OU2 Soil Invertebrate Sampling Work Plan

Kerr-McGee Chemical Corp – Navassa Superfund Site

Navassa, North Carolina

EPA ID #NCD980557805

Prepared for:

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Acronyms and Abbreviations

°C	degrees Celsius
bgs	below ground surface
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
cm	centimeter
cm^2	square centimeter
DNAPL	dense non-aqueous phase liquid
DQO	data quality objective
EarthCon	EarthCon Consultants of North Carolina, P.C.
ERA	ecological risk assessment
GPS	global positioning system
HMW	high molecular weight
LMW	low molecular weight
m^2	meter squared
mg/kg	milligrams per kilogram
OU1	Operable Unit 1
OU2	Operable Unit 2
PAH	polycyclic aromatic hydrocarbons
PPE	personal protective equipment
PVC	polyvinyl chloride
Ramboll	Ramboll US Corporation
RI	remedial investigation
SESD	Science and Ecosystem Support Division
SOP	standard operating procedure
SRI	Supplemental Remedial Investigation
SVOC	semi-volatile organic compound
TOC	total organic carbon
USEPA	U.S. Environmental Protection Agency

1.0 INTRODUCTION

This 2020 Operable Unit 2 (OU2) Soil Invertebrate Sampling Work Plan presents the technical approach for the collection of additional Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) data to support a remedial alternative evaluation at the Kerr-McGee Chemical Corp – Navassa Superfund Site [U.S. Environmental Protection Agency (USEPA) ID# NCD980557805], referred to herein as the Site, located in Navassa, North Carolina (Figure 1-1). Additional data includes the collection of terrestrial invertebrates and co-located soil samples for the calculation of a site-specific uptake factor for use in a refined OU2 ecological risk assessment (ERA). This Work Plan is being submitted by EarthCon Consultants of North Carolina, P.C. (EarthCon) and Ramboll US Corporation (Ramboll) on behalf of Greenfield Environmental Multistate Trust LLC, not individually but solely in its representative capacity as Trustee of the Multistate Environmental Response Trust (the Multistate Trust).

The Site operated as a creosote-based wood treating facility from 1936 to 1974. A Site plan showing the property boundary, Process Area, Wood Storage Areas and other prominent Site features is provided as Figure 1-2. Previous investigations have indicated that soil, groundwater, surface water and sediment were impacted by historical Site operations. The USEPA conducted a screening level ecological risk assessment (ERA) for Operable Unit 1 (OU1) using polycyclic aromatic hydrocarbon (PAH) soil data collected from previous investigations.The objective of this 2020 investigation is to collect sufficient data from OU2 to calculate a site-specific uptake factor to use in a refined ERA to inform the remedial/risk management decision for OU2. OU2 includes surface soil in the Treated Wood Storage Area and the Untreated Wood Storage Area along the northwestern portion of the Site.

2.0 INVESTIGATION STATUS

Beginning in the 1980s, multiple parties performed pre-CERCLA environmental investigations at the Site and surrounding areas. Beginning in 2006, remedial investigation (RI) activities were performed, including dense non-aqueous phase liquid (DNAPL) investigations; soil, groundwater, marsh sediment, and surface water sampling activities; and vapor intrusion assessments. Based on results of the investigations to date, creosote-related semi-volatile organic compounds (SVOCs), including PAHs, were detected in soil samples from OU2. The USEPA used site-specific high molecular weight (HMW) PAH and low molecular weight (LMW) PAH soil concentrations in a screening level ERA to characterize risk to birds in OU1 (USEPA R4, 2019). The screening level ERA for OU1 identified potential risk to birds due to PAH concentrations; therefore, USEPA recommended sampling invertebrates (earthworms and insects as separate samples) in OU2 to reduce the uncertainty in the risk estimate.

3.0 DATA QUALITY OBJECTIVES

The Site-specific objectives presented in this section have been developed using the USEPA data quality objective (DQO) process, as presented in USEPA's *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA, 2006) and *Data Quality Objectives Process for Hazardous Waste Site Investigations* (USEPA, 2000). The approved DQO for the investigation is provided in Appendix A.

3.1 Objective 1 - Refinement of Potential Ecological Risks to Foraging Birds using Site-Specific Invertebrate Tissue Samples from OU2

DQO 1 was developed to collect sufficient terrestrial invertebrate and co-located soil data to calculate a site-specific uptake factor to refine the risk to birds due to PAHs in surficial soil at OU2. Appendix A describes the problem statement, the goals, decision inputs, and study boundary for this objective. As described in Appendix A, locations selected for sampling will include five HMW PAH concentration ranges (in milligram/kilogram [mg/kg]) within OU2, as identified from previous soil investigations at the Site. Three locations from each of the five HMW PAH ranges are selected for terrestrial invertebrate and soil sampling for a total of 15 locations. Two types of invertebrates (earthworm/soil-dwelling and surface-dwelling) and co-located composite soil samples will be collected at each location. Figure 3-1, Table 3-1, and Table 3-2 summarize the areas selected for sampling; however, sampling locations within each area will be decided in the field based on field conditions at the time of the sampling event.

The terrestrial invertebrate and soil data will be used to calculate a Site-specific uptake factor to refine the risk to birds due to PAHs at OU2. If there is an an unacceptable risk to foraging bird populatinos, then additional investigation and/or soil removal will be discussed with the Beneficiaries, as necessary. The decision rules and the decision error limits for this objective are described in Appendix A.

4.0 PROPOSED DATA COLLECTION ACTIVITIES

This section provides a summary of the proposed data collection activities associated with OU2. The data collection activities include collection of two types of invertebrates and co-located composite soil samples from 15 locations within OU2. A summary of the proposed samples for each objective is provided in Table 4-1.

4.1 Terrestrial Invertebrate and Surficial Soil Sampling for DQO 1

The objective of the terrestrial invertebrate and co-located surficial soil sampling is to provide a sufficient data set to calculate a Site-specific uptake factor for PAHs into invertebrates to refine the ecological risk to birds due to PAHs in surficial soils at OU2 and to support remedial/risk management decisions. To that end, terrestrial invertebrates and co-located soil samples will be collected from five HMW PAH concentration ranges (in mg/kg), as identified in Appendix A and summarized below:

Σ10 HMW PAH Concentration Range (mg/kg)	**# of Earthworm /Soil- Dwelling Invertebrate Samples**	**# of Surface-Dwelling Invertebrate Samples**
Σ10 HMW PAHs < 1 mg/kg	3	3
1 < Σ10 HMW PAHs < 10 mg/kg	3	3
10 < Σ10 HMW PAHs < 50 mg/kg	3	3
50 < Σ10 HMW PAHs < 100 mg/kg	3	3
Σ10 HMW PAHs > 100 mg/kg	3	3

Previous soil data was used to divide the Site into Thiessen polygons based on HMW PAH data (Figure 3-1). Terrestrial invertebrates will be collected from three polygons (or locations) within each HMW PAH category, and grouped into two categories (soil-dwelling/earthworms and surface-dwelling) for a total of 15 samples per invertebrate group. The minimum tissue mass needed for analysis to achieve the lowest detection limit possible was calculated to be 10 grams (minimum) of invertebrate tissue per sample as described in Attachment A of Appendix A, " Although 10 grams is the minimum amount necessary for analysis, Ramboll will attempt to collect more than 10 grams of tissue per sample. Because multiple organisms will be required to achieve the minimum mass requirements, each analytical sample will be a composite of many individual organisms. Proposed sample polygons are shown on Figure 3-1; however, specific sampling locations within each polygon will be selected in the field based on field conditions at the time of sampling.

As described in Appendix A, depurated earthworms will also be collected from 5 locations at OU2 to quantify the uncertainty due to biovailability of chemicals in soil in the gut of a soil-dwelling invertebrate versus the bioavailability of chemicals in the tissue of a soil-dwelling invertebrate. Each depurated sample will only be collected from areas where sufficient tissue mass is available to collect both the depurated and non-depurated samples.

Both non-depurated and depurated terrestrial invertebrate samples will be analyzed for alkylated and nonalkylated PAHs by modified USEPA Method EPA 8270-PAH-ALK-SIM and USEPA Method 8270-PAH-SIM (Table 4-1), respectively. Non-depurated invertebrate samples will also be analyzed for lipids and moisture content by SGS Axys Standard Operating Procedure (SOP) 020 and 015, respectively. The laboratory SOPs are provided in Appendix B.

A total of 15 surface soil samples will be collected from the same locations soil-dwelling invertebrates were collected. Each soil sample will be a composite of the soil encountered at the specific locations soil-dwelling invertebrates were collected. The size of the area for soil collection will vary between sample locations as it will be dependent on the size of the area needed to collect the invertebrates. The surface soil found in the areas of invertebrate collection will be collected at a depth of 0 to 6-inches below ground surface

(bgs) using a stainless steel spade and composited so that there is one surface soil sample for each location. The surface soil samples will be analyzed for alkylated and nonalkylated PAHs by USEPA Method EPA 8270D-SIM and USEPA Method 8270D (Table 4-1), respectively. Soil samples will also be analyzed for total organic carbon (TOC) and moisture content by USEPA Method 9060 and ASTM D2974, respectively.

5.0 FIELD AND LABORATORY PROCEDURES

Field and laboratory procedures are described in the following sections.

5.1 Field Procedures

Field activities will be conducted in general accordance with the most recent USEPA Region 4 Science and Ecosystem Support Division (SESD) operating procedures (USEPA, 2020), and other procedures described in the Supplemental Remedial Investigation (SRI) Work Plans dated September 2015 (CH2M Hill, 2015) and December 2016 (EarthCon, 2016).

5.1.1 GPS Navigation

The sampling team will navigate to a designated polygon sampling area using an Arrow Gold Real-Time Kinematic (RTK) Global Positioning System (GPS) unit with a real-time inch-level accuracy. The polygon shapefile will be uploaded to the unit prior to the field event so that the sampling team will know where they are in relation to the polygon boundaries while in the field. Coordinates of previous soil sampling locations used to generate the Thiessen polygons will also be uploaded to the GPS unit and are provided in Table 5-1. The sampling team will first navigate to the location of the previous soil sampling location to collect invertebrates. If an insufficient volume is collected for analysis at the previous soil sampling location, the sampling team will then select other areas within the polygon to sample for invertebrates biasing the locations towards the areas with the highest likelihood of having soil invertebrate collection success based on habitat and field observations. Efforts will be made to collect the samples within the proposed sampling polygon; however, if needed, the area can be extended onto an adjacent polygon provided that the PAH concentrations and soil characteristics are similar.

5.1.2 Terrestrial Invertebrate Sampling

Terrestrial invertebrate sampling locations within each sampling polygon will be selected in the field based on field conditions at the time of the sampling event. Terrestrial invertebrate samples may be collected using a variety of methods and equipment depending on the target taxa and habitats of interest. Two groups of terrestrial invertebrates (soil-dwelling invertebrates/earthworms and surface-dwelling invertebrates) will be collected per sampling polygon for a total of 15 samples per group. To facilitate a systematic and thorough approach to sampling each polygon, and if conditions allow, a 1-square meter (m^2) grid may be placed within the selected polygon at or as near as possible

to the previous soil sample location and at a location with the highest likelihood of having soil invertebrate collection success based on habitat and field observations. At each grid, surface-dwelling invertebrates will be collected first (with the exception of those surface-dwelling invertebrates collected by pit-trapping), as described in Section 5.1.2.1, followed by soil-dwelling invertebrates/earthworms, as described in Section 5.1.2.2. If sufficient mass (minimum of 10 grams) is available in that 1-m^2 grid, then one composite sample of soil-dwelling invertebrates and one composite sample of surface-dwelling invertebrates will be collected from the 1-m^2 grid area. If sufficient mass is not available in the 1-m^2 grid area, then the sampling area will be incrementally and systematically expanded within the remainder of the polygon, and, if necessary, into adjacent polygons until sufficient mass is reached for the invertebrate group. The expanded area will focus on habitats where soil invertebrates will likely be present and associated soil can be collected for the composite soil sample.

5.1.2.1 Surface-Dwelling Invertebrates

Surface-dwelling invertebrates will be collected within the 1-m^2 grid using a variety of methods such as hand sorting, pitfall trapping, and sweep netting; however, alternate methods may be employed by the sampling team based on field conditions at the time of the sampling event to achieve the required sampling volume. The surface-dwelling invertebrate tissue will be composited into one sample. If sufficient mass (minimum of 10 grams) is available in that 1-m^2 grid, then one composite sample of surface-dwelling invertebrates will be collected from the 1-m^2 grid area. If sufficient mass is not available for collection in the 1-m^2 grid area, then the sampling area will be incrementally and systematically expanded, as described above, until sufficient mass is reached. In addition to samples collected withn the 1-m^2 grid area, aboveground invertebrates may be collected in areas adjacent to the 1-m^2 grid area using sweep netting or other methods. The inclusion of aboveground invertebrates, such as banana spiders, with surface-dwelling invertebrates in each composite sample was agreed upon with the Beneficiaries in a meeting on February 13, 2020.

Once the sampling volume is achieved soil and detritus will be removed from each sample. Samples will be weighed and photographed. General observations of the number and types of organisms in the composite sample will be recorded. Each sample will be placed in either a labeled amber sampling jar placed within a labelled zip-top bag. The samples will be frozen or stored at 4 degrees Celsius (°C) for shipping. Special care will be taken to avoid crushing or compacting the samples.

<u>Hand Sorting</u>

All plant litter overlying the soil will be collected within the 1-m^2 grid. The collected litter will be placed on a mesh screen and gently shaken over a tray to dislodge any surface-dwelling invertebrates. Invertebrates will be placed in an intermediary sampling jar or zip-top bag until organisms can be composited and weighed, as described above. Depending

on the ambient temperature at the time of sampling, organisms may be temporarily stored in a cool location.

Pitfall Trapping

Pitfall traps may be used for sampling active arthropods that tend to be strictly surface dwellers, including many beetles and spiders. Using a stainless steel spade, a hole will be dug that is slightly deeper and wider than the container that will be used. The container will be placed in a hole so that the lid is flush with (or slightly below) the ground surface. The soil will then be firmly compacted around the container. If appropriate, a wooden or plastic plate or lid will be suspended approximately 2.5 centimeters (cm) above the trap to protect the trap from rain, debris, and other larger, unwanted organisms. The covers will be weighted with a stone and supported by 7.5 cm to 15 cm squared (cm^2) wooden or hollow aluminum rods, set at right angles around the trap mouth to form an "X." These support rods will touch the mouth of the trap for efficient capture of specimens. Support rods arranged in this fashion not only support the cover firmly, but also intercept animals moving near the trap and funnel them into the trap. The pit traps, if needed, will be installed after the hand sorting of the plant litter for any surface-dwelling invertebrates and the soil-sieving for soil-dwelling invertebrates.

The traps will be monitored 1 to 2 times daily (morning and evening). If invertebrates are captured, they will be removed from the trap, and placed in an intermediary sampling jar or zip-top bag until organisms can be composited and weighed, as described above. Depending on the ambient temperature at the time of sampling, organisms may be temporarily stored in a cool location.

Sweep Netting

Sweep netting is used to capture flying insects or insects that live on bushes, high grasses, or low trees. Insect groups readily captured by sweep netting include grasshoppers, butterflies, flies, bees, and wasps. Sweep nets come in two basic designs. Lighter butterfly nets are designed to be easily moved about in pursuit of an active single prey that is capable of effective evasive action. Heavier nets are used to sweep through vegetation, capturing all insects that are present. The choice of nets depends on the targeted group of insects and will be decided upon by the sampling team based on field conditions at the time of the sampling event and the invertebrates expected to be encountered based on the available habitat.

For either type of net, the net is swept quickly in an arc through the vegetation or to capture a targeted insect (e.g., a butterfly), and the netting is then flipped quickly over the mouth of the net so the insects cannot escape. The net is then maneuvered to place the insect in an intermediary container such as a jar or plastic bag (special care is taken with bees and wasps) until organisms can be composited and weighed, as described above. Depending on the ambient temperature at the time of sampling, organisms may be temporarily stored in a cool location. Sweep netting, if needed, will occur in areas near the 1-m^2 grid.

5.1.2.2 Soil-Dwelling Invertebrates/Earthworms

After the collection of surface-dwelling invertebrates from each 1-m^2 grid, a decontaminated stainless-steel spade/trowel or shovel will be used to excavate soil to a depth of 6 inches bgs. Excavated soil will be placed in a sieve over a decontaminated stainless steel tray or bowl. The soil will be gently broken apart and any soil-dwelling invertebrates encountered will be removed and placed in an intermediary sampling jar or zip-top bag until organisms can be composited and weighed. Depending on the ambient temperature at the time of sampling, organisms may be temporarily stored in a cool location. Once the sampling volume is achieved, organisms will be gently rinsed with deionized water to remove excess soil found on the exterior portions of the invertebrate and lightly blotted dry with paper towels. Samples will be weighed and photographed. General observations of the number and types of organisms in the composite sample will be recorded. Each sample will be placed in either a labeled amber sampling jar or wrapped in aluminum foil (with the nonreactive or dull side against the invertebrates) and placed within a labelled zip-top bag. The samples will be frozen or stored at 4 degrees Celsius (°C) for shipping. Special care will be taken to avoid crushing or compacting the samples.

If there is sufficient soil-dwelling invertebrate mass at a sampling polygon, then a subset of the soil-dwelling invertebrates will be randomly selected for depuration. For depuration, living organisms will be gently rinsed with deionized water, lightly blotted dry, and then collectively weighed. The organisms will then be placed in a sorting pan containing moistened filter paper (using deionized water) for 24 hours to allow ingested soil to be excreted. After 24 hours, the organisms will be weighed again before being placed in a labeled amber samping jar and placed within a labelled zip-top bag. The samples will be frozen or stored at 4°C for shipping. Special care will be taken to avoid crushing or compacting the samples.

5.1.3 Surficial Soil Sampling

Composite soil sampling will be conducted at the same locations as the soil-dwelling invertebrate sampling so that the samples are co-located. For every 1-m^2 area where soil-dwelling invertebrates are collected, the soil sieved from that 1-m^2 area will be placed in a stainless steel bowl (or similar container to hold the maximum volume of soil collected).

The size of the area for soil collection will be a minimum of 1-m^2 to correspond with the grid used for the soil-dwelling invertebrate collection but may vary between sample locations as it will be dependent on the size of the area needed to collect the invertebrates. Once the minimum mass for the soil-dwelling invertebrate sample is achieved, the sieved soil associated with the invertebrate sample will be homogenized using a stainless-steel spoon. A subsample of the homogenized soil will be collected and placed in a laboratory-supplied container for analysis. Remaining leftover sieved soil will be used to fill any voids in the ground left by excavation for sampling. The procedures for sieving and compositing of soil from the invertebrate collection areas was agreed upon with the Beneficiaries in a meeting on February 13, 2020.

5.2 Field Photographs and Field Observations

Photographs will be taken of each location during field activities associated with DQO 1. Photographs will also be taken of each sample so that the invertebrates comprising the sample will be photo-documented. Field observations as well as other information such as, but not limited to, sample ID; coordinates; weather; chemistry analysis; date; time; habitat, taxonomic group, sampling equipment used, depth of soil excavated, and any other miscellaneous notes will be recorded on field forms. In particular, the field team will record observations of creosote staining or potential creosote odors. An example of a field form that may be used is included in Appendix C.

5.3 Sample Shipment

The samples will be placed in labeled amber samping jars and placed within a labeled zip-top bag. The samples will be frozen or stored at 4°C for shipping. Special care will be taken to avoid crushing or compacting samples. Sample handling will occur under standard chain of custody protocols, as described in the SRI Work Plans dated September 2015 (CH2M Hill, 2015) and December 2016 (EarthCon, 2016).

5.4 Equipment Decontamination

Reusable sampling equipment will be decontaminated before and immediately after each use as described in the USEPA Region 4 SESD operating procedure, SESDPROC-205, as updated (USEPA, 2020). Solids and liquids generated by decontamination operations will be containerized in 55-gallon drums and disposed off-Site in accordance with the SRI Waste Management Plan dated September 2015 (CH2M Hill, 2015).

5.5 Terrestrial Invertebrate and Soil Sample Analysis

A total of 15 composite terrestrial invertebrate samples will be collected from OU2 at the polygons identified in Figure 3-1. Each composite terrestrial invertebrate sample will be divided into two groups: earthworm/soil dwelling-invertebrates and surface-dwelling invertebrates In addition, 5 composite samples will consist of depurated earthworms. The

invertebrates will be analyzed for alkylated and nonalkylated PAHs by EPA Method 8270-AH-SIM and EPA Method 8270-PAH-ALK-SIM. Lipids and moisture content will be measured by SGS Axys Standard Operating Procedure (SOP) 020 and 015, respectively. A summary of the terrestrial invertebrate samples and laboratory analyses is provided in Table 4-1. A summary of the laboratory detection limits is provided in Attachment C of Appendix A.

A total of 15 composite soil samples will be collected from OU2 at the polygons identified in Figure 3-1. The soil samples will be analyzed for alkylated and nonalkylated PAHs by EPA Method 8270D-SIM and EPA Method 8270D, respectively. Total organic carbon (TOC) and moisture content will be analyzed by EPA Method 9060 and ASTM Method D2974, respectively. A summary of the soil samples and laboratory analyses is provided in Table 4-1. A summary of the laboratory detection limits is provided in Attachment C of Appendix A.

5.6 Quality Control

Field quality control samples will be collected as follows for both invertebrate and soil samples:

- Field duplicates will be collected at a rate of 1 per 10 samples
- Matrix spike/matrix spike duplicates (MS/MSD) will be collected at a rate of 1 per 20 samples
- Field blanks will be collected at a rate of one per week
- Equipment blanks will be collected at a rate of one blank per reusable equipment (stainless steel trowels, hand augers, etc.) per media, per 20 samples collected; or one per week, whichever is more frequent

Laboratory analyses and reporting will be conducted in accordance with the SRI Quality Assurance Project Plan dated September 2015 (CH2M Hill, 2015). Table 4-1 provides a summary of the QC samples to be collected. According to the analytical laboratory, MS/MSD samples aren't typically prepared for tissue samples analyzed with an isotope dilution method. Level III (EPA Stage 2A) validation of 90 percent of the data and a Level IV validation (EPA Stage 4) of 10 percent of the laboratory data will be performed as described in the SRI Quality Assurance Plan (CH2M Hill, 2015).

5.7 Investigation-derived Waste Sampling and Management

The following waste streams may be generated during this investigation:

- Used personal protective equipment (PPE), trash, and sampling materials; and,
- Decontamination fluids.

Used PPE, trash, and sampling materials will be placed in a 55-gallon drum pending off-Site disposal. Investigation-derived waste will be managed in accordance with the SRI Waste Management Plan dated September 2015 (CH2M Hill, 2015).

6.0 REPORTING

A technical memorandum will be submitted to document the terrestrial invertebrate and soil sampling activities and will include photographs and field notes. The technical memorandum will include the results of the refined ERA for OU2. Results of the terrestrial invertebrate and soil samples will be incorporated in the risk evaluation and presented in the technical memoradum.

