# DATA VALIDATION REPORT

## Portland Harbor Superfund Site
## March 2023 Storm Water Sampling

**SDG: K2302917**

Chemical Analyses Performed by:

**ALS Environmental, Kelso**

Prepared by:

**ENVIRONMENTAL DATA SERVICES, LTD.**

Prepared for:

**HGL**

**May 26, 2023**

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ENVIRONMENTAL DATA SERVICES, LTD.

# EXECUTIVE NARRATIVE

**Sample Delivery Group**: K2302917
**Laboratory:** ALS Kelso
**Site:** Portland Harbor Superfund Site
**Sampling dates**: 03/09/2023
**Number of Samples:** 6
**Test Method:** EPA SW846 8151A
**Analysis:** MCPP

**Validation Level:** Level 4

**Quality Assurance Project Plan:** Uniform Federal Policy-Quality Assurance Project Plan, Revision 3, Remedial Design Services, Swan Island Basin Project Area, Portland Harbor Superfund Site, Portland Multnomah County, Oregon, May 2022 (QAPP).

**Validation Guidelines:** United States Environmental Protection Agency (USEPA) National Functional Guidelines for Organic Superfund Methods Data Review, OLEM 9240.0-51, EPA-540-R-20-005, (November 2020).

| Client Sample ID | Laboratory Sample ID |
|---|---|
| SIB-SW-WR198-030923 | K2302917-001 |
| SIB-SW-WR186-030923 | K2302917-002 |
| SIB-SW-WR15-030923 | K2302917-003 |
| SIB-SW-WR428-030923 | K2302917-004 |
| SIB-SW-WR71-030923 | K2302917-005 |
| SIB-SW-WR30-030923 | K2302917-006 |

Table 1 provides a summary of the major and minor data quality issues identified in this data set. All data are acceptable except those results which have been qualified with “R”, rejected. Data validation qualifiers along with associated descriptions are provided in Table 2. All data qualification related to this group of samples is detailed on the attached sheets.

All data users should note two facts. First, an "R" flag means that the associated value is unusable due to significant quality control (QC) problems, the data is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on any data tables even as a last resort.

# DATA ASSESSMENT

## 1. NARRATIVE AND COMPLETENESS REVIEW:

*The case narrative was reviewed, and the data package was checked for completeness.*

## 2. SAMPLE DELIVERY AND CONDITION:

*The samples arrived at the laboratory in acceptable condition. Proper custody was documented.*

## 3. HOLDING TIME:

**The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detect results will be flagged as not detected at an estimated quantitation limit, "UJ", unless the holding time is grossly exceeded (by more than two times the holding time specified), in which case non-detect results are flagged "R", rejected. Qualifications were applied to the samples and analytes as shown below.**

*All sample analyses were within the validation guidance.*

## 4. CALIBRATION:

**Satisfactory instrument calibration is established to ensure that the instrument can produce acceptable quantitative data. An initial calibration demonstrates that the instrument can give acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.**

**Percent Relative Standard Deviation and Percent Difference**

**Percent relative standard deviation (%RSD) is calculated from the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent difference (%D) compares the response factor of the continuing calibration check to the mean response factor (RRF) from the initial calibration. Percent difference is a measure of the instrument's daily performance. If %RSD exceeds quality control criteria for target analytes, qualify all associated positive results "J". If the %D exceeds 20% qualify all associated positive results "J" and non-detects "UJ". If %RSD and %D grossly exceed QC criteria, non-detect data may be qualified "R".**

*No problems were found for this criterion requiring sample result qualification.*

## 5. BLANK CONTAMINATION:

**Quality assurance (QA) blanks, i.e., method, trip, field, or rinse blanks are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field, equipment and rinse blanks measure cross-contamination of samples during field operations.**

### A) Method blank contamination

*No problems were found for this criterion.*

### B) Field/Equipment blank contamination

*No sample was submitted as an equipment blank sample in association with the samples in this sample delivery group (SDG).*

## 6. SURROGATES / SYSTEM MONITORING COMPOUNDS:

**All samples are spiked with surrogate/system monitoring compounds (SMC) prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate/SMC concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 7. COMPOUND IDENTIFICATION:

**The retention times (RTs) of reported compounds must fall within the calculated retention time windows for the two chromatographic columns. Additionally, the %Relative Percent Difference (RPD) of the positive results obtained on the two GC columns must be ≤ 40%.**

**Retention Time**

*Criteria was reviewed at the Stage 4 level. No problems were found for this criterion.*

**Relative Percent Difference**

*Criteria was not reviewed at the Stage 4 level. No problems were found for this criterion.*

## 8. COMPOUND QUANTIFICATION:

*Target compound result quantitation was reviewed at the Stage 4 level. No anomalies were identified.*

*Manual integrations were reviewed at the Stage 4 level. No anomalies were identified.*

## 9. MATRIX SPIKE / MATRIX SPIKE RECOVERY:

**Matrix spike / matrix spike duplicate (MS/MSD) data is generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD data may be used in conjunction with other quality control criteria for additional qualification of data.**

*No sample was submitted for MS/MSD pair evaluations in association with this SDG.*

## 10. FIELD DUPLICATES:

**Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. An aqueous matrix control limit of 30% RPD shall be used when both the original and duplicate sample values are greater than or equal to five times the LOQ. A control limit of absolute difference of two times the LOQ shall be used if either the sample or duplicate value is less than five times the LOQ. For field duplicate analyses that do not meet the technical criteria, the action was applied to only the parent sample and its duplicate.**

*No samples were submitted as a field duplicate pair in association with this SDG.*

## 11. LABORATORY CONTROL SAMPLES:

**The Laboratory Control Sample (LCS) and LCS duplicates (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and quality assurance/quality control (QA/QC) procedures as employed for the samples. All LCS percent recoveries and relative percent differences (RPDs) must fall within the control limits. Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 12. OTHER PROBLEMS:

*None.*

## 13. DILUTIONS, RE-EXTRACTIONS & REANALYSIS:

**Samples may be re-analyzed for dilution, re-extraction and for other QC reasons. In such cases, the best result values are used.**

*No problems were found for this criterion.*

*Note: chromatographic interference was observed for samples SIB-SW-WR186-030923 and SIB-SW-WR428-030923 that resulted in elevated reporting limits for these samples.*

**Table 1 Major and Minor Findings**

| | Were acceptance criteria met? | | |
|---|---|---|---|
| | Yes | No | |
| MCPP | | Major | Minor |
| Holding Time | x | | |
| Calibration | x | | |
| Method Blank | x | | |
| Equipment/Field Blank | NA | | |
| Surrogates/System Monitoring Compounds | x | | |
| Compound Identification | x | | |
| Compound Quantitation | x | | |
| Matrix Spike/Matrix Spike Duplicate | NA | | |
| Field Duplicate | NA | | |
| Laboratory Control Samples | x | | |
| Other Quality Control Data out of Specification | x | | |

Major = Major data quality issue identified resulting in rejection of data.
Minor = Minor data quality issue identified resulting in the qualification of data. Data qualification should be used to inform the data users of data limitations.
NA = Not applicable

**Table 2 Data Validation Qualifiers**

| Data Qualifier | Definition |
|---|---|
| **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. |
| **UJ** | The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |
| **R** | The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample. |

ENVIRONMENTAL DATA SERVICES, LTD.

## EXECUTIVE NARRATIVE

**Sample Delivery Group**: K2302917
**Laboratory:** ALS Environmental
**Site:** Portland Harbor Superfund Site
**Sampling dates:** 03/09/2023
**Number of Samples:** 6
**Test Method:** SW-846 6020A
**Analysis:** Arsenic, Chromium, Copper, Zinc

**Validation Level:** Level 4

**Quality Assurance Project Plan:** Sampling and Analysis Plan; Non-Time Critical Removal Action Design Argonaut Mine Superfund Site Amador County, California; August 2022 Revision 00 (SAP).

**Validation Guidelines:** United States Environmental Protection Agency (USEPA) National Functional Guidelines for Inorganic Superfund Methods Data Review, OLEM 9240.1-66 EPA-542-R-20-006, November 2020, (USEPA 2020).

| Client Sample ID | Laboratory Sample ID |
|---|---|
| SIB-SW-WR198-030923 | K2302917-001 |
| SIB-SW-WR186-030923 | K2302917-002 |
| SIB-SW-WR15-030923 | K2302917-003 |
| SIB-SW-WR428-030923 | K2302917-004 |
| SIB-SW-WR71-030923 | K2302917-005 |
| SIB-SW-WR30-030923 | K2302917-006 |

Table 1 provides a summary of the major and minor data quality issues identified in this data set. All data are acceptable except those results which have been qualified with “R”, rejected. Data validation qualifiers along with associated descriptions are provided in Table 2. All data qualification related to this group of samples is detailed on the attached sheets.

All data users should note two facts. First, an "R" flag means that the associated value is unusable due to significant quality control (QC) problems, the data is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on any data tables even as a last resort. Second, no analyte concentration, even if it passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data, but any value potentially contains error.

# DATA ASSESSMENT

## 1. NARRATIVE AND COMPLETENESS REVIEW:

*The case narrative was reviewed, and the data package was checked for completeness.*

## 2. SAMPLE DELIVERY AND CONDITION:

*The samples arrived at the laboratory in acceptable condition. Proper custody was documented.*

## 3. HOLDING TIME:

**The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detect results are flagged "R", rejected. Qualifications were applied to the samples and analytes as shown below.**

*All samples were within the validation guidance.*

## 4. INSTRUMENT TUNING:

**The Inductively Coupled Plasma/Mass Spectrometer (ICP/MS) must be tuned on a daily basis prior to calibration. The ICP/MS tune serves as an initial demonstration of instrument stability and precision.**

*No problems were found for this criterion.*

## 5. CALIBRATION:

**Method requirements for satisfactory instrument calibration are established to ensure that the instrument can produce acceptable quantitative data. Initial calibration verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing calibration verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.**

### Initial and Continuing Calibration Verification

**Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s). The CCV standard shall be analyzed at a frequency of every two hours during an analytical run, at the beginning of the run, and again after the last analytical sample. The percent recovery acceptable limits for ICV/CCV are 90-110% for metals. The percent recovery acceptable limits for ICV/CCV for mercury and cyanide and are 85-115%. Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 6. BLANK CONTAMINATION:

**Quality assurance blanks (i.e. instrument, preparation, field, or rinse blanks) are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Both initial calibration and continuing calibration blanks (ICB and CCB) are used to ensure a stable instrument baseline before and during the analysis of analytical samples. Preparation blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the analytes as shown below.**

### A) Calibration Blank

*No problems were found for this criterion.*

### B) Method Blank

*Method blanks were analyzed with appropriate frequency. No problems were found for this criterion.*

### C) Field / Equipment Blank

*No sample submitted as a field / equipment blank in association with this sample delivery group (SDG).*

## 7. METAL QUANTIFICATION:

*Metals quantification was evaluated for a Level 4 review. No anomalies were identified.*

## 8. INTERFERENCE CHECK SAMPLE:

**The Interference Check Sample (ICS) is used to verify the analytical instrument's ability to overcome interferences typical of those found in samples. The laboratory analyzed and reported ICS results for all elements being reported from the analytical run and for all interferents (target and non-target) for these reported elements. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. Results for the analysis of the ICS solution must fall within the control limits of ±15% or ± 2 times the quantitation limit (whichever is greater) of the true value for the analytes and interferents included in the solution. If results that are greater than or equal to the method detection limit (MDL) are observed for analytes that are not present in the ICS solution, the possibility of false positives exists. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is greater than or equal to MDL, the possibility of false negatives in the samples exists. In general, sample data can be accepted if the concentrations of Al, Ca, Fe, and Mg in the sample are found to be less than or equal to their respective concentrations in the ICS. Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 9. LABORATORY CONTROL SAMPLE:

**The Laboratory Control Sample (LCS) and LCS duplicates (LCSD) serve as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and quality assurance/quality control (QA/QC) procedures as employed for the samples. All LCS percent recoveries and relative percent differences (RPDs) must fall within the control limits. Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 10. MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY:

**The matrix spike/matrix spike duplicate (MS/MSD) sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike percent recovery must fall within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥4x the spike added.**

**The matrix duplicate (laboratory duplicate) sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The relative percent difference (RPD) values between the pair must fall within +/- 20 or the absolute difference between the matrix duplicate results must be less than the reporting limit.**

*No sample was submitted for MS/MSD and/or matrix duplicate evaluations in association with this SDG.*

## 11. ICP SERIAL DILUTION:

**The serial dilution determines whether significant physical or chemical interferences exist due to sample matrix. If the analyte concentration is sufficiently high (concentration in the original sample is greater than 50 times the MDL, the percent difference between the original determination and the serial dilution analysis (a five-fold dilution) after correction for dilution shall be less than 20.**

*No sample was submitted for serial dilution evaluation in association with this SDG.*

## 12. INTERNAL STANDARDS PERFORMANCE:

**Internal standards were added to all sample and quality assurance evaluation digestates prior to analysis to monitor analytical performance and sample matrix effects. All samples and associated quality assurance analyses are verified to ensure percent recoveries are within validation acceptance criteria of 60-125%.**

*No problems were found for this criterion.*

## 13. FIELD DUPLICATES:

**Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 30% for aqueous sample and 50% for solid samples for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values greater than or equal to five times the LOQ. A control limit of two times the LOQ shall be used if either the sample or duplicate value is less than five times the LOQ. For field duplicate analyses that do not meet the technical criteria, the action was applied to only the field sample and its duplicate.**

*No samples were submitted as a field duplicate pair in association with this SDG.*

## 14. REPORTING LIMITS, DILUTIONS, RE-EXTRACTIONS & REANALYSIS:

**Samples may be re-analyzed for dilution, re-extraction and for other QC reasons. In such cases, the best result values are used.**

*No dilutions, re-extractions, or other re-analyses performed on any sample associated with this SDG were reported by the laboratory.*

## 15. OTHER PROBLEMS:

*None.*

**Table 1 Major and Minor Findings**

| | Were acceptance criteria met? | | |
|---|---|---|---|
| | Yes | No | |
| **Metals** | | Major | Minor |
| Holding Time | x | | |
| Tune | x | | |
| Calibration | x | | |
| Blank Contamination | x | | |
| Interference Check Samples | x | | |
| Laboratory Control Samples | x | | |
| Matrix Spike/Matrix Spike Duplicate/Matrix Duplicate | NA | | |
| ICP Serial Dilution | NA | | |
| Internal Standards Performance | x | | |
| Field Duplicate | NA | | |
| Reporting Limits | x | | |
| Other Quality Control Data out of Specification | x | | |

Major = Major data quality issue identified resulting in rejection of data.
Minor = Minor data quality issue identified resulting in the qualification of data. Data qualification should be used to inform the data users of data limitations.
NA = Not applicable

**Table 2 Data Validation Qualifiers**

| Data Qualifier | Definition |
|---|---|
| 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. |
| UJ | The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |
| R | The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample. |

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## EXECUTIVE NARRATIVE

**Sample Delivery Group**: K2302917
**Laboratory:** ALS Environmental
**Site:** Portland Harbor Superfund Site
**Sampling dates**: 03/09/2023
**Number of Samples:** 6
**Test Method:** SW 9060 and SM2540D
**Analysis:** Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), and Total Suspended Solids (TSS)

**Validation Level:** Level 2A (TOC/DOC) and Level 4 (TSS)

**Quality Assurance Project Plan:** Uniform Federal Policy-Quality Assurance Project Plan, Revision 3, Remedial Design Services, Swan Island Basin Project Area, Portland Harbor Superfund Site, Portland Multnomah County, Oregon, May 2022 (QAPP).

