15-0347 TPH/SHC QA/QC Summary

Project:	ANIMIDA III
Parameters:	TPH and SHC
Laboratory:	Battelle, Norwell, MA
Matrix:	Tissue
Data Set:	DP-15-0311
Analytical SOP:	5-202
Method Reference:	Modified EPA Method 8015C

Sample Custody	Receipt Date	Temp (°C)
	8/11/2015	0.9, 1.2, 0.3

Corrective Actions	Sample L4815 was listed on the COC as QAH-122 with a collection time of 8:40 on 8/6/15. There was no jar that had matching collection information but there was a jar that had the correct station information that belongs to that sample. The ID on the jar was QAH-207 with a collection date of 8/6/15 @ 10:00am. Logged in as the COC states but I believe it should be the QAH-207.
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. Samples were prepared for analysis by weighing approximately 5-20 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	Maintenance work was being performed on the lab roof and somebody was smoking on the roof. The odor came through vents in the lab. Several samples had low sample volume and the sample weights had to be restricted. See the sample prep comments for the exact samples. Due to column bleed some of the F2 analytes were transferred into the F1 fractions. Since these analytes were confirmed by FID analysis to be present in the F1 splits the F1 fraction was recombined and cleaned up by GPC. After GPC cleanup the F1 fraction was blown down and recombined with the F2 fraction, blown down to a PIV of 1000uL and no RIS was added. The sample was then submitted for PAH analysis and surrogates were hand calculated based on the

	process described above.
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. All data is reported as surrogate corrected versus dry weight. The NSC and CO are reported as not surrogate corrected versus oil weight.
Analysis comments	None.

Holding Times	Extraction Date(s)	Analysis Date(s)
	8/27/2015 and 9/3/2015	9/5-6/2015 and 9/11/2015

Procedural Blank (PB)	Two PB samples were prepared with this analytical batch to ensure the sample extraction and analysis methods are free of contamination.
PB <5 X MDL	Eleven exceedances noted.
Samples must be >5x PB	Comments: The blank had some "J" qualified data. This led to some "B" qualified data (decane and hentriacontane).

Laboratory Control Spike (LCS)	Two LCS samples were prepared with this analytical batch. The percent recoveries of target analytes were calculated to measure accuracy.
Recovery of 70-130%	No exceedances noted.
Nonane: 50-130%	Comments: None.

North Slope Crude (NSC) and CO (Control Oil)	A NSC Reference Oil and Control 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 exceedances noted.
	Comments: None.

Standard Reference Material (SRM)	An SRM was prepared with this analytical batch.
% Difference <30% for analytes above 5XMDL	No exceedances noted.
	Comments: There were no certified values for the target analytes.

Surrogate Recovery	Surrogate compounds were added prior to extraction. The surrogate recoveries are calculated to measure extraction efficiency.
Recovery of 40-120%	No exceedances 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%	No exceedances 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 exceedances 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 exceedances 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 exceedances noted.
	Comments: None.