

15-0350 PAH/Biomarker QA/QC Summary

Project:	ANIMIDA III
Parameters:	PAH and Biomarker
Laboratory:	Battelle, Norwell, MA
Matrix:	Sediment, Peat
Data Set:	DP-15-0317
Analytical SOP:	5-157
Method Reference:	Modified EPA Method 8270D

Sample Custody	Receipt Date	Temp (°C)
	8/11/2015 and 8/25/2015	0.9, 1.2, 0.3 and 2.8

Corrective Actions	<p>SHP-150811-03 had 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.</p> <p>SHP-150825-01 had missing COCs. The samples were confirmed with the project manager.</p>
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	<p>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.</p> <p>Other minor comments/observations can be found in the Sample Specific Comment section of the prep batch.</p>
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

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	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	None.	
Holding Times	Extraction Date(s)	Analysis Date(s)
	8/31/2015 & 9/2 and 9/2015	9/11-16/2015

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	No exceedances noted.
Samples must be >5x PB	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.

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.

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.

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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%	No exceedances noted.
Relative Percent Difference (RPD) < 30%	Comments: None.
Initial Calibration (ICAL)	The GC/MS is calibrated with a minimum 5 level curve for all compounds.
Individual RSD \leq 25%; Mean RSD \leq 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 \leq 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 \leq 25%; Mean RSD \leq 15%	No exceedances noted.
	Comments: None.