7.0 IMPLEMENTATION SCHEDULE

The following presents a summary of the approximate duration and constraints for implementation of field activities scheduled for July 2020:

Task Name	Estimated Duration	Notes
Terrrestrial Invertebrate and Co-location Soil Sampling	5 days	
Laboratory Analysis	15 days	Estimated from time of laboratory receipt of samples
Data Validation and Database Management	15 days	
Draft Technical Memorandum/Refined Food Web Model	25 days	Estimated date of draft submittal to the Beneficiaries is October 30, 2020 based on field activities beginning on 7/22/20

8.0 REFERENCES

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TABLES

Table 3-1. Polycyclic Aromatic Hydrocarbons (PAH) Concentrations in OU2
Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

		HMW PAHs (ND=0.5DL)	LMW PAHs (ND = 0.5DL)		Location Selected For Invertebrate Sampling
		(mg/kg)	(mg/kg)	(mg/kg)	Yes (range selected (a))
		Σ10 HMW PAHs	Σ8 LMW PAHs	Σ7 LMW PAHs	
OU2	SB-128	1.78	0.52	0.51	--
	SB-129	5.41	0.99	0.98	1. YES (1-10)
	SB-130	6.32	0.77	0.76	--
	SB-131	7.81	0.81	0.80	--
	SB-132	5.10	0.82	0.81	--
	SB-149	4.64	0.79	0.78	--
	SB-150	14.3	2.26	2.24	--
	SD021R	23.1	2.60	2.58	--
	SD021R-061419	38.7	4.02	4.00	--
	SS-110	161	6.93	6.91	1. YES (>100)
	SS-111	0.87	0.11	0.10	1. YES (<1)
	SS-112	6.53	1.14	1.13	--
	TB-08	95.4	10.6	10.6	1. YES (50-100)
	TB-09	17.1	2.73	2.71	--
	TB-10	0.57	0.09	0.08	2. YES (<1)
	TB-11	131	21.2	21.2	2. YES (>100)
	TB-12	273	68.0	67.9	--
	TB-13	1.45	0.40	0.38	--
	TB-14	16.0	2.36	2.34	1. Yes (11-50)
	TB-15	2.80	0.34	0.32	--
	TB-16	2017	661	659	--
	TB-17	95.9	11.3	11.3	--
	TB-18	100.7	9.36	9.29	--
	TB-19	7.37	1.24	1.23	--
	TB-20	5.61	0.76	0.74	--
	TB-21	13.6	0.75	0.73	--
	TB-22	8.63	1.48	1.46	--
	TWSB23	5.36	--	0.30	2. YES (1-10)
	SS-125	13.6	1.66	1.65	--
	SS-126	31.2	3.56	3.55	--
	TB-16A	59.5	7.46	7.40	--
	TB-16B	27.8	3.05	3.03	--
	TB-16C	133	16.3	16.2	--
	TB-16D	14.1	1.86	1.85	--
	TB-16F	33.2	4.75	4.70	2. Yes (11-50)
	TB-16G	1.99	0.28	0.27	--
	TB-16H	8.56	1.20	1.19	--
	TB-16E	5.15	0.84	0.83	--
	SB-133	48.7	2.56	2.47	--
	SB-134	1.85	0.64	0.63	--
	SB-135	7.69	1.63	1.62	--
	SB-136	6.63	1.41	1.40	--
	SB-151	2.89	0.47	0.46	--
	SB-152	4.37	0.39	0.38	--
	SB-153	9.04	1.44	1.42	--
	SS-113	96.3	14.0	14.0	--
	SS-114	108	21.0	20.97	--
	SS-115	19.7	2.61	2.59	--
	SS-116	36.9	4.76	4.72	--
	SS-117	131	21.2	21.1	3. YES (>100)
	SS-118	13.2	2.21	2.20	--
	SS-119	99.0	12.93	12.85	--
	SS-120	3.82	0.64	0.63	--
	SS-121	4.82	0.73	0.72	3. YES (1-10)
	SS-122	4.82	0.58	0.56	--

Table 3-1. Polycyclic Aromatic Hydrocarbons (PAH) Concentrations in OU2
Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

		HMW PAHs (ND=0.5DL)	LMW PAHs (ND = 0.5DL)		Location Selected For Invertebrate Sampling
		(mg/kg)	(mg/kg)	(mg/kg)	Yes (range selected (a))
		Σ10 HMW PAHs	Σ8 LMW PAHs	Σ7 LMW PAHs	
OU2	SS-123	0.72	0.12	0.11	**3. YES (<1)**
	SS-124	35.2	3.93	3.88	--
	TB-23	26.6	4.64	4.62	--
	TB-24	34.5	5.09	5.07	**3. Yes (11-50)**
	TB-25	0.80	0.14	0.13	--
	TB-26	2.11	0.29	0.28	--
	TWSB27	55.4	4.74	5.76	--
	RISB08	10.2			--
	RISB09	95.3			**2. YES (50-100)**
	RISB10	66.9			**3. YES (50-100)**
Summary	**Pre-excavation summary statistics for surficial soil:**				
	AVERAGE	**66.9**	**15.8**	**15.5**	--
	95th UCL (a)	**209**	**63.9**	**61.8**	--
	MAXIMUM	**2017**	**661.46**	**659.34**	--
	Post-excavation summary statistics for surficial soil:				
	AVERAGE	**31.0**	**4.67**	**4.59**	--
	95th UCL (a)	**60.7**	**10.8**	**10.5**	--
	MAXIMUM	**273**	**68.0**	**67.9**	--

Notes:

--	Not applicable
Σ10 HMW PAHs	Sum of 10 high molecular weight polycyclic aromatic hydrocarbons
Σ8 LMW PAHs	Sum of 8 low molecular weight polycyclic aromatic hydrocarbons
Σ7 LMW PAHs	Sum of 7 low molecular weight polycyclic aromatic hydrocarbons
DL	Detection limit (method)
ND	Not detected
mg/kg	Milligrams per kilogram
	Denotes descriptive statistics of grouped areas.
	Potential excavation area and so location was not selected for invertebrate sampling.

(a) ∑10 HMW PAH concentration range identified below

<1	∑10 HMW PAHs <1 mg/kg
1-10	1< ∑10 HMW PAHs <10 mg/kg
11-50	11< ∑10 HMW PAHs <50 mg/kg
51-100	51< ∑10 HMW PAHs <100 mg/kg
>100	∑10 HMW PAHs >100mg/kg

95th UCLs were calculated using the USEPA ProUCL software version 5.1. https://www.epa.gov/land-research/proucl-software (Accessed: November 14, 2019).

If there was at least one individual PAH that was detected, then the sum calculation was treated as a detected concentration.

All HMW and LMW sums calculated for each location had at least one detected PAH concentration; therefore, all sums were treated as detected concentrations.

(a) The ProUCL software selected a 95% H-statistic based UCL. Comments provided in the program said that the H-statistic UCL was not recommended for use but was provided for historical reasons only, and that the use of a nonparametric method was preferred for data not following a gamma distribution. This dataset did not appear to follow a gamma distribution based on the ProUCL results; therefore, the 95% Chebyshev nonparametric UCL was selected for all three PAH calculations.

Table 3-2. Invertebrate and Soil Sample Location Summary
Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

Location	∑10 HMW PAHs (ND=0.5DL) (mg/kg)	Location Selected For Invertebrate Sampling
SS-111	0.87	1. YES (<1)
TB-10	0.57	2. YES (<1)
SS-123	0.72	3. YES (<1)
TWSB23	5.36	1. YES (1-10)
SB-129	5.41	2. YES (1-10)
SS-121	4.82	3. YES (1-10)
TB-14	16.0	1. Yes (11-50)
TB-16F	33.2	2. Yes (11-50)
TB-24	34.5	3. Yes (11-50)
TB-08	95.4	1. YES (50-100)
RISB09	95.3	2. YES (50-100)
RISB10	66.9	3. YES (50-100)
SS-110	161	1. YES (>100)
TB-11	131	2. YES (>100)
SS-117	131	3. YES (>100)

Notes:

Σ10 HMW PAHs	Sum of 10 high molecular weight polycyclic aromatic hydrocarbons
DL	Detection limit (method)
ND	Not detected
mg/kg	Milligrams per kilogram

(a) ∑10 HMW PAH concentration range identified below

<1	∑10 HMW PAHs <1 mg/kg
1-10	1< ∑10 HMW PAHs <10 mg/kg
11-50	11< ∑10 HMW PAHs <50 mg/kg
51-100	51< ∑10 HMW PAHs <100 mg/kg
>100	∑10 HMW PAHs >100mg/kg

Table 4-1. Analytical Laboratories, Proposed Laboratory Methods, and Sample Summary
Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

Media	Analyte	Analytical Method	Lab-oratory	No. Samples	Type of Sample (a)	Field Dupes	Equip-ment Rinsate Blanks	MS	MSD	Total No. Samples	Min Sample Mass Per Sample (grams)	Sample Container	Pre-servative	Hold Times
Soil-Dwelling Invertebrates/Earthworms	Nonalkylated PAHs	EPA 8270-PAH-SIM	SGS Axys	15	Composite	1	--	1	1	18	10	Wide Mouth Amber Glass Jars	None, frozen	<1 year
	Alkylated PAHs	EPA 8270-PAH-ALK-SIM		15	Composite	1	--	1	1	18				
	Lipids	Axys SOP SLA-020		15	Composite	1	--	1	1	18				
	Moisture Content	Axys SOP SLA-015		15	Composite	1	--	1	1	18	1			
	Total Minimum Sample Mass Needed for Tissue Analysis per Sample:										**11**	**grams (b)**		
Depurated Earthworms	Nonalkylated PAHs	EPA 8270-PAH-SIM	SGS Axys	5	Composite	1	--	0	0	6	10	Wide Mouth Amber Glass Jars	None, frozen	<1 year
	Alkylated PAHs	EPA 8270-PAH-ALK-SIM		5	Composite	1	--	0	0	6				
	Lipids	Axys SOP SLA-020		5	Composite	1	--	0	0	6				
	Moisture Content	Axys SOP SLA-015		5	Composite	1	--	0	0	6	1			
	Total Minimum Sample Mass Needed for Tissue Analysis per Sample:										**11**	**grams (b)**		
Surface-Dwelling Invertebrates (e.g., beetles, ants, grubs)	Nonalkylated PAHs	EPA 8270-PAH-SIM	SGS Axys	15	Composite	1	--	0	0	16	10	Wide Mouth Amber Glass Jars	None, frozen	<1 year
	Alkylated PAHs	EPA 8270-PAH-ALK-SIM		15	Composite	1	--	0	0	16				
	Lipids	Axys SOP SLA-020		15	Composite	1	--	1	1	18				
	Moisture Content	Axys SOP SLA-015		15	Composite	1	--	1	1	18	1			
	Total Minimum Sample Mass Needed for Tissue Analysis per Sample:										**11**	**grams (b)**		
Soil	Nonalkylated PAHs	EPA 8270D	SGS Axys	15	Composite	1	1	1	--	18	30	4 ounce Wide Mouth Glass Jar	Cool to ≤ 6° Celsius	14 days extraction/40 days analysis (c)
	Alkylated PAHs	EPA 8270D-SIM		15	Composite	1	1	1	--	18	30			14 days extraction/40 days analysis (c)
	TOC (d)	EPA 9060		15	Composite	1	1	1	--	18	15			28 days
	Moisture Content	ASTM D2974		15	Composite	1	1	1	--	18	15			NA
	Total Minimum Sample Mass Needed for Soil Analysis per Sample:										**62**	**grams**		

Notes:

(a) Each composite sample will be comprised of terrestrial invertebrates collected from up to five individual 1-square meter (m^2) grids at each sample location. Composite soil samples will be comprised of the same areas that invertebrates were collected.

(b) Attachment A "Tissue Mass and Detection Limit Estimates" provides the calculations to support the collection of 10 grams of tissue for analysis.

(c) Holding time is 14 days from collection to extraction and 40 days from extraction to analysis.

ASTM ASTM International
Dupes Duplicate
EPA U.S. Environmental Protection Agency
MS Matrix spike
MSD Matrix spike duplicate
N/A Not applicable
No. Number
PAHs Polycyclic aromatic hydrocarbons
SIM Selected ion monitoring
SOP Standard operating procedure
TBD To be determined
TOC Total organic carbon

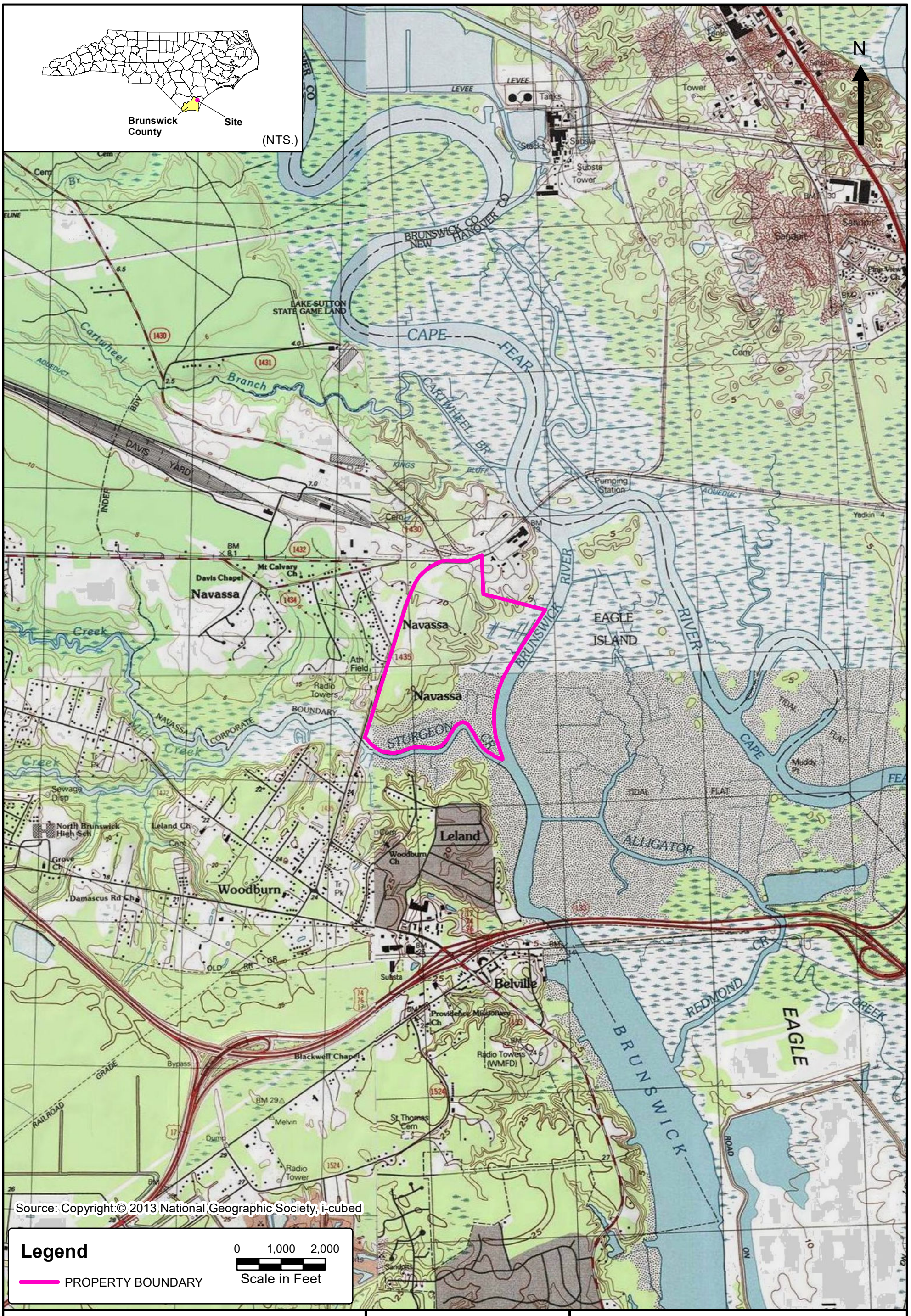
Table 5-1. GPS Coordinates of Terrestrial Invertebrate and Soil Sampling Polygons
Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

Sample Location	S10 HMW PAH (mg/kg) of Historical Soil Sample (a)	Historical Soil Sample Location		Polygon Acres	S10 HMW PAH Concentration Range (mg/kg)
		Northing	Easting		
RISB09	95.31	184077.06	2302782.60	0.29	50 < ∑10 HMW PAHs < 100 mg/kg
RISB10	66.91	184126.31	2303093.74	0.25	50 < ∑10 HMW PAHs < 100 mg/kg
SB-129	5.408	184613.10	2303201.89	0.52	1 < ∑10 HMW PAHs < 10 mg/kg
TWSB23	5.3565	184494.86	2302949.60	0.52	1 < ∑10 HMW PAHs < 10 mg/kg
TB-16F	33.16	184371.13	2303308.34	0.16	10 < ∑10 HMW PAHs < 50 mg/kg
TB-14	16.035	184478.90	2303375.09	0.22	10 < ∑10 HMW PAHs < 50 mg/kg
TB-08	95.35	184665.25	2302960.11	0.30	50 < ∑10 HMW PAHs < 100 mg/kg
TB-11	130.88	184607.51	2303380.11	0.43	∑10 HMW PAHs > 100 mg/kg
TB-10	0.565	184626.59	2303094.98	0.20	∑10 HMW PAHs < 1 mg/kg
TB-24	34.518	184125.25	2303141.71	0.09	10 < ∑10 HMW PAHs < 50 mg/kg
SS-117	131.35	184040.64	2302798.43	0.21	∑10 HMW PAHs > 100 mg/kg
SS-121	4.8162	183945.67	2302743.74	0.32	1 < ∑10 HMW PAHs < 10 mg/kg
SS-123	0.7241	183855.40	2303022.39	0.33	∑10 HMW PAHs < 1 mg/kg
SS-111	0.8671	184466.65	2303142.46	0.30	∑10 HMW PAHs < 1 mg/kg
SS-110	161.44	184738.96	2303099.12	0.31	∑10 HMW PAHs > 100 mg/kg

Notes:

S10 HMW PAHs	Sum of 10 high molecular weight polycyclic aromatic hydrocarbons
GPS	Global positioning system
mg/kg	Milligrams per kilogram
(a)	Concentration was used to create Thiessen polygons targeted for invertebrate and soil sampling.

FIGURES



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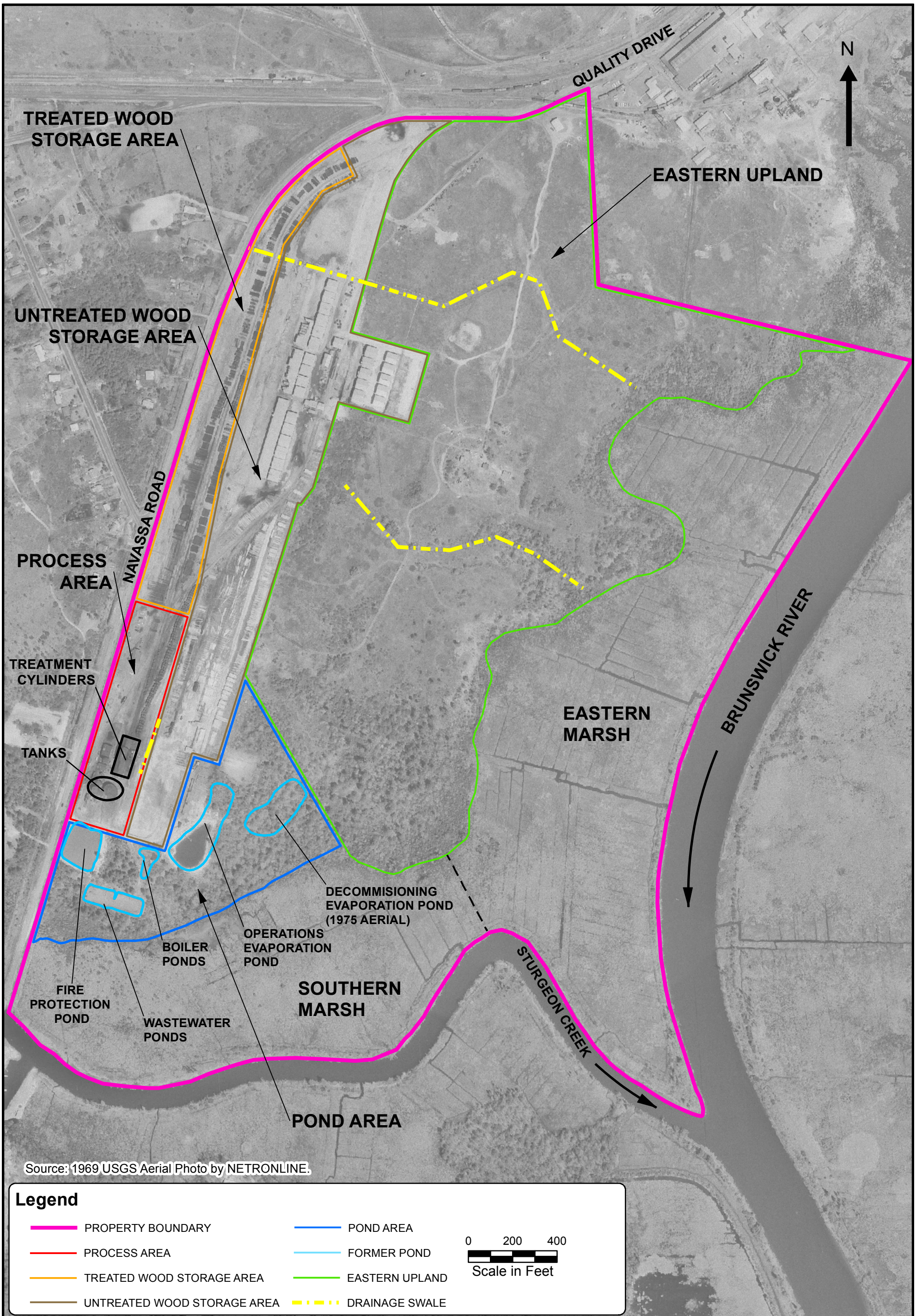
EARTHCON®

EARTHCON CONSULTANTS OF NORTH CAROLINA, P.C.

