

15-0347 PAH/Biomarker QA/QC Summary

Project:	ANIMIDA III
Parameters:	PAH and Biomarker
Laboratory:	Battelle, Norwell, MA
Matrix:	Tissue
Data Set:	DP-15-0312
Analytical SOP:	5-157
Method Reference:	Modified EPA Method 8270D

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	<p>Tissue samples were homogenized with titanium blades and split for metals analysis at Sequim and FIT.</p> <p>Samples were prepared for analysis by weighing approximately 3-20 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 alkylated PAH analysis. The extracts were concentrated and spiked with IS for analysis.</p> <p>There was a bleed through of the F2 fraction into the F1 which resulted in observed low SIS recoveries. As a corrective action the F1 fraction was combined (TPH and BIO splits) and run through the GPC. These cleaned up F1 fractions were then combined with the F2 fraction and re-submitted for PAH analysis. Additional IS was not added.</p>
Prep comments	<p>Maintenance work was being performed on the lab roof and somebody was smoking on the roof. The odor came through vents in the lab.</p> <p>A brief hint of smoke(camp fire) entered the lab while submitting samples for analysis(9/4/2015)</p>

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	<p>While recombining the F1s, the vials for MS portion was dry for L4819, L4820 and L4821. Samples were reconstituted and continued through prep.</p> <p>Several samples had low sample volume and the sample weights had to be restricted. See the sample prep comments for the exact samples.</p>	
Analysis	<p>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 and CO) is corrected based on surrogate recoveries. All data is reported on dry weight basis except the NSC and CO (oil weight).</p>	
Analysis comments	<p>The PAH portion needed additional processing (as explained in the Prep Section) which led to the internal standard amounts being manually entered for quantitation. The Excel spreadsheet is included in the MiscDoc section. The additional processing also led to numerous expected IS area failures of the PAH portion.</p>	
Holding Times	Extraction Date(s)	Analysis Date(s)
	8/27/2015 and 9/3/2015	9/4-6, 11, 12, 28-30/2015

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	Twenty Six exceedances noted.
Samples must be >5x PB	Comments: The blanks had some "J" qualified data. This led to some "B" qualified data (Naphthalene and Phenanthrene).

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.
	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.

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North Slope Crude (NSC) and 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.
Surrogate Recovery	Surrogate compounds were added prior to extraction. The surrogate recoveries are calculated to measure extraction efficiency.
Recovery of 40-120%	One exceedance noted. Comments: One surrogate was low in the duplicate analysis. The background sample passed all SIS recoveries and the PAH were similar in both indicating with was isolated to the one surrogate.
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/MS is calibrated with a minimum 5 level curve for all compounds.
Individual RSD \leq 25%; Mean RSD \leq 15	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 \leq 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 \leq 25%; Mean RSD \leq 15%	No exceedances noted. Comments: None.