**Validation Guidelines:** United States Environmental Protection Agency (USEPA) National Functional Guidelines for Organic Superfund Methods Data Review, OLEM 9240.0-51, EPA-540-R-20-005, (November 2020).

| Client Sample ID | Laboratory Sample ID | TOC | DOC | TSS |
|---|---|---|---|---|
| SIB-SW-WR198-030923 | K2302917-001 | X | X | X |
| SIB-SW-WR186-030923 | K2302917-002 | X | X | X |
| SIB-SW-WR15-030923 | K2302917-003 | X | X | X |
| SIB-SW-WR428-030923 | K2302917-004 | X | X | X |
| SIB-SW-WR71-030923 | K2302917-005 | X | X | X |
| SIB-SW-WR30-030923 | K2302917-006 | X | X | X |

Table 1 provides a summary of the major and minor data quality issues applied to this data set. All data are acceptable except those results which have been qualified with “R”, rejected. Data validation qualifiers along with associated descriptions are provided in Table 2. All data qualification related to this group of samples is detailed on the attached sheets.

All data users should note two facts. First, an "R" flag means that the associated value is unusable due to significant quality control (QC) problems, the data is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on any data tables even as a last resort. Second, no analyte concentration, even if it passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error.

# DATA ASSESSMENT

## 1. NARRATIVE AND COMPLETENESS REVIEW

*The case narrative was reviewed, and the data package was checked for completeness. No discrepancies were noted.*

## 2. SAMPLE DELIVERY AND CONDITION

*The samples arrived at the laboratory in acceptable condition. Proper custody was documented.*

## 3. HOLDING TIME

**The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". When holding times are exceeded the non-detects will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 4. BLANK CONTAMINATION

**Quality assurance (QA) blanks; i.e. method, trip, field, or rinse blanks; are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Trip blanks measure cross-contamination of samples during shipment. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.**

### A) Method blank contamination

*No problems were found for this criterion.*

### B) Field/Equipment blank contamination

*No sample was submitted as a field/equipment blank in association with the samples in this sample delivery group (SDG).*

## 5. ANALYTE QUANTIFICATION AND IDENTIFICATION

*Analyte quantitation was verified as part of the Level 4 data validation for TSS. No anomalies were identified.*

## 6. LABORATORY CONTROL SAMPLE

**The Laboratory Control Sample (LCS) serves to monitor the overall performance of each step during the analysis. Aqueous/water and soil/sediment LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and quality assurance/quality control procedures as employed for the samples. All LCS percent recoveries must fall within the QAPP established acceptance limits. Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 7. MATRIX SPIKE/MATRIX SPIKE DUPLICATE/MATRIX DUPLICATE

**The matrix spike/matrix spike duplicate (MS/MSD) sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike percent recovery and MS/MSD relative percent differences (RPDs) must fall within the QAPP established acceptance limits. However, spike recovery limits do not apply when the sample concentration is greater than or equal to four times the spike added.**

*Sample SIB-SW-WR198-030923 was submitted for MS and matrix duplicate pair evaluations in association with this SDG for TOC. Upon evaluation, all precision and accuracy indicators were favorable.*

## 8. FIELD DUPLICATES:

**Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 50% for the Relative Percent Difference (RPD) for solid samples and 30% RPD for liquid samples shall be used for original and duplicate sample values greater than or equal to five times the Project Quantitation Limit (PQL). A control limit of two times the PQL shall be used if either the sample or duplicate value is less than five times the PQL. For field duplicate analyses that do not meet the technical criteria, the action was applied to only the field sample and its duplicate.**

*No samples were submitted as a field duplicate pair in association with this SDG.*

## 9. PROJECT DEFINED REPORTING LIMITS:

*No problems were found for this criterion.*

## 10. OTHER PROBLEMS:

*None.*

**Table 1 Major and Minor Findings**

| | Were acceptance criteria met? | | |
|---|---|---|---|
| | Yes | No | |
| **TOC** | | **Major** | **Minor** |
| Holding Time | x | | |
| Method Blank | x | | |
| Field Blank | NA | | |
| Matrix Spike/Matrix Spike Duplicate | x | | |
| Laboratory Duplicate | x | | |
| Laboratory Control Sample | x | | |
| Field Duplicate | NA | | |
| Project Reporting Limits | x | | |
| Other | x | | |
| | **Were acceptance criteria met?** | | |
| | **Yes** | **No** | |
| **DOC** | | **Major** | **Minor** |
| Holding Time | x | | |
| Method Blank | x | | |
| Field Blank | NA | | |
| Matrix Spike/Matrix Spike Duplicate | NA | | |
| Laboratory Duplicate | NA | | |
| Laboratory Control Sample | x | | |
| Field Duplicate | NA | | |
| Project Reporting Limits | x | | |
| Other | x | | |
| | **Were acceptance criteria met?** | | |
| | **Yes** | **No** | |
| **TSS** | | **Major** | **Minor** |
| Holding Time | x | | |
| Method Blank | x | | |
| Field Blank | NA | | |
| Analyte Quantitation and Identification | x | | |
| Matrix Spike/Matrix Spike Duplicate | NA | | |
| Laboratory Duplicate | NA | | |
| Laboratory Control Sample | x | | |
| Field Duplicate | NA | | |
| Project Reporting Limits | x | | |
| Other | x | | |

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NA = Not applicable

**Table 2 Data Validation Qualifiers**

| Data Qualifier | Definition |
|---|---|
| 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. |
| UJ | The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |
| R | The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample. |
| N | The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification. |
| NJ | The analyte was tentatively identified, and the associated numerical value represents its approximate concentration. |

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## EXECUTIVE NARRATIVE

**Sample Delivery Group**: K2302917
**Laboratory:** ALS Environmental
**Site:** Portland Harbor Superfund Site
**Sampling dates**: 3/9/2023
**Number of Samples:** 6
**Test Method:** Method ALS SOP, Butyltins, Rev 16.0, SOC-BUTYL 12/2/2020
**Analysis:** Tributyltin

**Validation Level:** Level 4

**Quality Assurance Project Plan:** Uniform Federal Policy-Quality Assurance Project Plan, Revision 3, Remedial Design Services, Swan Island Basin Project Area, Portland Harbor Superfund Site, Portland Multnomah County, Oregon, May 2022 (QAPP).

**Validation Guidelines:** United States Environmental Protection Agency (USEPA) National Functional Guidelines for Organic Superfund Methods Data Review, OLEM 9240.0-51, EPA-540-R-20-005, (November 2020).

| Client Sample ID | Laboratory Sample ID |
|---|---|
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| SIB-SW-WR30-030923 | K2302917-006 |

Table 1 provides a summary of the major and minor data quality issues identified in this data set. All data are acceptable except those results which have been qualified with “R”, rejected. Data validation qualifiers along with associated descriptions are provided in Table 2. All data qualification related to this group of samples is detailed on the attached sheets.

All data users should note two facts. First, an "R" flag means that the associated value is unusable due to significant quality control (QC) problems, the data is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on any data tables even as a last resort.

Second, no analyte concentration, even if it passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data, but any value potentially contains error.

# ORGANIC DATA ASSESSMENT

## 1. NARRATIVE AND COMPLETENESS REVIEW:

*The case narrative was reviewed, and the data package was checked for completeness. No discrepancies were noted.*

## 2. SAMPLE DELIVERY AND CONDITION:

*The samples arrived at the laboratory in acceptable condition. Proper custody was documented. No qualification was required.*

## 3. HOLDING TIME:

**The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detect results are flagged "R", rejected. Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 4. CALIBRATION

**Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.**

**Percent relative standard deviation (%RSD) is calculated from the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent difference (%D) compares the response factor of the continuing calibration check to the mean response factor (RRF) from the initial calibration. Percent difference is a measure of the instrument's daily performance. For organotins, if %RSD exceeds limits outlined in validation guidance, qualify all associated positive results "J". If the %D exceeds 25% for any analyte, qualify all associated positive results "J" and non-detects "UJ". If %RSD and %D grossly exceed QC criteria, non-detect data may be qualified "R".**

*No problems were found for initial and continuing calibrations.*

## 5. BLANK CONTAMINATION:

**Quality assurance (QA) blanks; i.e. method, trip, field, or rinse blanks; are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Trip blanks measure cross-contamination of samples during shipment. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.**

### A) Method blank contamination:

*No problems were found for this criterion.*

### B) Field/Equipment blank contamination:

*No sample was submitted as a field/equipment blank in association with the samples in this sample delivery group (SDG).*

## 6. SURROGATES/SYSTEM MONITORING COMPOUNDS

**All samples are spiked with surrogate/system monitoring compounds (SMC) prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate/SMC concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below. Surrogate recovery should meet the limits established in the QAPP for this data set.**

*No problems were found for this criterion.*

## 7. COMPOUND QUANTIFICATION

*Target compound result quantitation was reviewed at the Stage 4 Level. No anomalies were identified.*

*Manual integrations were reviewed at the stage 4 validation level. No anomalies were identified.*

## 8. COMPOUND IDENTIFICATION:

**Organotin**

**The retention times (RTs) of reported compounds must fall within the calculated retention time windows for the two chromatographic columns. The percent difference (%D) of the positive results obtained on the two GC columns should be less than or equal to 40%.**

**Retention Time**

*No problems were found for this criterion.*

**Percent Difference**

*No positive results were reported for this SDG.*

## 9. MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY:

**Matrix spike/matrix spike duplicate (MS/MSD) data is generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD data may be used in conjunction with other quality control criteria for additional qualification of data. The spiking compound recoveries and relative percent differences (RPDs) should meet the limits established in the QAPP.**

*No sample was submitted for MS/MSD pair evaluation in association with this SDG.*

## 10. FIELD DUPLICATES:

**Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 50% for the Relative Percent Difference (RPD) for solid samples and 30% RPD for liquid samples shall be used for original and duplicate sample values greater than or equal to five times the Project Quantitation Limit (PQL). A control limit of two times the PQL for solid samples and +/- the PQL for liquid samples shall be used if either the sample or duplicate value is less than five times the PQL. For field duplicate analyses that do not meet the technical criteria, the action was applied to only the field sample and its duplicate.**

*No samples were submitted as a field duplicate pair in association with this SDG.*

## 11. LABORATORY CONTROL SAMPLES:

**The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and quality assurance/quality control (QA/QC) procedures as employed for the samples. All LCS percent recoveries must fall within the QAPP control limits. Qualifications were applied to the samples and analytes as shown below.**

*The LCS evaluations were performed at the appropriate frequency. No problems were found for this criterion.*

## 12. OTHER PROBLEMS:

*None.*

## 13. DILUTIONS, RE-EXTRACTIONS & REANALYSIS:

**Samples may be re-analyzed for dilution, re-extraction and for other QC reasons. In such cases, the best result values are used.**

*No problems were identified. Dilutions were performed as necessary to bring target analyte concentrations into calibration range.*

## 14. PROJECT DEFINED REPORTING LIMITS:

*No problems were found for this criterion.*

**Table 1 Major and Minor Findings**

| | Were acceptance criteria met? | | |
|---|---|---|---|
| | Yes | No | |
| **Tributyltin** | | Major | Minor |
| Holding Time | x | | |
| Percent Relative Standard Deviation and Percent Difference | x | | |
| Method Blank | x | | |
| Equipment/Field Blank | NA | | |
| Surrogates/System Monitoring Compounds | x | | |
| Compound Quantification | x | | |
| Compound Identification | x | | |
| Matrix Spike/Matrix Spike Duplicate | NA | | |
| Field Duplicate | NA | | |
| Laboratory Control Samples | x | | |
| Other Quality Control Data out of Specification | x | | |
| Project Reporting Limits | x | | |

Major = Major data quality issue identified resulting in rejection of data.
Minor = Minor data quality issue identified resulting in the qualification of data. Data qualification should be used to inform the data users of data limitations.
NA = Not applicable

**Table 2 Data Validation Qualifiers**

| Data Qualifier | Definition |
|---|---|
| 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. |
| UJ | The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |
| R | The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample. |

ENVIRONMENTAL DATA SERVICES, LTD.

## EXECUTIVE NARRATIVE

**Sample Delivery Group**: K2302917
**Laboratory:** ALS Environmental
**Site:** Portland Harbor Superfund Site
**Sampling dates**: 03/09/2023
**Number of Samples:** 6
**Test Method:** SW 846 8270D Low Level; SW 846 8270D SIM
**Analysis:** Bis(2-Ethylhexyl)phthalate and Pentachlorophenol; Polynuclear Aromatic Hydrocarbons (PAHs) SIM

**Validation Level:** Level 4

**Quality Assurance Project Plan:** Uniform Federal Policy-Quality Assurance Project Plan, Revision 3, Remedial Design Services, Swan Island Basin Project Area, Portland Harbor Superfund Site, Portland Multnomah County, Oregon, May 2022 (QAPP).

**Validation Guidelines:** United States Environmental Protection Agency (USEPA) National Functional Guidelines for Organic Superfund Methods Data Review, OLEM 9240.0-51, EPA-540-R-20-005, (November 2020).

| Client Sample ID | Laboratory Sample ID |
|---|---|
| SIB-SW-WR198-030923 | K2302917-001 |
| SIB-SW-WR186-030923 | K2302917-002 |
| SIB-SW-WR15-030923 | K2302917-003 |
| SIB-SW-WR428-030923 | K2302917-004 |
| SIB-SW-WR71-030923 | K2302917-005 |
| SIB-SW-WR30-030923 | K2302917-006 |

Table 1 provides a summary of the major and minor data quality issues identified this data set. All data are acceptable except those results which have been qualified with “R”, rejected. Data validation qualifiers along with associated descriptions are provided in Table 2. All data qualification related to this group of samples is detailed on the attached sheets.

All data users should note two facts. First, an "R" flag means that the associated value is unusable due to significant quality control (QC) problems, the data is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on any data tables even as a last resort. Second, no analyte concentration, even if it passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data, but any value potentially contains error.