SITE LOCATION
OU2 Soil Invertebrate Sampling Work Plan
Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

DRAWN	CHECKED	DATE	FIGURE
HVP	CDN	APR 2020	1-1



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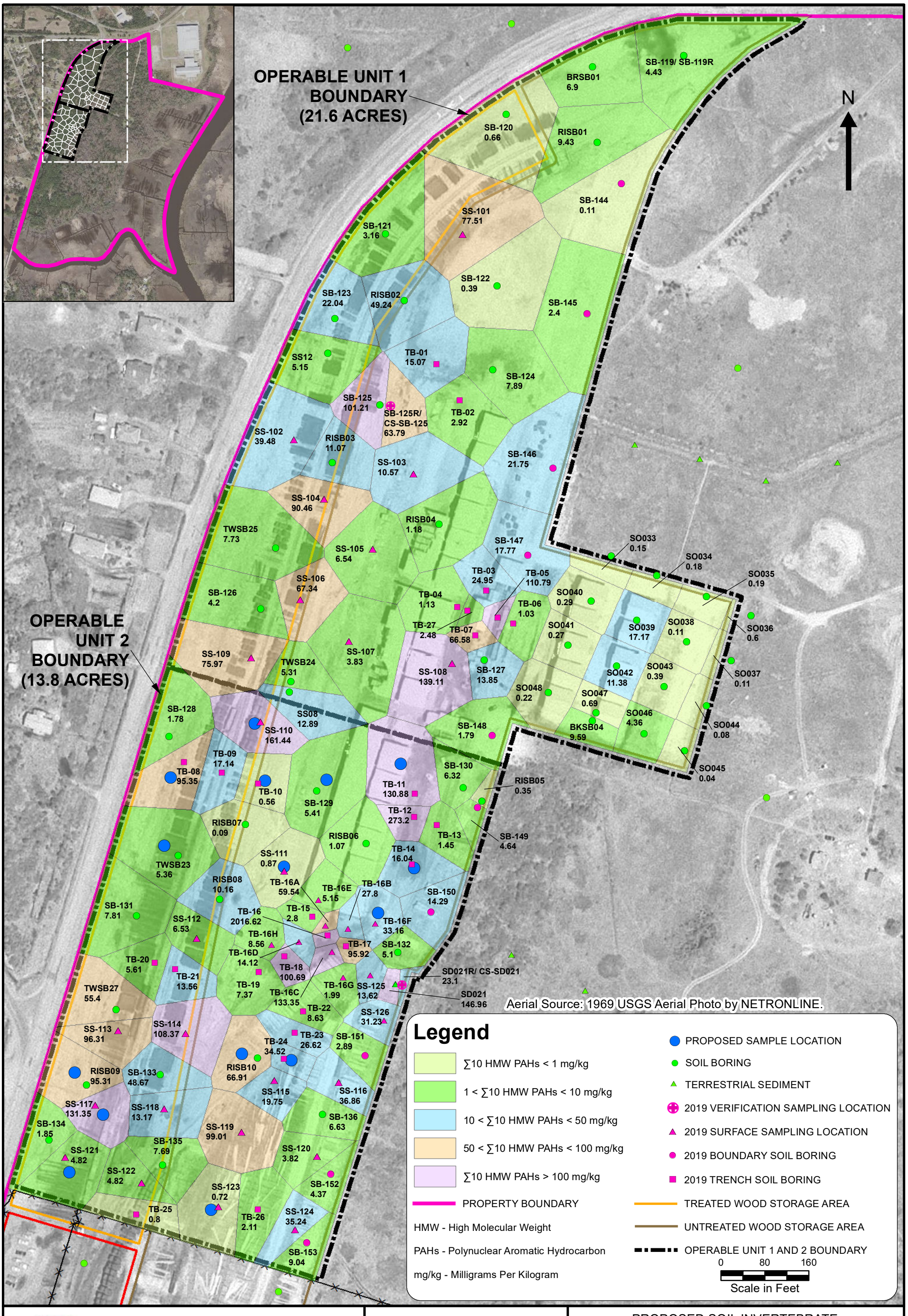
EARTHCON CONSULTANTS OF NORTH CAROLINA, P.C.

SITE PLAN

OU2 Soil Invertebrate Sampling Work Plan

Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

DRAWN	CHECKED	DATE	FIGURE
HVP	CDN	APR 2020	1-2



S:\Premier\Projects\Greenfield Environmental Multistate Trust\KMCC Navassa NC Superfund Site\CAD GIS Data\EARTHCON_GIS\MXDs\OU2 Soil Invertebrate Sampling Work Plan\Fig 3-1 Proposed Soil Invertebrate Sample Locations.mxd 5/1/2020 4:59:20 PM

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PROPOSED SOIL INVERTEBRATE SAMPLE LOCATIONS

OU2 Soil Invertebrate Sampling Work Plan

Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

DRAWN	CHECKED	DATE	FIGURE
HVP	CDN	APR 2020	3-1

APPENDICES

APPENDIX A

OU2 Soil Invertebrate Investigation Data Quality Objectives

2020 Operable Unit 2 Soil Invertebrate Investigation Data Quality Objectives

2020 OU2 Data Quality Objectives (DQOs)

- Objective 1: Refine the estimate of potential ecological risks to foraging birds by collecting site-specific invertebrate tissue and co-located soil samples from Operable Unit 2 (OU2) and estimating a site-specific ratio between invertebrate tissue polycyclic aromatic hydrocarbon (PAH) concentrations and soil PAH concentrations.

Steps Addressed for Each DQO

- Problem Statement
- Identify the Decisions
- Decision Inputs
- Study Boundary
- Error Limits
- Optimize Sampling Design

Tables

- Table 1A: Polycyclic Aromatic Hydrocarbons (PAH) Concentrations in OU2
- Table 1B: Soil Invertebrate and Soil Sampling Location Summary
- Table 2: Analytical Laboratories, Proposed Laboratory Methods, and Sample Summary

Figures

- Figure 1: OU2 Proposed Soil Invertebrate Sample Locations

Attachments

- Attachment A: Estimation of Tissue Mass to Achieve Required Detection Limits
- Attachment B: Summary of SGS Axys Method MLA-021 Rev 12 Ver 05
- Attachment C: Typical Detection Limits, Method Detection Limits and Low Calibration Limits for Parent PAHs and Alkylated PAHs by GC/MS

Objective 1
Refinement of Potential Ecological Risks to Foraging Birds using Site-Specific Invertebrate Tissue Samples from OU2

Problem Statement

Historical activities at the Site have resulted in residual polycyclic aromatic hydrocarbons (PAHs) in surface soils [0 to 12 inches below ground surface (bgs)] in OU2 (total approximate acreage of 13.8). The maximum high molecular weight (HMW) and low molecular weight (LMW) PAH concentrations[1] are 2,020 milligrams per kilogram (mg/kg) and 660 mg/kg, respectively (Table 1A). Excavation in the area of highest PAH concentrations in OU2 has been proposed. These areas are identified in Table 1A.

United States Environmental Protection Agency Region 4 (USEPA R4) has requested the collection of site-specific invertebrate tissue and co-located soil samples for PAH analysis. These data will be used to refine the screening level ecological risk assessment presented by USEPA R4 in the memo, "Semi-Screening Level Ecological Risk Assessment Calculations for Upland Areas 1A, 1B and 2 of the Kerr-McGee Chemical Company Site" (USEPA R4, 2019). The food chain model assumes that the PAH concentration in earthworms is 2.6 times the PAH concentration in soil (this is the default uptake factor as presented in USEPA, 2007). The food chain model also assumes that the insect portion of the foraging bird diet is 100% earthworms. The 2019 memo recommended sampling invertebrates (earthworms and insects as separate samples) to reduce the uncertainty in the risk estimate. The USEPA R4 intends to use the data collected under this objective to develop a soil cleanup level that would be protective of foraging birds using a site-specific uptake factor. This additional data may also be used to evaluate bird diets that contain both soil-dwelling and surface-dwelling insects.

Identify the Decisions

The following primary principal study questions have been identified:

(1) Do PAH concentrations in soil and terrestrial invertebrate tissue (if present) in OU2 pose an unacceptable risk to foraging birds in an industrial or commercial land use scenario?

(2) If there is an ecological risk to foraging bird populations, does it require additional investigation and/or soil removal?

To answer these questions, the following tasks have been identified:

(1) Refine the estimate of ecological risks to foraging birds in the food chain model by using site-specific uptake factors derived from the collection and analysis of soil and invertebrates at OU2.
   a. Collect and divide terrestrial invertebrates into two categories (soil-dwelling and surface-dwelling) for alkylated and nonalkylated PAH tissue analysis.
   b. Collect composite soil samples from the same locations as the invertebrate samples such that each invertebrate sample has a co-located soil sample.

(2) Estimate a site-specific uptake factor for both soil-dwelling and surface-dwelling invertebrates based on co-located tissue and soil PAH concentrations to calculate risk to foraging birds after excavation using the food chain model developed by the USEPA R4 (USEPA R4, 2019).

Decision Inputs

Type of information needed:

The primary inputs needed to support the decision-making process are:

- PAH presence and concentrations in invertebrate tissue samples
- PAH presence and concentrations in surface soil samples

Analytical results used in the decision-making process will be generated by SGS North America Inc.-EHS Laboratory in Orlando, Florida, for soil samples, and from Pace Analytical Services (Pace), in Green Bay, Wisconsin (or similar laboratory) for soil invertebrate tissue (Table 2).

Results used in the decision-making process will come from the following:

[1] The LMW PAH concentration reported here is for the sum of 7 LMW PAHs.

- Laboratory analysis of alkylated and nonalkylated PAHs (Σ34 PAHs), moisture content, and total organic carbon (TOC) in surface soil by the analytical methods provided in Table 2;
- Laboratory analysis of alkylated and nonalkylated PAHs (Σ34 PAHs), lipid content, and moisture content in terrestrial invertebrate tissue, which will be grouped into the following categories: soil-dwelling invertebrates (e.g., earthworms and grubs) and surface-dwelling invertebrates (e.g., crickets, beetles) by the analytical methods provided in Table 2; and
- Calculation of summary statistics including 95th upper confidence limit (UCL) for alkylated and nonalkylated HMW and LMW PAHs for surface soil and invertebrate tissue for use in an ecological risk assessment (ERA) for foraging birds (modeled after the ERA conducted by the USEPA R4 for upland Areas 1A, 1B, and 2 of OU1 at the Site as presented in USEPA R4, 2019).

Identification of sampling and analysis methods:

(1) Collection of terrestrial invertebrates

a. Up to 15 composite samples of 2 types of terrestrial invertebrates (earthworm/soil-dwelling and surface-dwelling) will be collected from five HMW PAH concentration ranges (in mg/kg) within OU2, as follows and as summarized on Tables 1A and 1B:

Σ10 HMW PAH Concentration Range (mg/kg)	**# of Earthworm /Soil-Dwelling Invertebrate Samples**	**# of Surface-Dwelling Invertebrate Samples**
Σ10 HMW PAHs < 1 m g/kg	3	3
1 < Σ10 HMW PAHs < 10 m g/kg	3	3
10 < Σ10 HMW PAHs < 50 m g/kg	3	3
50 < Σ10 HMW PAHs < 100 m g/kg	3	3
Σ10 HMW PAHs > 100 m g/kg	3	3

b. Sampling locations will be selected in the field based on field conditions at the time of the sampling event. At least three composite samples of each type of invertebrate per HMW PAH concentration range will be collected. Figure 1 depicts the different PAH concentrations as polygons and shows the potential sample locations. Previous soil sample results were used to estimate the PAH concentration for the polygons selected (Table 1A and Table 1B). Sampling techniques will depend on the conditions in the field but could include pit traps, soil grabs, soil sieving, and "ground foraging."

c. The target collection area will be a 1-m^2 grid placed within the selected polygon at a location with the highest likelihood of having soil invertebrate collection success based on habitat and field observations. If sufficient mass is available in that 1-m^2 grid, one composite sample of soil-dwelling invertebrates and one composite sample of surface-dwelling invertebrates will be collected from the 1-m^2 grid area. Soil-dwelling invertebrates can include any invertebrates found in the soil column while sieving such as earthworms and grubs. Surface-dwelling invertebrates can include any invertebrate that lives on the surface of the soil such as crickets, grasshoppers, and spiders.

d. Ramboll has coordinated with SGS Axys, the analytical laboratory analyzing the invertebrate tissue, and has calculated that the minimum amount of tissue needed per sample is 10 grams per sample per PAH concentration range to achieve detection limits of 0.0001 and 0.0002 mg/kg. Attachment A provides the minimum sample mass calculations verifying that 10 grams of tissue will be sufficient for SGS Axys for use in their tissue analysis. In addition, Attachment A takes into account various scenarios (e.g., low PAHs, dilution factors, and low sample mass) in the calculations to show that the lab can still achieve the low detection limits requested by the EPA. SGS Axys uses Method MLA-021 for their tissue analysis, which is provided in Attachment B. A table showing the analyte detection limits provided by SGS Axys using this method is provided as Attachment C.

e. If sufficient mass is not available for collection in the 1-m^2 grid area, then the sampling area will be incrementally and systematically expanded within the polygon until either sufficient mass is reached or the entire polygon has been sampled. The expanded area will focus on habitats where soil invertebrates will likely be present.

f. Efforts will be made to collect the sample within the proposed sampling polygon; however, if needed, the 1-m^2 grid area can be extended onto an adjacent polygon for sampling to reach sufficient mass provided that the PAH concentrations and soil characteristics are similar between the two polygons. At no time will the two different groups of invertebrates (surface-dwelling and soil-dwelling) collected at OU2 be combined.

g. Samples will be weighed in the field during collection to verify that the minimum mass needed for analysis is achieved. Because multiple organisms will be required to achieve the minimum mass requirements, each analytical sample will be a composite of many individual organisms; however, photos will be taken of each sample so that the invertebrates within a sample can be identified. Given the unpredictable nature of biota tissue collection, the exact compositing scheme will be determined by the onsite field personnel based on the objective of obtaining representative samples of potential prey for foraging birds and maximizing the numbers of samples for PAH analysis.

h. Invertebrates samples will not be depurated prior to analysis (i.e. the tissues collected will not include a step where the soil from the gut content is excluded in the tissue residue analysis). The use of non-depurated soil invertebrate tissue data reflects wildlife exposure in the food web, as birds and mammals that consume invertebrates in the natural environment eat the soil invertebrates as they are found (i.e., non-depurated).

(2) Collection of composite soil samples from the same locations as the invertebrate samples

a. Composite surface soil samples will be collected from the same 1-m^2 areas from which the invertebrate samples were collected so that the soil and tissue samples will be co-located. For each invertebrate sample location, the soil from the area sampled for invertebrates will be sieved and composited so that a single sample is collected that will be representative of the soil that invertebrates at that location will encounter. The size of the area for soil collection will be a minimum of 1-m^2 to correspond with the grid used for the invertebrate collection but may vary between sample locations as it will be dependent on the size of the area needed to collect the invertebrates.
b. The sieving and compositing of soil from the area in which the invertebrates are collected was agreed upon with the Beneficiaries in a meeting on February 13, 2020.

(3) Characterization of risk to terrestrial birds at OU2 using site-specific uptake factor

a. USEPA ProUCL Version 5.0 software will be used to calculate the 95% UCLs for the HMW and LMW PAH concentrations in surface soil and tissue samples.
b. The 95% UCLs for HMW and LMW alkylated and nonalkylated PAH concentrations will be screened against relevant USEPA R4 (2018) soil screening criteria.
c. Site-specific invertebrate uptake factors will be calculated for each group of invertebrates using co-located soil and non-depurated invertebrate samples.
d. To reduce uncertainty in the assessment of potential ecological risks to foraging birds, HMW and LMW PAH 95th UCLs will be used in an ERA model for foraging birds, which will be similar to the model created for Areas 1A, 1B, and 2 in OU1 (USEPA R4, 2019).

Uncertainty Analysis:

Chemicals in the soil from the gut of soil invertebrates are often less bioavailable than chemicals in the tissues of the soil invertebrates (Stafford and McGrath, 1986; Beyer and Stafford, 1993). This is particularly the case for earthworms, which can contain 20 to 30 percent of soil in their gut (Stafford and McGrath, 1986; Beyer and Stafford, 1993). To quantify this uncertainty, additional terrestrial invertebrate tissue samples will be collected for depurated analysis at OU2 where non-depurated tissues are collected, as follows:

a. This only applies to earthworm and potentially grubs (i.e., this does not apply to the surface invertebrates).

b. Each non-depurated sample will only be collected from areas where sufficient tissue mass is available to collect the non-depurated sample.
c. Each non-depurated sample will be a similar composition of organisms as the non-depurated sample at any given location.
d. The samples will be collected at the same time and in the same manner as the non-depurated samples except that these samples will be allowed to depurate for 24 hours prior to shipment to the laboratory.
e. To the greatest extent possible, these samples will be co-located with a non-depurated tissue and surface soil sample.
f. Depurated samples will be collected from a subset of sample locations, with effort to collect at least one depurated sample from each of the sample concentration ranges already discussed.

Lab performance:
The environmental data will be reported to the analyte's laboratory-specific method detection limit (MDL); i.e., positive results below the reporting limit (RL), but greater than the MDL, will be reported by the laboratory and flagged as estimated (J). MDLs will be adjusted on a sample-by-sample basis, as necessary, based on dilutions, sample volume, and percent moisture. Ramboll will coordinate with the laboratory and use the information provided by the laboratory to calculate the minimum tissue volume needed for analysis of each proposed sample location using previous soil data (Table1A).

Study Boundary
The media of interest is surficial soil and invertebrates which may be encountered by foraging birds. The study boundaries include the spatial boundaries of OU2, a sample depth of 0 to 6 inches bgs, and temporal boundaries such as field investigation dates and turnaround times on analytical results. The sample locations selected will be representative of a range of soil PAH concentrations such that OU2 is effectively characterized. Proposed sample locations are shown on Figure 1.

The sampling depth interval proposed (0 to 6 inches bgs) is the depth interval likely to be encountered by foraging birds probing the soil with their beaks for food. In addition, the 0 to 6-inch depth interval is also the depth interval of highest biological activity (USEPA, 2015). The two bird species proposed by the USEPA R4 for use in the upland ERA to represent foraging birds at the site are the American woodcock and the American robin.

The study boundary for this investigation is shown on Figure 1.

Field investigation activities will be scheduled after approval of this DQO table and the completion of a Field Sampling Plan. Field investigation activities are expected to be completed within one week. A 21-day turnaround time is anticipated for PAH analytical results from the laboratories for both soil and invertebrate tissue samples. Another 10 days is anticipated for validation of laboratory analytical results. One week is anticipated for calculation of HMW and LMW PAHs for use in the ERA, and two weeks is anticipated for the preparation of a draft ERA report for OU2.

Decision Rule
If the actual PAH concentration in soil-dwelling invertebrates, such as earthworms. is less than 2.6 times the PAH concentration in soil, then the uptake factor used in the food chain model is overconservative.

If the actual PAH concentration in surface-dwelling insects, such as crickets and grasshoppers, is less than the concentration in soil-dwelling invertebrates, then the assumption of a 100% earthworm diet is overconservative for birds that also consume surface-dwelling insects.

A hazard quotient for foraging birds in the sample areas will be calculated using the actual soil and invertebrate PAH concentrations in the food chain model.

The EPA will develop a site-specific uptake factor for both soil-dwelling and surface-dwelling invertebrates. This site-specific uptake factor may be used to determine a risk-based ecological cleanup level based on soil PAH concentration.

Error Limits
This sample effort is designed to sample in a study area that has elevated PAH concentrations based on previous investigations (Table 1A). However, random and systematic errors could be introduced during sample collection, sampling handling and storage, sample analysis and data reduction. The QC measures set forth in the approved Quality Assurance Project Plan (CH2M Hill 2015) and the specific analytical methods will serve to reduce these errors. QC samples will be used to monitor the accuracy and precision of the sampling activity as well as the analytical process.

Optimize Sampling Design
The data collection activities will focus on identifying PAH concentrations in soil and tissue samples from OU2. Details on the sampling design will be provided in the Field Sampling Plan.

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TABLES

Table 1A. Summary of HMW and LMW PAH Calculations for Upland Area 2
Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

		HMW PAHs (ND=0.5DL)	LMW PAHs (ND = 0.5DL)		Location Selected For Invertebrate Sampling
		(mg/kg)	(mg/kg)	(mg/kg)	Yes (range selected (a))
		Σ10 HMW PAHs	Σ8 LMW PAHs	Σ7 LMW PAHs	
OU2	SB-128	1.78	0.52	0.51	--
	SB-129	5.41	0.99	0.98	1. YES (1-10)
	SB-130	6.32	0.77	0.76	--
	SB-131	7.81	0.81	0.80	--
	SB-132	5.10	0.82	0.81	--
	SB-149	4.64	0.79	0.78	--
	SB-150	14.3	2.26	2.24	--
	SD021R	23.1	2.60	2.58	--
	SD021R-061419	38.7	4.02	4.00	--
	SS-110	161	6.93	6.91	1. YES (>100)
	SS-111	0.87	0.11	0.10	1. YES (<1)
	SS-112	6.53	1.14	1.13	--
	TB-08	95.4	10.6	10.6	1. YES (50-100)
	TB-09	17.1	2.73	2.71	--
	TB-10	0.57	0.09	0.08	2. YES (<1)
	TB-11	131	21.2	21.2	2. YES (>100)
	TB-12	273	68.0	67.9	--
	TB-13	1.45	0.40	0.38	--
	TB-14	16.0	2.36	2.34	1. Yes (11-50)
	TB-15	2.80	0.34	0.32	--
	TB-16	2017	661	659	--
	TB-17	95.9	11.3	11.3	--
	TB-18	100.7	9.36	9.29	--
	TB-19	7.37	1.24	1.23	--
	TB-20	5.61	0.76	0.74	--
	TB-21	13.6	0.75	0.73	--
	TB-22	8.63	1.48	1.46	--
	TWSB23	5.36	--	0.30	2. YES (1-10)
	SS-125	13.6	1.66	1.65	--
	SS-126	31.2	3.56	3.55	--
	TB-16A	59.5	7.46	7.40	--
	TB-16B	27.8	3.05	3.03	--
	TB-16C	133	16.3	16.2	--
	TB-16D	14.1	1.86	1.85	--
	TB-16F	33.2	4.75	4.70	2. Yes (11-50)
	TB-16G	1.99	0.28	0.27	--
	TB-16H	8.56	1.20	1.19	--
	TB-16E	5.15	0.84	0.83	--
	SB-133	48.7	2.56	2.47	--
	SB-134	1.85	0.64	0.63	--
	SB-135	7.69	1.63	1.62	--
	SB-136	6.63	1.41	1.40	--
	SB-151	2.89	0.47	0.46	--
	SB-152	4.37	0.39	0.38	--
	SB-153	9.04	1.44	1.42	--
	SS-113	96.3	14.0	14.0	--
	SS-114	108	21.0	20.97	--
	SS-115	19.7	2.61	2.59	--
	SS-116	36.9	4.76	4.72	--
	SS-117	131	21.2	21.1	3. YES (>100)
	SS-118	13.2	2.21	2.20	--
	SS-119	99.0	12.93	12.85	--
	SS-120	3.82	0.64	0.63	--
	SS-121	4.82	0.73	0.72	3. YES (1-10)
	SS-122	4.82	0.58	0.56	--

Table 1A. Summary of HMW and LMW PAH Calculations for Upland Area 2
Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

		HMW PAHs (ND=0.5DL)	LMW PAHs (ND = 0.5DL)		Location Selected For Invertebrate Sampling
		(mg/kg)	(mg/kg)	(mg/kg)	Yes (range selected (a))
		Σ10 HMW PAHs	Σ8 LMW PAHs	Σ7 LMW PAHs	
OU2	SS-123	0.72	0.12	0.11	**3. YES (<1)**
	SS-124	35.2	3.93	3.88	--
	TB-23	26.6	4.64	4.62	--
	TB-24	34.5	5.09	5.07	**3. Yes (11-50)**
	TB-25	0.80	0.14	0.13	--
	TB-26	2.11	0.29	0.28	--
	TWSB27	55.4	4.74	5.76	--
	RISB08	10.2			--
	RISB09	95.3			**2. YES (50-100)**
	RISB10	66.9			**3. YES (50-100)**
Summary	**Pre-excavation summary statistics for surficial soil:**				
	AVERAGE	**66.9**	**15.8**	**15.5**	--
	95th UCL (a)	**209**	**63.9**	**61.8**	--
	MAXIMUM	**2017**	**661.46**	**659.34**	--
	Post-excavation summary statistics for surficial soil:				
	AVERAGE	**31.0**	**4.67**	**4.59**	--
	95th UCL (a)	**60.7**	**10.8**	**10.5**	--
	MAXIMUM	**273**	**68.0**	**67.9**	--

Notes:

--	Not applicable
Σ10 HMW PAHs	Sum of 10 high molecular weight polycyclic aromatic hydrocarbons
Σ8 LMW PAHs	Sum of 8 low molecular weight polycyclic aromatic hydrocarbons
Σ7 LMW PAHs	Sum of 7 low molecular weight polycyclic aromatic hydrocarbons
DL	Detection limit (method)
ND	Not detected
mg/kg	Milligrams per kilogram
	Denotes descriptive statistics of grouped areas.
	Potential excavation area and so location was not selected for invertebrate sampling.

