15-0349 PAH/Biomarker QA/QC Summary

Project:	ANIMIDA III
Parameters:	PAH and Biomarker
Laboratory:	Battelle, Norwell, MA
Matrix:	Sediment
Data Set:	DP-15-0315
Analytical SOP:	5-157
Method Reference:	Modified EPA Method 8270D

Sample Custody	Receipt Date	Temp (°C)
	8/14/2014 and 8/11/2015	4.0 and 0.9, 1.2, 0.3

Corrective Actions	Sample L4815 was listed on the COC as QAH-122 with a collection time of 8:40 on 8/6/15. There was no jar that had matching collection information but there was a jar that had the correct station information that belongs to that sample. The ID on the jar was QAH-207 with a collection date of 8/6/15 @ 10:00am. Logged in as the COC states but I believe it should be the QAH-207.
Sample Storage	The samples were stored in an access-limited freezer until sample preparation could begin.

METHOD SUMMARIES

Sample Preparation	Samples were prepared for analysis by weighing approximately 30 grams of sample material into a pre-cleaned extraction vessel and dried using sodium sulfate. Each sample was spiked with PAH, Biomarker and SHC surrogates and extracted 3 times using methylene chloride by shaker table. The combined extracts were dried over sodium sulfate and concentrated by Kuderna-Danish (KD) and nitrogen evaporation techniques. Sample clean-up was performed on the extracts using alumina columns. Extracts were further cleaned up and fractionated using silica gel columns. The F1 fraction was collected and split for TPH/SHC and biomarker analyses. The F2 fraction was collected for PAH and alkylated PAH analysis. The extracts were concentrated and spiked with IS for analysis.
Prep comments	All samples had overlying water layer. Overlying water was poured off before being weighed out. After samples were weighed out to 30g, overlying water layer still existed. The samples were centrifuged and then poured off again to remove existing water layer. Two comments in relation to the PB: Sample concentrated to approximately 1 mL pre silica clean-up. During silica columns, the F2 fractionation was eluted with 36 mL of DCM/Hexane(1:1) instead of 21.
Analysis	PAH, alkylated PAH (F2 fraction) and Biomarkers (F1 fraction) were measured by gas chromatography-mass spectrometry (GC/MS) in the selected ion mode (SIM). An initial calibration consisting of target analytes was analyzed prior to analysis to demonstrate the linear range of analysis. Calibration verification was performed every 24 hours in which samples were analyzed. Concentrations of target compounds were calculated versus internal standards. Target PAH were quantified using the average response factors (RF) generated from the initial calibration. The alkyl homologue PAH series were assigned the RF of the parent PAH. Biomarkers

	used RFs from the single individual biomarkers within the calibration standard curve. All reported data (except NSC and CO) is corrected based on surrogate recoveries. All data is reported on dry weight basis except the NSC and CO (oil weight).
Analysis comments	All data are reported as surrogate corrected and on a dry wt. basis. The NSC and CO are reported as not surrogate corrected on an oil weight basis.

Holding Times	Extraction Date(s)	Analysis Date(s)
	8/25/2015 & 9/2/2015	9/3-6/2015

Procedural Blank (PB)	A PB was prepared with this analytical batch to ensure the sample extraction and analysis methods are free of contamination.
PB <5 X MDL Samples must be >5x PB	No exceedances noted.
	Comments: Comments: None.

Laboratory Control Spike (LCS)	A LCS was prepared with this analytical batch. The percent recoveries of target analytes were calculated to measure accuracy.
Recovery of 70-130%	No exceedances noted.
	Comments: None.

North Slope Crude (NSC) and Control Oil	A NSC Reference Oil and Control Oil was prepared with this batch to evaluate the instrumental accuracy and also provide petroleum pattern information, aiding in the qualitative identification of target analytes.
< 30% RPD for 90% of analytes	No exceedances noted.
	Comments: None.

Surrogate Recovery	Surrogate compounds were added prior to extraction. The surrogate recoveries are calculated to measure extraction efficiency.
Recovery of 40-120%	No exceedance noted.
	Comments: None.

Standard Reference Material (SRM)	An SRM was prepared with this analytical batch.
% Difference <30% for analytes above 5XMDL	No exceedances noted.
	Comments: There were no certified values for the target analytes.

Matrix Spike/Matrix Spike Duplicate (MS/MSD)	A MS/MSD was prepared with this analytical batch. The percent recoveries of target analytes were calculated to measure accuracy. The RPD of target analytes were calculated to measure data quality in terms of accuracy.
Recovery of 70-130% Relative Percent Difference (RPD) < 30%	No exceedances noted.
	Comments: None.

Initial Calibration (ICAL)	The GC/MS is calibrated with a minimum 5 level curve for all compounds.
Individual RSD ≤25%; Mean RSD ≤15%	No exceedances noted.
	Comments: None.

Independent Calibration Check (ICC)	The independent check was run after each initial calibration to verify the calibration. This standard is from a different source than the ICAL.
Individual and Mean PD ≤25%	No exceedances noted.
	Comments: None.

Continuing Calibration Verification (CCV)	Continuing calibration standards were run every 24 hours to ensure that initial calibration is still valid.
Individual RSD ≤25%; Mean RSD ≤15%	No exceedances noted.
	Comments: None.