# DATA ASSESSMENT

## 1. NARRATIVE AND COMPLETENESS REVIEW:

*The case narrative was reviewed, and the data package was checked for completeness. No discrepancies were noted.*

## 2. SAMPLE DELIVERY AND CONDITION:

*The samples arrived at the laboratory in acceptable condition. Proper custody was documented.*

## 3. HOLDING TIME:

**The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detect results are flagged "R", rejected. Qualifications were applied to the samples and analytes as shown below.**

*All sample analyses reported were within the validation guidance.*

## 4. MASS SPECTROMETER TUNING:

**Tuning and performance criteria are established to ensure adequate mass resolution, proper identification of compounds and to some degree, sufficient instrument sensitivity. These criteria are not sample specific. Instrument performance is determined using standard materials. Therefore, these criteria should be met in all circumstances. The tuning standard for semi-volatile organics is decafluorotriphenylphosphine. If the mass calibration is in error, all associated data will be classified as unusable "R". Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 5. CALIBRATION:

**Satisfactory instrument calibration is established to ensure that the instrument can produce acceptable quantitative data. An initial calibration demonstrates that the instrument can give acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.**

### A) Response Factor

**The response factor measures the instrument's response to specific chemical compounds. All analytes for initial and continuing calibration should meet the minimum relative response factor (RRF) criteria as listed in the USEPA National Functional Guidelines for Superfund Organic Methods Data Review. If the RRF is less than minimum RRF specified, use professional judgment and all detects in the sample will be qualified as "J" or "R". All non-detects for that compound will be rejected "R". Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

**B) Percent Relative Standard Deviation and Percent Difference**

**Percent relative standard deviation (%RSD) is calculated for the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent difference (%D) compares the response factor of the continuing calibration check to the mean RRF from the initial calibration.**

**Percent RSD must be less than maximum %RSD listed in the USEPA National Functional Guidelines for Superfund Organic Methods Data Review for all target analytes. In cases where linear and non-linear regressions are used, correlation coefficients must be greater than 0.995. For the opening or closing continuing calibration verification (CCV) the %D must be within the inclusive opening or closing maximum %D limits for all target compounds. A value outside of these limits indicates potential detection and quantitation errors. If the %RSD exceeds quality control criteria, detects may be qualified as "J" and professional judgment is used to qualify non-detects. If the %D exceeds quality control criteria, the positive results are flagged as estimated, "J" and non-detects are flagged "UJ". Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

**6. BLANK CONTAMINATION:**

**Quality assurance (QA) blanks (i.e. method, trip, field, or rinse blanks) are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.**

**A) Method blank contamination**

*No problems were found for this criterion with the following exceptions. The target analytes listed below were positively identified in the method associated with all the samples in this sample delivery group (SDG).*

| | | |
|---|---|---|
| *2-methylnaphthalene* | *benzo(a)anthracene* | *naphthalene* |
| *acenaphthene* | *fluoranthene* | *phenanthrene* |
| *anthracene* | *fluorene* | *pyrene* |

*Positive sample results for the impacted analytes have been evaluated and qualified per validation guidance.*

**B) Field/Equipment blank contamination**

*No sample was submitted for the field/equipment blank in association with the samples in this sample delivery group (SDG).*

**7. SURROGATES:**

**All samples are spiked with system monitoring compounds prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate recovery limits were outside quality control limits established in the QAPP, qualifications were applied to all the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 8. COMPOUND IDENTIFICATION AND QUANTIFICATION:

### Compound Identification

**The compounds are identified on the GC/MS by using the analytes relative retention time (RRT) and ion spectra. For the results to be a positive hit the sample peak must be within ±0.06 RRT units of the standard compound and have an ion spectrum which has a ratio of the primary and secondary m/e intensities within 20% of that in the standard compound. In the cases where there is not an adequate ion spectrum match, the laboratory may have provided false positive identifications.**

*Target compound identifications were reviewed at the Stage 4 level. No anomalies were identified.*

*Tentatively Identified Compounds (TICs) were not reported.*

### Compound Quantification

*Target compound result quantitation was reviewed at the Stage 4 level. No anomalies were identified.*

*Manual integrations were reviewed at the Stage 4 level. No anomalies were identified.*

## 9. MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY:

**Matrix spike/matrix spike duplicate (MS/MSD) data is generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD data may be used in conjunction with other quality control criteria for additional qualification of data.**

*No sample was submitted for MS/MSD pair evaluation in association with this SDG.*

## 10. INTERNAL STANDARDS PERFORMANCE:

**Internal standard performance criteria are meant to ensure that the gas chromatograph/mass spectrometer (GC/MS) sensitivity and response are stable during every experimental run.**

**The internal standard area count must not vary by more than a factor of two from the associated continuing calibration standard. The retention time of the internal standard must not vary by more than ±30 seconds from the associated continuing calibration standard. The area count must be within a (50-200%) range of the associated standard. If the area count is greater than 200%, non-detected results are not qualified, and positive results are flagged as estimated "J-". If the area count is less than 50%, positive results are flagged as estimated "J+" and non-detected results are flagged “UJ”. If the area count is less than 20%, positive results are flagged as estimated "J+" and non-detected results will be classified as unusable "R". Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 11. FIELD DUPLICATES:

**Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 50% for the Relative Percent Difference (RPD) for solid samples and 30% RPD for liquid samples shall be used for original and duplicate sample values greater than or equal to five times the Project Quantitation Limit (PQL). A control limit of two times the PQL shall be used for solid samples and +/- the PQL for liquid samples if either the sample or duplicate value is less than five times the PQL. For field duplicate analyses that do not meet the technical criteria, the action was applied to only the field sample and its duplicate.**

*No samples were submitted as a field duplicate pair in association with this SDG.*

## 12. LABORATORY CONTROL SAMPLES:

**The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and quality assurance/quality control (QA/QC) procedures as employed for the samples. All LCS percent recoveries must fall within the QAPP control limits. Qualifications were applied to the samples and analytes as shown below.**

*No problems were found for this criterion.*

## 13. DILUTIONS, RE-EXTRACTIONS & REANALYSIS:

**Samples may be re-analyzed for dilution, re-extraction and for other QC reasons. In such cases, the best result values are used.**

*No problems were found for this criterion.*

## 14. PROJECT DEFINED REPORTING LIMITS:

*No problems were found for this criterion.*

## 15. OTHER PROBLEMS:

*None.*

**Table 1 Major and Minor Findings**

| | Were acceptance criteria met? | | |
|---|---|---|---|
| | Yes | No | |
| **Bis(2-Ethylhexyl)phthalate/Pentachlorphenol** | | Major | Minor |
| Holding Time | x | | |
| Mass Spectrometer Tuning | x | | |
| Calibration | x | | |
| Response Factor | x | | |
| Percent Relative Standard Deviation and Percent Difference | x | | |
| Internal Standards | x | | |
| Method Blank | x | | |
| Equipment/Rinse Blank | NA | | |
| Surrogates | x | | |
| Matrix Spike/Matrix Spike Duplicate | NA | | |
| Field Duplicate | NA | | |
| Laboratory Control Samples | x | | |
| Project Reporting Limits | x | | |
| Other Quality Control Data out of Specification | x | | |
| | **Were acceptance criteria met?** | | |
| | **Yes** | **No** | |
| **PAHs SIM** | | **Major** | **Minor** |
| Holding Time | x | | |
| Mass Spectrometer Tuning | x | | |
| Response Factor | x | | |
| Percent Relative Standard Deviation and Percent Difference | x | | |
| Internal Standards | x | | |
| Method Blank | | | x |
| Equipment/Rinse Blank | NA | | |
| Surrogates | x | | |
| Matrix Spike/Matrix Spike Duplicate | NA | | |
| Field Duplicate | NA | | |
| Laboratory Control Samples | x | | |
| Project Reporting Limits | x | | |
| Other Quality Control Data out of Specification | x | | |

Major = Major data quality issue identified resulting in rejection of data.
Minor = Minor data quality issue identified resulting in the qualification of data. Data qualification should be used to inform the data users of data limitations.
NA = Not applicable

**Table 2 Data Validation Qualifiers**

| Data Qualifier | Definition |
|---|---|
| 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. |
| NJ | The analyte has been “tentatively identified” or “presumptively” as present and the associated numerical value is the estimated concentration in the sample. |
| UJ | The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |
| R | The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample. |

# Qualified Sample Result Summaries

Summary of Qualified Results

[1] Validated results assigned by validator that are different than those assigned by the laboratory have been indicated in bold blue font.

| Sample | Lab ID | Analyte | Validated Result[1] | lab_qualifiers | validator_qualifiers | interpreted_qualifiers | unit | Reason Codes |
|---|---|---|---|---|---|---|---|---|
| SIB-SW-WR198-030923 | K2302917-001 | ANTHRACENE | 1.3 | J | U | U | ng/l | MBL |
| SIB-SW-WR198-030923 | K2302917-001 | BENZO(A)ANTHRACENE | 0.9 | J | U | U | ng/l | MBL |
| SIB-SW-WR198-030923 | K2302917-001 | ACENAPHTHENE | 1.7 | J | U | U | ng/l | MBL |
| SIB-SW-WR198-030923 | K2302917-001 | PHENANTHRENE | 10 | | U | U | ng/l | MBL |
| SIB-SW-WR198-030923 | K2302917-001 | FLUORENE | 2.1 | J | U | U | ng/l | MBL |
| SIB-SW-WR198-030923 | K2302917-001 | NAPHTHALENE | 6.2 | | U | U | ng/l | MBL |
| SIB-SW-WR186-030923 | K2302917-002 | ANTHRACENE | 1.5 | J | U | U | ng/l | MBL |
| SIB-SW-WR186-030923 | K2302917-002 | BENZO(A)ANTHRACENE | 1.4 | J | U | U | ng/l | MBL |
| SIB-SW-WR186-030923 | K2302917-002 | PHENANTHRENE | 13 | | U | U | ng/l | MBL |
| SIB-SW-WR186-030923 | K2302917-002 | FLUORENE | 2.2 | J | U | U | ng/l | MBL |
| SIB-SW-WR186-030923 | K2302917-002 | NAPHTHALENE | 7.4 | | U | U | ng/l | MBL |
| SIB-SW-WR15-030923 | K2302917-003 | BENZO(A)ANTHRACENE | 1.3 | J | U | U | ng/l | MBL |
| SIB-SW-WR15-030923 | K2302917-003 | ACENAPHTHENE | 2.3 | J | U | U | ng/l | MBL |
| SIB-SW-WR15-030923 | K2302917-003 | NAPHTHALENE | 9.4 | | U | U | ng/l | MBL |
| SIB-SW-WR428-030923 | K2302917-004 | BENZO(A)ANTHRACENE | 3.1 | J | U | U | ng/l | MBL |
| SIB-SW-WR428-030923 | K2302917-004 | ACENAPHTHENE | 1.9 | J | U | U | ng/l | MBL |
| SIB-SW-WR428-030923 | K2302917-004 | FLUORENE | 3.5 | | U | U | ng/l | MBL |
| SIB-SW-WR428-030923 | K2302917-004 | NAPHTHALENE | 8.1 | | U | U | ng/l | MBL |
| SIB-SW-WR428-030923 | K2302917-004 | 2-METHYLNAPHTHALENE | 3.4 | | U | U | ng/l | MBL |
| SIB-SW-WR71-030923 | K2302917-005 | PYRENE | 3.8 | | U | U | ng/l | MBL |
| SIB-SW-WR71-030923 | K2302917-005 | FLUORANTHENE | 1.8 | J | U | U | ng/l | MBL |
| SIB-SW-WR71-030923 | K2302917-005 | PHENANTHRENE | 3.3 | | U | U | ng/l | MBL |
| SIB-SW-WR71-030923 | K2302917-005 | NAPHTHALENE | 3.9 | | U | U | ng/l | MBL |
| SIB-SW-WR30-030923 | K2302917-006 | ANTHRACENE | 1.6 | J | U | U | ng/l | MBL |
| SIB-SW-WR30-030923 | K2302917-006 | ACENAPHTHENE | 3.1 | J | U | U | ng/l | MBL |
| SIB-SW-WR30-030923 | K2302917-006 | PHENANTHRENE | 13 | | U | U | ng/l | MBL |

# ATTACHMENT E
# Data Qualification Reason Codes

| QC Element | Reason Code | Definition |
|---|---|---|
| Ambient Blank | ABH | Ambient blank result ≥ limit of quantitation (LOQ) |
| Ambient Blank | ABHB | Result is judged to be biased high based on associated ambient blank result |
| Ambient Blank | ABL | Ambient blank result <LOQ |
| Analyte Quantitation | ACR | Result above the upper end of the calibrated range |
| Analyte Quantitation | EXC | Result excluded; another data point for this analyte was selected for use (use with X-qualified results) |
| Analyte Quantitation | RTW | Target analyte outside retention time window |
| Analyte Quantitation | PSL | Solid matrix sample with percent solids less than 50% |
| Analyte Quantitation | PSLX | Solid matrix sample with percent solids less than 10% |
| Analyte Quantitation | TR | Result between the detection limit and LOQ |
| Calibration Blank | CBH | Initial or continuing calibration blank result ≥LOQ |
| Calibration Blank | CBHB | Result is judged to be biased high based on associated continuing calibration blank result |
| Calibration Blank | CBL | Initial or continuing calibration blank result <LOQ |
| Calibration Blank | CBN | Negative initial or continuing calibration blank result with absolute value <LOQ |
| Calibration Blank | CBNH | Negative initial or continuing calibration blank result with absolute value ≥LOQ |
| Continuing Calibration | CCCC | Calibration check compound did not meet percent difference (%D) criterion in continuing calibration standard |
| Continuing Calibration | CCVD | Continuing calibration standard did not meet %D criterion |
| Continuing Calibration | CRFL | Continuing calibration RRF below acceptance criterion |
| Continuing Calibration | CSPC | System performance check compound did not meet minimum RRF criterion in continuing calibration |
| Continuing Calibration | CVDX | Continuing calibration standard did not meet %D criterion, extreme discrepancy |
| Confirmation | CF | Confirmation precision exceeded acceptance criterion |
| Cyanide Method | DSH | High-level distillation standard did not meet %D criterion |
| Cyanide Method | DSL | Low-level distillation standard did not meet %D criterion |
| Equipment Blank | EBH | Equipment blank result ≥LOQ |
| Equipment Blank | EBHB | Result is judged to be biased high based on associated equipment blank result |
| Equipment Blank | EBL | Equipment blank result <LOQ |
| Field Duplicate | FDPA | Field duplicate results did not meet absolute difference criterion |
| Field Duplicate | FDPR | Field duplicate results did not meet RPD criterion |
| Holding Time | HTA | Analytical holding time exceeded |
| Holding Time | HTAX | Analytical holding time exceeded, extreme discrepancy |
| Holding Time | HTP | Preparation holding time exceeded |
| Holding Time | HTPX | Preparation holding time exceeded, extreme discrepancy |
| Initial Calibration | ICCC | Calibration check compound did not meet percent relative standard deviation (%RSD) criterion in initial calibration |

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## ATTACHMENT E (continued)
## Data Qualification Reason Codes

| QC Element | Reason Code | Definition |
|---|---|---|
| Initial Calibration | ICLS | Initial calibration low-level standard >LOQ |
| Initial Calibration | ICR2 | Initial calibration $r^2$ below acceptance criterion |
| Initial Calibration | ICRD | Initial calibration %RSD above acceptance criterion |
| Initial Calibration | ICRX | Initial calibration %RSD above acceptance criterion, extreme discrepancy |
| Initial Calibration | IRFL | Initial calibration RRF below acceptance criterion |
| Initial Calibration | ISPC | System performance check compound did not meet minimum mean RRF criterion in initial calibration |
| Initial Calibration | LQSH | LOQ check standard above acceptance criteria |
| Initial Calibration | LQSL | LOQ check standard below acceptance criteria |
| Initial Calibration | SSVD | Second-source standard did not meet %D criterion |
| Initial Calibration Verification | ICVD | Continuing calibration standard did not meet %D criterion |
| Initial Calibration Verification | ICVX | Continuing calibration standard did not meet %D criterion, extreme discrepancy |
| Interference Check Standard | ICAH | Non-spiked concentration above acceptance criterion in ICSA |
| Interference Check Standard | ICAN | Negative concentration with absolute value above acceptance criterion in ICSA |
| Interference Check Standard | ICHX | Non-spiked concentration above acceptance criterion in ICSA, extreme discrepancy |
| Interference Check Standard | ICNX | Negative concentration with absolute value above acceptance criterion in ICSA, extreme discrepancy |
| Interference Check Standard | ICSH | ICSA or ICSAB spiked analyte with high percent recovery (%R) |
| Interference Check Standard | ICSL | ICSA or ICSAB spiked analyte with low %R |
| Internal Standards | IRH | Internal standard peak area above upper limit |
| Internal Standards | IRL | Internal standard peak area below lower limit |
| Internal Standards | IRLX | Internal standard peak area below lower limit, extreme discrepancy |
| Internal Standards | ISRT | Internal standard retention time outside window |
| Labeled Standards | LSH | Labeled standard %R above acceptance criterion |
| Labeled Standards | LSL | Labeled standard %R below acceptance criterion |
| Labeled Standards | LSLX | Labeled standard %R below acceptance criterion, extreme discrepancy |
| Laboratory Control Sample | LCLX | LCS and/or LCSD %R below acceptance criterion, extreme discrepancy |
| Laboratory Control Sample | LCSH | LCS and/or LCSD %R above acceptance criterion |
| Laboratory Control Sample | LCSL | LCS and/or LCSD %R below acceptance criterion |
| Laboratory Control Sample | LCSP | LCS/LCSD RPD above acceptance criterion |
| Laboratory Duplicate | LDPA | Laboratory duplicate results did not meet absolute difference criterion |
| Laboratory Duplicate | LDPR | Laboratory duplicate results did not meet RPD criterion |

