Experimental Design for PCB Pilot Study
2Apr08 Draft Prepared by Deana Crumbling, USEPA TIFSD
(Note: all tables & figures are at the end of this document)

USEPA TIFSD Soil PCB PILOT Study

1.0 Purpose of this Pilot Study

1.1 This pilot study will evaluate the PCB and TEQ-relevant analyte concentrations of the soils in each of the 20 1-kg bags and selected extracts. This information will be used to prepare the 4 concentration levels for use in the main study; and refine the experimental design of the main study plan and reduce the number of expensive analyses if sufficient analyte is not present at detection limits in some bagged samples.

1.2 The pilot study will also provide a preliminary assessment of the amount of within-bag matrix heterogeneity and the sample processing effort needed to control it.

1.3 The pilot offers the chance for the operator to become familiar with the lab space and supplies; and with the Abraxis immunoassay kits.

1.4 The study will also provide PCB results to the KRCEE effort to allow them to assess short-scale matrix heterogeneity (over distances of 1 meter), and allow them to assess whether a predictive relationship exists between uranium concentration and PCB concentrations.

2.0 Pilot Study Structure

2.1 Overview

Each of the 20 1-kg sample bags of Paducah soil will be sampled using a multi-increment (MI) strategy. These MI samples will be analyzed for total PCBs by the Abraxis PCB-HC kit. Processed soil samples will also be pooled to create sufficient volume for analysis for PAHs and PCB Aroclors by the ERT-Edison laboratory. Methanol extracts from Abraxis extractions will be analyzed by the ERT-Edison lab, the Abraxis lab (using PCB-LC and co-planar kits), and the XDS laboratory for TEQ analytes (D/F, co-planar PCBs, and PAHs).

2.2 Detailed Pilot Study Plan

2.2.1 Soil and Extract Processing and Analysis

Refer to Table 1 and Figure 1. The following text describes the activities to be performed in the Las Vegas laboratory for soil processing, soil analysis, and extract processing & analysis.

2.2.1.1. Soil Sample Processing in the Las Vegas ORD Lab

All 20 1-kg bags of Paducah soil will be processed and analyzed. These 20 bags represent 5 positions from each of 4 locations assumed to have PCB concentrations that range from background (non-detect?) to medium and high concentrations.

- Each 1-kg bagged sample will be emptied into a lab pan and spread to an even layer to facilitate cleaning it of extraneous material. It will also be moderately disaggregated (to mimic minimal field sample preparation).
- Under the guidance of John Nocerino, each 1-kg sample (in its pan) will then be subsampled twice using MI sampling and a Gy-correct technique to create two distinct 25-g soil subsamples (designated as "MIS" in Figure 1) intended to be representative of the bulk average for the 1-kg sample. Therefore, there will be a total of 5 pairs of MIS subsamples (10 MISs) per Paducah sampling location.
- Also under the guidance of John Norcerino, each 25-g MIS will be further processed to reduce matrix heterogeneity within the 25-g jarred subsample, possibly by using a mortar and pestle with sieving, which is easily adapted to field analysis.
- Each 25-g jarred sample will then be subsampled to create a 10-g analytical sample, which will be extracted using the Abraxis extraction reagents. An extract is designated with an "E" in Figure 1. The extract will be analyzed with the Abraxis PCB-HC kit.
- The Abraxis PCB-HC results for each pair of 25-g subsamples (a pair is from the same 1-kg bag) will be assessed for precision (using %RPD or another appropriate precision measure). The pair with the poorest duplicate precision in each location group will be selected to undergo further evaluation to ascertain the source of data variability.
- The variability evaluation will be conducted as illustrated in Figure 1. The additional samples & analyses for the variability evaluation are halo'd in purple. Both 25-g jars will again be subsampled for a 10-g analytical sample, which will be extracted and analyzed using the Abraxis PCB-HC kit.
- The remainder of the prepared soils from the location-specific MIS jars will be pooled and thoroughly mixed. Four pooled soils (one for each of the Paducah locations) will be prepared. The pooled soil samples will be Gy-correctly split (under the direction of John Nocerino) into 4 30-g jarred aliquots.
- Two of the pooled jars from each location will be analyzed with the Abraxis HC kit in the Las Vegas Lab. The other 2 pooled jars will be sent to the EPA Edison Lab.

2.2.1.2. Soil Sample Processing in the EPA ERT-Edison, NJ Lab

The replicate pooled soil jars (2 per location for a total of 8 jars) that are sent to the ERT lab will be analyzed for PAHs, PCB Aroclors and total PCBs. These data will be used to determine what potentially confounding analytes are in the Paducah soil samples, and their approximate concentrations. This information will be used to streamline the main study, and for estimating the Abraxis detection capability. If any of the pooled samples

are of an appropriate PCB concentration, it may also be possible to estimate the detection capabilities of the different methods being evaluated in the study. The results will be reported to Deana Crumbling.

