14-0421 PAH/BIOMARKERs QA/QC Summary

Project:	ANIMIDA III
Parameters:	PAH and Biomarkers
Laboratory:	Battelle, Norwell, MA
Matrix:	Tissue
Data Set:	DP-14-0587
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	Tissue samples were homogenized with titanium blades and split for metals analysis at Sequim and FIT. The tissue samples were extracted following a modified EPA Method 3510C. Samples were prepared for analysis by weighing approximately 20 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 tissuemizer. 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 alkyated PAH analysis. The extracts were concentrated and spiked with IS for analysis.
Prep comments	The batch average dry weight is being applied to samples M5856, M5857, M5859,M5860, M5862, M5863, M5865, M5866, M5867, M5869, M5870, M5871, M5880, M5903 and M5904 as there was not enough material to perform separate dry weights. Another NSC for MS was added to batch because original NSC was made with ID61. 1 mL of IC44 used to make NSC.
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) is corrected based on surrogate recoveries. All data is reported on dry weight basis except the SRM (wet weight) and NSC and CO (oil weight).
Analysis comments	None.

Holding Times	Extraction Date(s)	Analysis Date(s)
	10/17, 29/2014 & 11/19, 20/2014	11/7-9, 19, 21-24/2014

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	Many exceedences noted.
	Comments: There were thirty-three exceedences for analytes detected in samples at less than five times the blank concentration. Reanalysis of the PB confirmed results. These were mostly Naphthalene, C1-Naphthalenes, and C2-Naphthalenes.

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%	Three exceedences noted.
	Comments: Surrogate recoveries for 5B(H)-Cholane were high in the CO and in samples M5856 and M5880. Reanalysis on another instrument confirmed the results. Prep records and integrations were verified. All other surrogate recoveries were acceptable.

Sample Duplicate (QADUP)	A QADUP was prepared with this analytical batch. The RPD of target analytes were calculated to measure data quality in terms of accuracy.
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	One exceedence noted.
	Comments: Benzo(a)anthracene was recovered low in the SRM. Results were confirmed by reanalysis on another instrument. Prep records and integrations were verified. Recoveries for this analyte were acceptable in all CCVs as well as the LCS for this batch. No

MQO to apply	further action was taken.
North Slope Crude (NSC)	A NSC Reference 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. The control oil also run in this batch has no associated target values and is not evaluated.
< 30% RPD for 90% of analytes	No exceedences 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 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 ≤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 ≤25%; Mean RSD ≤15%	No exceedences noted.
	Comments: CCVs Q9609, method MQ0378C, had a mean PD above 15%. Individual PDs were acceptable and no further action was taken.