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| QC Element | Reason Code | Definition |
|---|---|---|
| Low-Level Calibration Check | LLCH | Low-level calibration check above the upper limit |
| Low-Level Calibration Check | LLCL | Low-level calibration check below the lower limit |
| Low-Level Calibration Check | LLXL | Low-level calibration check below the lower limit, extreme discrepancy |
| Method Blank | MBH | Method blank result ≥LOQ |
| Method Blank | MBHB | Result is judged to be biased high based on associated method blank result |
| Method Blank | MBL | Method blank result <LOQ |
| Matrix Spike | MSH | MS and/or MSD %R above acceptance criterion |
| Matrix Spike | MSL | MS and/or MSD %R below acceptance criterion |
| Matrix Spike | MSLX | MS and/or MSD %R below acceptance criterion, extreme discrepancy |
| Matrix Spike | MSP | MS/MSD RPD above acceptance criterion |
| Post-Digestion Spike | PDH | Post-digestion spike recovery high |
| Post-Digestion Spike | PDL | Post-digestion spike recovery low |
| Post-Digestion Spike | PDLX | Post-digestion spike recovery low, extreme discrepancy |
| Post-Digestion Spike | PDN | Post-digestion spike not performed or not applicable and serial dilution result not performed or not applicable |
| Sample Delivery and Condition | BUB | Bubbles >5 millimeters in volatile organic compounds vial |
| Sample Delivery and Condition | DAM | Sample container damaged |
| Sample Delivery and Condition | PRE | Sample not properly preserved |
| Sample Delivery and Condition | TEMP | Sample received at elevated temperature |
| Sample Delivery and Condition | TMPX | Sample received at elevated temperature, extreme discrepancy |
| Serial Dilution | SDIL | Serial dilution did not meet %D criterion |
| Serial Dilution | SDN | Serial dilution not performed |
| Surrogate | SSH | Surrogate %R high |
| Surrogate | SSL | Surrogate %R low |
| Surrogate | SSLX | Surrogate %R low, extreme discrepancy |
| Surrogate | SSN | Surrogate compound not spiked into sample |
| Trip Blank | TBH | Trip blank result ≥LOQ |
| Trip Blank | TBL | Trip blank result <LOQ |
| Validator Judgment | VJ | Validator judgment (see validation narrative) |

ICS = interference check sample
MS = matrix spike
MSD = matrix spike duplicate
QC = quality control
RPD = relative percent difference
RRF = relative response factor

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# Data Validation Worksheet

**CLIENT:** HGL

**PROJECT:** Portland Harbor Superfund Site

**SDG:** K2302917

**LAB:** ALS Kelso

**Validated by:** DLW

**Validation date:** 5/26/23

**Reviewed by: PMH**

**Review date: 5/26/23**

Herbicide – MCPP only

**Sample Receipt / Hold times (COC, receipt logs, case narrative)**

✓Temperature upon receipt (<6◦C)

✓HT

## WORKSHEET #19 AND 30.1 (CONTINUED)
## SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

| Matrix | Parameter | Analytical and Preparation Method/SOP Reference | Containers | Preservation Requirements | Maximum Holding Time |
|---|---|---|---|---|---|
| Centrifuged HVS Stormwater | VOCs | 8260C (VOC-8260) | NA[4] | NA[4] | 14 days |
| | SVOCs | 8270D (SVM-8270L) and 3510C (EXT-3510) | NA[4] | NA[4] | 7 days to prepare and 40 days from extraction to analysis |
| | PAHs | 8270D-SIM (SVM-8270S) and 3520C (EXT-3520) | NA[4] | NA[4] | 7 days to prepare and 40 days from extraction to analysis |
| | Herbicides | 8151A (SOC-8151) | NA[4] | NA[4] | 7 days to prepare and 40 days from extraction to analysis |

**Pesticides Table 1. Preservation and Holding Time Actions**

| Matrix | Preservation | Criteria | Action | |
|---|---|---|---|---|
| | | | Detect | Non-detect |
| Aqueous/Non-aqueous | Samples received at a temperature > 6°C | | J | UJ |
| Aqueous/Non-aqueous | Cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared within the 14-day technical holding time | No qualification | No qualification |
| | Not cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared within the 14-day technical holding time | Use professional judgment | Use professional judgment |
| | Cooled/not cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared outside the 14-day technical holding time | J | R |

| Matrix | Preservation | Criteria | Action | |
|---|---|---|---|---|
| | | | Detect | Non-detect |
| Aqueous | Cooled at temperature ≤ 6°C | Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time | No qualification | No qualification |
| | | Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time | J | R |
| | | Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time | J | R |
| Aqueous | Not cooled at temperature ≤ 6°C | Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time | Use professional judgment | Use professional judgment |
| | | Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time | J | R |
| | | Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time | J | R |

Collected: 3/9/23
Extracted: 3/15/23
Analyzed: 4/12/23 and 4/13/23

All ok

## WORKSHEET #12.2A
## MEASUREMENT PERFORMANCE CRITERIA: PCBS AS AROCLORS, HERBICIDES, AND TRIBUTYLTIN

Matrix: Solid (PCBs and tributyltin) and aqueous (PCBs, herbicides, and tributyltin)
Analytical Group/Method: GC/ECD / SW-846 Methods 8082A and 8151A; GC/FPD / Laboratory-developed method based on Krone-Unger
Concentration Level: Low

| DQI | QC Sample or Measurement Performance Activity | Measurement Performance Criteria |
|---|---|---|
| Precision (overall) | Field Duplicate | RPD ≤50% (solid matrix) or ≤30% (aqueous matrix) if both results ≥5x PQL[1] |
| Precision (analytical) | LCS/LCSD[2] | RPD limits presented in Worksheet #12.2b (PCBs), Worksheet #12.2c (herbicides), and Worksheet #12.2d (tributyltin) |
| Precision (analytical, matrix interference) | MS/MSD | |
| Accuracy (analytical) | LCS and LCSD[2] | %R limits presented in Worksheet #12.2b, (PCBs), Worksheet #12.2c (herbicides), and Worksheet #12.2d (tributyltin) |
| Accuracy (analytical, matrix interference) | MS and MSD | |
| Accuracy (analytical, matrix interference) | Surrogate Spike | |
| Representativeness (field) | EB ((Rinse) | Not detected > PQL |
| Representativeness (transport) | Temperature Blank | 0°C to 6°C |
| Representativeness (analytical system) | Laboratory MB | No analytes detected > ½ the PQL |
| Representativeness (matrix interference) | Method-specified matrix cleanup | Performed as required/requested to address challenging sample matrices and chromatographic interference |
| Sensitivity | MDL and PQL | Adjusted correctly for sample-specific factors (subsample size, moisture content, dilution) |
| Completeness | Calculation of field and analytical completeness | >90% completeness |
| Accuracy (analytical, matrix interference) | Second column/second detector quantification[3] | RPD ≤40% |
| Sensitivity | MDL | MDL at or below the analyte-specific PALs listed in Worksheets #15.4a and b, #15.5, and #15.6 |
| | | Conducted and updated at least annually[3] |
| Sensitivity | PQL[3] | Established at or above the low point of calibration curve |

[1] For low-level results (one or both detected values ≤5x PQL) or when one result is a non-detection, the control limit is absolute difference ≤2x PQL (solid matrix) or ≤PQL (aqueous matrix). Non-detected values will be assigned the nominal value of the MDL for making this comparison.
[2] LCSDs are not a method requirement; however, if LCSD results are reported, the LCSD%Rs and LCS/LCSD RPD will be evaluated.
[3] This method requirement will only be reviewed to complete definitive (Stage 4) data validation.

**Initial Calibration and Verification (Form 6, run log)**

- ✓ Individual standards at 5 concentrations
- ✓ % RSDs of CFs ≤20% or $r^2 \geq 0.99$

ICAL both columns

INST K-GC-34

3/21/23 %RSD ok

ICV KC2300197-09 PENTA02-67D ICV 100PPB 03/21/2023 14:26

Continuing Calibration Verification KQ2306916-01 4/12/2023 19:27:22

Continuing Calibration Blank KQ2306916-02 4/12/2023 19:53:13

Samples -1 through -5

Continuing Calibration Verification KQ2306916-03 4/13/2023 00:37:47

Continuing Calibration Blank KQ2306916-04 4/13/2023 01:03:43

Sample -6

Continuing Calibration Verification KQ2306916-05 4/13/2023 02:21:21

col 1 ok col 2 out report sample from col 1 no Q

Continuing Calibration Blank KQ2306916-06 4/13/2023 02:47:14

All ok unless noted above

**Pesticides Table 3. Initial Calibration Action**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Initial Calibration not performed at specified frequency and sequence | R | R |
| Initial Calibration not performed at specified concentrations | J | UJ |
| RT windows incorrect<br>Or<br>Chromatogram criteria not met | J | R |
| %RSD for target analyte outside specified acceptance limits* | J | No qualification<br>or<br>UJ |
| %RSD for target analyte within specified acceptance limits* | No qualification | No qualification |

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* %RSD ≤ 20.0% for single component target analytes except alpha-BHC and delta-BHC.
%RSD ≤ 25.0% for alpha-BHC and delta-BHC.
%RSD ≤ 30.0% for Toxaphene peaks.
%RSD ≤ 20.0% for surrogates (TCX and DCB).

| | | | | |
|---|---|---|---|---|
| | analysis sequence | | control CCV | |
| Five-point initial calibration for target analytes (six points required for quadratic); lowest concentration standard at or below the PQL. | Initial calibration prior to sample analysis | Each analyte %RSD for each analyte ≤20%;<br>Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$;<br>Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$. | 1) Evaluate system<br>2) Recalibrate as necessary | SOC-8151 |
| ICV (must be from a second source) | Following ICAL | %R = 80 to 120% | 1) Evaluate system<br>2) Recalibrate as necessary | SOC-8151 |
| Retention time verification | Update at start of run or daily | Standards within retention time window | 1) Correct problem<br>2) Reanalyze samples analyzed since the last retention time check | SOC-8151 |
| CCV | Before sample analysis, after every 10 samples, and at the end of the analysis sequence | %R = 80 to 120% | 1) Evaluate system<br>2) Clean system<br>3) Reanalyze affected samples since the last in-control CCV | SOC-8151 |

**Continuing calibration Verification (run log, Form 7)**

- ✓ CCV before sample analysis, after every 10 samples, and at the end of the analysis sequence
- ✓ %Ds ± 20%
- ✓ RTs withing established windows

**Pesticides Table 4. CCV Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| CCV PEM or CCV standard not performed at specified frequency and sequence | J or R | UJ or R |
| CCV PEM not performed at specified concentration | No qualification or J | No qualification or UJ |
| CCV standard not performed at specified concentration | No qualification or J | No qualification or UJ |
| RT outside specified RT window | J | No qualification or UJ |



| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| CCV PEM %D for target analyte outside specified limits | J | UJ |
| CCV standard %D for target analyte outside specified limits | J | UJ |
| RT, CCV PEM %D, CCV %D, and time elapsed within specified limits | No qualification | No qualification |
| Time elapsed between opening CCV instrument blank and closing CCV exceeds 14 hours | Use professional judgment | Use professional judgment |
| Time elapsed between opening CCV instrument blank and last sample or blank exceeds 12 hours | Use professional judgment | Use professional judgment |

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

All ok unless noted above

**Blanks (Form 4, Form 1)**

- ✓ evaluate storage, method, clean up, field blanks
- ✓ method blank were prepared and analyzed in association with all samples in SDG

**Pesticides Table 5. Blank Actions**

| Blank Type | Blank Result | Sample Result | Action |
|---|---|---|---|
| | Not analyzed at specified frequency or sequence | Detect | J |
| | | Non-detect | No qualification |
| Method, TCLP/SPLP LEB, Sulfur cleanup, Instrument, Field (including Equipment and Rinse) | Detects | Non-detect | No qualification |
| | < QL | < QL | Report at QL and qualify U |
| | | ≥ QL | Report at sample result and qualify J+ or No qualification |
| | ≥ QL | < QL | Report at QL and qualify U |
| | | ≥ QL but < Blank Result | Report at sample result and qualify U |
| | | ≥ QL and ≥ Blank Result | Report at sample result and qualify J+ or No qualification |
| | Gross contamination | Detect | Report at sample result and qualify R |

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

KQ2304730-01 ND

**Surrogates (Form 2)**

**WORKSHEET #12.2C**
**ANALYTE-SPECIFIC LIMITS FOR HERBICIDES (8151A)**

| Chemical | CASRN | Solid Matrix Accuracy Control Limits (%R) | Solid Matrix Precision Control Limits (RPD) | Aqueous Matrix Accuracy Control Limits (%R) | Aqueous Matrix Precision Control Limits (RPD) |
|---|---|---|---|---|---|
| MCPP | 93-65-2 | NA | NA | 16-141 | 30 |
| *Surrogates* | | | | | |
| 2,4-Dichlorophenylacetic acid | 19719-28-9 | NA | NA | 17-113 | NA |

MCPP = methylchlorophenoxypropionic acid

All ok

**Pesticides Table 6. Surrogate Actions**

| Criteria | Action* | |
|---|---|---|
| | Detect | Non-detect |
| Surrogate not present or not at specified concentration | J or R | UJ or R |
| RT out of specified RT window | Use professional judgment | Use professional judgment |
| RT within specified RT window | No qualification | No qualification |
| %R < Expanded Lower Acceptance Limit (10%, undiluted sample) | J- | R |
| %R < Expanded Lower Acceptance Limit (10%, diluted sample) | Use professional judgment | Use professional judgment |
| Expanded Lower Acceptance Limit (10%) ≤ %R < specified Lower Acceptance Limit | J- | UJ |
| %R within specified Acceptance Limits | No qualification | No qualification |





| Criteria | Action* | |
|---|---|---|
| | Detect | Non-detect |
| Upper Acceptance Limit < %R ≤ Expanded Upper Acceptance Limit (200%) | J+ | No qualification |
| %R > Expanded Upper Acceptance Limit (200% ) | J+ | Use professional judgment |

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

* Use professional judgment in qualifying data, as surrogate recovery problems may not directly apply to target analytes.

