



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10 LABORATORY
7411 Beach Dr. East
Port Orchard, Washington 98366

**QUALITY ASSURANCE MEMORANDUM
FOR ORGANIC CHEMICAL ANALYSES**

Date: October 5, 2016

To: Helen Bottcher, RPM
Office of Environmental Cleanup, USEPA Region 10

From: Chris Pace, Chemist
Office of Environmental Assessment, USEPA Region 10 Laboratory

Subject: Quality Assurance Review for the Polycyclic Aromatic Hydrocarbon Analysis of Samples from the Wyckoff/ Eagle Harbor SF Site Clam Tissue Sampling Project.

Project Code: WEH-021C
Account Code: 2016T10P302DD210S1LA00

CC: Cathy Martin - USACE

The following is a quality assurance review of the data for polycyclic aromatic hydrocarbon analysis of clam tissue samples from the above referenced site. The analyses were performed by the US EPA Region 10 Laboratory in Port Orchard, WA, following US EPA and Laboratory guidelines.

This review was conducted for the following samples:

16274200	16274201	16274202	16274203	16274204	16274208
16274209	16274210	16274211	16274212	16274213	16274214
16274216					

Data Qualifications

Comments below refer to the quality control specifications outlined in the Laboratory's current Quality Assurance Manual, Standard Operating Procedures (SOPs) and the Quality Assurance Project Plan (QAPP). No excursions were required from the method Standard Operating Procedure.

The quality control measures which did not meet Laboratory/QAPP criteria are annotated in the title of each affected subsection with "*Laboratory/QAPP Criteria Not Met*".

For those tests for which the EPA Region 10 Laboratory has been accredited by The NELAC Institute (TNI), all requirements of the current TNI Standard have been met.

1. Sample Transport and Receipt

Upon sample receipt, no conditions were noted that would impact data quality.

2. Sample Holding Times

The concentration of an analyte in a sample or extract of a sample may increase or decrease over time depending on the nature of the analyte. The holding time maximum criteria applied for the extraction of frozen tissue samples is one year from the time of collection. Extracts have a holding time maximum of 40 days from the time of preparation. All samples were extracted and analyzed within these criteria.

3. Sample Preparation

Samples were prepared according to the method/SOP.

4. Initial Calibration - Laboratory/QAPP Criteria Not Met

Initial calibration was performed on 9/19/16 for the target and surrogate compounds. Percent relative standard deviations (%RSDs) of the relative response factors (RRFs) met the criteria of $\leq 15\%$ or correlation coefficients met the criteria of ≥ 0.99 except for the following.

Benzo(b)fluoranthene resulted with a correlation coefficient < 0.99 . The percent difference of the individual benzo(b)fluoranthene calibration levels did not vary by more than $\pm 20\%$ from their true values when processed vs. the calibration curve as samples. None of the benzo(b)fluoranthene results were qualified on this basis.

The second source verifications (SSV) percent accuracies were 70-130% of the true values.

5. Continuing Calibration Verification (CCV) - Laboratory/QAPP Criteria Not Met

The CCV met the criteria for frequency of analysis and relative retention time (RRT) windows for all target and surrogate compounds. The percent accuracies were 80-120% of the true values for all reported results except for the following.

Benzo(a)anthracene, indeno(1,2,3-cd)pyrene and dibenzo(a,h)anthracene resulted with $> 120\%$ accuracy in the CCV analyzed on 9/21/16. The detected benzo(a)anthracene, indeno(1,2,3-cd)pyrene and dibenzo(a,h)anthracene results in the associated samples were qualified estimated. "J". The non-detected results did not require qualification on this basis. Associated samples: 16274201 and 16274202.

6. LCS/LCSD

Data for laboratory control sample/laboratory control sample duplicates (LCS/LCSD) are generated to provide information on the accuracy and precision of the analytical method and the laboratory performance. The LCS/LCSD recoveries met the criteria of 50-150% with a RPD of ≤ 30 .

7. Blank Analysis

Method blanks were analyzed with each sample batch to evaluate the potential for laboratory contamination and effects on the sample results. Target analytes detected in samples were reported without qualification if the responses were ≥ 5 times that of the blank(s). Detected sample results were qualified 'U' if the results were below this criteria. The sample concentration or the sample quantification limit, whichever is greater, was reported as the qualified result.

8. Surrogate Spikes

Surrogate recoveries are used to help in the evaluation of laboratory performance on individual samples. The surrogate recoveries in all samples met the criteria of 50-150%.

9. Matrix Spike/Matrix Spike Duplicate Analysis (MS/MSD) - Laboratory/QAPP Criteria Not Met

Data for MS/MSD are generated to provide information on the accuracy and precision of the analytical method and the laboratory performance. An MS/MSD analysis was performed using samples 16274201. The MS/MSD recoveries met the criteria of 50-150% with a RPD of ≤ 30 except for the following.

Benzo(a)anthracene resulted with $>150\%$ recovery in the matrix spike of 16274201. Benzo(a)anthracene, indeno(1,2,3-cd)pyrene and dibenzo(a,h)anthracene resulted with $>150\%$ recovery in the matrix spike duplicated of 16274201. The detected benzo(a)anthracene and indeno(1,2,3-cd)pyrene in the native sample were qualified as estimated, "J". The non-detected dibenzo(a,h)anthracene did not require qualification on this basis.

10. Internal Standard Performance

Internal standards performance criteria ensure that GC/MS sensitivity and response are stable during every analytical run. The retention time variations of all internal standards were within 30 seconds of the continuing calibration standard. The percent areas of all the internal standards were within the specified 50% to 200% of the continuing calibration standard for all reported results.

11. Compound Quantitation

The initial calibration functions were used for calculations. Reported quantitation limits were based on the initial calibration standards and sample size used for the analysis. Due to background levels of PAHs and/or chromatographic interference found in the blank(s), PAH results were report to the reporting limit. The background levels prevented reporting "J" values for detected PAHs between the limit of detection and reporting limit.

All manual integrations have been reviewed and found to comply with acceptable integration practices.

12. Identification

All of the compounds detected in the analyses were within the RRT windows, met the USEPA spectral matching criteria and/or were judged to be acceptable.

13. Data Qualifiers

All requirements for data qualifiers from the preceding sections were accumulated. Each sample data summary sheet and each compound was checked for positive or negative results. From this, the overall need for data qualifiers for each analysis was determined. In cases where more than one of the preceding sections required data qualifiers, the most restrictive qualifier has been added to the data.

The usefulness of qualified data should be treated according to the severity of the qualifier in light of the project's data quality objectives. Should questions arise regarding the data, contact Chris Pace at the Region 10 Laboratory, phone number (360) 871 - 8703.

Qualifier	Definition
U	The analyte was not detected at or above the reported value.
J	The identification of the analyte is acceptable; the reported value is an estimate.
UJ	The analyte was not detected at or above the reported value. The reported value is an estimate.
R	The presence or absence of the analyte can not be determined from the data due to severe quality control problems. The data are rejected and considered unusable. <u>No value is reported with this qualification.</u>
NA	Not Applicable, the parameter was not analyzed for, or there is no analytical result for this parameter. <u>No value is reported with this qualification.</u>