(a) ∑10 HMW PAH concentration range identified below

<1	∑10 HMW PAHs <1 mg/kg
1-10	1< ∑10 HMW PAHs <10 mg/kg
11-50	11< ∑10 HMW PAHs <50 mg/kg
51-100	51< ∑10 HMW PAHs <100 mg/kg
>100	∑10 HMW PAHs >100mg/kg

95th UCLs were calculated using the USEPA ProUCL software version 5.1. https://www.epa.gov/land-research/proucl-software (Accessed: November 14, 2019).

If there was at least one individual PAH that was detected, then the sum calculation was treated as a detected concentration.

All HMW and LMW sums calculated for each location had at least one detected PAH concentration; therefore, all sums were treated as detected concentrations.

(a) The ProUCL software selected a 95% H-statistic based UCL. Comments provided in the program said that the H-statistic UCL was not recommended for use but was provided for historical reasons only, and that the use of a nonparametric method was preferred for data not following a gamma distribution. This dataset did not appear to follow a gamma distribution based on the ProUCL results; therefore, the 95% Chebyshev nonparametric UCL was selected for all three PAH calculations.

Table 1B. Invertebrate and Soil Sample Location Summary
Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

Location	∑10 HMW PAHs (ND=0.5DL) (mg/kg)	Location Selected For Invertebrate Sampling
SS-111	0.87	1. YES (<1)
TB-10	0.57	2. YES (<1)
SS-123	0.72	3. YES (<1)
TWSB23	5.36	1. YES (1-10)
SB-129	5.41	2. YES (1-10)
SS-121	4.82	3. YES (1-10)
TB-14	16.0	1. Yes (11-50)
TB-16F	33.2	2. Yes (11-50)
TB-24	34.5	3. Yes (11-50)
TB-08	95.4	1. YES (50-100)
RISB09	95.3	2. YES (50-100)
RISB10	66.9	3. YES (50-100)
SS-110	161	1. YES (>100)
TB-11	131	2. YES (>100)
SS-117	131	3. YES (>100)

Notes:

Σ10 HMW PAHs	Sum of 10 high molecular weight polycyclic aromatic hydrocarbons
DL	Detection limit (method)
ND	Not detected
mg/kg	Milligrams per kilogram

(a) ∑10 HMW PAH concentration range identified below

<1	∑10 HMW PAHs <1 mg/kg
1-10	1< ∑10 HMW PAHs <10 mg/kg
11-50	11< ∑10 HMW PAHs <50 mg/kg
51-100	51< ∑10 HMW PAHs <100 mg/kg
>100	∑10 HMW PAHs >100mg/kg

Table 2. Analytical Laboratories, Proposed Laboratory Methods, and Sample Summary
Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

Media	Analyte	Analytical Method	Lab-oratory	No. Samples	Type of Sample (a)	Field Dupes	Equip-ment Rinsate Blanks	MS/ MSD	Total No. Samples	Min Sample Mass Per Sample (grams)	Sample Container	Pre-servative	Hold Times
Earthworms	Nonalkylated PAHs	EPA 8270-PAH-SIM	SGS Axys	15	Composite	1	--	1	17	10	Foil wrapped, zip-top bag	None, frozen	<1 year
	Alkylated PAHs	EPA 8270-PAH-ALK-SIM		15	Composite	1	--	1	17				
	Lipids	Axys SOP SLA-020		15	Composite	1	--	1	17				
	Moisture Content	Axys SOP SLA-015		15	Composite	1	--	1	17	1			
	Total Minimum Sample Mass Needed for Tissue Analysis:									**11**	**grams (b)**		
Depurated Earthworms	Nonalkylated PAHs	EPA 8270-PAH-SIM	SGS Axys	5	Composite	1	--	1	7	10	Foil wrapped, zip-top bag	None, frozen	<1 year
	Alkylated PAHs	EPA 8270-PAH-ALK-SIM		5	Composite	1	--	1	7				
	Lipids	Axys SOP SLA-020		5	Composite	1	--	1	7				
	Moisture Content	Axys SOP SLA-015		5	Composite	1	--	1	7	1			
	Total Minimum Sample Mass Needed for Tissue Analysis:									**11**	**grams (b)**		
Soil Invertebrates (e.g., beetles, ants, grubs)	Nonalkylated PAHs	EPA 8270-PAH-SIM	SGS Axys	15	Composite	1	--	1	17	10	Foil wrapped, zip-top bag	None, frozen	<1 year
	Alkylated PAHs	EPA 8270-PAH-ALK-SIM		15	Composite	1	--	1	17				
	Lipids	Axys SOP SLA-020		15	Composite	1	--	1	17				
	Moisture Content	Axys SOP SLA-015		15	Composite	1	--	1	17	1			
	Total Minimum Sample Mass Needed for Tissue Analysis:									**11**	**grams (b)**		
Soil	Nonalkylated PAHs	EPA 8270D	SGS Axys	15	Composite	1	1	1	18	30	4 ounce Wide Mouth Glass Jar	Cool to ≤ 6° Celsius	14 days extraction/40 days analysis (c)
	Alkylated PAHs	EPA 8270D-SIM		15	Composite	1	1	1	18	30			14 days extraction/40 days analysis (c)
	TOC	EPA 9060		15	Composite	1	1	1	18	15			28 days
	Moisture Content	ASTM D2974		15	Composite	1	1	1	18	15			NA
	Total Minimum Sample Mass Needed for Soil Analysis:									**62**	**grams**		

Notes:

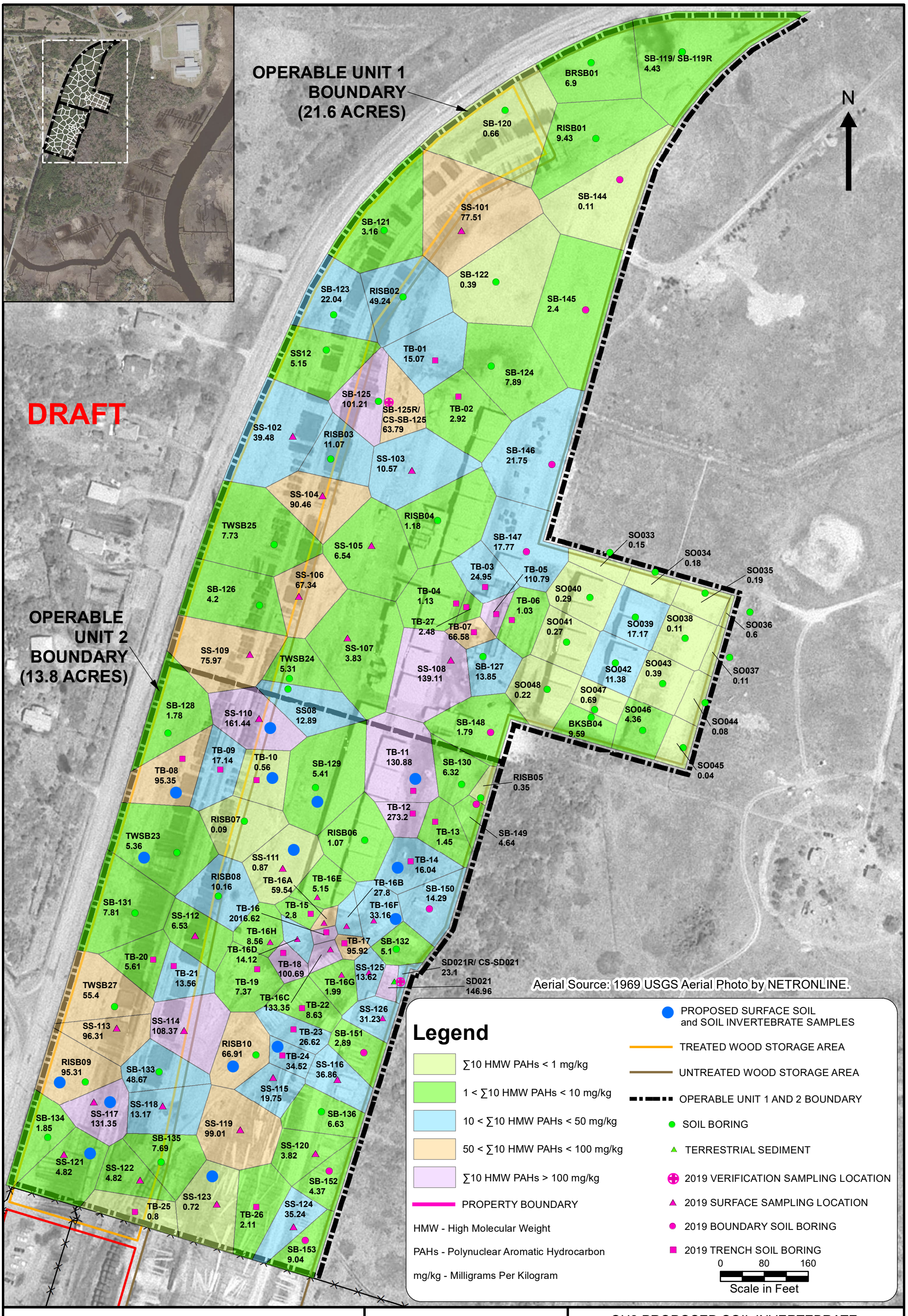
(a) Each composite sample will be comprised of terrestrial invertebrates collected from up to five individual 1-square meter (m^2) grids at each sample location. Composite soil samples will be comprised of the same areas that invertebrates were collected.

(b) Attachment A "Tissue Mass and Detection Limit Estimates" provides the calculations to support the collection of 10 grams of tissue for analysis.

(c) Holding time is 14 days from collection to extraction and 40 days from extraction to analysis.

ASTM	ASTM International
Dupes	Duplicate
EPA	U.S. Environmental Protection Agency
MS/MSD	Matrix spike/matrix spike duplicate
N/A	Not applicable
No.	Number
PAHs	Polycyclic aromatic hydrocarbons
SIM	Selected ion monitoring
SOP	Standard operating procedure
TBD	To be determined
TOC	Total organic carbon

FIGURES



S:\Premier\Projects\Greenfield Environmental Multistate Trust\KMCC Navassa NC Superfund Site\CAD GIS Data\EARTHCON_GIS\MXDs\OU1\Proposed Plan OU1\Fig 1 OU2 Proposed Soil Invertebrate Sample Locations.mxd 3/16/2020 3:39:13 PM

Greenfield Environmental Multistate Trust LLC
Trustee of the Multistate Environmental Response Trust

PREPARED BY:
EARTHCON
EARTHCON CONSULTANTS OF NORTH CAROLINA, P.C.

OU2 PROPOSED SOIL INVERTEBRATE SAMPLE LOCATIONS

Operable Unit 2

Kerr-McGee Chemical Corp - Navassa Superfund Site
Navassa, North Carolina

DRAWN	CHECKED	DATE	FIGURE
HVP	CDN	FEB 2020	1

ATTACHMENTS

ATTACHMENT A

Estimation of Tissue Mass to Achieve Required Detection Limits

Greenfield Environmental Multistate Trust, LLC
Trustee of the Multistate Environmental Response Trust

EARTHCON

RAMBOLL

Attachment A
Estimation of Tissue Mass to Achieve Required Detection Limits

The soil invertebrate sampling at OU2 will cover the range of concentrations used in the July 2019 EPA food web model for the upland area (EPA 2019). The PAH concentrations and calculated earthworm concentrations from the upland food web model generated by the EPA are shown in Table 1. As shown here, the estimated earthworm tissue concentrations ranged from 21.06 to 160.94 mg/kg dry weight.

Table 1. Estimated HMW PAH Concentrations in Earthworms for OU2 (from EPA Screening ERA)

Receptor/diet	HMW PAH conc in soil (mg/kg DW)	Uptake factor to earthworms (a)	HMW PAH concentration in earthworm DW tissue (mg/kg DW) (b)	HWM PAH concentration in earthworm WW tissue (mg/kg WW) (c)
Robin (100% earthworms)	61.9	2.6	160.94	25.75
Robin (100% earthworms)	25.9	2.6	67.34	10.77
Robin (100% earthworms)	8.1	2.6	21.06	3.37

Notes:
(a) Uptake factor used in the EPA food web model is from Eco-SSL 2007.
(b) Concentration in earthworm was calculated using the following formula: Cworm = Csoil * uptake factor
(c) The HMW PAH concentration in dry weight earthworm tissue (mg/kg DW) was converted to show the HMW PAH concentration in wet weight tissue using 84% moisture and the following formula: Cworm (mg PAH/kg DW worm) * 0.16 kg DW worm/kg WW worm

Csoil = Concentration in soil
Cworm = Concentration in earthworm
DW = Dry weight
EPA = Environmental Protection Agency
EPA = Environmental Protection Agency
HMW PAH = High molecular weight polycyclic aromatic hydrocarbons
mg/kg = Milligram per kilograms
WW = Wet weight

EPA estimated a range of tissue mass that may be needed with varying ranges of PAHs in soils, with the goal of meeting a detection limit of 0.05 mg/kg and an uptake factor of 0.1 for a typical 30 gram soil sample. Ramboll recreated EPA's estimated tissue concentration using a template provided by EPA in an email dated February 14, 2020. The calculations are shown in Table 2.

Table 2. Estimated Tissue Mass Needed for a 30 gram soil aliquot with a detection limit of 0.05 mg/kg and an uptake factor of 0.1

Soil concentration	Uptake Factor	Invert Concentration (a)	Detection Limit	Soil Aliquot	Invert Mass Needed (b)	Invert Mass Needed (c)
mg/kg soil	Unitless DW	mg/kg DW invert	mg/kg	g soil	g DW	g WW
assumed	assumed	calculated	assumed	assumed	calculated	calculated
1	0.1	0.1	0.05	30	15	93.8
10	0.1	1	0.05	30	1.5	9.4
50	0.1	5	0.05	30	0.3	1.9
100	0.1	10	0.05	30	0.15	0.94
500	0.1	50	0.05	30	0.03	0.19

(a) Concentration in earthworm was calculated using the following formula: Cworm = Csoil * uptake factor
(b) Invertebrate mass needed was calculated with the following equation:
Mass Needed (grams DW) = (PAH detection limit in mg/kg * soil aliquot in grams)/Detection limit needed for Inverts in mg/kg

(c) Invertebrate mass needed was converted to wet weight from dry weight using a 84% moisture.

DW = Dry weight
g = grams
Invert = Invertebrate
Gray cells are formula
mg/kg = Milligrams per kilogram
WW = Wet weight

Attachment A
Estimation of Tissue Mass to Achieve Required Detection Limits

Following the February 13 2020 meeting between the Multistate Trust and Beneficiaries, Ramboll looked for low detection limit options for use in the soil invertebrate analysis. Low detection limit analysis can be conducted using SGS Axys Parent PAH and Alkylated PAHs by GC/MS via Method GSG AXYS MLA-021.12 VER 05, which is the method used for USEPA 8270C with modification. Attachment B provides the method in detail. Attachment C provides the PAH detection limits SGS Axys can provide. According to information provided by SGS Axys, the lab can obtain low detection limits for alkylated and nonalkylated PAHs, as shown in Table 3A.

Table 3A. SGS Axys SDLs at 10 gram of tissue mass wet weight

SDLs (ng/g)	SDLs (mg/kg)
0.1	0.0001
0.2	0.0002

Notes:
Biological tissue sample size of 10g tissue is standard for use at SGS Axys to achieve very low SDLs.
Units in ng/g = µg/kg, which were converted to mg/kg.
ng/g = Nanogram per gram
µg/kg = Microgram per kilogram
SDL = Sample detection limit

Ramboll used the information provided by SGS Axys in Table 3A, to calculate the minimum mass needed (in grams) for collection. Table 3B presents the calculations for a 0.1 uptake factor using both a 0.0001 and 0.0002 mg/kg detection limit.

Table 3B. Estimated Tissue Mass Needed for a 1.6 gram aliquot with detection limits from Table 3A and an uptake factor of 0.1 and 0.2

Soil concentration mg/kg soil assumed	Uptake Factor Unitless DW assumed	Invert Conc (a) mg/kg DW Invert calculated	Detection Limit mg/kg SGS Axys	Tissue Aliquot (b) g tissue DW SGS Axys	Invert Mass Needed (c) g tissue DW calculated	Invert Mass Needed (d) g WW calculated
0.05	0.1	0.005	0.0001	1.6	0.032	0.2
0.1	0.1	0.01	0.0001	1.6	0.016	0.1
0.5	0.1	0.05	0.0001	1.6	0.0032	0.02
1	0.1	0.1	0.0001	1.6	0.0016	0.01
10	0.1	1	0.0001	1.6	0.00016	0.001
50	0.1	5	0.0001	1.6	0.000032	0.0002
100	0.1	10	0.0001	1.6	0.000016	0.0001
500	0.1	50	0.0001	1.6	0.0000032	0.00002

Soil Concentration mg/kg soil assumed	Uptake Factor Unitless DW assumed	Invert Conc mg/kg DW Invert calculated	Detection Limit mg/kg SGS Axys	Tissue Aliquot (b) g tissue DW SGS Axys	Invert Mass Needed (c) g tissue DW calculated	Invert Mass Needed (d) g WW calculated
0.05	0.1	0.005	0.0002	1.6	0.064	0.4
0.1	0.1	0.01	0.0002	1.6	0.032	0.2
0.5	0.1	0.05	0.0002	1.6	0.0064	0.04
1	0.1	0.1	0.0002	1.6	0.0032	0.02
10	0.1	1	0.0002	1.6	0.00032	0.002
50	0.1	5	0.0002	1.6	0.000064	0.0004
100	0.1	10	0.0002	1.6	0.000032	0.0002
500	0.1	50	0.0002	1.6	0.0000064	0.00004

Attachment A
Estimation of Tissue Mass to Achieve Required Detection Limits

(a) Concentration in earthworm was calculated using the following formula: Cworm = Csoil * uptake factor
(b) As stated in Table 3A, SGS Axys only needs 10 grams of wet weight tissue to achieve their low detection limits. This value was converted to dry weight using a 84% moisture content as follows:
10 g WW tissue * 0.16 g DW tissue/1 g WW tissue

(c) Invertebrate mass needed was calculated with the following equation:
Mass Needed (grams DW) = (PAH detection limit in mg/kg * soil aliquot in grams)/Detection limit needed for Inverts in mg/kg
(d) Invertebrate mass needed was converted to wet weight from dry weight using a 84% moisture.

DW = Dry weight
g = grams
Invert = Invertebrate
Gray cells are formula
mg/kg = Milligrams per kilogram
WW = Wet weight

If less than 10 grams of tissue is provided, then the detection limits provided by SGS Axys for analysis are adjusted but are still low. Table 3C provides the detection limits with SGS Axys that can be achieved if less than 10 grams of biological tissue is collected for analysis.

Table 3C. SGS Axys SDLs if Insufficient Tissue Mass is available

SDLs using 10 grams of tissue mass (mg/kg)	Adjusted SDL if <10 grams of tissue (a)	Target tissue mass (grams)	Actual amount (in grams) of tissue available	Target Uptake Factor	Estimated worm tissue conc (mg/kg)	Csoil (mg/kg)
0.0001	0.0002	10	5	0.1	0.5	5
0.0001	0.0005	10	2	0.1	1	10
0.0002	0.0004	10	5	0.1	5	50
0.0002	0.001	10	2	0.1	10	100
0.0002	0.0004	10	5	0.1	20	200

Notes: (a) Adjusted Tissue SDL = SDL (at 10 grams of tissue) x Percent of Tissue Available (Target Tissue Amount/Amount Collected)
(b) Uptake Factor = Concentration in worm (mg/kg)/Concentration in soil (mg/kg)
Gray cells are formula

If a tissue or soil sample has elevated PAHs that exceed the calibration range, then the sample will need to be diluted. Table 3D show the detection limit adjustments if dilution occurs.

Table 3D. SGS Axys Adjusted SDLs to Account for Dilutions

SDLs	Adjusted SDL	Dilution Factor
0.0001	0.0002	2
0.0001	0.001	10
0.0001	0.005	50
0.0002	0.0004	2
0.0002	0.002	10
0.0002	0.01	50

Notes: If elevated PAH concentration are found in worms, the SDL will increase, as follows: SDL= Sample Detection Limit x Dilution Factor
Gray cells are formula

Conclusion: SGS Axys provides low detection limits for invertebrate analysis for alkylated and nonalkylated PAHs. Even accounting for low PAHs, dilution factors, and low sample mass, it is possible to obtain the detection limits the EPA has requested using a 10 gram invertebrate mass sample.

References:

EPA Region 4. 2019. Semi-Screening Level Ecological Risk Assessment Calculations for Upland Areas 1A, 1B, and 2 of the Kerr-McGee Chemical Company Site in Navassa, North Carolina. Memorandum from Brett Thomas, EPA, to Erik Spalvins, Remedial Project Manager - EPA. July 30.

USEPA. 2007. Guidance for Developing Ecological Soil Screening Levels (OSWER Directive 9285.7-55). Washington, D.C.: USEPA, Office of Solida Waste and Emergency Response. November.

ATTACHMENT B

Summary of SGS Axys Method MLA-021 Rev 12 Ver 05

SGS AXYS Analytical Services Ltd.

SUMMARY OF SGS AXYS METHOD MLA-021 REV. 12 VER. 05:

ANALYTICAL METHOD FOR THE DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS (PAH), ALKYLATED POLYCYCLIC AROMATIC HYDROCARBONS, AND ALKANES

SGS AXYS Method MLA-021 describes the determination of concentrations of PAHs, alkylated PAHs and alkanes in solid (sediment, soil, ash), tissue (including blood), aqueous, XAD-2 column (resin and filters), air, and oil samples and in solvent extracts such as those produced by dialysis of SPMD (Semipermeable Membrane Device) samplers.

The method may be used for analysis of samples where USEPA Methods 1625B or 8270C/D have been requested **provided the modifications described in this document are permitted by contract**.

This summary document covers only the analysis of PAHs and alkylated PAHs.