**LCS (Form 3, batch worksheet)**

- ✓ LCS for each matrix
- ✓ if qualification is required, qualified all samples prepped with the LCS

**WORKSHEET #12.2C**
**ANALYTE-SPECIFIC LIMITS FOR HERBICIDES (8151A)**

| Chemical | CASRN | Solid Matrix Accuracy Control Limits (%R) | Solid Matrix Precision Control Limits (RPD) | Aqueous Matrix Accuracy Control Limits (%R) | Aqueous Matrix Precision Control Limits (RPD) |
|---|---|---|---|---|---|
| MCPP | 93-65-2 | NA | NA | 16-141 | 30 |
| ***Surrogates*** | | | | | |
| 2,4-Dichlorophenylacetic acid | 19719-28-9 | NA | NA | 17-113 | NA |

MCPP = methylchlorophenoxypropionic acid



**Pesticides Table 8. LCS Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| LCS not performed at specified frequency or concentration | Use professional judgment | Use professional judgment |
| LCS %R limits not specified | Use professional judgment | Use professional judgment |
| %R < specified Lower Acceptance Limit | J- | R |
| %R within specified Acceptance Limits | No qualification | No qualification |
| %R > specified Upper Acceptance Limit | J+ | No qualification |

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

**KQ2304730-02** and **KQ2304730-03** all ok

**MS/MSD (Form 3)**

- ✓ evaluate accuracy (%R) and precision (RPD)
- ✓ qualify parent sample only

**WORKSHEET #12.2C**
**ANALYTE-SPECIFIC LIMITS FOR HERBICIDES (8151A)**

| Chemical | CASRN | Solid Matrix Accuracy Control Limits (%R) | Solid Matrix Precision Control Limits (RPD) | Aqueous Matrix Accuracy Control Limits (%R) | Aqueous Matrix Precision Control Limits (RPD) |
|---|---|---|---|---|---|
| MCPP | 93-65-2 | NA | NA | 16-141 | 30 |
| ***Surrogates*** | | | | | |
| 2,4-Dichlorophenylacetic acid | 19719-28-9 | NA | NA | 17-113 | NA |

MCPP = methylchlorophenoxypropionic acid

**Pesticides Table 7. MS/MSD Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| MS/MSD not analyzed at specified frequency or concentration | Use professional judgment | Use professional judgment |
| MS/MSD not prepared from field sample | Use professional judgment | Use professional judgment |
| %R or RPD limits not specified | Use professional judgment | Use professional judgment |
| %R < Expanded Lower Acceptance Limit (20%) | J | R |
| Expanded Lower Acceptance Limit (20%) ≤ %R < specified Lower Acceptance Limit | J | UJ |
| %R or RPD within specified Acceptance Limits | No qualification | No qualification |
| %R or RPD > specified Upper Acceptance Limit | J | No qualification |

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

None

**Target Compound Identification (Form1, raw data)**

- ✓ Form 10 present for all detected analytes
- ✓ %Ds <25% ( if RPD reported, used 40% per method 8082)
- ✓ RTs within window on both columns

OK all ND

**Target Analyte Quantitation (Form 1, run log, prep log, EDD)**

✓ verify all positive results are reported

✓ evaluate dilutions, re-extractions and re-analyses to confirm best results is reported and all other results are not reportable in EDD

✓ all solid sample % solids > 10% (or % moisture <90%)

All ok

**Field Duplicates**

✓ If both original sample and duplicate sample results are ≥ 5x the LOQ and the RPD is > 50%, qualify detects as estimated "J", and qualify non-detects as estimated "UJ". List samples and results effected below.

✓ If the original sample or duplicate sample result is < 5x the LOQL (including non-detects) and the absolute difference between sample and duplicate > 2X LOQ, qualify detects as estimated "J" and non-detects as estimated "UJ".

✓ see field duplicate worksheet

none

**WORKSHEET #15.5**
**PALS AND LABORATORY-SPECIFIC MDLS/PQLS – HERBICIDES IN WATER BY GC/ECD (8151A)**

| Analyte | CASRN | Category | ALS-Kelso Sensitivity Limits (µg/L) | | Surface Water CUL (µg/L) |
|---|---|---|---|---|---|
| | | | MDL | PQL | |
| MCPP | 93-65-2 | COC | 14 | 100 | 16 |

**TSS Validation Checklist**

| | |
|---|---|
| **Site:** | Portland Harbor-Stormwater |
| **Laboratory:** | ALS Environmental |
| **SDG:** | K2302917 |
| **Data Validator:** | PMH |
| **Validation Date:** | 5/24/2023 |
| **Reviewer:** | DLW |
| **Review Date:** | 5/24/2023 |
| **Method:** | SM2540D |

| | |
|---|---|
| **Date Collected:** | 3/9/2023 |
| **Date of Analysis:** | 3/15/2023 |

## Data Completeness and Deliverables

***Have any missing deliverable been recieved and added to the data package?*** ☐ Yes ☑ No

**ACTION:** Call lab for explanation/resubmittal of any missing deliverables. If the lab cannot probive them, note the effect on review of the data in the non-compliance section of the data assessment narrative.

Notes and validation action:

## Custody Documents and Narratives

***Are chains of custody present and complete for all samples?*** ☑ Yes ☐ No

**ACTION:** Contract lab for replacement of missing documents.

***Do chains of custody or lab narratives indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?*** ☐ Yes ☑ No

Notes and validation action:

**Temperature:**

***Sample coolers received @ 0-6 ℃ ?*** ☑ Yes ☐ No

**Preservation:**

***none required*** ☑ Yes ☐ No

**ACTION:** Samples improperly preserved shall be noted in the data validation report. Professional judgement may result in the results of an analysis of an improperly preserved sample by the laboratory being qualified as estimated "J" or "UJ".

**ACTION**: If samples were not iced upon receipt, flag all positive results as estimated, an all non-detects “UJ”.

Notes and validation action:

**Holding Time**

***Does the holding time exceed 7 days?*** ☐ Yes ☑ No

**ACTION:** If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J" or “UJ” as appropriate. When holding times are exceeded by more than twice the time specified, the non-detects will be flagged as unusable, "R”.

Notes and validation action:

**Blanks**

***Has a method blank analysis been reported per twenty samples of a similar matrix or concentration level, and for each extraction batch?*** ☑ Yes ☐ No

***Upon examination of laboratory and field blank data, do any blanks contain positive results?*** ☐ Yes ☑ No

**ACTION:** If yes, qualify associated results as follows: If the sample result is greater than the laboratory reporting limit but less than 5 times the blank concentration, flag sample result as non-detect ("U"). If the sample result is reported as detected at a concentration less than the reporting limit and less than 5 times the blank concentration, qualify the sample result as non-detectable at the laboratory reporting limit. For aqueous blanks applied to soil/sediment samples, compare the sample result to the equivalent concentration of the blank.

***Are there field/rinsepment blanks associated with every sample*** ☐ Yes ☑ No

Notes and validation action:

| blank | positives | Flag | assocated samples |
|---|---|---|---|
| K2302917-MB1 | ND | | |
| K2302917-MB2 | ND | | |

**Matrix Spike**

***Is a matrix spike/matrix spike duplicate summary present?*** ☐ Yes ☑ No

**ACTION:** If any matrix spike data are missing, call the lab for explanation/re-submittal. If information is not available, document the effect in narrative notes.

***Are all matrix spike and/or matrix spike duplicate %Rs or RPDs within acceptance range? Use lab limits*** ☐ Yes ☐ No

Notes and validation action:

| Sample | MS %R | MSD %R | RPD | Flag |
|---|---|---|---|---|
| N/A to this method | | | | |

**Laboratory Duplicate**

***Is a laboratory duplicate summary present?*** ☐ Yes ☑ No

***Are the RPDs within the control limit?*** ☐ Yes ☐ No

Notes and validation action: no QAPP limits, use lab limits

| Sample | RPD |
|---|---|
| none | |

**Laboratory Control Sample**

***Were LCS samples evaluated with each batch of 20 samples or less and were observed percent recoveries within the laboratory defined limits?*** ☑ Yes ☐ No

Notes and validation action: no QAPP limits, use lab limits

| LCS or LCSD | %R | Flag | Associated samples: |
|---|---|---|---|
| K2302917-LCS2 | ok | | |

**Field Duplicates**

***Were field duplicates submitted for TSS analysis?***

**ACTION**: For field duplicate analyses that do not meet the technical criteria, apply action to only the parent sample and its duplicate.

☐ Yes ☑ No

Notes and validation action: no field dups present

**Review Level**

☑ Yes ☐ No

List samples reviewed and bold samples used for calculations:
all

**TOC Validation Checklist**

| | |
|---|---|
| **Site:** | Portland Harbor- Stormwater |
| **Laboratory:** | ALS Environmental |
| **SDG:** | K2302917 |
| **Data Validator:** | PMH |
| **Validation Date:** | 5/24/2023 |
| **Reviewer:** | DLW |
| **Review Date:** | 5/24/2023 |
| **Method:** | SM9060A |

| | | |
|---|---|---|
| **Date Collected:** | 3/9/2023 | |
| **Date of Analysis:** | 3/17/2023 | |

**Data Completeness and Deliverables**

***Have any missing deliverable been recieved and added to the data package?*** ☐ Yes ☑ No

**ACTION:** Call lab for explanation/resubmittal of any missing deliverables. If the lab cannot provide them, note the effect on review of the data in the non-compliance section of the data assessment narrative.

Notes and validation action:

**Custody Documents and Narratives**

***Are chains of custody present and complete for all samples?*** ☑ Yes ☐ No

**ACTION:** Contract lab for replacement of missing documents.

***Do chains of custody or lab narratives indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?*** ☐ Yes ☑ No

***Per NFG: Are the % solids >30% for all solids samples?***

**ACTION**: If the %Solids for a soil/sediment sample is < 10.0%, use professional judgment to qualify detects and non-detects.
If the %Solids for a soil/sediment sample is ≥ 10.0% and < 30.0%, use professional judgment to qualify detects and non-detects.

Notes and validation action:

**Temperature:**

***Sample coolers received @ 0-6 ℃ ?*** ☑ Yes ☐ No

**Preservation:**

***Sulfuric or hydrochloric acid pH of 2?*** ☑ Yes ☐ No

**ACTION:** Samples improperly preserved shall be noted in the data validation report. Professional judgement may result in the results of an analysis of an improperly preserved sample by the laboratory being qualified as estimated "J-" or "R".

**ACTION**: If samples >6°C but <10° C flag all positive results as estimated "J" an all non-detects "UJ".

Notes and validation action: samples received unpreserved, preserved by the lab prior to analysis

**Holding Time**

***Does the holding time exceed 28 days?***
***or***
***Freeze to −10°C***
***for up to 1 year*** ☐ Yes ☑ No

**ACTION:** If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J" or non detects "R" as

Notes and validation action:

**Calibration Verification**

***Are raw data and summary sheets present for both inital and continuing calibrations?*** ☐ Yes ☐ No

***At least the number of standards requried by QAPP and a blank?*** ☐ Yes ☐ No

***Correlation coeff > 0.995?*** ☐ Yes ☐ No

***Are the %D values between lab limits?*** ☐ Yes ☐ No

***Are the ICV/CCV %r within 80-120%?*** ☐ Yes ☐ No
**ACTION:** If criteria not met, qualify with bias per NFG

Notes and validation action:

90-110

Not validated at Level 2A

**Blanks**

***Has a method blank analysis been reported per twenty samples of a similar matrix or concentration level, and for each extraction batch?*** ☑ Yes ☐ No

***Upon examination of laboratory and field blank data, do any blanks contain positive results?*** ☐ Yes ☑ No

***Have calibration blanks been analyzed at the proper frequency?*** ☐ Yes ☐ No

***Are there field/rinse/equipment blanks associated with every sample?*** ☐ Yes ☑ No

**ACTION:** If contamination is present, qualify per NFG.

Notes and validation action:

| blank | positives | Flag | associated samples |
|---|---|---|---|
| ICB/CCBs not evaluated at stage 2A | | | |
| K2302917-MB1 | ND | | |
| K2302917-MB2 | ND | | |

Equipment Blank
none

**Matrix Spike**

***Is a matrix spike/matrix spike duplicate summary present?*** ☐ Yes ☑ No

**ACTION:** If any matrix spike data are missing, call the lab for explanation/re-submittal. If information is not available, document the effect in narrative notes.

***Are all matrix spike and/or matrix spike duplicate %Rs or RPDs within 70-130%?*** ☐ Yes ☐ No

USe QAPP Limits 72-122%R RPD 20 (solid) 83-117% RPD 17 (aqueous)

**ACTION:** If no, qualify with bias per NFG.

Notes and validation action:

| Sample | MS %R | MSD %R | RPD | Flag |
|---|---|---|---|---|
| K2302917-001 | OK | n/a | n/a | SIB-SW-WR198-030923 |

**Laboratory Duplicate**

***Is a laboratory duplicate summary present?*** ☑ Yes ☐ No

***Are the RPDs within 20%?*** USe QAPP Limits ☑ Yes ☐ No

**ACTION:** If orginal and duplicate results are ≥ 5X CRQL and RPD is >20% or original and duplicate results are <5X CRQL and the absolute difference between sample and duplicate is >CRQL then qualify as "J" and/or "UJ."

Notes and validation action:

| Sample | RPD | |
|---|---|---|
| K2302917-001 | OK | SIB-SW-WR198-030923 |

**Laboratory Control Sample**

***Were LCS samples evaluated with each batch of 20 samples or less and were observed percent recoveries within the laboratory defined limits of (75-125%)?*** ☑ Yes ☐ No

Use QAPP Limits: 72-122 %R, RPD ≤ 20% (solid) ; 83-117 %R, RPD ≤ 17% (aqueous)

**ACTION**: If LCS %R fall outside the control limits, qualify per NFG

Notes and validation action:

| LCS or LCSD | %R | Flag | Associated samples: |
|---|---|---|---|
| K2302917-LCS2 | ok | | |
| K2302917-LCS3 | ok | | |

**Field Duplicates**

***Were field duplicates submitted for TOC analysis?*** ☐ Yes ☑ No

**ACTION**: For field duplicate analyses that do not meet the technical criteria as required by QAPP, apply action to only the parent sample and its duplicate.

QAPP: RPD 50% (solid) 30% (aqueous) if both results ≥ 5X PQL

Notes and validation action:
none

| **Review Level** | | |
|---|---|---|
| ***Is a level 4 review required for this project?*** | ☐ Yes | ☑ No |

List samples reviewed and bold samples used for calculations:
**SDG validated at level 2A**

**TSS Validation Checklist**

| | |
|---|---|
| **Site:** | Portland Harbor-Stormwater |
| **Laboratory:** | ALS Environmental |
| **SDG:** | K2302917 |
| **Data Validator:** | PMH |
| **Validation Date:** | 5/24/2023 |
| **Reviewer:** | DLW |
| **Review Date:** | 5/24/2023 |
| **Method:** | SM2540D |

| | |
|---|---|
| **Date Collected:** | 3/9/2023 |
| **Date of Analysis:** | 3/15/2023 |

## Data Completeness and Deliverables

***Have any missing deliverable been recieved and added to the data package?*** ☐ Yes ☑ No

**ACTION:** Call lab for explanation/resubmittal of any missing deliverables. If the lab cannot probive them, note the effect on review of the data in the non-compliance section of the data assessment narrative.

Notes and validation action:

## Custody Documents and Narratives

***Are chains of custody present and complete for all samples?*** ☑ Yes ☐ No

**ACTION:** Contract lab for replacement of missing documents.

***Do chains of custody or lab narratives indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?*** ☐ Yes ☑ No

Notes and validation action:

**Temperature:**

***Sample coolers received @ 0-6 ℃ ?*** ☑ Yes ☐ No

**Preservation:**

***none required*** ☑ Yes ☐ No

**ACTION:** Samples improperly preserved shall be noted in the data validation report. Professional judgement may result in the results of an analysis of an improperly preserved sample by the laboratory being qualified as estimated "J" or "UJ".

**ACTION**: If samples were not iced upon receipt, flag all positive results as estimated, an all non-detects “UJ”.

Notes and validation action:

**Holding Time**

***Does the holding time exceed 7 days?*** ☐ Yes ☑ No

**ACTION:** If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J" or “UJ” as appropriate. When holding times are exceeded by more than twice the time specified, the non-detects will be flagged as unusable, "R”.

