14-0507 TPH/SHC QA/QC Summary

Project:	ANIMIDA III
Parameters:	TPH and SHC
Laboratory:	Battelle, Norwell, MA
Matrix:	Sediment
Data Set:	DP-14-0810
Analytical SOP:	5-202
Method Reference:	Modified EPA Method 8015C

	Receipt Date	Temp (°C)
Sample Custody	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, 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 alkyated PAH analysis. The extracts were concentrated and spiked with IS for analysis.
Prep comments	Sample M5921 was lost during pre-silica column cleanup. The sample will be re-extracted in batch 14-0508.
Analysis	TPH/SHC was measured by gas chromatography with flame ionization detection (GC/FID). An initial calibration consisting of target analytes was completed prior to analysis to demonstrate the linear range of analysis. Calibration verification was performed at the beginning and end of each 24 hour period (or 10 samples) in which samples were analyzed. Concentrations of TPH/SHC were calculated by the internal standard method. Normal alkanes were quantified using the average RF generated from the initial calibration. TPH concentrations were quantified using the average RF of nC9 through nC40.
Analysis comments	All data are reported as surrogate corrected versus dry wt. The NSC and CO are reported as not surrogate corrected versus oil weight.
Holding Times	Extraction Date(s)	Analysis Date(s)

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	10/16/2014 & 11/4, 18/2014	11/5-7/2014 and 11/18/2014

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.

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.
< 30% RPD for 90% of analytes	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 sample was re-run with similar 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/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.

Initial Calibration (ICAL)	The GC/FID is calibrated with a minimum 5 level curve for all compounds.
Individual RSD ≤25%; Mean RSD ≤20%	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 ≤25%; Mean RSD ≤20%	No exceedences noted.
	Comments: None.