Target Analytes

PAHs and alkylated PAHs - determined by multi-point calibration [2]

List 1 Compounds

Naphthalene [1]
Acenaphthylene
Acenaphthene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene
Benz(a)anthracene
Chrysene
Benzo(b)fluoranthene
Benzo(j/k)fluoranthenes
Benzofluoranthenes
Benzo(e)pyrene
Benzo(a)pyrene
Perylene
Dibenzo(ah)anthracene
Indeno(1,2,3-cd)pyrene
Benzo(ghi)perylene
Dibenzothiophene
2-Methylnaphthalene [1]
2,6-Dimethylnaphthalene [1]
2,3,5-Trimethylnaphthalene [1]
1-Methylphenanthrene

List 2 Compounds

1-Methylnaphthalene [1]
C1-Naphthalenes [1]
1,2-Dimethylnaphthalene [1]
C2-Naphthalenes [1]
2,3,6-Trimethylnaphthalene [1]
C3-Naphthalenes [1]
1,4,6,7-Tetramethylnaphthalene [1]
C4-Naphthalenes [1]
2-Methylphenanthrene
C1-Phenanthrenes/Anthracenes
1,7-Dimethylphenanthrene
1,8-Dimethylphenanthrene
2,6-Dimethylphenanthrene
3,6-Dimethylphenanthrene
C2-Phenanthrenes/Anthracenes
1,2,6-Trimethylphenanthrene
C3-Phenanthrenes/Anthracenes
Retene

3-Methylphenanthrene
9/4-Methylphenanthrenes
2-Methylanthracene
C4-Phenanthrenes/Anthracenes
Biphenyl

List 3 Compounds

C1-Biphenyls
C2-Biphenyls
C1-Acenaphthenes
2-Methylfluorene
C1-Fluorenes
1,7-Dimethylfluorene
C2-Fluorenes
C3-Fluorenes
2/3-Methyldibenzothiophenes
C1-Dibenzothiophenes
2,4-Dimethyldibenzothiophene
4,6-Dimethyldibenzothiophene
C2-Dibenzothiophenes
C3-Dibenzothiophenes
C4-Dibenzothiophenes
3-Methylfluoranthene/Benzo(a)fluorene
C1-Fluoranthenes/Pyrenes
C2-Fluoranthenes/Pyrenes
C3-Fluoranthenes/Pyrenes
C4-Fluoranthenes/Pyrenes
1-Methylchrysene
5/6-Methylchrysenes
C1-Benz(a)anthracenes/Chrysenes
5,9-Dimethylchrysene
C2-Benz(a)anthracenes/Chrysenes
C3-Benz(a)anthracenes/Chrysenes
C4-Benz(a)anthracenes/Chrysenes
7-Methylbenzo(a)pyrene
C1-Benzofluoranthenes/Benzopyrenes
C2-Benzofluoranthenes/Benzopyrenes

[1] Maximum value when determined in XAD-2 adsorbent.

[2] Optionally calibration may be performed by single-point bracketing calibration.

1.0 EXTRACTION AND CLEANUP PROCEDURES

All samples are spiked with deuterated surrogate standards prior to extraction and extracted as per the table below. Optional extraction procedures are shown within parentheses.

Sample Extraction

Matrix	Extraction
Aqueous	Sample with ≤1% suspended solids - Liquid-liquid extraction with dichloromethane. Sample with >1% suspended solids - sample is centrifuged prior to extraction and the particulate fraction separately extracted by Soxhlet extraction with dichloromethane. The two extracts are then combined. (Optional: the supernatant can be added to the extraction solvent for the particulate)
Solid (sediment, soil, sludge, particles on filter paper)	Soxhlet extraction with dichloromethane (Optional: Base digestion and liquid-liquid extraction with hexane)

Matrix	Extraction
Solid (ash)	Soxhlet extraction with toluene
Solid (fly ash)	Sonication with hydrochloric acid and filtering. Liquid-liquid extraction of filtrate using dichloromethane, Soxhlet extraction of particulate using toluene:acetone 80:20. The two extracts are combined.
Tissue	Soxhlet extraction with dichloromethane. (Optional: Base digestion and liquid-liquid extraction with pentane)
Whole blood/serum/plasma	Liquid-liquid extraction with ethanol:hexane:saturated ammonium sulphate.
XAD-2 column and filter	XAD-2 columns and filters are usually co-extracted for multiple analyses (For example: PCB, pesticides) and the resulting extracts are split with a portion being used for PAH analysis
Ambient air (PUF and filter)	The PUF and filter(s) are Soxhlet extracted together using dichloromethane
Stationary Source Air Samples (Stack Gas sample trains)	The filter is sonicated with dilute hydrochloride acid and filtered. Equipment rinsates are collected, filtered, dried and/or extracted depending on sampling conditions.
SPMD samples	SPMD tubes are dialyzed with hexane.

After extraction the extracts are routinely cleaned up using the following procedures:

- column chromatography on silica
- gel permeation (Biobeads) column chromatography
- treatment with activated copper (except tissues)

The extracts may be cleaned up further, as necessary, using some or all of the following procedures:

- washing with base
- column chromatography on Biobeads
- column chromatography on alumina

2.0 INSTRUMENTATION

Instrumental analysis is performed by low-resolution mass spectrometry (LRMS) using an RTX-5 capillary GC column. The LRMS is operated at a unit mass resolution in the electron impact (EI) ionization mode using multiple ion detection (MID) acquiring at least one characteristic ion for each target analyte and surrogate standard.

Benzo(b)fluoranthene Correction to Remove Surrogate Interference

At low concentrations native benzo(b)fluoranthene results are affected by an interference from the surrogate standard D_{12}-benzo(k)fluoranthene. D_{12}-benzo(k)fluoranthene has the same chromatographic retention time as benzo(b)fluoranthene and produces an artifactual response in the quantifying ion channel m/z 252. To remedy this interference the estimated peak area contribution from D_{12}-benzo(k)fluoranthene is subtracted from the benzo(b)fluoranthene peak area before quantification.

The peak area of d_{12}-benzo(b)fluoranthene in ion channel *m/z* 252 of the procedural blank is divided by the area of the same compound in ion channel *m/z* 264 to calculate the ion abundance ratio, usually around 0.003. (D_{12}-benzo(b)fluoranthene is used rather than d_{12}-benzo(k)fluoranthene because it doesn't coelute with any known native compound.) The corrected peak area of the native benzo(b)fluoranthene is then calculated as:

$$A^{corr.}_{benzo(b)fluoranthene(252)} = A^{sample}_{benzo(b)fluoranthene(252)} - A^{sample}_{d12\text{-}benzo(k)fluoranthene(264)} \left(\frac{A^{blank}_{d12\text{-}benzo(b)fluoranthene(252)}}{A^{blank}_{d12\text{-}benzo(b)fluoranthene(264)}} \right)$$

where $A^{corr.}_{benzo(b)fluoranthene(252)}$ = Corrected peak area for benzo(b)fluoranthene in the sample

$A^{sample}_{benzo(b)fluoranthene(252)}$ = Measured peak area for benzo(b)fluoranthene in the sample

$A^{sample}_{d12\text{-}benzo(k)fluoranthene(264)}$ = Peak area for d_{12}-benzo(k)fluoranthene in the sample

$A^{blank}_{d12\text{-}benzo(b)fluoranthene(252)}$ = Area for d_{12}-benzo(b)fluoranthene (m/z 252) in the blank

$A^{blank}_{d12\text{-}benzo(b)fluoranthene(264)}$ = Area for d_{12}-benzo(b)fluoranthene (m/z 264) in the blank

This correction is performed by the instrument quantification software and the LIMS software.

3.0 CALIBRATION

Initial calibration is performed using a five point calibration series of solutions that encompass the working concentration range. Initial calibration solutions contain the suite of labelled surrogate and recovery standards and authentic target PAHs listed as "PAHs and alkylated PAHs determined by multi-point calibration". Calibration procedures use the mean RRFs determined from the initial calibration to calculate analyte concentrations. Calibration is verified at least once every 12 hours by analysis of a mid-level calibration solution.

Upon the request of the client single-point bracketing calibration protocols may be followed. Bracketing calibration procedures use mean RRFs from the analysis of the mid-level calibration solution to calculate analyte concentration.

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Concentration of PAHs/Alkylated PAHs Calibration Standard Solutions

TARGET ANALYTE	Level A (Sens. Std) (ng/mL)	Concentration of Calibration Standard Solutions (ng/mL)					Conc. of Native Std (Low Level) (ng/mL)	Conc. of Native Std (High Level) (ng/mL)
		Level B	Level C	Level D	Level E (Mid-level)	Level F		
Acenaphthene	10	50	100	500	2000	5000	2000	20 000
Acenaphthylene	10	50	100	500	2000	5000	2000	20 000
Anthracene	10	50	100	500	2000	5000	2000	20 000
Benz[a]anthracene	10	50	100	500	2000	5000	2000	20 000
Benzo[b]fluoranthene	10	50	100	500	2000	5000	2000	20 000
Benzo[k]fluoranthene	10	50	100	500	2000	5000	2000	20 000
Benzo[ghi]perylene	10	50	100	500	2000	5000	2000	20 000
Benzo[a]pyrene	10	50	100	500	2000	5000	2000	20 000
Benzo[e]pyrene	10	50	100	500	2000	5000	2000	20 000
Biphenyl	10	50	100	500	2000	5000	2000	20 000
Chrysene	10	50	100	500	2000	5000	2000	20 000
Dibenzo[ah]anthracene	10	50	100	500	2000	5000	2000	20 000
2,6-Dimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
Fluoranthene	10	50	100	500	2000	5000	2000	20 000
Fluorene	10	50	100	500	2000	5000	2000	20 000
Indeno[1,2,3-cd]pyrene	10	50	100	500	2000	5000	2000	20 000
1-Methylnaphthalene	10	50	100	500	2000	5000	2000	20 000
2-Methylnaphthalene	10	50	100	500	2000	5000	2000	20 000
1-Methylphenanthrene	10	50	100	500	2000	5000	2000	20 000
Naphthalene	10	50	100	500	2000	5000	2000	20 000
Perylene	10	50	100	500	2000	5000	2000	20 000
Phenanthrene	10	50	100	500	2000	5000	2000	20 000
Pyrene	10	50	100	500	2000	5000	2000	20 000
2,3,5-Trimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
Dibenzothiophene	10	50	100	500	2000	5000	2000	20 000
3,6-Dimethylphenanthrene	10	50	100	500	2000	5000	2000	20 000
Retene	10	50	100	500	2000	5000	2000	20 000
2-Methylanthracene	10	50	100	500	2000	5000	2000	20 000
1,2-Dimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
2-Methylphenanthrene	10	50	100	500	2000	5000	2000	20 000
1,2,6-Trimethylphenanthrene	10	50	100	500	2000	5000	2000	20 000
2,3,6-Trimethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
1,7-Dimethylphenanthrene	10	50	100	500	2000	5000	2000	20 000
1,4,6,7-Tetramethylnaphthalene	10	50	100	500	2000	5000	2000	20 000
2-Methylfluorene	10	50	100	500	2000	5000	2000	20 000
1,7-Dimethylfluorene	10	50	100	500	2000	5000	2000	20 000
2-Methyldibenzothiophene	10	50	100	500	2000	5000	2000	20 000
2,4-Dimethyldibenzothiophene	10	50	100	500	2000	5000		
4,6-Dimethyldibenzothiophene	10	50	100	500	2000	5000	2000	20 000
5,9-Dimethylchrysene	10	50	100	500	2000	5000	2000	20 000
7-Methylbenzo(a)pyrene	10	50	100	500	2000	5000	2000	20 000
3-Methylfluoranthene	10	50	100	500	2000	5000	2000	20 000
6-Methylchrysene	10	50	100	500	2000	5000	2000	20 000
1-Methylchrysene	10	50	100	500	2000	5000	2000	20 000

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LABELLED SURROGATE STANDARDS	Level A (Sens. Std) (ng/mL)	Concentration of Calibration Standard Solutions					Conc. of Surrogate Std (Low Level) (ng/mL)	Conc. of Surrogate Std (High Level) (ng/mL)
		Level B	Level C	Level D	Level E	Level F		
d_8-Naphthalene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-2-Methylnaphthalene	2000	2000	2000	2000	2000	2000	2000	20 000
d_8-Acenaphthylene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-Phenanthrene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-Fluoranthene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benz[a]anthracene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Chrysene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-2,6-Dimethylnaphthalene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[b]fluoranthene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[k]fluoranthene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[a]pyrene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Perylene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Indeno[1,2,3-cd]pyrene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{14}-Dibenzo[ah]anthracene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{12}-Benzo[ghi]perylene	2000	2000	2000	2000	2000	2000	2000	20 000
d_{10}-Biphenyl	2000	2000	2000	2000	2000	2000	2000	20 000
d_8-Dibenzothiophene	2000	2000	2000	2000	2000	2000	2000	20 000
LABELLED RECOVERY STANDARDS							**Conc. of Recovery Std (ng/mL)**	
d_{10}-Acenaphthene	2000	2000	2000	2000	2000	2000	20 000	
d_{10}-Pyrene	2000	2000	2000	2000	2000	2000	20 000	
d_{12}-Benzo[e]pyrene	2000	2000	2000	2000	2000	2000	20 000	

4.0 QUANTIFICATION PROCEDURES

Concentrations of target PAHs are calculated using the isotope dilution method of quantification. Compounds are quantified by comparing the area of the quantification ion to that of the corresponding deuterium-labelled standard and correcting for response factors. Response factors are determined daily using authentic PAHs. Calculations are carried out using HP EnviroQuant and Prolab MS-Extended for targeting and quantification.

$$\text{Concentration of Target (ng/g or ng/L)} = \left(\frac{\text{area of Target}}{\text{area of Surr Std}}\right) \times \left(\frac{\text{weight of Surr Std (ng)}}{\text{RRF}}\right) \times \left(\frac{1}{\text{weight of sample (g or L)}}\right)$$

$$\textit{where} \quad \text{RRF} = \left(\frac{\text{area of Target}}{\text{area of Surr Std}}\right) \times \left(\frac{\text{concentration of Surr Std}}{\text{concentration of Target}}\right)$$

and the Surr Std is either the surrogate or the internal standard

4.1 Reporting Limits

Concentrations and detection limits for the target PAHs are reported. Typical reporting units for all data are ng/g, ng/L, or ng/sample. Concentrations for solids are reported on a dry weight basis. Concentrations in tissues (including blood and milk) are reported on a wet weight basis and/or on a lipid weight basis when requested. Concentrations in aqueous are reported on a volume basis. Concentrations in XAD-2 resin, filters and stack gas samples are reported on a per sample basis or a per volume basis. Concentrations in particulate filters are reported on a per sample basis.

The following are commonly requested reporting limits:

Sample Specific Detection Limit or Sample Detection Limit (SDL) – determined individually for every sample analysis run by converting the area equivalent of 3.0 times (2.5 times for EPA 1600 series methods) the estimated chromatographic noise height to a concentration in the same manner that target peak responses are converted to final concentrations. The SDL accounts for any effect of matrix on the detection system and for recovery achieved through the analytical work-up. Equivalent term(s): Estimated Detection Limit (EDL) from EPA method 8290.

Method Detection Limit (MDL) - determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B (no iteration option). The 99% confidence level MDL is determined based on analysis of a minimum of 7 replicate matrix spikes fortified at 1-10 times the estimated detection limit. MDL is determined as required based on accreditation, contract and workload requirements.

Lower Method Calibration Limit (LMCL) - determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used. ((lowest level cal conc.) x (extract volume))/sample size. Typical extract volume for aqueous and tissue samples is 100 µL for all other matrices typical extract volume is 500 µL.

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For the analysis of PAHs SGS AXYS standard is to report sample concentrations using the SDL as the reporting limit.

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Analyte Ions Monitored, Surrogates Used and RRF Determination For PAH

TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
Naphthalene	128	102	0.064	d_8-Naphthalene	6.84	Naphthalene
Acenaphthylene	152	151	0.222	d_8-Acenaphthylene	10.83	Acenaphthylene
Acenaphthene	154	153	1.18	d_8-Acenaphthylene	11.33	Acenaphthene
Fluorene	166	165	1.01	d_{10}-Phenanthrene	12.63	Fluorene
Phenanthrene	178	176	0.202	d_{10}-Phenanthrene	15.04	Phenanthrene
Anthracene	178	176	0.196	d_{10}-Phenanthrene	15.15	Anthracene
Fluoranthene	202	200	0.214	d_{10}-Fluoranthene	18.06	Fluoranthene
Pyrene	202	200	0.219	d_{10}-Fluoranthene	18.60	Pyrene
Benz[a]anthracene[6]	228	226	0.281	d_{12}-Benz[a]anthracene	21.68	Benz[a]anthracene
Chrysene[1]	228	226	0.312	d_{12}-Chrysene	21.79	Chrysene
Benzo[b]fluoranthene	252	253	0.218	d_{12}-Benzo[b]fluoranthene	25.21	Benzo[b]fluoranthene
Benzo[j,k]fluoranthenes	252	253	0.215	d_{12}-Benzo[k]fluoranthene	25.30	Benzo[k]fluoranthene
Benzo[e]pyrene	252	253	0.213	d_{12}-Benzo[a]pyrene	26.36	Benzo[e]pyrene
Benzo[a]pyrene	252	253	0.217	d_{12}-Benzo[a]pyrene	26.58	Benzo[a]pyrene
Perylene	252	253	0.212	d_{12}-Perylene	27.00	Perylene
Dibenzo[ah]anthracene[2]	278	139	0.144	d_{14}-Dibenzo[ah]anthracene	31.86	Dibenz[ah]anthracene
Indeno[1,2,3-cd]pyrene	276	138	0.179	d_{12}-Indeno[1,2,3,cd]pyrene	31.71	Indeno[1,2,3-cd]pyrene
Benzo[ghi]perylene	276	138	0.194	d_{12}-Benzo[ghi]perylene	32.53	Benzo[ghi]perylene
Biphenyl[3]	154	152	0.304	d_{10}- Biphenyl	9.81	Biphenyl
Dibenzothiophene[3]	184	152	0.073	d_8-Dibenzothiophene	14.72	Dibenzothiophene
1-Methylnaphthalene[3]	142	141	0.962	d_{10}-2-Methylnaphthalene	8.81	1-Methylnaphthalene
2-Methylnaphthalene[3]	142	141	0.930	d_{10}-2-Methylnaphthalene	8.55	2-Methylnaphthalene
1-Naphthalenes[3]	142	[4]	[4]	d_{10}-2-Methylnaphthalene	[5]	1- & 2-Methylnaphthalene
2,6-Dimethylnaphthalene[3]	156	141	0.666	d_{12}-2,6 Dimethylnaphthalene	10.17	2,6-Dimethylnaphthalene
1,2-Dimethylnaphthalene	156	141	1.26	d_{12}-2,6 Dimethylnaphthalene	10.90	1,2-Dimethylnaphthalene
C2-Naphthalenes[3]	156	[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	2,6- & 1,2-Dimethylnaphthalene

[1] Coelutes with Triphenylene
[2] Coelutes with Dibenz[ac]anthracene
[3] These compounds are in addition to the regular suite of analytes, and are analyzed by client request only.
[4] Secondary ion confirmation procedures do not apply
[5] RRT ranges apply to alkylated PAH Totals
[6] Benz(a)anthracene coelutes with cyclopenta(cd)pyrene, which may contribute response in the second ion to cause a failing ion abundance ratio. Therefore quantification of benz(a)anthracene should use response from the first ion only.

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
2,3,5-Trimethylnaphthalene[3]	170	155	0.873	d_{12}-2,6 Dimethylnaphthalene	12.35	2,3,5- Trimethylnaphthalene
2,3,6-Trimethylnaphthalene	170	155	0.876	d_{12}-2,6 Dimethylnaphthalene	12.17	2,3,6- Trimethylnaphthalene
C3-Naphthalenes[3]	170			d_{12}-2,6 Dimethylnaphthalene	[5]	2,3,5- & 2,3,6-Trimethylnaphthalene
1,4,6,7-Tetramethylnaphthalene	184	139	0.027	d_{12}-2,6 Dimethylnaphthalene	13.89	1,4,6,7-Tetramethylnaphthalene
C4-Naphthalene		[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	1,4,6,7-Tetramethylnaphthalene
2-Methylanthracene	192	191	0.531	d_{10}-Phenanthrene	16.45	2-Methylanthracene
3-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.27	1- & 2-Methylphenanthrene & 2-Methylanthracene
2-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.36	2-Methylphenanthrene
9/4-Methylphenanthrenes	192	191	0.634	d_{10}-Phenanthrene	16.59	1- & 2-Methylphenanthrene & 2-Methylanthracene
1-Methylphenanthrene[3]	192	191	0.634	d_{10}-Phenanthrene	16.64	1-Methylphenanthrene
C1-Phenanthrenes/Anthracenes[3]	192	[4]	[4]	d_{10}-Phenanthrene	[5]	1- & 2-Methylphenanthrene & 2-Methylanthracene
3,6-Dimethylphenanthrene[3]	206	191	0.342	d_{10}-Fluoranthrene	17.46	3,6-Dimethylphenanthrene
2,6-Dimethylphenanthrene	206	191	0.342	d_{10}-Fluoranthrene	17.54	3,6- & 1,7-Dimethyl-phenanthrenes
1,7-Dimethylphenanthrene	206	191	0.332	d_{10}-Fluoranthrene	17.89	1,7-Dimethylphenanthrene
1,8-Dimethylphenanthrene	206	191	0.332	d_{10}-Fluoranthrene	18.13	3,6- & 1,7-Dimethyl-phenanthrenes
C2-Phenanthrenes/Anthracenes[3]	206	[4]	[4]	d_{10}-Fluoranthrene	[5]	3,6- & 1,7-Dimethyl-phenanthrenes
1,2,6-Trimethylphenanthrene	220	205	0.581	d_{10}-Fluoranthrene	19.41	1,2,6-Trimethylphenanthrene
C3-Phenanthrenes/Anthracenes				d_{10}-Fluoranthrene	[5]	1,2,6-Trimethylphenanthrene
Retene[3]	234	219	1.63	d_{10}-Fluoranthene	19.53	Retene
C4-Phenanthrenes/Anthracenes	234	[4]	[4]	d_{10}-Fluoranthrene	[5]	Retene
C1-Biphenyls	168	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C2-Biphenyls	182	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C1-Acenaphthenes	168	[4]	[4]	d_8-Acenaphthylene	[5]	Acenaphthene
2-Methylfluorene	180	165	1.23	d_{10}-Phenanthrene	14.06	2-Methylfluorene
C1-Fluorenes	180	[4]	[4]	d_{10}-Phenanthrene	[5]	2-Methylfluorene
1,7-Dimethylfluorene	194	177	0.092	d_{10}-Phenanthrene	15.49	1,7-Dimethylfluorene
C2-Fluorenes	194	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
C3-Fluorenes	208	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene
2/3-Methyldibenzothiophenes	198	197	0.738	d_8-Dibenzothiophene	16.07	2/3-Methyldibenzothiophenes
C1-Dibenzothiophenes	198	[4]	[4]	d_8-Dibenzothiophene	[5]	2/3-Methyldibenzothiophenes
2,4-Dimethyldibenzothiophene	212	197	0.514	d_8-Dibenzothiophene	17.08	2,4-Dimethyldibenzothiophene
4,6-Dimethyldibenzothiophene	212	197	0.160	d_8-Dibenzothiophene	16.94	4,6-Dimethyldibenzothiophene
C2-Dibenzothiophenes	212	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
C3-Dibenzothiophenes	226	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
C4-Dibenzothiophenes	240	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
3-Methylfluoranthene/Benzo(a)fluorene	216	215	0.880	d_{10}-Fluoranthrene	19.53	3-Methylfluoranthene
C1-Fluoranthenes/Pyrenes	216	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
C2-Fluoranthenes/Pyrenes	230	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
C3-Fluoranthenes/Pyrenes	244	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
C4-Fluoranthenes/Pyrenes	258	[4]	[4]	d_{10}-Fluoranthrene	[5]	3-Methylfluoranthene
5/6-Methylchrysenes	242	[4]	[4]	d_{12}-Chrysene	23.15	6-Methylchrysene
1-Methylchrysene	242	[4]	[4]	d_{12}-Chrysene	23.32	1-Methylchrysene
C1-Benz(a)anthracenes/Chrysenes	242	[4]	[4]	d_{12}-Chrysene	[5]	1- & 6-Methylchrysenes
5,9-Dimethylchrysene	256	[4]	[4]	d_{12}-Chrysene	24.49	5,9-Dimethylchrysene
C2-Benz(a)anthracenes/Chrysenes	256	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C3-Benz(a)anthracenes/Chrysenes	270	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C4-Benz(a)anthracenes/Chrysenes	284	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
7-Methylbenzo(a)pyrene	266	[4]	[4]	d_{12}-Benzo[a]pyrene	29.35	7-Methylbenzo(a)pyrene
C1-Benzofluoranthenes/Benzopyrenes	266	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo(a)pyrene
C2-Benzofluoranthenes/Benzopyrenes	280	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo(a)pyrene
LABELLED SURROGATE STANDARDS	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**		**RECOVERY CALCULATED AGAINST**		
d_8-Naphthalene	136	134	0.095	d_{10}-Acenaphthene	6.80	
d_{10}-2-Methylnaphthalene	152	151	0.195	d_{10}-Acenaphthene	8.47	
d_{10}-Biphenyl	164	[4]	[4]	d_{10}-Acenaphthene	9.75	
d_{12}-2,6-Dimethylnaphthalene	168	150	0.747	d_{10}-Acenaphthene	10.07	
d_8-Acenaphthylene	160	158	0.159	d_{10}-Acenaphthene	10.80	
d_8-Dibenzothiophene	192	160	0.085	d_{10}-Pyrene	14.67	
d_{10}-Phenanthrene	188	184	0.143	d_{10}-Pyrene	14.97	
d_{10}-Fluoranthene	212	208	0.173	d_{10}-Pyrene	18.02	
d_{12}-Benz[a]anthracene	240	236	0.250	d_{10}-Pyrene	21.63	