Notes and validation action:

**Blanks**

***Has a method blank analysis been reported per twenty samples of a similar matrix or concentration level, and for each extraction batch?*** ☑ Yes ☐ No

***Upon examination of laboratory and field blank data, do any blanks contain positive results?*** ☐ Yes ☑ No

**ACTION:** If yes, qualify associated results as follows: If the sample result is greater than the laboratory reporting limit but less than 5 times the blank concentration, flag sample result as non-detect ("U"). If the sample result is reported as detected at a concentration less than the reporting limit and less than 5 times the blank concentration, qualify the sample result as non-detectable at the laboratory reporting limit. For aqueous blanks applied to soil/sediment samples, compare the sample result to the equivalent concentration of the blank.

***Are there field/rinsepment blanks associated with every sample*** ☐ Yes ☑ No

Notes and validation action:

| blank | positives | Flag | assocated samples |
|---|---|---|---|
| K2302917-MB1 | ND | | |
| K2302917-MB2 | ND | | |

**Matrix Spike**

***Is a matrix spike/matrix spike duplicate summary present?*** ☐ Yes ☑ No

**ACTION:** If any matrix spike data are missing, call the lab for explanation/re-submittal. If information is not available, document the effect in narrative notes.

***Are all matrix spike and/or matrix spike duplicate %Rs or RPDs within acceptance range? Use lab limits*** ☐ Yes ☐ No

Notes and validation action:

| Sample | MS %R | MSD %R | RPD | Flag |
|---|---|---|---|---|
| N/A to this method | | | | |

**Laboratory Duplicate**

***Is a laboratory duplicate summary present?*** ☐ Yes ☑ No

***Are the RPDs within the control limit?*** ☐ Yes ☐ No

Notes and validation action: no QAPP limits, use lab limits

| Sample | RPD |
|---|---|
| none | |

**Laboratory Control Sample**

***Were LCS samples evaluated with each batch of 20 samples or less and were observed percent recoveries within the laboratory defined limits?*** ☑ Yes ☐ No

Notes and validation action: no QAPP limits, use lab limits

| LCS or LCSD | %R | Flag | Associated samples: |
|---|---|---|---|
| K2302917-LCS2 | ok | | |

**Field Duplicates**

***Were field duplicates submitted for TSS analysis?***

**ACTION**: For field duplicate analyses that do not meet the technical criteria, apply action to only the parent sample and its duplicate.

☐ Yes ☑ No

Notes and validation action: no field dups present

**Review Level**

☑ Yes ☐ No

List samples reviewed and bold samples used for calculations:
all

Validated on: 5/25/2023

Validate by: GAP

Reviewed by: PMH

Reviewed on: 5/26/2023

**Client requested to not use qualifier bias**

ICP Checklist 6020

Project: Portland Harbor

SDG: K2302917

Analytes: As, Cr, Cu, Zn

Matrix: aqueous

Temp. (0-6 degrees C) ok

**Preservation Holding Time** –

Date collected : 3/9/2023

pH < 2 yes

6 months HT yes

---

**Tune Analysis-**

Was tune performed before ICAL (yes/no) yes

Resolution within <0.1 amu

%RSD < 5%

ok

---

Calibration

Instrument: **K-ICP-MS-06**

Date: 3/20/2023

Blank and 1 Standard yes

---

PQL or CRQL Std. per QAPP requirements (80-120%) ok

Correlation coefficient met QAPP requirement or <0.995

Initial calibration %Ds ±30%

ICV/CCV %RSD

**Initial Calib Verif**. ICV (90-110 %R) ok

**Continuing Calib. Verif**. CCV (90-110 %R) ok

**Initial Calib Blank ICB** (ND or U) ok

**Continuing Calib. Blank CCB** (ND or U) ok

Ran a frequency designated in QAPP or every 2 hours

---

As, Cr, Cu, Zn

ICV 1 03/20/23 16:29

CCV 1 03/20/23 16:31

ICB 1 03/20/23 16:34

CCB 1 03/20/23 16:36

Full list

CCV 1 03/20/23 17:12

CCB 1 03/20/23 17:15

All samples

CCV 1 03/20/23 17:40

CCB 1 03/20/23 17:43

---

**Blanks: (Remember never qualify a blank for a blank and always use highest blank value) see Table 4 below**

**Method/Preparation Blank (ND or U)**

**Client requested to not use qualifier bias**

KQ2304570-01

**All ND**

**Field Blanks/Equipment Blanks/Rinse Blanks (ND or U)**

none

ICP-MS Table 4. Blank Actions

| Blank Type | Blank Result | Sample Result | Action |
|---|---|---|---|
| ICB/CCB | Not analyzed at the specified frequency | Non-detect | UJ |
| | | Detect | J |
| ICB/CCB | Detect < QL | Non-detect | No qualification |
| | | Detect < QL | Report at QL and qualify U |

**Inorganic Data Review** **ICP-MS**

| Blank Type | Blank Result | Sample Result | Action |
|---|---|---|---|
| | | ≥ QL | J+ or no qualification |
| ICB/CCB | ≤ (-MDL) but > (-QL) | Non-Detect | UJ |
| | | Detect | J- or no qualification |
| ICB/CCB | ≥ QL | Non-detect | No qualification |
| | | Detect < QL | Report at QL and qualify U |
| | | ≥ QL but < ICB/CCB Result | Report at ICB/CCB Result and qualify U |
| | | ≥ ICB/CCB Result | J+ or no qualification |
| ICB/CCB | ≤ (-QL) | Non-detect | UJ or R |
| | | Detect < QL | J- |
| | | ≥ QL | J- |
| Preparation Blank | Not analyzed at specified frequency | Non-detect | UJ |
| | | Detect | J |
| Preparation Blank/Field Blank | Detect < QL | Non-detect | No qualification |
| | | Detect < QL | Report at QL and qualify U |
| | | ≥ QL | J+ or no qualification |
| Preparation Blank/Field Blank | ≤ (-MDL) but > (-QL) | Non-detect | UJ |
| | | Detect | J- or no qualification |
| Preparation Blank/Field Blank | ≥ QL | Non-detect | No qualification |
| | | Detect < QL | Report at QL and qualify U |
| | | ≥ QL but < 10x the Preparation Blank Result | Report at Preparation Blank Result and qualify J+ or R |
| | | ≥ 10x the Preparation Blank Result | No qualification |
| Preparation Blank/Field Blank | ≤ (-QL) | Non-detect | UJ |
| | | Detect < QL | J- |
| | | ≥ QL but < 10x QL | J- |
| | | ≥ 10x QL | No qualification |

**Interference Check Sample (ICS)**

**ICSA (+/- LOD or 85-115 %R)** ok

**ICSAB (+/- LOD or 85-115 %R)** ok

**Confirm no false positives or false negatives** ok

**Analysis date: 3/20/2023 ok**

**ICP-MS Table 5. Interference Check Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| ICS not analyzed | R | R |
| ICS not analyzed in specified sequence | J | UJ |
| ICSAB %R < 50% | J- | R |
| ICS %R 50-84% [or ICS found value is < (true value – 2x QL), whichever is lower] | J- | UJ |
| ICS %R 85-115% | No qualification | No qualification |
| ICS %R 116-150% [or ICS true value is > (true value + 2x QL), whichever is greater] | J+ | No qualification |
| ICS %R > 150% | J+ | No qualification |
| ICSA results ≥ DLs or MDLs, but not present in ICS (potential false positives) | J+ | No qualification |
| Negative ICSA results, but not present in ICS (potential false negatives) | J- for results < 10x (\|negative sample result\|) | UJ |

---

**Laboratory Control Sample (LCS) 70-130%**

KQ2304570-02 **ok**

---

**Duplicates: 1 in 20**

**Yes/No** **no**

none

**RPD < 20%.** **Yes**

---

**Matrix Spike (MS) (only if sample concentration is less than 4x spike added)**

**Apply action to all sample of same matrix**

**Yes/No** **no**

**If yes, sample name:** none

| Metal | MS %R | MSD %R | RPD | PDS | FLAG |
|---|---|---|---|---|---|
| All | | | | | |

**%R 75-125%**

**%RPD < 20%**

**PDS 75-125%R**

**ICP-MS Table 8. Spike Sample Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Matrix Spike analysis not performed at the specified frequency | J | UJ |
| Matrix Spike not prepared from field sample | J | UJ |
| Matrix Spike %R < 30%<br>Post-digestion spike %R < 75% | J- | R |
| Matrix Spike %R < 30%<br>Post-digestion spike %R ≥ 75% | J | UJ |
| Matrix Spike %R 30-74%<br>Post-digestion spike %R < 75% | J- | UJ |
| Matrix Spike %R 30-74%<br>Post-digestion spike %R ≥ 75% | J | UJ |
| Matrix Spike %R > 125%<br>Post-digestion spike %R > 125% | J+ | No qualification |



| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Matrix Spike %R > 125%Post-digestion spike %R ≤ 125% | J | No qualification |
| Matrix Spike %R < 30%<br>No post-digestion spike performed | J- | R |
| Matrix Spike %R 30-74%<br>No post-digestion spike performed | J- | UJ |
| Matrix Spike %R 75-125%<br>No post-digestion spike is required | No qualification | No qualification |
| Matrix Spike %R > 125%<br>No post-digestion spike performed | J+ | No qualification |

**Serial Dilution**

**Apply action to all sample of same matrix**

**Yes/No** no

**If yes, sample name:** none

**%D < 20% (only if undiluted result is > 50x IDL)** N/A

---

**Internal Standards (IS)**

**Present (Y/N)** Y

**IS intensity within 60-125% of intensity of the IS in the ICAL (Y/N)** Yes

---

**Field Duplicates (use project specific limit as defined in QAPP)**

**List Field Duplicates:**

none

**Precision acceptable (Y/N)** N/A

---

# Tributyltin

**CLIENT:** HGL

**PROJECT:** Portland Harbor Superfund Site

**SDG:** K2302917 (Stormwater)

**LAB:** ALS Kelso WA

**Validated by:** PMH

**Validation date:** 5/24/2023

**Reviewed by:** **DLW**

**Review date:** **5/25/23**

**ALS SOP**

**Sample Receipt / Hold times (COC, receipt logs, case narrative)**

| | | | | | | |
|---|---|---|---|---|---|---|
| Tributyltin **SOLID** | Laboratory-modified Krone-Unger (SOC-BUTYL/ EXT-OSWT) | 4 oz glass jar | Cool ≤6°C | 14 days to prepare and 40 days from extraction to analysis | Freeze to −10°C for up to 1 year | ALS-Kelso |
| Tributyltin **LIQUID** | Laboratory-modified Krone-Unger (SOC-BUTYL/ EXT-OSWT) | 2 x L amber glass | Cool ≤6°C | 7 days to prepare and 40 days from extraction to analysis | NA | ALS-Kelso |

✓Temperature upon receipt (<6◦C) ok

✓HT

**Pesticides Table 1. Preservation and Holding Time Actions**

| Matrix | Preservation | Criteria | Action | |
|---|---|---|---|---|
| | | | Detect | Non-detect |
| Aqueous/Non-aqueous | Samples received at a temperature > 6°C | | J | UJ |
| Aqueous/Non-aqueous | Cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared within the 14-day technical holding time | No qualification | No qualification |
| | Not cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared within the 14-day technical holding time | Use professional judgment | Use professional judgment |
| | Cooled/not cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared outside the 14-day technical holding time | J | R |

| Matrix | Preservation | Criteria | Action | |
|---|---|---|---|---|
| | | | Detect | Non-detect |
| Aqueous | Cooled at temperature ≤ 6°C | Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time | No qualification | No qualification |
| | | Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time | J | R |
| | | Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time | J | R |
| Aqueous | Not cooled at temperature ≤ 6°C | Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time | Use professional judgment | Use professional judgment |
| | | Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time | J | R |
| | | Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time | J | R |

| Matrix | Preservation | Criteria | Action | |
|---|---|---|---|---|
| | | | Detect | Non-detect |
| Non-aqueous | Cooled at temperature ≤ 6°C | Samples extracted within the 14-day and analyzed within the 40-day technical holding time | No qualification | No qualification |
| | | Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time | J- | R |
| | | Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time | J- | R |
| Non-aqueous | Not cooled at temperature ≤ 6°C | Samples extracted within 14- day and analyzed within the 40-day technical holding time | Use professional judgment | Use professional judgment |
| | | Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time | J | R |
| | | Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time | J | R |

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Sampled: 3/9/2023
Prepped: 3/16/2023
Analyzed: 4/7/2023

All ok

**Initial Calibration and Verification (Form 6, run log)**

- ✓ Individual standards at 5 concentrations
- ✓ five-point ICAL for Toxaphene
- ✓ % RSDs of CFs ≤25% or

Option 2: linear least squares regression for each analyte: r2 ≥0.99;

Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.

- ✓ ICV second source %R = 75 to 125% RTs withing established windows

**Pesticides Table 3. Initial Calibration Action**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Initial Calibration not performed at specified frequency and sequence | R | R |
| Initial Calibration not performed at specified concentrations | J | UJ |
| RT windows incorrect<br>Or<br>Chromatogram criteria not met | J | R |
| %RSD for target analyte outside specified acceptance limits* | J | No qualification<br>or<br>UJ |
| %RSD for target analyte within specified acceptance limits* | No qualification | No qualification |

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* %RSD ≤ 20.0% for single component target analytes except alpha-BHC and delta-BHC.
%RSD ≤ 25.0% for alpha-BHC and delta-BHC.
%RSD ≤ 30.0% for Toxaphene peaks.
%RSD ≤ 20.0% for surrogates (TCX and DCB).

**Continuing calibration Verification (run log, Form 7)**

- ✓ CCV analyzed at the beginning of day and end of run (one set every 10 samples)
- ✓ %Ds ± 25%
- ✓ RTs withing established windows (See Diane)

**Pesticides Table 4. CCV Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| CCV PEM or CCV standard not performed at specified frequency and sequence | J or R | UJ or R |
| CCV PEM not performed at specified concentration | No qualification or J | No qualification or UJ |
| CCV standard not performed at specified concentration | No qualification or J | No qualification or UJ |
| RT outside specified RT window | J | No qualification or UJ |



| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| CCV PEM %D for target analyte outside specified limits | J | UJ |
| CCV standard %D for target analyte outside specified limits | J | UJ |
| RT, CCV PEM %D, CCV %D, and time elapsed within specified limits | No qualification | No qualification |
| Time elapsed between opening CCV instrument blank and closing CCV exceeds 14 hours | Use professional judgment | Use professional judgment |
| Time elapsed between opening CCV instrument blank and last sample or blank exceeds 12 hours | Use professional judgment | Use professional judgment |

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Instrument ID: K-GC-26
Primary col = RTX-1

ICAL 03/29/23 all ok
**ICV** 50PPB J:\GC26\DATA\032923CAL\0329F013.D 03/29/2023 19:00 ok
CCVs: all ok
J:\GC26\DATA\040623\0406F087.D\ Continuing Calibration Verification KQ2307277-01 4/7/2023 10:42:00
J:\GC26\DATA\040623\0406F102.D\ Continuing Calibration Verification KQ2307277-03 4/7/2023 15:09:00
J:\GC26\DATA\040623\0406F109.D\ Continuing Calibration Verification KQ2307277-05 4/7/2023 17:17:00

**Blanks (Form 4, Form 1)**

- ✓ evaluate storage, method, clean up, field blanks
- ✓ method blank were prepared and analyzed in association with all samples in SDG

**Pesticides Table 5. Blank Actions**

| Blank Type | Blank Result | Sample Result | Action |
|---|---|---|---|
| | Not analyzed at specified frequency or sequence | Detect | J |
| | | Non-detect | No qualification |
| Method, TCLP/SPLP LEB, Sulfur cleanup, Instrument, Field (including Equipment and Rinse) | Detects | Non-detect | No qualification |
| | < QL | < QL | Report at QL and qualify U |
| | | ≥ QL | Report at sample result and qualify J+ or No qualification |
| | ≥ QL | < QL | Report at QL and qualify U |
| | | ≥ QL but < Blank Result | Report at sample result and qualify U |
| | | ≥ QL and ≥ Blank Result | Report at sample result and qualify J+ or No qualification |
| | Gross contamination | Detect | Report at sample result and qualify R |

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

KQ2304849-01 ND

No equipment blank associated with this SDG

**Surrogates (Form 2)**

- ✓ Tripropyltin added to all samples
- ✓ %Rs within 10-120% solids
- ✓ %Rs within 31-137% liquids

**Pesticides Table 6. Surrogate Actions**

| Criteria | Action* | |
|---|---|---|
| | Detect | Non-detect |
| Surrogate not present or not at specified concentration | J or R | UJ or R |
| RT out of specified RT window | Use professional judgment | Use professional judgment |
| RT within specified RT window | No qualification | No qualification |
| %R < Expanded Lower Acceptance Limit (10%, undiluted sample) | J- | R |
| %R < Expanded Lower Acceptance Limit (10%, diluted sample) | Use professional judgment | Use professional judgment |
| Expanded Lower Acceptance Limit (10%) ≤ %R < specified Lower Acceptance Limit | J- | UJ |
| %R within specified Acceptance Limits | No qualification | No qualification |





| Criteria | Action* | |
|---|---|---|
| | Detect | Non-detect |
| Upper Acceptance Limit < %R ≤ Expanded Upper Acceptance Limit (200%) | J+ | No qualification |
| %R > Expanded Upper Acceptance Limit (200% ) | J+ | Use professional judgment |

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

* Use professional judgment in qualifying data, as surrogate recovery problems may not directly apply to target analytes.

