualifier — Indicates that the data are acceptable both qualitatively and quantitatively.

U — The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

J — The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample. The data are valid for project use to achieve project data quality objectives (DQOs).

J+ — The result is an estimated quantity, but the result may be biased high. The data are valid for project use to achieve project DQOs.

J- — The result is an estimated quantity, but the result may be biased low. The data are valid for project use to achieve project DQOs.

UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate. The data are valid for project use to achieve project DQOs.
R	The sample results are not usable to achieve project DQOs based on certain QC criteria outside of acceptance limits. The analyte may or may not be present in the sample.

4.0 DATA ASSESSMENT

One hundred percent of the analytical data in the SDGs WLC06, WLC07, WLC08, WLC10, WLC11, and WLC12 was reviewed for the criteria listed in Section 2.0. A discussion of the data is presented in this section.

4.1 Deliverables

The data package and electronic deliverable for the SDGs is complete.

4.2 Chain of Custody and Sample Receipt

The samples were received by Eurofins under appropriate chain of custody, in good condition and properly preserved, unless otherwise noted. The cooler temperatures were within the specified EPA guidelines of ≤6°C.

The methanol preservative did not fully cover the VOC soil samples ETF-WC-01 (SDG WLC10) and WPA-WC-02 (SDG WLC11). Methanol was added at the laboratory after the net weight was determined. The detected VOC results were qualified (J) and non-detect VOC results were qualified (UJ) in samples ETF-WC-01 and WPA-WC-02.

4.3 Holding Times

The samples were extracted and analyzed within the method-specific holding times, with the exceptions noted below.

- The VOC, SVOC, and mercury analyses for sample ETF-WC-01 (SDG WLC10) was performed outside the holding time. The sample was collected on 4/25/17. Additional analysis was requested on 5/31/17. The results are qualified as estimated (J).
- The VOC, SVOC, and mercury analyses for sample WPA-WC-02 (SDG WLC11) were performed outside the holding time. The sample was collected on 4/25/17. Additional analysis was requested on 5/31/17. The results are qualified as estimated (J).
- The VOC, SVOC, and mercury analyses for sample WPA-WC-01 (SDG WLC12) were performed outside the holding time. The sample was collected on 4/25/17. Additional analysis was requested on 6/13/17. The results are qualified as estimated (J).

Since the sample data are for waste characterization purposes the data are considered usable to achieve the project objectives.

4.4 Calibration Criteria

- The calibrations for the target analytes were within the control criteria as specified for each method with the exceptions noted below. The continuing calibration verification for the VOC analytes 1,1,1-trichloroethane and carbon tetrachloride exceeded the control limit in SDG WLC10 and WLC11. The associated sample results for sample ETF-WC-01 (SDG WLC10) and WPA-WC-02 (SDG WLC11) were qualified (UJ), estimated non-detect data.
- The response for the surrogate DCB in the continuing calibration verification standard for PCB analysis (SDG WLC08) was outside the acceptance limits. The laboratory repeated

the analysis, and the surrogate was also outside the acceptance limits for the reanalysisd. The PCB results in sample WPA-WC-01 (SDG WLC08) are qualified (UJ).

For PCB results, dual column analyses in which the calibration response is outside the acceptance criteria on one column and within criteria on the second column, the affected analytes are reported from the compliant column. The sample raw data identifies the column used to report each analyte.

4.5 Identification Criteria

The ion ratio was outside acceptable limits for detected concentrations of 2,3,7,8-tetrachlorodibenzofuran (TCDF), 2,3,7,8-tetrachlorobenzodioxin (TCDD), 2,3,4,7,8-pentachlorodibenzofuran (PeCDF), 1,2,3,7,8-pentachlorodibenzo-p-dioxin (PeCDD), 1,2,3,4,7,8-hexachlorodibenzodioxin (HxCDD), 1,2,3,7,8,9-hexachlorodibenzo-p-dioxin (HxCDD), 1,2,3,7,8,9-hexachlorodibenzofuran (HxCDF), and 1,2,3,4,6,7,8-heptachlorodibenzofuran (HpCDF) in sample ETF-WC-01 (SDG WLC06). The laboratory reported an estimated maximum possible concentration (EMPC) for these compounds as required by the method. These results are qualified as estimated (J).

The ion ratio was outside acceptable limits for detected concentrations of 2,3,7,8-TCDD, 1,2,3,7,8-PeCDF, 2,3,4,7,8-PeCDF, 1,2,3,7,8-PeCDD, 1,2,3,4,7,8-HxCDF, 1,2,3,6,7,8-HxCDF, 2,3,4,6,7,8-HxCDF, 1,2,3,7,8,9-HxCDF, 1,2,3,4,6,7,8-HpCDF, and octachlorodibenzofuran (OCDF) in sample WPA-WC-02 (SDG WLC07). The laboratory reported an EMPC for these compounds as required by the method. These results are qualified as estimated (J).