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
d_{12}-Chrysene	240	236	0.278	d_{10}-Pyrene	21.73	
d_{12}-Benzo[b]fluoranthene	264	260	0.216	d_{12}-Benzo[e]pyrene	25.11	
d_{12}-Benzo[k]fluoranthene	264	260	0.208	d_{12}-Benzo[e]pyrene	25.23	
d_{12}-Benzo[a]pyrene	264	260	0.216	d_{12}-Benzo[e]pyrene	26.47	
d_{12}-Perylene	264	260	0.256	d_{12}-Benzo[e]pyrene	26.88	
d_{12}-Indeno[1,2,3,cd]pyrene	288	284	0.192	d_{12}-Benzo[e]pyrene	31.63	
d_{14}-Dibenzo[ah]anthracene	292	288	0.260	d_{12}-Benzo[e]pyrene	31.75	
d_{12}-Benzo[ghi]perylene	288	284	0.205	d_{12}-Benzo[e]pyrene	32.45	
LABELLED RECOVERY STANDARDS	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**				
d_{10}-Acenaphthene	164	160	0.464		11.24	
d_{10}-Pyrene	212	208	0.176		18.56	
d_{12}-Benzo[e]pyrene	264	260	0.269		26.25	

5.0 QUALITY ACCEPTANCE CRITERIA

Samples are analyzed in batches consisting of a maximum of twenty samples, one procedural blank and one spiked matrix (OPR) sample. A duplicate is analyzed, provided there is sufficient sample, with batches containing 7-20 samples. Matrix spike/matrix spike duplicate (MS/MSD) pairs may be analyzed on an individual contract basis. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the established acceptance criteria presented on the analysis reports.

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PAHs - QC Acceptance Criteria

MATRIX	Typical Sample Specific * Detection Limits							Procedural Blank Level				Acceptable OPR
	Solid	Aqueous	Tissue	XAD-2 Column	PUF & SPMD Extract	Filter	Oil	Solid & SPMD Extract	Aqueous	Tissue	All other matrices	
Analyte:	ng/g	ng/L	ng/g	ng	ng	ng	ng/g	ng (tot)	ng (tot)	ng (tot)	ng (tot)	% Recovery
Naphthalene	0.5	1	0.1	5	5	5	50	≤ 10	≤ 10	≤ 50	≤ 10 [1]	70-130
Acenaphthylene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-140
Acenaphthene [5]	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Fluorene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	60-140
Phenanthrene	0.5	1	0.1	5	5	5	50	≤ 10	≤ 10	≤ 10	≤ 10	70-130
Anthracene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Fluoranthene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Pyrene	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Benz(a)anthracene	0.5	1	0.1	5	5	5	50	≤ 1.9	≤ 1	≤ 0.9	≤ 5	70-130
Chrysene	0.5	1	0.1	5	5	5	50	≤ 3.0	≤ 1	≤ 1.3	≤ 5	70-130
Benzo(b)fluoranthene	0.5	1	0.1	5	5	5	50	≤ 4.2	≤ 0.6	≤ 1.6	≤ 5	70-130
Benzo(j/k)fluoranthenes	0.5	1	0.1	5	5	5	50	≤ 2.1	≤ 0.4	≤ 1.2	≤ 5	70-130
Benzo(e)pyrene [5]	0.5	1	0.1	5	5	5	50	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Benzo(a)pyrene	0.5	1	0.1	5	5	5	50	≤ 3.6	≤ 0.9	≤ 0.7	≤ 5	70-130
Perylene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	70-130
Dibenzo(ah)anthracene	1.0	2	0.2	10	10	10	100	≤ 2.8	≤ 0.7	≤ 0.7	≤ 5	70-130
Indeno(1,2,3-cd)pyrene	1.0	2	0.2	10	10	10	100	≤ 2.8	≤ 0.8	≤ 1.5	≤ 5	70-130
Benzo(ghi)perylene	1.0	2	0.2	10	10	10	100	≤ 2.4	≤ 0.9	≤ 0.7	≤ 5	70-130
Biphenyl	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	70-130
Dibenzothiophene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	60-140
1-Methylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5 [1]	70-130
2-Methylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5 [1]	70-130
2,6-Dimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	70-130
1,2-Dimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	60-140
2,3,5-Trimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	50-150
2,3,6-Trimethylnaphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	50-150

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MATRIX	Typical Sample Specific * Detection Limits							Procedural Blank Level				Acceptable OPR
	Solid	Aqueous	Tissue	XAD-2 Column	PUF & SPMD Extract	Filter	Oil	Solid & SPMD Extract	Aqueous	Tissue	All other matrices	
Analyte:	ng/g	ng/L	ng/g	ng	ng	ng	ng/g	ng (tot)	ng (tot)	ng (tot)	ng (tot)	% Recovery
1,4,6,7-Tetramethyl-naphthalene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	50-200
2-Methylanthracene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
3-Methylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
2-Methylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
9/4-Methylphenanthrenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
1-Methylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
3,6-Dimethylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
2,6-Dimethylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
1,7-Dimethyl-phenanthrene [2]	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
1,8-Dimethylphenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	N.A.
1,2,6-Trimethyl-phenanthrene	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
Retene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
2-Methylfluorene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
1,7-Dimethylfluorene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
2/3-Methyldibenzo-thiophenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	50-150
2,4-Dimethyl-dibenzothiophene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
4,6-Dimethyl-dibenzothiophene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
3-Methylfluoranthene [3]/ Benzo(a)fluorene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
5/6-Methylchrysenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
1-Methylchrysene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
5,9-Dimethylchrysene [4]	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
7-Methylbenzo(a)pyrene	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	50-150
C1-Biphenyls	1.0	2	0.2	10	10	10	100	≤ 50	≤ 50	≤ 50	≤ 50	

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MATRIX	Typical Sample Specific * Detection Limits							Procedural Blank Level				Acceptable OPR
	Solid	Aqueous	Tissue	XAD-2 Column	PUF & SPMD Extract	Filter	Oil	Solid & SPMD Extract	Aqueous	Tissue	All other matrices	
Analyte:	ng/g	ng/L	ng/g	ng	ng	ng	ng/g	ng (tot)	ng (tot)	ng (tot)	ng (tot)	% Recovery
C2-Biphenyls	1.0	2	0.2	10	10	10	100	≤ 250	≤ 250	≤ 250	≤ 250	
C1-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10 [1]	
C2-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 50	≤ 50	≤ 50	≤ 50 [1]	
C3-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15 [1]	
C4-Naphthalenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25 [1]	
C1-Acenaphthenes	0.5	1	0.1	5	5	5	50	≤ 10	≤ 10	≤ 10	≤ 10	
C1-Fluorenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	
C2-Fluorenes	1.0	2	0.2	10	10	10	100	≤ 50	≤ 50	≤ 50	≤ 50	
C3-Fluorenes	1.0	2	0.2	10	10	10	100	≤ 20	≤ 20	≤ 20	≤ 20	
C1-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	
C2-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	
C3-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C4-Dibenzothiophenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	
C1-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C2-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C3-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	
C4-Phenanthrenes/ Anthracenes	1.0	2	0.2	10	10	10	100	≤ 25	≤ 25	≤ 25	≤ 25	
C1-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C2-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C3-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	
C4-Fluoranthenes/ Pyrenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	

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MATRIX	Typical Sample Specific * Detection Limits							Procedural Blank Level				Acceptable OPR
	Solid	Aqueous	Tissue	XAD-2 Column	PUF & SPMD Extract	Filter	Oil	Solid & SPMD Extract	Aqueous	Tissue	All other matrices	
Analyte:	ng/g	ng/L	ng/g	ng	ng	ng	ng/g	ng (tot)	ng (tot)	ng (tot)	ng (tot)	% Recovery
C1-Benz(a)anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 5	≤ 5	≤ 5	≤ 5	
C2-Benz(a)anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	
C3-Benz(a)anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 10	≤ 10	≤ 10	≤ 10	
C4-Benz(a)anthracenes/ Chrysenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C1-Benzofluoranthenes/ Benzopyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
C2-Benzofluoranthenes/ Benzopyrenes	1.0	2	0.2	10	10	10	100	≤ 15	≤ 15	≤ 15	≤ 15	
Typical Sample Size	10 g (dry)	1 L	10 g (wet)	1 col	1 sample	1 filter	0.1 g					
Typical Final Volume (µL)	500	100	100	500	500	500	500					

* Detection limits quoted are those routinely achieved. Detection limits for alkylated PAH Compound Totals are based on detection of a single component.

[1] Procedural blank level limits don't apply for naphthalene and alkylated naphthalene from XAD-2 resin.

[2] Due to limited availability of 1,7-dimethylphenanthrene standard, this compound may be absent from OPR samples.

[3] Due to limited availablilty of 3-methylfluoranthene standard, this compound may be absent from OPR samples. It may be substituted by 2-methylfluoranthene as the exemplar alkylfluoranthene in the OPR.

[4] Due to limited availablilty of 5,9-dimethylchrysene standard, this compound may be absent from OPR samples. It may be substituted by other alternative dimethylchrysene isomers as the exemplar C2/C3/C4-benz(a)anthracene/chrysene in the OPR; in such cases the recovery specification for 5,9-dimethylchrysene would not apply.

[5] For solid samples extracted by the optional hexane:acetone 1:1 Soxhlet extraction the OPR method limits are 70 – 170 % recovery for acenaphthene and 70-160 % for benzo(e)pyrene.

NOTE: Reference samples are unavailable for Alkylated PAH Compound Totals, data acceptability evaluation is based on recoveries of the associated individual alkylated PAH compounds

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SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES ALL MATRICES
d_8-naphthalene	15 – 130
d_8-acenaphthylene	20 – 130
d_{10}-phenanthrene	30 – 130
d_{10}-fluoranthene *	30 – 130
d_{12}-benz[a]anthracene	30 – 130
d_{12}-chrysene	30 – 130
d_{12}-benzo[b]fluoranthene	30 – 130
d_{12}-benzo[k]fluoranthene	30 – 130
d_{12}-benzo[a]pyrene	30 – 130
d_{12}-perylene	30 – 130
d_{14}-dibenz[ah]anthracene *	30 – 130
d_{12}-indeno[1,2,3-cd]pyrene	30 – 130
d_{12}-benzo[ghi]perylene	30 – 130
d_{10}-2-methylnaphthalene	20 – 130
d_{12}-2,6-dimethylnaphthalene	20 – 130
d_{10}-biphenyl	15 – 130
d_8-dibenzothiophene	30 – 130

* Compound not used as surrogate standard for SPMD analysis. Surrogate recovery specifications do not apply for SPMD samples.

Performance Reference Compound (PRC) and Photolysis Standard Recovery Range Specifications for SPMD OPR samples: These ranges can also be used as guidance limits for any Client Day 0 and Field or Trip Blank Samples.

PRC	%RECOVERY RANGES
d_{10}-fluoranthene	70 – 130
d_{14}-dibenz[ah]anthracene	70 – 130
d_{10}-anthracene	70 – 130

QC Specification Table: Instrumental Analysis, and Analyte Quantification

Parameter	Acceptance Specification
Procedural Blank	Refer to Table "QC Specification Table: Authentic and Surrogate Standard Recoveries, OPR and Samples" above, or 5 times lower than analogous analyte value detected in the samples.
Analysis Duplicate	Duplicates must fall within ±20% of the mean (applicable to concentrations >10 times the DL) These are guidelines – departures based on professional judgement allowed. (Note that ±20% of the mean is equivalent to 40 relative percent difference)
Instrument Sensitivity	S/N 3:1 for 10 pg of acenaphthene, dibenzo(a,h)anthracene.
Instrument Resolution	Calibration gas PFTBA (FC43) unit mass resolution at m/e 69/70 and 219/220, Unit mass resolution is demonstrated by the presence of a resolved peak at m/z 70 and m/e 220.
Instrument Linearity	Linearity is demonstrated by a 5-point calibration over the working concentration range with a relative standard deviation of the RRFs ≤20% for targets with a labelled analog present and all labelled compounds, ≤35% for targets with no labelled analog present.
Continuing Cal Ver	Opening Cal Ver: Concentrations of native compounds and labelled surrogates must be within ±25% of expected values for all targets. Closing Cal Ver: Concentrations of native compounds must be within ±25% of expected values. Concentrations of labelled surrogates must be within ±25% of expected values, with any two (2) values allowed to be within ±40%
Bracketing Cal (optional)	RRFs for the opening and closing calibrations over a 12 hour period must agree to within ±20% of the mean (i.e., ≤40 RPD between RRFs and for the opening and closing calibrations, which is equivalent to ≤28.3 % RSD).
GC Resolution	Benzo[b] & [k]fluoranthene valley height must be ≤75% for equal concentrations. Phenanthrene/anthracene valley height must be ≤30% for equal concentrations.
Chromatogram Quality	Maximum peak width must be ≤15 seconds for benzo[ghi]perylene peak at 10% peak height.
Retention Time Window for Target Compounds	RT within ± 3 seconds of the predicted retention time determined from the calibration standard and adjusted relative to the peak retention time reference (i.e. labelled surrogate). A second requirement is that an authentic elute after its labelled analog.
Ion Abundance Ratios	CAL VER: Ion ratios for authentic and labelled dibenz[ah]anthracene, indeno[1,2,3-cd]pyrene and benzo[ghi]perylene must be within ±35% of the mid-point of the I-CAL All other native analytes and labelled surrogates must be within ±20% of the mid-point of the I-CAL Samples: Ion ratios for authentic dibenz[ah]anthracene, indeno[1,2,3-cd]pyrene and benzo[ghi]perylene must be within ±35% of the 12 hour CAL VER (or bracketing) calibration standard. All other native analytes and labelled surrogates must be within ±20% of the 12 hour CAL VER (or bracketing) calibration standard.

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APPENDIX 1 – SUMMARY OF KEY ATTRIBUTES OF METHODS MLA-021, EPA 8270C, 8270D AND 1625B

Analysis by GC/LRMS, Key Attributes of SGS AXYS MLA-021, EPA 8270C/D and EPA 1625B				
	MLA-021	**EPA 8270C**	**EPA 8270D**	**EPA 1625B**
MS acquisition mode	SIM[1]	Full Scan or SIM[1]	Full Scan or SIM[1]	Full Scan[1]
Qualitative Identification Criteria	Retention time & ratio of 2 ions	Retention time & ratio of 3 [2] ions	Retention time & ratio of 3 [2] ions	Retention time & ratio of characteristic ions
MS Ion Ratio Criteria	20 %	30 %	30 %	-50 % to +200 %
MS Tuning Type and Check Frequency	PTFBA, daily	DFTTP[1], 12 hrs	DFTTP[1], 12 hrs	DFTTP[1], 8 hrs
Quantification References	Isotopically labeled standards added prior to extraction	Internal standards added prior to instrumental analysis	Internal standards added prior to instrumental analysis	Isotopically labeled standards added prior to extraction
Recovery correction of results	YES	NO	NO	YES
Initial Calibration, # levels	5	5	5	5
Initial Calibration Limit (% RSD)	20 % (35 % if no labeled analog)	15 %	20 %	20 % (35 % if no labeled analog)
Calibration Verification Frequency	12 hrs	12 hrs	12 hrs	8 hrs
Calibration Verification Relative Response Limit (% diff.)	< 25 % of I-CAL	< 20 % of I-CAL	< 20 % of I-CAL	Various; most stringent is -20% to +25% of I-CAL
Calibration Verification IS area (% of I-CAL midpoint)	50-200 %	50-200 %	50-200 %	n.a.
Calibration verification IS RT (diff. from I-CAL midpoint)	n.a.	30 sec.	30 sec.	n.a.
Extraction	DCM (L/L (aqueous) DCM, Soxhlet (solids)	Options specified externally	Options specified externally	DCM, L/L (aqueous), pH>11 or pH other [3]

Notes:

1 SIM (Selected Ion Monitoring) acquisition protocol is permitted by EPA8270 and Federal Register Vol.77 Iss. 97 (May 18, 2012) Part 136

2 Based on availability, use of fewer ions is permitted

3 Modifications are permitted under Federal Register Vol.77 Iss.97 (May 18, 2012) Part 136.6

APPENDIX 2 – IDENTIFICATION AND QUANTIFICATION OF ALKYLATED PAH COMPOUND TOTALS

RT windows for Alkylated PAH Compound Totals are determined from a retention time reference extract run at the beginning of the instrumental run list; assignment of the sample RT windows is made based on comparison of the peak retention times and peak patterns in samples to that of the retention time reference standard. Table 13 lists estimated 'typical' values.

Analyte Ions Monitored, Surrogate Used and RRF Determination for Alkylated PAH Compound Totals

Alkylated PAH total parameter	Compounds/peaks	Typical peak/ window RT, min	Quant. ion	Conf. ion	Typical ion ratio	RRF reference	RT reference	Typical RT ref., min
C1-Biphenyls	(2 peaks)	11.22 - 11.44	168			Biphenyl	d10-Biphenyl	9.73
C2-Biphenyls	(6 or 7 peaks)	12.42 - 12.96	182			Biphenyl	d10-Biphenyl	9.73
C1-Naphthalenes	1-methylnaphthalene	8.80	142	141	0.965	1-methylnaphthalene	d_{10}-2-Methylnaphthalene	8.46
	2-methylnaphthalene	8.53	142	141	0.930	2-methylnaphthalene		
C2-Naphthalenes	Ethyl/dimethylnaphthalenes	9.96 - 10.75	156	141		Average of 1,2-dimethylnaphthalene and 2,6-dimethylnaphthalene	d12-2,6-dimethyl-naphthalene	10.05
	1,2-dimethylnaphthalene	10.88	156	141	1.320	1,2-dimethylnaphthalene		
C3-Naphthalenes	(≥2 peaks)	11.65 - 12.95	170			Average of 2,3,5-Trimethylnaphthalene and 2,3,6-Trimethylnaphthalene	d12-2,6-dimethyl-naphthalene	10.05
C4-Naphthalenes	(≥2 peaks)	13.05 - 14.65	184			1,4,6,7-Tetramethylnaphthalene	d12-2,6-dimethyl-naphthalene	10.05
C1-Acenaphthenes	(≥2 peaks)	11.60 - 11.70	168			Acenaphthene	d10-Acenaphthene	11.23
C1-Fluorenes	(≥2 peaks)	13.73 - 14.23	180			2-Methylfluorene	d10-Phenanthrene	14.96
C2-Fluorenes	(≥2 peaks)	14.96 - 16.01	194			1,7-Dimethylfluorene	d10-Phenanthrene	14.96
C3-Fluorenes	(≥2 peaks)	16.25 - 17.70	208			1,7-Dimethylfluorene	d10-Phenanthrene	14.96
C1-Dibenzothiophenes	C1-dibenzothiophenes-1	15.84	198			2/3-Methyldibenzothiophenes	d8-Dibenzothiophene	14.67
	2/3-methyldibenzo-thiophenes	16.05	198					
	C1-dibenzothiophenes-2	16.32	198					
C2-Dibenzothiophenes	(≥2 peaks)	16.35 - 17.95	212			2,4-Dimethyldibenzothiophene	d8-Dibenzothiophene	14.67
C3-Dibenzothiophenes	(≥2 peaks)	17.70 - 19.20	226			2,4-Dimethyldibenzothiophene	d8-Dibenzothiophene	14.67
C4-Dibenzothiophenes	(≥2 peaks)	18.80 - 20.50	240			2,4-Dimethyldibenzothiophene	d8-Dibenzothiophene	14.67
C1-Phenanthrenes/ Anthracenes	3-methylphenanthrene	16.27	192	191	0.618	Average of 1-Methylphenanthrene, 2-Methylphenanthrene and 2-Methylanthracene	d10-Phenanthrene	14.96

Alkylated PAH total parameter	Compounds/peaks	Typical peak/ window RT, min	Quant. ion	Conf. ion	Typical ion ratio	RRF reference	RT reference	Typical RT ref., min
	2-methylphenanthrene	16.33	192	191	0.610	2-methylphenanthrene		
	2-methylanthracene	16.44	192	191	0.531	2-methylanthracene		
	9/4-methylphenanthrenes	16.56	192	191	0.621	Average of 1-Methylphenanthrene, 2-Methylphenanthrene and 2-Methylanthracene		
	1-methylphenanthrene	16.61	192	191	0.629	1-methylphenanthrene		
C2-Phenanthrenes/ Anthracenes	Ethyl/dimethyl-phenanthrenes/anthracenes	17.22 - 17.95	206			Average of 3,6-Dimethylphenanthrene and 1,7-Dimethylphenanthrene	d10-Fluoranthene	17.99
	1,8-dimethylphenanthrene	18.10	206	191	0.365	Average of 3,6-Dimethylphenanthrene and 1,7-Dimethylphenanthrene		
C3-Phenanthrenes/ Anthracenes	(≥2 peaks)	18.40 - 19.40	220			1,2,6-Trimethylphenanthrene	d10-Fluoranthene	17.99
C4-Phenanthrenes/ Anthracenes	(≥2 peaks)	19.45 - 21.55	234			Retene	d10-Fluoranthene	17.99
C1-Fluoranthenes /Pyrenes	C1-Fluoranthenes/Pyrenes-1 (2 peaks)	19.17 - 19.58	216			3-Methylfluoranthene	d10-Fluoranthene	17.99
	C1-Fluoranthenes/Pyrenes-2 (3 or 4 peaks)	19.70 - 20.07	216			3-Methylfluoranthene		
C2-Fluoranthenes/ Pyrenes	(≥2 peaks)	20.50 - 21.50	230			3-Methylfluoranthene	d10-Fluoranthene	17.99
C3-Fluoranthenes/ Pyrenes	(≥2 peaks)	21.80 - 23.15	244			3-Methylfluoranthene	d10-Fluoranthene	17.99
C4-Fluoranthenes/ Pyrenes	(≥2 peaks)	23.20 - 24.50	258			3-Methylfluoranthene	d10-Fluoranthene	17.99
C1-Benz(a)anthracenes/ Chrysenes	(7 or more peaks)	22.60 - 23.38	242			Average of 5/6-methylchrysenes and 1-methylchrysene	d12-Chrysene	21.70
C2-Benz(a)anthracenes/ Chrysenes	(≥2 peaks)	23.70 - 25.00	256			5,9-Dimethylchrysene	d12-Chrysene	21.70
C3-Benz(a)anthracenes/ Chrysenes	(≥2 peaks)	25.20 - 26.20	270			5,9-Dimethylchrysene	d12-Chrysene	21.70
C4-Benz(a)anthracenes/ Chrysenes	(≥2 peaks)	26.20 - 27.90	284			5,9-Dimethylchrysene	d12-Chrysene	21.70
C1-Benzofluoranthenes/ Benzopyrenes	(≥2 peaks)	27.26 - 29.80	266			7-Methylbenzo(a)pyrene	d12-Benzo(a)pyrene	26.40
C2-Benzofluoranthenes/ Benzopyrenes	(≥2 peaks)	29.80 - 30.85	280			7-Methylbenzo(a)pyrene	d12-Benzo(a)pyrene	26.40

APPENDIX 3: ANALYSIS OF 2-CHLORONAPHTHALENE BY GC-HRMS

The analysis of 2-chloronaphthalene in solid and ash samples may be carried out by GC-HRMS analysis of the F2 extract cleanup fraction. Method MLA-021 applies for 2-chloronaphthalene analysis with the following modifications:

Quality Assurance/Quality Control:

QC Acceptance Criteria for 2-Chloronaphthalene

Compound	Procedural Blank Level (ng/sample)	OPR and Sample Recovery Range (%)
2-Chloronaphthalene	< 1	50 – 150
d7-2-Chloronaphthalene	n.a.	30 – 150

Cleanup procedures: Fraction F2 from the silica column cleanup is used for the analysis.