All ok

**LCS (Form 3, batch worksheet)**

- ✓ LCS for each matrix
- ✓ if qualification is required, qualified all samples prepped with the L
- ✓ %Rs withing 10-122% solids RPD </= 40
- ✓ %Rs withing 32-122% liquids RPD </= 30



**Pesticides Table 8. LCS Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| LCS not performed at specified frequency or concentration | Use professional judgment | Use professional judgment |
| LCS %R limits not specified | Use professional judgment | Use professional judgment |
| %R < specified Lower Acceptance Limit | J- | R |
| %R within specified Acceptance Limits | No qualification | No qualification |
| %R > specified Upper Acceptance Limit | J+ | No qualification |

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

LCS KQ2304849-02 ok

LCSD KQ2304849-03 ok

RPD OK

**MS/MSD (Form 3)**

- ✓ %Rs withing 10-122% solids RPD </= 40
- ✓ %Rs withing 32-122% liquids RPD </= 30
- ✓ qualify parent sample only

**Pesticides Table 7. MS/MSD Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| MS/MSD not analyzed at specified frequency or concentration | Use professional judgment | Use professional judgment |
| MS/MSD not prepared from field sample | Use professional judgment | Use professional judgment |
| %R or RPD limits not specified | Use professional judgment | Use professional judgment |
| %R < Expanded Lower Acceptance Limit (20%) | J | R |
| Expanded Lower Acceptance Limit (20%) ≤ %R < specified Lower Acceptance Limit | J | UJ |
| %R or RPD within specified Acceptance Limits | No qualification | No qualification |
| %R or RPD > specified Upper Acceptance Limit | J | No qualification |

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

MS MSD RPD

None

**Target Compound Identification (Form1, raw data)**

✓ Form 10 present for all detected analytes

✓ %RPDs <40%

✓ RTs within window on both columns LEVEL 4 ONLY (use raw data flagging to assess)

Used +/- 0.1 minutes as RT window guidance for evaluation purposes

**Pesticides Table 11. Target Analyte Identification Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Detected target analyte RT outside specified RT window (false positive) | Report at QL and qualify U | Not applicable |
| Detected target analyte peak exhibits an interference with the potential detection of another target peak (false positive) | R | Not applicable |
| Reported non-detect target analyte RT within specified RT windows on both GC columns (false negative) | Use professional judgment to report results | Not applicable |
| Toxaphene peak RT windows overlap with single component target analytes or chromatographic interferences exist | Use professional judgment | Use professional judgment |
| Toxaphene peaks exhibit a marginal pattern-matching quality | Use professional judgment<br>or<br>Report results and qualify NJ | Use professional judgment |





| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Evident chromatographic interference or co-elution for the detected target analyte | Use professional judgment to report result at lower value and qualify as NJ<br>or<br>Report at QL and qualify U | Not applicable |
| %D for any target analyte ≥ 25% | J | Not applicable |

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

All samples ND - ok

**Target Analyte Quantitation (Form 1, run log, prep log, EDD) LEVEL 4 ONLY**

✓ verify all positive results are reported

✓ evaluate dilutions, re-extractions and re-analyses to confirm best results is reported and all other results are not reportable in EDD

✓ all solid sample % solids > 10% (or % moisture <90%)

**Pesticides Table 13. Percent Solids Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| %Solids < 10.0% | Use professional judgment | Use professional judgment |
| 10.0% ≤ %Solids < 30.0% | Use professional judgment | Use professional judgment |
| %Solids ≥ 30.0% | No qualification | No qualification |

Criteria listed in the Table are the EPA NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

ok

**Field Duplicates** Not specified in NFG

✓ If both original sample and duplicate sample results are ≥ 5x the PQL and the RPD is > 50% for solid samples or 30% for liquid samples, qualify detects as estimated "J", and qualify non-detects as estimated "UJ". List samples and results effected below.

✓ If the original sample or duplicate sample result is < 5x the PQL (including non-detects) and the absolute difference between sample and duplicate > 2X PQL for solid samples and the PQL for liquid samples, qualify detects as estimated "J" and non-detects as estimated "UJ".

✓ see field duplicate worksheet

None

**CLIENT:** HGL

**PROJECT:** Portland Harbor Superfund Site

**SDG:** K2302917

**Validated by:** LL 5/24/23

**Reviewed by:** **DLW 5/25/23**

**LAB:** ALS Kelso, WA

bis(2-ethylhexyl)phthalate and pentachlorophenol

**Sample Receipt / Hold times** (COC, receipt logs, case narrative)

✓received at <10% ◦C

✓HT (aqueous– 7 days to extract, 40 days extraction to analysis; solids – 14 days to extraction, 40 days extraction to analysis)

Collected: 3/9
Extracted: 3/16
Analyzed: 3/20

All ok

**Semivolatiles Table 1. Preservation and Holding Time Actions**

| Matrix | Preservation | Criteria | Action | |
|---|---|---|---|---|
| | | | Detect | Non-detect |
| Aqueous/Non-aqueous | Samples received at a temperature > 6°C | | J | UJ |
| Aqueous/Non-aqueous | Cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared within the 14-day technical holding time | No qualification | No qualification |
| | Not cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared within the 14-day technical holding time | Use professional judgment | Use professional judgment |
| | Cooled/not cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared outside the 14-day technical holding time | J | R |
| Aqueous | Cooled at temperature ≤ 6°C | Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time | No qualification | No qualification |
| | | Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time | J- | R |
| | | Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time | J- | R |

| Matrix | Preservation | Criteria | Action | |
|---|---|---|---|---|
| | | | Detect | Non-detect |
| Aqueous | Not cooled at temperature ≤ 6°C | Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time | Use professional judgment | Use professional judgment |
| | | Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time | J | R |
| | | Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time | J | R |
| Non-aqueous | Cooled at temperature ≤ 6°C | Samples extracted within the 14-day and analyzed within the 40-day technical holding time | No qualification | No qualification |
| | | Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time | J- | R |
| | | Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time | J- | R |
| Non-aqueous | Not cooled at temperature ≤ 6°C | Samples extracted within 14- day and analyzed within the 40-day technical holding time | Use professional judgment | Use professional judgment |
| | | Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time | J | R |
| | | Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time | J | R |

**Surrogates (Form II)**

✓ %Rs within limit. Used QAPP limits p-Terphenyl-d14 solid 30-102, aqueous 48-109; 2,4,6-Tribromophenol aqueous 35-132

If any surrogate is out, qualify all results as follows in Table 7.

**Semivolatiles Table 7. Surrogate Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Surrogate not present or not at specified concentration | J or R | UJ or R |
| %R < Expanded Lower Acceptance Limit (10%, excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis) | J- | R |
| Expanded Lower Acceptance Limit (10%) ≤ %R (excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis) < specified Lower Acceptance Limit | J- | UJ |
| %R < specified Lower Acceptance Limit (diluted sample analysis) | Use professional judgment | Use professional judgment |
| %R within specified Acceptance Limits | No qualification | No qualification |
| %R > specified Upper Acceptance Limit | J+ | No qualification |

All ok

**Tune Check, Calibration and Verification (Form V, run log)**

✓ DFTPP before calibrations and verifications and every 12 hours

**Semivolatiles Table 2. Instrument Performance Check Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Instrument Performance Check not analyzed at specified frequency and sequence | R | R |
| Base peak mass assignment incorrect | R | R |
| Ion abundance criteria not met | J or R | UJ or R |

✓ all ion abundance criteria within range

All ok

**Calibration and Verification (run log, Form VI, Form VII)**

- ✓ Initial Calibration RSD met criteria RSD <20%
- ✓ minimum RRF met criteria Note EDS used RRF >0.05 UJ <0.05 R
- ✓ ICV after calibration
- ✓ ICV/opening 70-130%

CCV %D and minimum RRF met criteria %D <20%

- ✓ CCV every 12 hours
- ✓ closing CCV %D and minimum RRF met criteria

**Semivolatiles Table 3. Initial Calibration Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Initial Calibration not performed at specified frequency and sequence | R | R |
| Initial Calibration not performed at specified concentrations | J | UJ |
| RRF for target analyte < specified Minimum RRF | J+ or R | UJ or R |
| RRF for target analyte ≥ specified Minimum RRF | No qualification | No qualification |
| %RSD for target analyte > specified Maximum %RSD | J | No qualification or UJ |

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| %RSD for target analyte ≤ specified Maximum %RSD | No qualification | No qualification |

**Semivolatiles Table 4. ICV Actions**

| Criteria for ICV | Action | |
|---|---|---|
| | Detect | Non-detect |
| ICV not performed at specified frequency and sequence | J | UJ |
| ICV not performed at specified concentration | No qualification<br>or<br>J | No qualification<br>or<br>UJ |
| ICV not from alternate source or different lot than the ICAL standards | J | No qualification |
| RRF for target analyte < specified Minimum RRF | J or R | UJ or R |
| RRF for target analyte ≥ specified Minimum RRF | No qualification | No qualification |
| %D for target analyte not within specified %D acceptance limits | J | UJ |

| Criteria for ICV | Action | |
|---|---|---|
| | Detect | Non-detect |
| %D for target analyte within specified %D acceptance limits | No qualification | No qualification |

**Semivolatiles Table 5. CCV Actions for Semivolatiles Analysis**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| CCV not performed at specified frequency and sequence | J or R | UJ or R |
| CCV not performed at specified concentration | No qualification<br>or<br>J | No qualification<br>or<br>UJ |
| RRF for target analyte < specified Minimum RRF | J | UJ or R |
| RRF for target analyte ≥ specified Minimum RRF | No qualification | No qualification |
| %D for target analyte not within specified %D acceptance limits | J | UJ |
| %D for target analyte within specified %D acceptance limits | No qualification | No qualification |

Inst: K-MS-28

ICAL 7/7/22

ICV 7/7/22 2039

CCV 3/20/23 1342

All ok

**Blanks (Form IV, Form 1)**

- ✓ evaluate storage, method, and field blanks
- ✓ method blank were prepared and analyzed in association with all samples in SDG
- ✓ blanks may not be qualified because of contamination in another blank

**Semivolatiles Table 6. Blank Actions**

| Blank Type | Blank Result | Sample Result | Action |
|---|---|---|---|
| | Not analyzed at specified frequency or sequence | Detect | J |
| | | Non-detect | No qualification |
| Method, TCLP/SPLP LEB, Field (including Equipment and Rinse) | Detect | Non-detect | No qualification |
| | < QL | < QL | Report at QL and qualify U |
| | | ≥ QL | Report at sample result and qualify J+ or No qualification |
| | ≥ QL | < QL | Report at QL and qualify U |
| | | ≥ QL but < Blank Result | Report at sample result and qualify U |
| | | ≥ QL and ≥ Blank Result | Report at sample result and qualify J+ or No qualification |

| Blank Type | Blank Result | Sample Result | Action |
|---|---|---|---|
| | Gross contamination | Detect | Report at sample result and qualify R |
| | TICs concentrations ≥ QLs | Detect | Use professional judgment |

Method Blanks

KQ2304868-01 all ND

Equipment Blanks

none

**LCS (Form III, batch worksheet)**

- ✓ use QAPP limits to evaluate accuracy (%R) aqueous 42-147%R, solid 39-113%R; Pentachlorophenol aqueous 27-112%R

- ✓ if qualification is required, qualified all samples in prep batch

Semivolatiles Table 9. LCS Actions

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| LCS not performed at specified frequency or concentration | Use professional judgment | Use professional judgment |
| LCS %R limits not specified | Use professional judgment | Use professional judgment |
| %R < specified Lower Acceptance Limit | J- | R |
| %R within specified Acceptance Limits | No qualification | No qualification |
| %R > specified Upper Acceptance Limit | J+ | No qualification |

Per EDS guidance-do not Reject results unless %R is <10% recovery.

KQ2304868-02 and KQ2304868-03 all ok

**MS/MSD (Form III)**

- ✓ use QAPP limits to evaluate accuracy (%R) and precision (RPD)

aqueous 42-147%R RPD 30, solid 39-113%R RPD 40

- ✓ qualify parent sample only
- ✓ remember 4X rule

Semivolatiles Table 8. MS/MSD Actions

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| MS/MSD not analyzed at specified frequency | Use professional judgment | Use professional judgment |
| MS/MSD not prepared from field sample | Use professional judgment | Use professional judgment |
| %R or RPD limits not specified | Use professional judgment | Use professional judgment |
| %R < Expanded Lower Acceptance Limit (10%, excluding spiked analyte with %R lower limit of 10% or less) | J | R |
| Expanded Lower Acceptance Limit (10%, excluding spiked analyte with %R lower limit of 10% or less) ≤ %R < specified Lower Acceptance Limit | J | UJ |
| %R or RPD within specified Acceptance Limits | No qualification | No qualification |
| %R or RPD > specified Upper Acceptance Limit | J | No qualification |

none

**Internal Standards (Form VIII)**

✓ peaks within -50-200%

✓ RTs within 30 seconds

**Semivolatiles Table 11. Internal Standard Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Internal standard compound not present in sample or blank as specified | R | R |
| Internal standard compound not analyzed at specified concentration | Use professional judgment | Use professional judgment |
| Area response < Expanded Lower Acceptance Limit (20%) of the opening CCV or ICV in the same analytical sequence | J+ | R |
| Expanded Lower Acceptance Limit (20%) ≤ Area response < Lower Acceptance Limit (50%) of the opening CCV or ICV in the same analytical sequence | J+ | UJ |
| Lower Acceptance Limit (50%) ≤ Area response ≤ Upper Acceptance Limit ( 200%) of the opening CCV or ICV in the same analytical sequence | No qualification | No qualification |
| Area response > Upper Acceptance Limit (200%) of the opening CCV or ICV in the same analytical sequence | J- | No qualification |
| RT shift between sample/blank and opening CCV or ICV in the same analytical sequence > 30 seconds | J | R |
| RT shift between sample/blank and opening CCV or ICV in the same analytical sequence < 30 seconds | No qualification | No qualification |

All ok

**Target Analytes (Form 1, raw data)**

- ✓ RRTs for all positive results withing ±0.06 of standard
- ✓ primary and secondary ion intensities within 20% of that in the standard
- ✓ acceptable baseline stability, resolution, peak shape, graph

**Semivolatiles Table 12. Target Analyte Identification Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Mass spectral ion abundance criteria specified for target analyte not met | J or R<br>or<br>Report the result at QL and qualify U | Not applicable |
| Target analyte RRT outside specified RRT windows | R<br>or<br>Report the result at QL and qualify U | Not applicable |

All ok

**TICs (Form 1)**

✓ match quality >85% for tentatively identified - NJ

✓ match quality >85% - for unknown - J

✓ match quality <85% - for tentatively identified – change to unknow and J

None

**Compound Quantitation (Form 1, run log, prep log, EDD)**

✓ evaluate dilutions, re-extractions and re-analyses to confirm best results is reported and all other results are not reportable in EDD

All ok

**Field Duplicates**

✓ RPD ≤50% (solid matrix) or ≤30% (aqueous matrix) if both results ≥5x PQL

For low-level results (one or both detected values ≤5x PQL) or when one result is a non-detection, the control limit is absolute difference ≤2x PQL (solid matrix) or ≤PQL (aqueous matrix). Non-detected values will be assigned the nominal value of the MDL for making comparison.