The ion ratio was outside acceptable limits for detected concentrations of 2,3,7,8-TCDF, 2,3,7,8-TCDD, 1,2,3,7,8-PeCDF, 2,3,4,7,8-PeCDF, 1,2,3,4,7,8-HxCDF, 1,2,3,6,7,8-HxCDF, 2,3,4,6,7,8-HxCDF, 1,2,3,7,8,9-HxCDF, 1,2,3,4,7,8,9-HpCDF, and OCDF in sample WPA-WC-01 (SDG WLC08). The laboratory reported an EMPC for these compounds as required by the method. These results are qualified as estimated (J).

4.6 Blank Detections

Method blank samples were prepared and analyzed with each analytical method batch in addition to project samples. No analytical data were qualified based on blank detections with the exceptions noted below.

- Dioxin/furan compounds were reported in the method blanks sample in SDG WLC08. The following results were qualified (U) in sample WPA-WC-01 due to detections in the method blank: 2,3,7,8-TCDD, 2,3,7,8-TCDF, 1,2,3,7,8-PeCDF, 2,3,4,7,8-PeCDF, 1,2,3,4,7,8-HxCDF, 1,2,3,7,8,9-HxCDF, 2,3,4,6,7,8-HxCDF, and 1,2,3,4,7,8,9-HpCDF.
- Chromium was detected in one of the associated continuing calibration blanks. The chromium result was qualified (UJ) in sample WPA-WC-02 (SDG WLC07).

4.7 Laboratory Control Samples

The laboratory control samples (LCS) were prepared and analyzed as recommended by the analytical method and project requirements. The percent recoveries (%R) were within the method control limits for all methods with the exceptions noted below.

- VOC compounds carbon tetrachloride, dibromochloromethane, and bromoform recovered below the control limit in the LCS for SDG WLC10 and WLC11. The associated results were qualified as estimated (UJ) in the samples ETF-WC-01 and WPA-WC-02.
- The SVOC compound pentachlorophenol recovered below the control limit in the LCS associated with SDGs WLC10, WLC11, and WLC12. The pentachlorophenol result was qualified (UJ) in the samples ETF-WC-01, WPA-WC-01, and WPA-WC-02.

4.8 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) Samples

MS/MSD analyses were prepared for each analysis. No data were qualified based on the MS/MSD recoveries.

4.9 Surrogate Recoveries

Surrogate analyses were performed for the organic methods. The surrogate recoveries were within acceptable limits applicable to the specific methods with exception of the

TPH surrogate orthoterphenyl which recovered above the control limit in sample WPA-WC-02 (SDG WLC07). The (C6-C12), (C12-C28), (C28-C35) hydrocarbon results were qualified (J+). The total (C6-C28) and (C6-C35) hydrocarbons were also qualified (J+).

4.10 Labeled Compound Recoveries

Labeled compound recoveries were within the laboratory acceptance limits for the dioxin/furan analyses with the exception of the labeled compound 13C12-ODCF which recovered below the method control limit in the project sample ETF-WC-01 (SDG WLC06). The OCDF result in sample ETF-WC-01 was qualified as estimated low (J-).

4.11 Field and Laboratory Duplicate Samples

Field duplicate samples were not required to be collected for the waste characterization samples. Laboratory duplicates were analyzed for the metals analyses. Relative percent difference (RPDs) were calculated for detections greater than five times the detection limit. No data qualification was required based on the laboratory duplicate sample analyses, with the exception of the RPD for lead which exceeded the control limit in the laboratory duplicate associated with SDG WLC12. The lead result was qualified as estimated (J) in sample WPA-WC-01.

4.12 Serial Dilution

Serial dilution analysis was performed for the metals analyses. The calculated percent difference (%D) was within the method requirements where applicable.

4.13 Sample Quantitation and Reported Detection Limits

Project samples were analyzed at the appropriate concentrations to achieve the project-required reporting limits. Reporting limits were elevated due to matrix interference in sample ETF-WC-01 (SDG WLC06, WLC10) and WPA-WC-02 (SDG WLC07, WLC11). The sample results were verified for 10 percent of the project samples.

4.14 Overall Assessment of Data

Project deliverables were reviewed for completeness and compliance with the SAP. Based on the data review, the completeness of deliverables is 100 percent. Results were qualified as estimated due to the following indicator criteria exceptions: 1) holding time exceedances for VOCs, SVOCs and mercury, 2) methanol preservation for VOCs, 3) continuing calibration verification for VOCs and PCBs, 4) dioxin/furan ion ratios, 5) method blank and calibration blank detections for dioxin/furans and metals, 6) LCS recoveries for VOCs and SVOCs, 7) surrogate recoveries for TPH, and 8) laboratory duplicate sample precision for metals. No analytical data were qualified (R) signifying rejected or unusable data. The analytical data achieve greater than the 90 percent data completeness objective and the project data quality objectives. The April 2017 sample data are usable to support the Wilcox Oil Company Superfund Site RI/FS.

REFERENCES

EA Engineering, Science, and Technology, Inc., PBC (EA). 2016. *Sampling and Analysis Plan for Remedial Investigation/Feasibility Study Wilcox Oil Company Superfund Site, Bristow, Creek County, Oklahoma.* September.

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EPA. 1986. *Test Methods for Evaluating Solid Waste, SW-846, Third Edition and Updates.* November.