GC-HRMS Analysis: High resolution gas chromatography/high resolution mass spectrometry analysis of the F2 fraction is performed using a capillary gas chromatograph coupled to a high-resolution mass spectrometer. A J&W DB-5 chromatography column (60 m, 0.25 mm i.d., 0.10 µm film thickness)) is coupled directly to the HRMS source. The HRMS is operated at a static (8000) mass resolution (10% valley) in the electron ionization (EI) mode using multiple ion detection (MID) acquiring two characteristic ions for each target analyte and surrogate standard. Sample concentrations are determined by bracketing calibration using the SAR solution.

Analyte Ions Monitored, Surrogates Used and RRF Determination for 2-Chloronaphthalene Analysis

Compound	Quantification and RRT Standard	Typical Retention Time (min.)	Quant. Ion (m/z)	Conf. Ion (m/z)	Typical Ion Ratio (Quant./Conf.)	Ion Ratio Tolerance
2-Chloronaphthalene	d7-2-Chloronaphthalene	11:53	162.0236	164.0207	3.12	20 %
d7-2-Chloronaphthalene	d10-Acenaphthene	11:50	169.0676	171.0646	3.12	20 %
d10-Acenaphthene		13:21	162.1269	164.1410	1.1	20 %
Lock-mass			168.9888			

APPENDIX 4: QUANTIFICATION OF CLIENT, FIELD (PRC) AND PHOTOLYSIS STANDARDS

This section is applicable for determination of PAHs in PUF (polyurethane foam) and in SPMD (semipermeable membrane device) samples.

Because PUF and SPMD samples require the use of d_{10}-anthracene and d_{10}-fluoranthene as client or field standards, and SPMD samples require the use of d_{14}-dibenzo(ah)anthracene as photolytic standard, the default quantification scheme (the table between sections 4.1 and 5.0) must be modified as per the table below.

Analyte Ions Monitored, Surrogates Used and RRF Determination for PAH using Client, Field, PRC and Photolysis Standards

TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
Naphthalene	128	102	0.064	d_8-Naphthalene	6.84	Naphthalene
Acenaphthylene	152	151	0.222	d_8-Acenaphthylene	10.83	Acenaphthylene
Acenaphthene	154	153	1.18	d_8-Acenaphthylene	11.33	Acenaphthene
Fluorene	166	165	1.01	d_{10}-Phenanthrene	12.63	Fluorene
Phenanthrene	178	176	0.202	d_{10}-Phenanthrene	15.04	Phenanthrene
Anthracene	178	176	0.196	d_{10}-Phenanthrene	15.15	Anthracene
Fluoranthene	202	200	0.214	d_{10}-Phenanthrene	18.06	Fluoranthene
Pyrene	202	200	0.219	d_{10}-Phenanthrene	18.60	Pyrene
Benz[a]anthracene [6]	228	226	0.281	d_{12}-Benz[a]anthracene	21.68	Benz[a]anthracene
Chrysene [1]	228	226	0.312	d_{12}-Chrysene	21.79	Chrysene
Benzo[b]fluoranthene	252	253	0.218	d_{12}-Benzo[b]fluoranthene	25.21	Benzo[b]fluoranthene
Benzo[j,k]fluoranthenes	252	253	0.215	d_{12}-Benzo[k]fluoranthene	25.30	Benzo[k]fluoranthene
Benzo[e]pyrene	252	253	0.213	d_{12}-Benzo[a]pyrene	26.36	Benzo[e]pyrene
Benzo[a]pyrene	252	253	0.217	d_{12}-Benzo[a]pyrene	26.58	Benzo[a]pyrene
Perylene	252	253	0.212	d_{12}-Perylene	27.00	Perylene
Dibenzo[ah]anthracene [2]	278	139	0.144	d_{12}-Indeno[1,2,3,cd]pyrene	31.86	Dibenz[ah]anthracene
Indeno[1,2,3-cd]pyrene	276	138	0.179	d_{12}-Indeno[1,2,3,cd]pyrene	31.71	Indeno[1,2,3-cd]pyrene
Benzo[ghi]perylene	276	138	0.194	d_{12}-Benzo[ghi]perylene	32.53	Benzo[ghi]perylene

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
Biphenyl [3]	154	152	0.304	d_{10}- Biphenyl	9.81	Biphenyl
Dibenzothiophene [3]	184	152	0.073	d_8-Dibenzothiophene	14.72	Dibenzothiophene
1-Methylnaphthalene [3]	142	141	0.962	d_{10}-2-Methylnaphthalene	8.81	1-Methylnaphthalene
2-Methylnaphthalene [3]	142	141	0.930	d_{10}-2-Methylnaphthalene	8.55	2-Methylnaphthalene
C1-Naphthalenes [3]	142	[4]	[4]	d_{10}-2-Methylnaphthalene	[5]	1- & 2-Methylnaphthalene
2,6-Dimethylnaphthalene [3]	156	141	0.666	d_{12}-2,6 Dimethylnaphthalene	10.17	2,6-Dimethylnaphthalene
1,2-Dimethylnaphthalene	156	141	1.26	d_{12}-2,6 Dimethylnaphthalene	10.90	1,2-Dimethylnaphthalene
C2-Naphthalenes [3]	156	[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	2,6- & 1,2-Dimethylnaphthalene
2,3,5-Trimethylnaphthalene [3]	170	155	0.873	d_{12}-2,6 Dimethylnaphthalene	12.35	2,3,5- Trimethylnaphthalene
2,3,6-Trimethylnaphthalene	170	155	0.876	d_{12}-2,6 Dimethylnaphthalene	12.17	2,3,6- Trimethylnaphthalene
C3-Naphthalenes [3]	170			d_{12}-2,6 Dimethylnaphthalene	[5]	2,3,5- & 2,3,6-Trimethylnaphthalene
1,4,6,7-Tetramethylnaphthalene	184	139	0.027	d_{12}-2,6 Dimethylnaphthalene	13.89	1,4,6,7-Tetramethylnaphthalene
C4-Naphthalene	184	[4]	[4]	d_{12}-2,6 Dimethylnaphthalene	[5]	1,4,6,7-Tetramethylnaphthalene
2-Methylanthracene	192	191	0.531	d_{10}-Phenanthrene	16.45	2-Methylanthracene
3-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.27	1- & 2-Methylphenanthrene & 2-Methylanthracene
2-Methylphenanthrene	192	191	0.608	d_{10}-Phenanthrene	16.36	2-Methylphenanthrene
9/4-Methylphenanthrenes	192	191	0.634	d_{10}-Phenanthrene	16.59	1- & 2-Methylphenanthrene & 2-Methylanthracene
1-Methylphenanthrene [3]	192	191	0.634	d_{10}-Phenanthrene	16.64	1-Methylphenanthrene
C1-Phenanthrenes/Anthracenes [3]	192	[4]	[4]	d_{10}-Phenanthrene	[5]	1- & 2-Methylphenanthrene & 2-Methylanthracene
3,6-Dimethylphenanthrene [3]	206	191	0.342	d_{10}-Phenanthrene	17.46	3,6-Dimethylphenanthrene
2,6-Dimethylphenanthrene	206	191	0.342	d_{10}-Phenanthrene	17.54	3,6- & 1,7-Dimethyl-phenanthrenes
1,7-Dimethylphenanthrene	206	191	0.332	d_{10}-Phenanthrene	17.89	1,7-Dimethylphenanthrene
1,8-Dimethylphenanthrene	206	191	0.332	d_{10}-Phenanthrene	18.13	3,6- & 1,7-Dimethyl-phenanthrenes
C2-Phenanthrenes/Anthracenes [3]	206	[4]	[4]	d_{10}-Phenanthrene	[5]	3,6- & 1,7-Dimethyl-phenanthrenes
1,2,6-Trimethylphenanthrene	220	205	0.581	d_{10}-Phenanthrene	19.41	1,2,6-Trimethylphenanthrene
C3-Phenanthrenes/Anthracenes	220	[4]	[4]	d_{10}-Phenanthrene	[5]	1,2,6-Trimethylphenanthrene
Retene [3]	234	219	1.63	d_{10}-Phenanthrene	19.53	Retene

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
C4-Phenanthrenes/Anthracenes	234	[4]	[4]	d_{10}-Phenanthrene	[5]	Retene
C1-Biphenyls	168	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C2-Biphenyls	182	[4]	[4]	d_{10}- Biphenyl	[5]	Biphenyl
C1-Acenaphthenes	168	[4]	[4]	d_8-Acenaphthylene	[5]	Acenaphthene
2-Methylfluorene	180	165	1.23	d_{10}-Phenanthrene	14.06	2-Methylfluorene
C1-Fluorenes	180	[4]	[4]	d_{10}-Phenanthrene	[5]	2-Methylfluorene
1,7-Dimethylfluorene	194	177	0.092	d_{10}-Phenanthrene	15.49	1,7-Dimethylfluorene
C2-Fluorenes	194	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene
C3-Fluorenes	208	[4]	[4]	d_{10}-Phenanthrene	[5]	1,7-Dimethylfluorene
2/3-Methyldibenzothiophenes	198	197	0.738	d_8-Dibenzothiophene	16.07	2/3-Methyldibenzothiophenes
C1-Dibenzothiophenes	198	[4]	[4]	d_8-Dibenzothiophene	[5]	2/3-Methyldibenzothiophenes
2,4-Dimethyldibenzothiophene	212	197	0.514	d_8-Dibenzothiophene	17.08	2,4-Dimethyldibenzothiophene
4,6-Dimethyldibenzothiophene	212	197	0.160	d_8-Dibenzothiophene	16.94	4,6-Dimethyldibenzothiophene
C2-Dibenzothiophenes	212	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
C3-Dibenzothiophenes	226	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
C4-Dibenzothiophenes	240	[4]	[4]	d_8-Dibenzothiophene	[5]	2,4-Dimethyldibenzothiophene
3-Methylfluoranthene/Benzo(a)fluorene	216	215	0.880	d_{10}-Phenanthrene	19.53	3-Methylfluoranthene
C1-Fluoranthenes/Pyrenes	216	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
C2-Fluoranthenes/Pyrenes	230	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
C3-Fluoranthenes/Pyrenes	244	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
C4-Fluoranthenes/Pyrenes	258	[4]	[4]	d_{10}-Phenanthrene	[5]	3-Methylfluoranthene
5/6-Methylchrysenes	242	[4]	[4]	d_{12}-Chrysene	23.15	6-Methylchrysene
1-Methylchrysene	242	[4]	[4]	d_{12}-Chrysene	23.32	1-Methylchrysene
C1-Benz(a)anthracenes/Chrysenes	242	[4]	[4]	d_{12}-Chrysene	[5]	1- & 6-Methylchrysenes
5,9-Dimethylchrysene	256	[4]	[4]	d_{12}-Chrysene	24.49	5,9-Dimethylchrysene
C2-Benz(a)anthracenes/Chrysenes	256	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C3-Benz(a)anthracenes/Chrysenes	270	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
C4-Benz(a)anthracenes/Chrysenes	284	[4]	[4]	d_{12}-Chrysene	[5]	5,9-Dimethylchrysene
7-Methylbenzo(a)pyrene	266	[4]	[4]	d_{12}-Benzo[a]pyrene	29.35	7-Methylbenzo(a)pyrene
C1-Benzofluoranthenes/Benzo-pyrenes	266	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo(a)pyrene

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TARGET ANALYTES	Quantification Ion (m/z)	Confirmation Ions (m/z)	Typical Ion Ratio (Conf./Quant.)	SURROGATE	Typical Retention Time (minutes)	RRF DETERMINED FROM
C2-Benzofluoranthenes/Benzo-pyrenes	280	[4]	[4]	d_{12}-Benzo[a]pyrene	[5]	7-Methylbenzo(a)pyrene
LABELLED CLIENT/FIELD STANDARDS	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**	**Typical Ion Ratio (Conf./Quant.)**	**RECOVERY CALCULATED AGAINST**	**Typical Retention Time (minutes)**	**RRF DETERMINED FROM**
d_{10}-Anthracene	188	184		d_{10}-Phenanthrene	15.1	
d_{10}-Fluoranthene	212	208	0.173	d_{12}-Benz(a)anthracene	18.02	
LABELLED PHOTOLYSIS STANDARD	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**	**Typical Ion Ratio (Conf./Quant.)**	**RECOVERY CALCULATED AGAINST**	**Typical Retention Time (minutes)**	**RRF DETERMINED FROM**
d_{14}-Dibenzo[ah]anthracene	292	288	0.260	d_{12}-Indeno[1,2,3-cd]pyrene	31.75	
LABELLED SURROGATE STANDARDS	**Quantification Ion (m/z)**	**Confirmation Ions (m/z)**	**Typical Ion Ratio (Conf./Quant.)**	**RECOVERY CALCULATED AGAINST**	**Typical Retention Time (minutes)**	
d_8-Naphthalene	136	134	0.095	d_{10}-Acenaphthene	6.80	
d_{10}-2-Methylnaphthalene	152	151	0.195	d_{10}-Acenaphthene	8.47	
d_{10}-Biphenyl	164			d_{10}-Acenaphthene	9.75	
d_{12}-2,6-Dimethylnaphthalene	168	150	0.747	d_{10}-Acenaphthene	10.07	
d_8-Acenaphthylene	160	158	0.159	d_{10}-Acenaphthene	10.80	
d_8-Dibenzothiophene	192	160	0.085	d_{10}-Pyrene	14.67	
d_{10}-Phenanthrene	188	184	0.143	d_{10}-Pyrene	14.97	
d_{12}-Benz[a]anthracene	240	236	0.250	d_{10}-Pyrene	21.63	
d_{12}-Chrysene	240	236	0.278	d_{10}-Pyrene	21.73	
d_{12}-Benzo[b]fluoranthene	264	260	0.216	d_{12}-Benzo[e]pyrene	25.11	
d_{12}-Benzo[k]fluoranthene	264	260	0.208	d_{12}-Benzo[e]pyrene	25.23	
d_{12}-Benzo[a]pyrene	264	260	0.216	d_{12}-Benzo[e]pyrene	26.47	
d_{12}-Perylene	264	260	0.256	d_{12}-Benzo[e]pyrene	26.88	
d_{12}-Indeno[1,2,3,cd]pyrene	288	284	0.192	d_{12}-Benzo[e]pyrene	31.63	
d_{12}-Benzo[ghi]perylene	288	284	0.205	d_{12}-Benzo[e]pyrene	32.45	

[1] Coelutes with triphenylene

[2] Coelutes with dibenz[ac]anthracene

[3] These compounds are in addition to the regular suite of analytes, and are analyzed by client request only.

[4] Secondary ion confirmation procedures do not apply

[5] RRT ranges apply to alkylated PAH Totals

[6] Benz(a)anthracene coelutes with cyclopenta(cd)pyrene, which may contribute response in the second ion to cause a failing ion abundance ratio. Therefore quantification of benz(a)anthracene should use response from the first ion only

ATTACHMENT C

Typical Detection Limits, Method Detection Limits and Low Calibration Limits for Parent PAHs and Alkylated PAHs by GC/MS

Attachment C

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TYPICAL DETECTION LIMITS, METHOD DETECTION LIMITS AND LOW CALIBRATION LIMITS for Parent PAHs and Alkylated PAHs by GC/MS

SGS AXYS Method: MLA-021
Instrument Type: Low Resolution GC/MS
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B Rev.1 (or * = MDLs determined according to Rev. 2, [2017])
Quantification: SGS AXYS default is linearity. Full bracketing calibration option is available

Matrix		**SOIL/SEDIMENT/BIOSOLIDS**				**TISSUE**			
Units/Sample Size		ng/g based on 10g dry weight sample				ng/g based on 10g sample			
Default Extract Volume		500uL				100uL			
Analyte ***List 1 - Standard PAH Parents and Select Alkylated PAHs determined by linearity method***	**Analyte Type**	**Typical SDL**	**MDL ***	**LOQ**	**LMCL based on Low Cal.**	**Typical SDL**	**MDL ***	**LOQ**	**LMCL based on Low Cal.**
Naphthalene	LMW	0.5	1.18	2.96	2.5	0.1	0.646	0.7	0.7
Acenaphthylene	LMW	0.5	0.49	2.95	2.5	0.1	0.099	0.5	0.5
Acenaphthene	LMW	0.5	0.43	3.01	2.5	0.1	0.119	0.5	0.5
Fluorene	LMW	0.5	0.50	2.95	2.5	0.1	0.286	0.5	0.5
Phenanthrene	LMW	0.5	0.97	3.03	2.5	0.1	0.473	0.5	0.5
Anthracene	LMW	0.5	0.33	3.01	2.5	0.1	0.099	0.5	0.5
Fluoranthene	HMW	0.5	0.31	2.97	2.5	0.1	0.144	0.5	0.5
Pyrene	HMW	0.5	0.36	2.97	2.5	0.1	0.144	0.5	0.5
Benz(a)anthracene	HMW	0.5	0.51	3.02	2.5	0.1	0.094	0.5	0.5
Chrysene	HMW	0.5	0.22	3.01	2.5	0.1	0.076	0.5	0.5
Benzo(b)fluoranthene	HMW	0.5	0.63	3.02	2.5	0.1	0.101	0.5	0.5
Benzo(j/k)fluoranthenes	HMW	0.5	0.25	3.02	2.5	0.1	0.093	0.5	0.5
Benzo(e)pyrene	HMW	0.5	0.60	3.00	2.5	0.1	0.187	0.5	0.5
Benzo(a)pyrene	HMW	0.5	0.34	3.00	2.5	0.1	0.076	0.5	0.5
Perylene	HMW	1.0	0.28	3.00	2.5	0.2	0.070	0.5	0.5
Dibenzo(ah)anthracene	HMW	1.0	0.30	2.96	2.5	0.2	0.094	0.5	0.5
Indeno(1,2,3-cd)pyrene	HMW	1.0	0.29	3.02	2.5	0.2	0.194	1.0	1.0
Benzo(ghi)perylene	HMW	1.0	0.27	2.96	2.5	0.2	0.077	0.5	0.5
2-Methylnaphthalene	LMW	1.0	0.70	2.99	2.5	0.2	0.366	0.5	0.5
2,6-Dimethylnaphthalene	LMW	1.0	0.52	3.01	2.5	0.2	0.109	0.5	0.5
2,3,5-Trimethylnaphthalene	LMW	1.0	1.36	3.00	2.5	0.2	0.217	0.5	0.5
1-Methylphenanthrene	LMW	1.0	0.77	3.00	2.5	0.2	0.131	0.5	0.5
Dibenzothiophene	LMW	1.0	0.29	3.00	2.5	0.2	0.125	0.5	0.5

TYPICAL DETECTION LIMITS, METHOD DETECTION LIMITS AND LOW CALIBRATION LIMITS for Parent PAHs and Alkylated PAHs by GC/MS

SGS AXYS Method: MLA-021
Instrument Type: Low Resolution GC/MS
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B Rev.1 (or * = MDLs determined according to Rev. 2, [2017])
Quantification: SGS AXYS default is linearity. Full bracketing calibration option is available

List 2 - Alkylated PAHs determined by linearity method		**Typical SDL**	**MDL ***	**LOQ**	**LMCL based on Low Cal.**	**Typical SDL**	**MDL ***	**LOQ**	**LMCL based on Low Cal.**
1-Methylnaphthalene	LMW	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C1-Naphthalenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
1,2-Dimethylnaphthalene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C2-Naphthalenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
2,3,6-Trimethylnaphthalene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C3-Naphthalenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
1,4,6,7-Tetramethylnaphthalene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C4-Naphthalenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
2-Methylphenanthrene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
3-Methylphenanthrene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
9/4-Methylphenanthrenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
2-Methylanthracene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C1-Phenanthrenes/Anthracenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
1,7-Dimethylphenanthrene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
1,8-Dimethylphenanthrene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
2,6-Dimethylphenanthrene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
3,6-Dimethylphenanthrene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C2-Phenanthrenes/Anthracenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
1,2,6-Trimethylphenanthrene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C3-Phenanthrenes/Anthracenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
Retene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C4-Phenanthrenes/Anthracenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
Biphenyl	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5

TYPICAL DETECTION LIMITS, METHOD DETECTION LIMITS AND LOW CALIBRATION LIMITS for Parent PAHs and Alkylated PAHs by GC/MS

SGS AXYS Method: MLA-021
Instrument Type: Low Resolution GC/MS
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B Rev.1 (or * = MDLs determined according to Rev. 2, [2017])
Quantification: SGS AXYS default is linearity. Full bracketing calibration option is available

List 3 - Extended alkylated PAHs determined by linearity		**Typical SDL**	**MDL ***	**LOQ**	**LMCL based on Low Cal.**	**Typical SDL**	**MDL ***	**LOQ**	**LMCL based on Low Cal.**
C1-Biphenyls	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
C2-Biphenyls	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
C1-Acenaphthenes	Alkylated PAHs	0.5	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
2-Methylfluorene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C1-Fluorenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
1,7-Dimethylfluorene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C2-Fluorenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
C3-Fluorenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
2/3-Methyldibenzothiophenes	Alkylated PAHs	1.0	NA [3]	2.5	2.5	0.2	NA [3]	0.5	0.5
C1-Dibenzothiophene	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
2,4-Dimethyldibenzothiophene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C2-Dibenzothiophene	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
C3-Dibenzothiophene	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
C4-Dibenzothiophene	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
3-Methylfluoranthene/Benzo(a)fluorene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C1-Fluoranthenes/Pyrenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
C2-Fluoranthenes/Pyrenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
C3-Fluoranthenes/Pyrenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
C4-Fluoranthenes/Pyrenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
1-Methylchrysene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
5/6-Methylchrysenes	Alkylated PAHs	1.0	NA [3]	2.5	2.5	0.2	NA [3]	0.5	0.5
C1-Benz(a)anthracenes/Chrysenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
5,9-Dimethylchrysene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C2-Benz(a)anthracenes/Chrysenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
C3-Benz(a)anthracenes/Chrysenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
C4-Benz(a)anthracenes/Chrysenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
7-Methylbenzo(a)pyrene	Alkylated PAHs	1.0	NA	2.5	2.5	0.2	NA	0.5	0.5
C1-Benzofluoranthenes/Benzopyrenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]
C2-Benzofluoranthenes/Benzopyrenes	Alkylated PAHs	1.0	NA [3]	NA [4]	NA [4]	0.2	NA [3]	NA [4]	NA [4]

Note: Sample Detection limits (SDL) provided are for demonstration purposes only.

