

14-0420 TPH/SHC QA/QC Summary

Project:	ANIMIDA III
Parameters:	TPH and SHC
Laboratory:	Battelle, Norwell, MA
Matrix:	Tissue
Data Set:	DP-14-0584
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	<p>Tissue samples were homogenized with titanium blades and split for metals analysis at Sequim and FIT.</p> <p>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 tissue mixer. 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.</p>
Prep comments	<p>Sample M5883 was noted to contain water after the post column cleanup. Sodium sulfate was added and the prep continued with the rest of the batch. Also, the GC/MS fraction went dry. 250uL of hexane was added to the vial before re-combining for the FID dilutions.</p> <p>The batch average dry weight is being applied to sample M5901 as there was not enough material to perform a separate dry weight.</p>
Analysis	<p>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</p>

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	were quantified using the average RF of nC9 through nC40. All data is reported as surrogate corrected versus dry wt. The NSC and CO are reported as not surrogate corrected versus oil weight.	
Analysis comments	<p>Sample M5901 exhibited septum contamination during initial run, re-ran on Sequence SO0400 with the diluted samples. No septum contamination was noted in second run of sample indicating it was isolated to the original injection.</p> <p>n-Pentatriacontane concentrations were ME qualified in samples M5889, M5891, M5894, M5897, M5898, M5899, M5900 and M5901. Analyte concentrations were anomalous with surrounding analyte concentrations. In each case an inflection point was detected in the peak, a vertical integration was performed inside the peak to better represent the detected alkane. No further corrective action taken.</p> <p>n-Pentatriacontane concentrations were ME qualified in samples M5892, and M5895, M5896. Analyte concentrations were anomalous with surrounding analyte concentrations. In each case no inflection point was detected in the peak, the whole peak was integrated. No further corrective action taken.</p> <p>n-Nonatriacontane concentration was ME qualified in sample M5883. Analyte concentrations were anomalous with surrounding analyte concentrations. In this case an inflection point was detected in the peak, a vertical integration was performed inside the peak to better represent the detected alkane. No further corrective action taken.</p>	
Holding Times	Extraction Date(s)	Analysis Date(s)
	10/8,15/2014 & 11/20/2014	10/16-18, 20/2014 and 11/12-15,20/2014

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	<p>No exceedences noted.</p> <p>Comments: None.</p>
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%	<p>One exceedence noted.</p> <p>Comments: The LCS failed for Nonane below the MQO criteria. It was re-analyzed on a different instrument with similar results. No further actions taken.</p>
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	<p>No exceedences noted.</p> <p>Comments: None.</p>
Surrogate Recovery	Surrogate compounds were added prior to extraction. The surrogate

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Recovery of 40-120%	recoveries are calculated to measure extraction efficiency.
	No exceedences noted. Comments: None.
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%	One exceedence noted.
	Comments: The RPD of the diluted background and duplicate samples for Pristane is greater than 30%. All other analytes pass within acceptable criteria. No further corrective action taken.
Initial Calibration (ICAL)	The GC/FID is calibrated with a minimum 5 level curve for all compounds.
Individual RSD \leq 25%; Mean RSD \leq 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 \leq 25%; Mean RSD \leq 20%	No exceedences noted.
	Comments: None.