2.2.1.3. Additional Abraxis Extract Processing in the Las Vegas ORD Lab

Each Abraxis extraction of a soil sample will generate about 15 mL of recoverable extract. After analysis, extracts generated from a single location (including the 2 pooled soil sample analyses) will be pooled. This will generate roughly 210 mL of pooled extract per location. A pooled extract will be aliquoted into 2 40-mL potions, 1 60-mL portion, and 1 20-mL portion (a total of 160 mL). The remainder of the 4 pooled extracts will be archived.

2.2.1.4. Abraxis Extract Processing in the EPA ERT-Edison, NJ Lab

Eight sample extracts in MeOH will be submitted to the ERT lab for PAH, PCB Aroclors and total PCB analyses. In conjunction with the pooled soil samples, this information will help determine whether and how much of these analytes may be left behind in the Paducah soils by the Abraxis MeOH extraction. The results will be reported to Deana Crumbling.

2.2.1.5 Abraxis Extract Processing in the Abraxis Lab

The 4 extracts sent to the Abraxis company laboratory will be analyzed using the Abraxis PCB-LC and co-planar test systems. Abraxis will report the results to Deana Crumbling. This will determine whether analytes appropriate to each method are present to warrant carrying these 2 Abraxis tests through the main study for all samples.

2.2.1.6 Abraxis Extract Processing in the XDS Lab

The 4 extracts sent to the XDS company laboratory will be analyzed using the CALUX cell-receptor assay method for dioxin-like compounds that interact with the cell's Ah receptor. The extracts will be processed to separate 3 fractions: a PAH fraction, a co-planar PCB fraction, and a dioxin fraction. This will determine whether analytes appropriate to each method are present to warrant carrying the CALUX testing through the main study for all samples. It will also help determine whether PAHs and dioxins are present (which could complicate interpretation of the CALUX results for co-planar PCBs). XDS will report the results to Deana Crumbling.

2.2.2 Description of Information from the Variability Evaluation

2.2.2.1 The data from the duplicated MISs (from a single 1-kg bag), will be used to estimate the within-sample variability (when minimal processing is performed).

2.2.2.2 The data from the duplicate analysis of the same well-prepared soil (within a single jar), will be used to estimate the within-sample variability for maximal field-friendly preparation.

2.2.2.3 The data from the duplicated extract analyses will be used to estimate the analytical (determinative method) variability on real sample extracts. The analytical precision of "real" sample extracts will be compared to the precision of spiked kit standards and the positive control. If the precision of the real samples is significantly different from the precision on standards, this may be an indication that matrix interferences with inconsistent behavior in the Abraxis test system are being extracted from the sample along with the target analytes. In addition, determination of analytical precision on matrix-free solutions permits partitioning of data uncertainty into its various sources.

2.2.2.4 The estimates of data variability from various sources along the sampling and analytical chain will be used to streamline the main study. This information will also be provided to the KRCEE team along with PCB data results.

- In addition to the knowledge of short-scale (1 meter) PCB concentration variability of the Paducah area under investigation, they will also have information about other sources of data variability. This information should help guide them in developing their field sampling plan for how to collect and process samples to minimize the effects of within-sample heterogeneity.
- For residual heterogeneity/data variability that cannot be removed through sample processing techniques, replicate analyses may be used to reduce data uncertainty to levels that are acceptable to support project decision-making. The KRCEE team should be able to use the information from this study to determine the number of field analysis replicates to control for precision.
- Although the main study will provide more definitive information, there will be an indication of whether significant bias is present for Paducah soils when analyzed by Abraxis immunoassay as opposed to traditional fixed lab analysis.
- The KRCEE team can also use this information to develop a strategy for selecting and preparing samples to be analyzed by fixed lab analysis to establish data comparability for the KRCEE field study.
- The information developed from this pilot study may also be used by KRCEE to develop the project-specific QC plans for both the immunoassay and fixed lab methods. This study will provide indications of what QC checks will be valuable to support the KRCEE study, and will indicate what kind of acceptance limits might be achievable.

3.0 Soil Sample Designations for the Pilot Study

This section will provide a listing of all anticipated samples and their labeling designations.
[to be completed]

Table 1: Samples and Analyses of the 20 1-kg Bagged Paducah Soil Samples					
Paducah Sample ID	Abraxis HC Soil Subsamples	Abraxis LC (send extract)	Abraxis co-planar (send extract)	ERT PAHs, Aroclors & total PCB	XDS CALUX extract analyses
Bkgd C(enter)	2				
Bkgd #1	2				
Bkgd #2	2				
Bkgd #3	2				
Bkgd #4	2				
Variability Eval	6				
Subsample Pool	2			2	
Extract Pool (2 analyses?)	1	1	1	2	1
Low C(enter)	2				
Low #1	2				
Low #2	2				
Low #3	2				
Low #4	2				
Variability Eval	6				
Subsample Pool	2			2	
Extract Pool (2 analyses?)	1	1	1	2	1
492 C(enter)	2				
492 #1	2				
492 #2	2				
492 #3	2				
492 #4	2				
Variability Eval	6				
Subsample Pool	2			2	
Extract Pool (2 analyses?)	1	1	1	2	1
H2 C(enter)	2				
H2 #1	2				
H2 #2	2				
H2 #3	2				
H2 #4	2				
Variability Eval	6				
Subsample Pool	2			2	
Extract Pool (2 analyses?)	1	1	1	2	1
# analyses	72			8	
# extracts for analyses	4	4	4	8	4