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DEFINITIONS

Method Detection Limit (MDL) - determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B (Rev.1 or Rev.2, as documented for each method/matrix). MDL is determined as required based on accreditation, contract and workload requirements**. The MDL determination is a general demonstration of method detection limit. It is performed at a particular time, using a set of sample prepared using clean matrix, and may not account for all matrix effects encountered in environmental samples.**

Sample Detection Limit (SDL) – determined by converting the area equivalent of 3.0 times (2.5 times for EPA 1600 series methods) the estimated chromatographic noise height to a concentration in the same manner that target peak responses are converted to final concentrations. Determined individually for every sample analysis run. The SDL accounts for any effect of matrix on the detection system and for recovery achieved through the analytical work-up. **It does not account for any lab background input.**

US DoD Detection Limit (DL) (only applied to work under US DoD accreditation) - the detection limit applicable when target compounds are detected, must be greater than the method MDL

US DoD Limit of Detection (LOD) (only applied to work under US DoD accreditation) - the detection limit reported when target compounds are not detected, 2-4 times higher than the US DoD DL

Reporting Limit (RL) – the **lowest** concentration value that AXYS routinely reports for the method. AXYS defines RLs for LC analyses as equal to the greater of lowest calibration standard or the SDL. For GC methods, RLs are equal to the SDL, or a value greater than the SDL determined to meet client needs.

Lower Method Calibration Limit (LMCL) - determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used. ((lowest level cal conc.) x (extract volume))/sample size

Quantification by Multi-level Calibration (Constant RRF or Regression)- A multi-point calibration series (linearity) is analyzed at a frequency that is determined by the method. Prior to analyzing samples, the mid-point calibration standard (CALVER) is analyzed. If the CALVER meets method acceptance criteria, demonstrating that the instrument is in a state of control, Relative Response Factors (RRFs) from the linearity are used for quantification. Quantification by linearity is appropriate for analyses where analytes are not comprised of mixtures or patterns and where there is not a large disparity in abundance of compounds.

Quantification by Single Level Bracketing Calibration - Bracketing calibration uses RRFs that are generated from analyzing a calibration standard immediately before the sample is run and confirmed immediately afterwards. These two must be within +/- 20% and the mean RRF of these two is used to quantify the analytes. A sensitivity calibration is also analyzed to ensure sensitivity of the instrument. Bracketing calibration quantification produces data with increased accuracy at a sample and batch level. Bracketing calibration quantification is appropriate for methods where analytes are comprised of patterns and for tests where there is a disparity in the abundance of compounds, such as sterols and hormones, as it allows for better control of analytes. A linearity has limited applicability for tests which are comprised of patterns or mixtures such as PAHs, Alkylphenols, Toxaphene and Naphthenic Acids, because the lower calibration standards will lose response of the minor peaks from the pattern.

Limit of Quantification (LOQ) - LOQ is the lowest concentration, at or above the LMCL, at which test accuracy (precision and bias) has been demonstrated. LOQs are established by analysis of replicate low level spiked samples of clean matrix. Alternatively, for tests not subject to NELAP/TNI or US DOD accreditation, LOQ may be defined as equal to the LMCL.

For NELAP/TNI and DoD accredited tests LOQs are verified quarterly or for each analysis batch on a schedule managed by the laboratory. The precision and bias of the method at the LOQ is included in data packages under DoD accreditation; for any other work the precision and bias at the LOQ is made available to clients and reported in data packages upon request.

Concentration results reported below the LOQ are flagged to denote increased quantitative uncertainty for work subject to NELAP/TNI or US DoD accreditation, or when requested by the client.

APPENDIX B

Summary of SGS Axys Standard Operating Procedures for Analysis

SGS AXYS Analytical Services Ltd.
Standard Operating Procedure

Purpose:

To determine the moisture content (expressed as a percentage of sample weight) of a solid or tissue sample in order to calculate a dry weight equivalent from a wet sample weight.

Scope:

These procedures are applicable to percent moisture determinations performed on tissue (including wood, vegetation), sediment, soil, ash, sludge and pulp samples. When available sample is limited, “micro-moisture” procedures, described herein, are carried out.

Although samples are usually analyzed wet, analytical results may be expressed on a wet or dry weight basis depending on the sample matrix and/or clients' request. Determination of moisture content allows the sample results to be reported on a dry weight basis even though the sample has been analyzed wet. Percent moisture is reported with the analytical data.

Drying oven temperatures are recorded immediately prior to use and immediately after use.

Solids/Sediment/Soil: Moisture determination is performed in duplicate on all solid samples (including freeze-dried sediments) with every analysis. The average of the duplicate moisture determinations is reported to the client.

Tissues: Moisture determination is performed on tissue samples upon client request. The moisture determination is carried out only once, even if the sample is analyzed for more than one set of contaminants by more than one method. Duplicate moisture determinations are not required, as tissue samples have been found to be highly homogenous. The Project Manager selects which analysis area performs the moisture determination.

Equipment and Materials:

- Drying oven (Fisher Isotemp, model 13-247-725F, or equivalent), with calibrated thermometer.
- Top loading balance (2 and 3-place)
- Aluminum weigh boats
- Spatulas (solvent-rinsed)
- Desiccator Cabinet (Boekel Scientific, mod. 134441), 110 L, with stainless steel shelves and automatic desiccant regeneration, or equivalent

Procedures:

Refer to SOP SLA-023 *Use of Balances* for instructions on using the balance. Refer to SOP SLA-022 *Use of Drying Ovens and Muffle Furnace* for instructions on using the drying oven.

I. % MOISTURE AS PART OF AN ANALYSIS

1. Refer to SLA-079 *Agricultural Hazard Protocols for Soils* and SLA-082 *Handling of Human Biohazardous Samples* for handling procedures for agricultural and biohazardous samples.
2. Refer to the Batch List for instructions regarding moisture determination. If “micro-moisture” procedures are indicated, follow instructions in Section III. Consult the Lab Supervisor or Project Manager if upon examination of the sample, it is determined there is insufficient sample for a routine moisture determination.
3. Using a lab marker pen, label an aluminum weigh boat with the SGS AXYS sample ID. Weigh the aluminum weigh boat and record the weight on the analysis worksheet or capture in LIMS. Record weights to two decimal places. Follow SLA-152 *Electronic Capturing of Balance Readings* for data entry.
4. Using a solvent-rinsed spatula, transfer a 1-5 g ***** (dependent on the moisture content and the amount of available sample) subsample from a well-stirred, homogenized sample into a tared weigh boat **. Accurately weigh the sample and record the weight on the sample worksheet or capture in LIMS. Carry this step out in duplicate for solid samples. Place all the weigh boats onto a disposable aluminum tray and cover with aluminum foil. Label with the workgroup ID and date on the foil.
   - ***** If samples are >50% water, as determined by the Sample Preparation group, and there is sufficient sample, use a 5 g subsample.
   - ****** If there is limited sample available, or if it is requested in the Project Notes, a micro moisture procedure may be carried out. Refer to Section III.
5. Just before placing the sample(s) into the drying oven verify that the oven temperature is 105°C ±5°C and record the temperature on the analysis worksheet.
6. Dry the subsample in the drying oven at 105 °C in oven for 16 - 24 hours. Record the date and time that the sample went into the oven on the analysis worksheet.
7. Just before removing the sample(s) from the oven record the oven temperature on the analysis worksheet.
8. Remove the subsample from the oven and record the date and time on the analysis worksheet. Place the sample in a desiccator, record entry into logbook LBK-188 *Desiccator Tracking*

Logbook, and allow it to cool to room temperature.

9. Weigh the dried subsample and aluminum weigh boat. Record the weight on the analysis worksheet or capture in LIMS.

10. Enter the percent moisture data into LIMS as described in Step 11 below. Do not dispose of the weigh boat with dried material until the calculations have completed and either all of the samples have passed (the RPD between the two determinations must be ≤20%) or the Lab Supervisor or Project Manager has been informed of any failures. Refer to SLA-079 *Agricultural Hazard Protocols for Soils*, SLA-082 *Handling of Human Biohazardous Samples*, and SAD-014 *Sample Disposal* for the disposal procedures.

11. To enter the data, log into LIMS → Sample Management → Workstat → Moist/TSS/Lipid. Enter the WG, select moistures and press OK. For each sample enter the extraction date, sample size, tare weight, tare + wet weight and tare + dry weight. The spreadsheet calculates the sample dry weight, the percent moisture, percent solids and relative percent difference (RPD) between duplicates (where applicable). Once all data is entered, press Save. To print a copy of the spreadsheet, press Print and an Excel spreadsheet will open. When asked to save press Cancel. Print this sheet and then close the spreadsheet without saving. The mass data summary screen can now be closed. Attach the printed results to the Batch List.

12. Where applicable, for sediments and soils, compare the percent moisture with the percent moisture calculated by the Sample Preparation group. The relative percent difference (RPD) between the mean percent moisture determined in the extraction lab and the mean percent moisture determined by the Sample Preparation group should be ≤20%. Report instances of poor replication to the Lab Supervisor who will assess the need for corrective action. Do not continue with the analysis until the Lab Supervisor has reviewed the data and determines a course of action.

13. The calculations carried out by the spreadsheet are as follows:

<u>Percent Moisture</u>

Wet wt of sample = (wt of wet sample and weigh boat) – (wt of weigh boat)
Dry wt of sample = (wt of dry sample and weigh boat) – (wt of weigh boat)
% moisture = [1-(dry wt of sample/wet wt of sample)] x 100

<u>Sample Dry Weight</u>

Sample Dry Wt = Wt. of Wet Sample for extraction x (dry wt of sample/wet wt of sample)

Relative Percent Difference (RPD)

$$RPD = \frac{|Result\ 1 - Result\ 2|}{(Result\ 1 + Result\ 2)/2} \times 100$$

II. INDEPENDENT % MOISTURE DETERMINATION, PRIOR TO ANALYSIS

The results are used to determine the size of the subsample used for analysis, where a specified dry weight is required.

1. Carry out the moisture analysis as described above, recording all weights and analysis information on the worksheet FWO-306 *Sample Weight and Moisture* or capture in LIMS. Do not carry out the analysis in duplicate. Enter the percent moisture as described in Section I Verify the entry of the data into the spreadsheet and make any necessary corrections. Document the verification on the bottom of the worksheet. Attach the printout of the calculated moistures to the worksheet.

III. LIMITED SAMPLE – MICRO-MOISTURE PROCEDURES

These procedures are to be used when the amount of sample is limited. Typically, the Batch List indicates if 'micro-moisture" procedures are to be carried out. Carry out the moisture analysis as described in Section I with the following modifications:

1. Use a balance capable of weighing to 3 decimal places and record or capture in LIMS all weights to 3 decimal places.
2. Weigh out subsamples that are between 100 and 500 mg.

IV. SINGLE MOISTURE DETERMINATION FOR MULTIPLE WORKGROUPS

These procedures may be used if there is one sub-sampling event for multiple analyses performed in separate workgroups (i.e. if the sub-sampling for extraction is carried out at the same time by the same analyst for multiple workgroups.) The moisture determination may be done once and the data copied in LIMS from one workgroup to another.

1. Carry out the moisture analysis as described in section I, recording all weight and analysis

information on the worksheet or capture in LIMS. For the workgroup(s) that will use copied moisture data record on the worksheet the workgroup number of the original moisture determination.

2. Enter moistures and extraction sub-sample weight into LIMS as described in section I. Save and print the Excel spreadsheet.
3. For subsequent workgroups, from the Moist/TSS/Lipid screen in LIMS, click the “Copy to new WG” radio button and enter the new workgroup number.
4. The extraction sub sample weight must be entered for the new workgroup, the moisture data will be copied automatically. Save and print the Excel spreadsheet – the moisture determination data will not appear on this spreadsheet, only the wet and dry weights of the extraction sub sample will appear.

References:

SLA-022	Use of Drying Ovens and Muffle Furnace
SLA-023	Use of Balances
SLA-079	Agricultural Hazard Protocols for Soils
SLA-082	Handling of Human Biohazardous Samples
SLA-152	Electronic Capturing of Balance Readings
SAD-014	Sample Disposal
FWO-306	Sample Weight and Moisture
LBK-188	Desiccator Tracking Logbook

Approval:

Approved 29-Oct-2018	John Cosgrove, Vice President and Senior Technical Director
	Shea Hewage, Director of Operations
	Dale Hoover, QA Manager

SGS AXYS Analytical Services Ltd.

Standard Operating Procedure

Purpose:

To determine the lipid content (expressed as a percent of sample weight) of a tissue sample.

Scope:

When required by contract, a lipid determination on a tissue sample is performed on a tissue sample extract, prior to any chromatographic clean-up procedures. A Chemist working in the Extraction Lab performs the analysis. The percent lipid determination for a tissue sample is carried out in duplicate on the same extract. The Project Manager selects the analysis in which the lipid determination is carried out if more than one analysis is being performed. The data (sample weights) are entered into LIMS and then, a spreadsheet (FWO-205 *Lipid Calculation Spreadsheet*) calculates the results. The data are saved in the LIMS (Laboratory Information Management System).

Equipment and Materials:

Top loading balance – accurate to 2 decimal places
Analytical balance – accurate to 4 decimal places
Drying oven (Fisher Isotemp Oven 200 Series, Lab-Line Heet-Cab No.3515) maintained at 105 °C, with calibrated thermometer.
Solvents - high purity, distilled in glass, either HPLC grade or pesticide residue grade. Each lot number of solvent must be checked for impurities by performing a solvent proof prior to use
Rotary evaporator - equipped with a water bath
Pasteur pipettes
Glass Petri dishes

Glassware Cleaning

All glassware must be organically clean. Glassware must be washed and baked using standard operating procedures (SLA-001 *Cleaning and Baking Glassware and Disposal of Laboratory Waste Solvent and Material*). If baked glassware is not available, glassware must be washed and solvent rinsed following standard operating procedures (SLA-018 *Solvent Rinsing of Glassware and Equipment for Organic Analysis*).

Procedure:

Record all weights on the sample worksheet or capture in LIMS

1. Prior to beginning the sample extraction, weigh each *labelled* round-bottom flask (including anti-bumping granules) that will be used to collect the sample extract on a top loading balance.

Record or capture each weight to two decimal places. Follow SLA-152 *Electronic Capturing of Balance Readings* for data entry.

2. If sodium sulphate is present in the extract after the extraction procedure has completed, carefully transfer the extract with rinses to another weighed, labelled round-bottom flask ensuring that no sodium sulphate or anti-bumping granules are transferred. Use hexane rinses for extracts in hexane or in 1:1 hexane:dichloromethane. Use dichloromethane for extracts in 100% dichloromethane.
3. When the extraction procedure is complete, concentrate the extract to 10-20 mL using rotary evaporation.
4. Label and accurately weigh two prebaked glass Petri dishes on the analytical balance. Record or capture the weight of each dish to four decimal places.
5. Allow the flask with the extract to achieve room temperature. Ensure the outside of the flask is dry. Place the flask on a top loading balance.
6. Add sufficient solvent* to bring the weight of the extract to 30 g. Record or capture the weight of the extract (Wte) to two decimal places.

   * Add hexane to extracts that are in hexane or in 1:1 hexane:dichloromethane.
   Add dichloromethane to extracts in 100% dichloromethane.

7. Place a Petri dish on the top loading balance and tare to zero. Transfer a 2 g subsample of extract to the Petri dish using a glass Pasteur pipette and bulb. Record or capture the weight (Wle) of the extract to two decimal places
8. Repeat Step 7 with the second Petri dish and subsample. The extract has now been subsampled twice, one subsample in each Petri dish.
9. Place the Petri dishes with the extracts in a fumehood at room temperature until the solvent is evaporated, approximately 15 minutes.
10. Just before placing the Petri dishes into the drying oven verify that the oven temperature is 105°C±5°C and record the temperature on the analysis worksheet.
11. Place the Petri dishes in the drying oven at 105 °C for 30 minutes.
12. Just before removing the Petri dishes from the oven record the oven temperature on the analysis worksheet.
13. Remove the Petri dishes from the oven and allow to cool.
14. Weigh the Petri dishes on the analytical balance. Record or capture the weight of the dish with lipid to four decimal places.
15. Concentrate the remaining 26 g of extract using the same procedure that would be used if lipid analysis on the extract had not been required.

Calculation of Results

Calculation of lipid content is completed by entering the recorded weights from FWO-308 *Lipid Sheet* into LIMS or capturing recorded weights in LIMS, which generates an Excel spreadsheet. The results for all samples in the batch are saved, printed and attached to the Batch List.

1. Login to LIMS, Sample Management, Workstat, and click the Moist/SS/Lipid radio button.
2. Enter the WG and select lipid. Click OK.
3. Clicking on the first line of the data sheet will pop up a data entry screen. For each sample, fill in the weighing data in the spreadsheet fields displayed in the Workup Sheet Information window and click on "OK". Repeat for all samples. OK will move to the next sample for data entry.
4. The spreadsheet calculates the above values using the formulas below. These can be used to double-check the calculated values if necessary. The spreadsheet displays the data entered and results for:

   - Extract weight;
   - Dry lipid;
   - Percent lipid for each aliquot and average percent lipid;
   - Duplicate test results;
   - Recovery correction factor;

   To calculate lipid weight:

   $$W_l = \text{(weight of dish and lipid)} - \text{(weight of dish)}$$

   To calculate the percent lipid:

   $$\%lipid = \frac{W_l}{W_s} \times \frac{W_{te}}{W_{le}} \times 100$$

   where W_l = weight of lipid (above);
   W_s = weight of sample taken for analysis;
   W_{te} = weight of total extract (Step 4); and
   W_{le} = weight of extract for lipid analysis (Step 6).

5. Average the percent lipid from the two analyses.

   To calculate the lipid factor:

$$Factor = \frac{W_{te}}{W_{te} - W_{le}}$$

where W_{te} = weight of extract
W_{le} = weight of both lipid extracts

To calculate Relative Percent Difference (RPD)

$$RPD = \frac{|\text{Result 1} - \text{Result 2}| \times 100}{\frac{(\text{Result 1} + \text{Result 2})}{2}}$$

6. Review the data for duplicate lipid determinations. Any discrepancies in entered data (i.e., {Dry Lipid + Tare} < Tare) are highlighted with a colour code on the spreadsheet. Duplicate test results with a RPD greater than 20% are also highlighted. Report RPD differences larger than 20% to the Lab Supervisor and/or Project Manager and halt the extraction procedures until a course of action has been determined. Historically, if the mean lipid weight is not greater than 0.0015 g, then the 20% RPD specification should not be expected to be met.
7. If the raw data has been entered correctly, click on “Save Samples”. The results are saved under the workgroup number in the LIMS database. A copy of the spreadsheet is printed at the default printer.

References:

SLA-001	Cleaning and Baking Glassware and Disposal of Laboratory Waste Solvent and Material
SLA-018	Solvent Rinsing of Glassware and Equipment for Organic Analysis
SLA-152	Electronic Capturing of Balance Readings
FWO-205	Lipid Calculation Spreadsheet)
FWO-308	Lipid Sheet

Approval:

Approved 27-May-2019	Shea Hewage, General Manager
	Dale Hoover, Senior Technical Director
	Rhonda Stoddard, QA Manager

APPENDIX C

Field Forms

Navassa OU2 Soil and Invertebrate Sampling

Location ID: _____

Start Date:_____ Time:_____ End Date:_____ Time:_____

Sampling Team:_____

Targeted PAH Concentration Range Sampled:_____

Was Polygon Expanded? (Y/N):_____

Other:_____

Weather

Weather (circle all that apply): heavy rain / overcast / steady rain / partly cloudy / intermittent rain / clear-sunny / Other_____

Inches of rain in the last 24 hours: _____ Temperature:_____

Other notes regarding weather:_____

Composite Invertebrate Samples

Surface/Aboveground-dwelling Sample:

Sample ID:_____ Final Sample Weight (10 g min):_____

Undepurated Soil-Dwelling Sample Weight (10 g min):_____

Sample ID:_____ Final Sample Weight (10 g min):_____

Depurated Subsample (if Sufficient Weight to Allow for This) (Yes/No?):_____

Sample ID:_____

Date/Time and Weight Start of Depuration:_____

Date/Time and Weight End of Depuration:_____

Composite Soil Sample

Sample ID:_____

Date and time: _____

Sampling personnel: _____

Methodology: _____

Soil description (0-6” bgs): _____

_____

Location ID:_____

Invertebrates Subsamples of Composite

Invert Type (Circle): Aboveground, surface/leaf, or soil

GPS (if needed):_____

Date/Time: _____

Habitat Description (general):_____

Methodology: _____

Species collected: _____

Notes: _____

_____

_____

Invert Type (Circle): Aboveground, surface/leaf, or soil

GPS (if needed):_____

Date/Time: _____

Habitat Description (general):_____

Methodology: _____

Species collected: _____

Notes: _____

_____

_____

Invert Type (Circle): Aboveground, surface/leaf, or soil

GPS (if needed):_____

Date/Time: _____

Habitat Description (general):_____

Methodology: _____

Species collected: _____

Notes: _____

_____

_____