Qualify detects as estimated "J" and qualify non-detects as estimated "UJ".

List samples and results effected below.

✓ see field duplicate worksheet

none

**CLIENT:** HGL

**PROJECT:** Portland Harbor

**SDG:** K2302911

**Validated by:** LL 5/24/23

**Reviewed by:** **DLW 5/25/23**

**LAB:** ALS Kelso, WA

Polynuclear Aromatic Hydrocarbons (PAHs) SIM

**Sample Receipt / Hold times** (COC, receipt logs, case narrative)

✓received at <10% ◦C

✓HT (aqueous– 7 days to extract, 40 days extraction to analysis; solids – 14 days to extraction, 40 days extraction to analysis)

Collected: 3/9

Extracted: 3/15

Analyzed: 4/14

All ok

**Semivolatiles Table 1. Preservation and Holding Time Actions**

| Matrix | Preservation | Criteria | Action | |
|---|---|---|---|---|
| | | | Detect | Non-detect |
| Aqueous/Non-aqueous | Samples received at a temperature > 6°C | | J | UJ |
| Aqueous/Non-aqueous | Cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared within the 14-day technical holding time | No qualification | No qualification |
| | Not cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared within the 14-day technical holding time | Use professional judgment | Use professional judgment |
| | Cooled/not cooled at temperature ≤ 6°C | TCLP/SPLP leachate samples prepared outside the 14-day technical holding time | J | R |
| Aqueous | Cooled at temperature ≤ 6°C | Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time | No qualification | No qualification |
| | | Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time | J- | R |
| | | Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time | J- | R |

| Matrix | Preservation | Criteria | Action | |
|---|---|---|---|---|
| | | | Detect | Non-detect |
| Aqueous | Not cooled at temperature ≤ 6°C | Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time | Use professional judgment | Use professional judgment |
| | | Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time | J | R |
| | | Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time | J | R |
| Non-aqueous | Cooled at temperature ≤ 6°C | Samples extracted within the 14-day and analyzed within the 40-day technical holding time | No qualification | No qualification |
| | | Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time | J- | R |
| | | Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time | J- | R |
| Non-aqueous | Not cooled at temperature ≤ 6°C | Samples extracted within 14- day and analyzed within the 40-day technical holding time | Use professional judgment | Use professional judgment |
| | | Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time | J | R |
| | | Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time | J | R |

**Surrogates (Form II)**

✓ %Rs within limit. Used lab limits

If any surrogate is out, qualify all results as follows in Table 7.

**Semivolatiles Table 7. Surrogate Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Surrogate not present or not at specified concentration | J or R | UJ or R |
| %R < Expanded Lower Acceptance Limit (10%, excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis) | J- | R |
| Expanded Lower Acceptance Limit (10%) ≤ %R (excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis) < specified Lower Acceptance Limit | J- | UJ |
| %R < specified Lower Acceptance Limit (diluted sample analysis) | Use professional judgment | Use professional judgment |
| %R within specified Acceptance Limits | No qualification | No qualification |
| %R > specified Upper Acceptance Limit | J+ | No qualification |

All ok

**Tune Check, Calibration and Verification (Form V, run log)**

- ✓ DFTPP before calibrations and verifications and every 12 hours

**Semivolatiles Table 2. Instrument Performance Check Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Instrument Performance Check not analyzed at specified frequency and sequence | R | R |
| Base peak mass assignment incorrect | R | R |
| Ion abundance criteria not met | J or R | UJ or R |

- ✓ all ion abundance criteria within range

All ok

**Calibration and Verification (run log, Form VI, Form VII)**

- ✓ Initial Calibration RSD met criteria RSD <20%
- ✓ minimum RRF met criteria Note EDS used RRF >0.05 UJ <0.05 R
- ✓ ICV after calibration
- ✓ ICV/opening CCV %D and minimum RRF met criteria %D <20%
- ✓ CCV every 12 hours
- ✓ closing CCV %D and minimum RRF met criteria

**Semivolatiles Table 3. Initial Calibration Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Initial Calibration not performed at specified frequency and sequence | R | R |
| Initial Calibration not performed at specified concentrations | J | UJ |
| RRF for target analyte < specified Minimum RRF | J+ or R | UJ or R |
| RRF for target analyte ≥ specified Minimum RRF | No qualification | No qualification |
| %RSD for target analyte > specified Maximum %RSD | J | No qualification or UJ |

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| %RSD for target analyte ≤ specified Maximum %RSD | No qualification | No qualification |

**Semivolatiles Table 4. ICV Actions**

| Criteria for ICV | Action | |
|---|---|---|
| | Detect | Non-detect |
| ICV not performed at specified frequency and sequence | J | UJ |
| ICV not performed at specified concentration | No qualification<br>or<br>J | No qualification<br>or<br>UJ |
| ICV not from alternate source or different lot than the ICAL standards | J | No qualification |
| RRF for target analyte < specified Minimum RRF | J or R | UJ or R |
| RRF for target analyte ≥ specified Minimum RRF | No qualification | No qualification |
| %D for target analyte not within specified %D acceptance limits | J | UJ |

| Criteria for ICV | Action | |
|---|---|---|
| | Detect | Non-detect |
| %D for target analyte within specified %D acceptance limits | No qualification | No qualification |

Semivolatiles Table 5. CCV Actions for Semivolatiles Analysis

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| CCV not performed at specified frequency and sequence | J or R | UJ or R |
| CCV not performed at specified concentration | No qualification or J | No qualification or UJ |
| RRF for target analyte < specified Minimum RRF | J | UJ or R |
| RRF for target analyte ≥ specified Minimum RRF | No qualification | No qualification |
| %D for target analyte not within specified %D acceptance limits | J | UJ |
| %D for target analyte within specified %D acceptance limits | No qualification | No qualification |

Inst: K-MS-20
ICAL 4/12/23
ICV 4/13/23 0905
CCV 4/14/23 0818 all samples
All ok

**Blanks (Form IV, Form 1)**

- ✓ evaluate storage, method, and field blanks
- ✓ method blank were prepared and analyzed in association with all samples in SDG
- ✓ blanks may not be qualified because of contamination in another blank

**Semivolatiles Table 6. Blank Actions**

| Blank Type | Blank Result | Sample Result | Action |
|---|---|---|---|
| | Not analyzed at specified frequency or sequence | Detect | J |
| | | Non-detect | No qualification |
| Method, TCLP/SPLP LEB, Field (including Equipment and Rinse) | Detect | Non-detect | No qualification |
| | < QL | < QL | Report at QL and qualify U |
| | | ≥ QL | Report at sample result and qualify J+ or No qualification |
| | ≥ QL | < QL | Report at QL and qualify U |
| | | ≥ QL but < Blank Result | Report at sample result and qualify U |
| | | ≥ QL and ≥ Blank Result | Report at sample result and qualify J+ or No qualification |

| Blank Type | Blank Result | Sample Result | Action |
|---|---|---|---|
| | Gross contamination | Detect | Report at sample result and qualify R |
| | TICs concentrations ≥ QLs | Detect | Use professional judgment |

Method Blanks

KQ2304866-01 SIM All samples

2-Methylnaphthalene 0.90 J 0.95 4.75
Sample 4 < 5X Q U @ result (MBL)
Acenaphthene 0.65 J 0.69 3.45
All <5X or ND Q U @ result (MBL)
Anthracene 0.39 J 0.41 2.05
All <5X or ND Q U @ result (MBL)
Benz(a)anthracene 0.67 J 0.71 3.55
All <5X or ND Q U @ result (MBL)
Fluoranthene 0.95 J 1.01 5.05
Sample 5 <5X Q U @ result (MBL)
Fluorene 0.70 J 0.74 3.7
Samples 1,2,4,5 <5X or ND Q U @ result (MBL)
Naphthalene 1.9 J 1.97 9.85
Samples 1-5 <5X Q U @ result (MBL)
Phenanthrene 3.0 J 3.23 16.15
Samples 1, 2, 5, 6 <5X Q U @ result (MBL)

Pyrene 0.92 J 0.98 4.9
Samples 5 <5X Q U @ result (MBL)

Equipment Blanks

None

**LCS (Form III, batch worksheet)**

- ✓ use lab limits to evaluate accuracy (%R)
- ✓ if qualification is required, qualified all samples in prep batch

**Semivolatiles Table 9. LCS Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| LCS not performed at specified frequency or concentration | Use professional judgment | Use professional judgment |
| LCS %R limits not specified | Use professional judgment | Use professional judgment |
| %R < specified Lower Acceptance Limit | J- | R |
| %R within specified Acceptance Limits | No qualification | No qualification |
| %R > specified Upper Acceptance Limit | J+ | No qualification |

| Chemical | CASRN | Solid Matrix Accuracy Control Limits (%R) | Solid Matrix Precision Control Limits (RPD) | Aqueous Matrix Accuracy Control Limits (%R) | Aqueous Matrix Precision Control Limits (RPD) |
|---|---|---|---|---|---|
| 2-Methylnaphthalene | 91-57-6 | 43-92 | 40 | 42-108 | 30 |
| Acenaphthene | 83-32-9 | 44-95 | 40 | 58-98 | 30 |
| Acenaphthylene | 208-96-8 | 44-93 | 40 | 61-102 | 30 |
| Anthracene | 120-12-7 | 46-100 | 40 | 65-98 | 30 |
| Benzo(a)anthracene | 56-55-3 | 52-105 | 40 | 67-96 | 30 |
| Benzo(a)pyrene | 50-32-8 | 52-111 | 40 | 68-107 | 30 |
| Benzo(b)fluoranthene | 205-99-2 | 52-114 | 40 | 69-104 | 30 |
| Benzo(g,h,i)perylene | 191-24-2 | 45-107 | 40 | 61-110 | 30 |
| Benzo(k)fluoranthene | 207-08-9 | 52-112 | 40 | 68-108 | 30 |
| Chrysene | 218-01-9 | 51-110 | 40 | 67-105 | 30 |
| Dibenz(a,h)anthracene | 53-70-3 | 44-110 | 40 | 54-118 | 30 |
| Fluoranthene | 206-44-0 | 49-102 | 40 | 63-106 | 30 |
| Fluorene | 86-73-7 | 45-98 | 40 | 59-97 | 30 |
| Indeno(1,2,3-cd)pyrene | 193-39-5 | 44-117 | 40 | 61-115 | 30 |
| Naphthalene | 91-20-3 | 42-88 | 40 | 59-95 | 30 |
| Phenanthrene | 85-01-8 | 41-99 | 40 | 61-100 | 30 |
| Pyrene | 129-00-0 | 48-104 | 40 | 64-104 | 30 |

SIM: KQ2304866-02 and KQ2304866-03 all ok

**MS/MSD (Form III)**

- ✓ use QAPP limits to evaluate accuracy (%R) and precision (RPD)

- ✓ qualify parent sample only
- ✓ remember 4X rule

none

**Semivolatiles Table 8. MS/MSD Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| MS/MSD not analyzed at specified frequency | Use professional judgment | Use professional judgment |
| MS/MSD not prepared from field sample | Use professional judgment | Use professional judgment |
| %R or RPD limits not specified | Use professional judgment | Use professional judgment |
| %R < Expanded Lower Acceptance Limit (10%, excluding spiked analyte with %R lower limit of 10% or less) | J | R |
| Expanded Lower Acceptance Limit (10%, excluding spiked analyte with %R lower limit of 10% or less) ≤ %R < specified Lower Acceptance Limit | J | UJ |
| %R or RPD within specified Acceptance Limits | No qualification | No qualification |
| %R or RPD > specified Upper Acceptance Limit | J | No qualification |

**Internal Standards (Form VIII)**

- ✓ peaks within -50-200%
- ✓ RTs within 30 seconds

**Semivolatiles Table 11. Internal Standard Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Internal standard compound not present in sample or blank as specified | R | R |
| Internal standard compound not analyzed at specified concentration | Use professional judgment | Use professional judgment |
| Area response < Expanded Lower Acceptance Limit (20%) of the opening CCV or ICV in the same analytical sequence | J+ | R |
| Expanded Lower Acceptance Limit (20%) ≤ Area response < Lower Acceptance Limit (50%) of the opening CCV or ICV in the same analytical sequence | J+ | UJ |
| Lower Acceptance Limit (50%) ≤ Area response ≤ Upper Acceptance Limit ( 200%) of the opening CCV or ICV in the same analytical sequence | No qualification | No qualification |
| Area response > Upper Acceptance Limit (200%) of the opening CCV or ICV in the same analytical sequence | J- | No qualification |
| RT shift between sample/blank and opening CCV or ICV in the same analytical sequence > 30 seconds | J | R |
| RT shift between sample/blank and opening CCV or ICV in the same analytical sequence < 30 seconds | No qualification | No qualification |

All ok

**Target Analytes (Form 1, raw data)**

- ✓ RRTs for all positive results withing ±0.06 of standard
- ✓ primary and secondary ion intensities within 20% of that in the standard
- ✓ acceptable baseline stability, resolution, peak shape, graph

**Semivolatiles Table 12. Target Analyte Identification Actions**

| Criteria | Action | |
|---|---|---|
| | Detect | Non-detect |
| Mass spectral ion abundance criteria specified for target analyte not met | J or R<br>or<br>Report the result at QL and qualify U | Not applicable |
| Target analyte RRT outside specified RRT windows | R<br>or<br>Report the result at QL and qualify U | Not applicable |

All ok

**TICs (Form 1)**

✓ match quality >85% for tentatively identified - NJ

✓ match quality >85% - for unknown - J

✓ match quality <85% - for tentatively identified – change to unknow and J

None

**Compound Quantitation (Form 1, run log, prep log, EDD)**

✓ evaluate dilutions, re-extractions and re-analyses to confirm best results is reported and all other results are not reportable in EDD

All ok

**Field Duplicates**

✓ RPD ≤50% (solid matrix) or ≤30% (aqueous matrix) if both results ≥5x PQL

For low-level results (one or both detected values ≤5x PQL) or when one result is a non-detection, the control limit is absolute difference ≤2x PQL (solid matrix) or ≤PQL (aqueous matrix). Non-detected values will be assigned the nominal value of the MDL for making comparison.

Qualify detects as estimated "J" and qualify non-detects as estimated "UJ".

List samples and results effected below.

✓ see field duplicate worksheet

none