

14-0507 PAH/BIOMARKERS QA/QC Summary

Project:	ANIMIDA III
Parameters:	PAH and Biomarkers
Laboratory:	Battelle, Norwell, MA
Matrix:	Sediment
Data Set:	DP-14-0779
Analytical SOP:	5-157
Method Reference:	Modified EPA Method 8270D

Sample Custody	Receipt Date	Temp (°C)
	8/14/2014	4.0

Corrective Actions	None.
Sample Storage	The samples were stored in an access-limited freezer until sample preparation could begin.

METHOD SUMMARIES

Sample Preparation	The sediment samples were extracted following a modified EPA Method 3510C. 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 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	None.
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

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	the NSC and CO (oil weight).	
Analysis comments	None.	
Holding Times	Extraction Date(s)	Analysis Date(s)
	10/16/2014, 11/4/2014 and 11/18/2014	11/8-14, 20,22/2014

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 exceedences noted. 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 exceedences 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%	One exceedence noted. Comments: Sample M5908 fails SIS area criteria below QC limits. The FID analysis confirms the results, and nothing was noted in the sample preparation records. Surrogate corrected data similar to other samples. No further corrective action was taken.

Matrix Spike and 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 exceedences noted. Comments: None.

Standard Reference Material (SRM)	An SRM was prepared with this batch to assess accuracy of the analytical procedures.
< 30 PD from target concentration and the 95% confidence level analyte concentration must be > 5x the MDL. Concentration must be certified and >5x the MDL for MQO to apply	No exceedences noted. Comments: None.

North Slope Crude (NSC)	A NSC Reference Oil was prepared with this batch to evaluate the
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	instrumental accuracy and also provide petroleum pattern information, aiding in the qualitative identification of target analytes. The control oil also run in this batch has no associated target values and is not evaluated.
< 30% RPD for 90% of analytes; Concentration must be >5x the MDL for MQO to apply.	No exceedences noted.
	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 exceedences 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 exceedences 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 exceedences noted.
	Comments: One PAH CCV had a mean percent difference of 16.2. That CCV passed all individual percent difference criteria and all batch quality control samples passed indicating the instrument was in control.