14-0508 TPH/SHC QA/QC Summary

Project:	ANIMIDA III
Parameters:	TPH and SHC
Laboratory:	Battelle, Norwell, MA
Matrix:	Sediment
Data Set:	DP-14-0811
Analytical SOP:	5-202
Method Reference:	Modified EPA Method 8015C

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, 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	Several notes about specific samples and the silica columns. Please see the sample specific notes in the Prep section of the package.
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 injections) 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 is reported on dry weight basis except the NSC and CO (oil weight). All reported data (except NSC and CO) are corrected based on surrogate recoveries.

Holding Times	Extraction Date(s)	Analysis Date(s)
	10/27/2014 & 11/3, 5, 18/2014	11/7-9, 20/2014 and 11/19/2014

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%	Three exceedences noted.
	Comments: CD641LCS failed Nonane and Decane below the lower allowable limit. CD642LCS failed Nonane below the lower allowable limit. They were re-analyzed on a different instrument with similar results. No further actions 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.
< 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%	No exceedences noted.
	Comments: None.

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%	One exceedence noted.
	Comments: One analyte (Nonane) is "N" qualified in the MS sample. All analytes pass in the MSD. The RPD for the MS and MSD are within the acceptable range. No further corrective action taken.

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 ≤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.