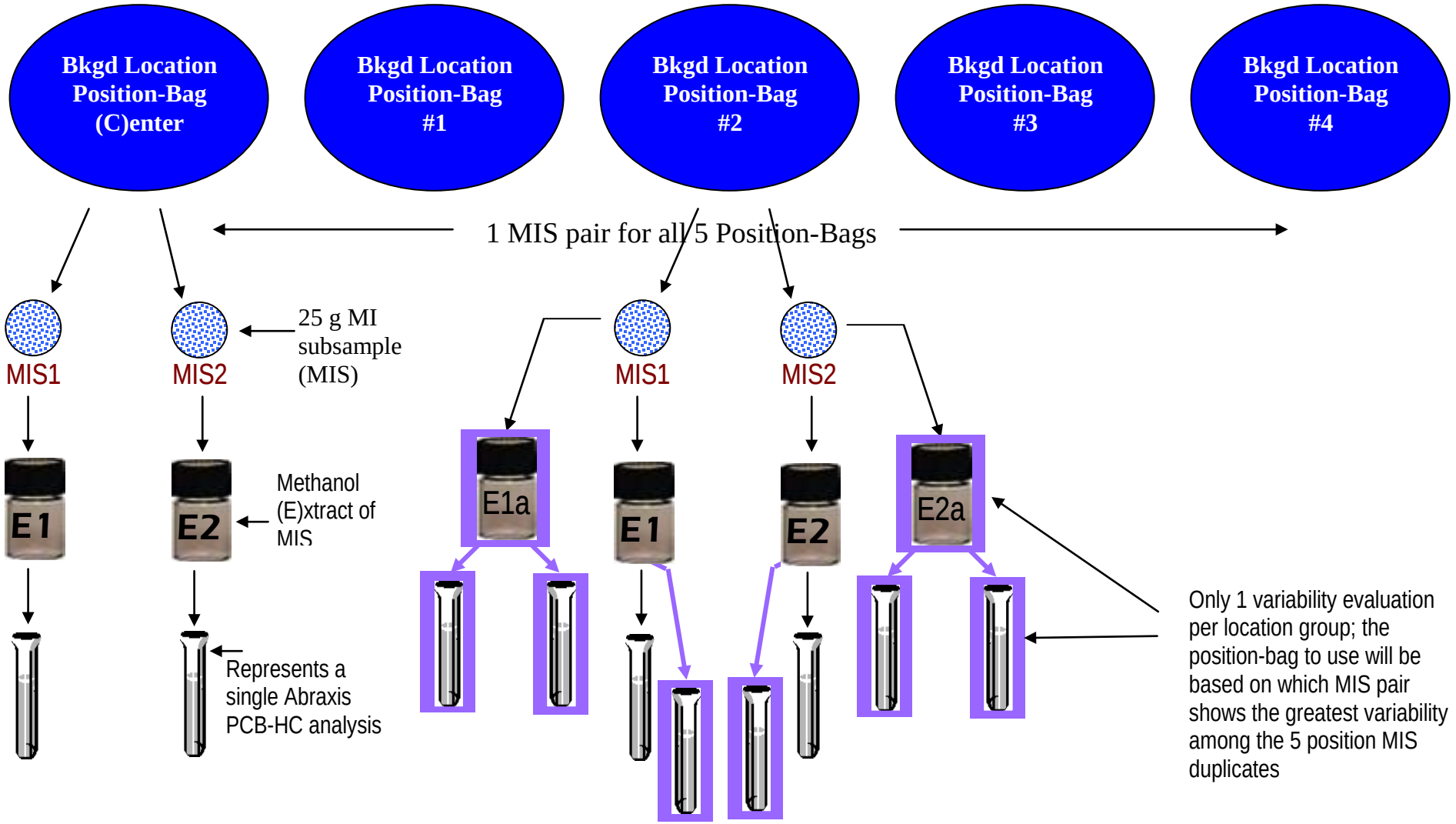

Figure 1: General pilot study strategy for analyzing each of the 20 bagged samples, illustrated using the 5 position bags for the Background (Bkgd) location group. Each 1-kg bagged sample will be prepared as described in the text. Each 1-kg sample is then subsampled twice using a multi-increment Gy-correct technique to create 2 25-g soil MIS subsamples representative of the bulk average for the 1-kg sample (as exemplified for the C-position bag). Each 25-g subsample will be further processed to reduce matrix heterogeneity within the 25-g MI-derived subsample. Each processed 25-g MI-derived soil will then be subsampled to produce a 10-g analytical sample, which will be extracted and analyzed with the Abraxis PCB-HC kit. Each position-specific MIS pair will be assessed for precision. The pair with the poorest precision in each location group will be selected for an evaluation of data variability sources (as depicted by the #2 position bag) by additional replicate analyses (additional extracts and analyses show in purple halo. See text for further discussion.