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Pilot Survey of Levels of Polychlorinated Dibenzo-*p*-dioxins, Polychlorinated Dibenzofurans, Polychlorinated Biphenyls, and Mercury in Rural Soils of the United States

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National Center for Environmental Assessment
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LIST OF ABBREVIATIONS AND ACRONYMS

ASE	Accelerated solvent extraction
BOD	Biological oxygen demand
CALUX	Chemical-Activated Luciferase Expression
CB	Chlorinated biphenyl
CDD	Chlorinated dibenzo-*p*-dioxin
CDDs/CDFs	Chlorinated dibenzo-*p*-dioxins and chlorinated dibenzofurans
CDF	Chlorinated dibenzofuran
GC	Gas chromatography
GPC	Gel permeation chromatography
HpCDD	Heptachlorodibenzo-*p*-dioxin
HpCDF	Heptachlorodibenzofuran
HRMS	High-resolution mass spectrometry
HxCDD	Hexachlorodibenzo-*p*-dioxin
HxCDF	Hexachlorodibenzofuran
ND	Nondetect
NDAMN	National Dioxin Air Monitoring Network
NR	Not reported
OCDD	Octachlorodibenzo-*p*-dioxin
OCDF	Octachlorodibenzofuran
PCB	Polychlorinated biphenyl
PCD/PCDF	Polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans
PeCDD	Pentachlorodibenzo-*p*-dioxin
PeCDF	Pentachlorodibenzofuran
QA	Quality assurance
QC	Quality control
r	Regression coefficient
RL	Reporting limit
RPD	Relative percent difference
SD	Standard deviation
SE	Standard error
SOP	Standard operating procedure
TCDD	Tetrachlorodibenzo-*p*-dioxin
TCDF	Tetrachlorodibenzofuran
TEF	Toxicity equivalence factor

LIST OF ABBREVIATIONS AND ACRONYMS (continued)

TEQ	Toxicity equivalent
TOC	Total organic carbon
WHO	World Health Organization
XDS	Xenobiotic Detection Systems, Inc.

UNITS

°C	degrees Centigrade
cm	centimeters
d	day
fg	femtograms
ft	feet
g	grams
kg	kilograms
L	liters
m	meters
mg	milligrams
mL	milliliters
mm	millimeters
ng	nanograms
oz	ounces
pg	picograms
µg	micrograms
µL	microliters

PREFACE

Many environmental contaminants accumulate in soils. Understanding the concentrations and spatial distribution of these chemicals is critical to determining how they may contribute to human exposure via direct contact or uptake through the food chain. The purpose of this document is to report the results of a pilot survey of the levels of polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), polychlorinated biphenyls (PCBs), and mercury in rural soils of the United States. All samples were collected during 2003. The study was conducted by the National Center for Environmental Assessment with contract support provided by Battelle.

AUTHORS, CONTRIBUTORS, AND REVIEWERS

AUTHORS

U.S. EPA
John Schaum
National Center for Environmental Assessment
Washington, DC

Battelle, Columbus, OH
Mary Schrock
Jane Chuang
Adam Abbgy
Basil Coutant
Karen Riggs
Jennifer Ickes
Karen Tracy
Pam Hartford
Nicole Iroz-Elardo

CONTRIBUTORS

Dwain Winters, U.S. EPA, Office of Pollution Prevention and Toxics
David Cleverly, U.S. EPA, National Center for Environmental Assessment

REVIEWERS

Internal Reviewers
Toney Baney, U.S. EPA, Office of Prevention, Pesticides and Toxic Substances
Andy Beliveau, U.S. EPA, Region 1
Marlene Berg, U.S. EPA, Office of Solid Waste and Emergency Response
Laura Casey, U.S. EPA, Office of Prevention, Pesticides and Toxic Substances
William Coakley, U.S. EPA, Office of Solid Waste and Emergency Response
Dan Duncan, U.S. EPA, Region 10
Matthew Lorber, U.S. EPA, Office of Research and Development
Carl Orazio, U.S. Geological Survey
Al Rubin, U.S. EPA, Office of Water
Robert Virta, U.S. Geological Survey
Dwain Winters, U.S. EPA, Office of Pollution Prevention and Toxics
Bruce Woods, U.S. EPA, Region 10

External Reviewers
Peter Adriaens, Department of Civil and Environmental Engineering, University of Michigan
Stephen Boyd, Department of Crop and Soil Sciences, Michigan State University
Pierre Goovaerts, PGeostat, LLC
Myrto Petreas, California Department of Toxic Substances Control, California Environmental Protection Agency
Richard Wenning, ENVIRON International Corporation

EXECUTIVE SUMMARY

This report provides a national-scale pilot survey of the levels of the following chemicals in rural/remote soils of the United States: chlorinated dibenzo-*p*-dioxins (CDDs), chlorinated dibenzofurans (CDFs), polychlorinated biphenyls (PCBs), and mercury.

Soils can serve as long-term reservoirs for CDDs, CDFs, PCBs, and mercury, with releases to both terrestrial and aquatic systems (U.S. EPA, 2003, 1997; Brzuzy and Hites, 1996). Understanding their distribution in soil is important because they are taken up by plants and animals through soil pathways and bioaccumulate through the human food chain. The relative importance of soil as a potential source for these chemicals is increasing as their point source emissions are being reduced. The final reason for conducting this study is that relatively few soil surveys of these compounds have been conducted.

The soil samples were collected in 2003 at 27 monitoring stations of the National Dioxin Air Monitoring Network (NDAMN) (U.S. EPA, 2005a). These stations are located in rural/remote areas, matching the areas of interest for the soil survey. Also they are distributed across the continental United States and Alaska, providing the nation-wide perspective desired for this study. Use of these sites provided the opportunity to study air-soil relationships using historical air concentration data available from NDAMN to compare with soil data collected under this study. Finally, NDAMN sites were a practical choice because site operators were already in place, facilitating logistics and reducing the costs of gathering soil samples.

The results presented pertain to the 27 sites sampled and should not be more broadly interpreted as statistically representative of all rural soils in the United States. These results, however, may be a plausible basis for a preliminary characterization of soils in rural/remote areas. The primary measurement results are summarized below.

- Total CDDs averaged 1,585 pg/g (standard deviation (SD) = 2945). Total CDFs averaged 47 pg/g (SD = 68). Levels of the tetrachlorodibenzo-*p*-dioxin (TCDD) homologues were the lowest, with an average concentration of 0.2 pg/g. Levels of the octachlorodibenzo-*p*-dioxin (OCDD) homologue were the highest, with an average concentration of 1,482 pg/g. The range of concentrations found here is similar to the range across five published studies on CDD/CDF levels in soils from rural areas of North America.

- Total PCBs averaged 3,089 pg/g (SE = 1,009, SD = 5,241). Levels of the deca-chlorinated biphenyl homologues were the lowest, with an average concentration of 29 pg/g. Levels of the penta-chlorinated biphenyl homologues were the highest, with an average concentration of 1,013 pg/g. The range of concentrations found here is similar to the range across three published studies on PCB levels in soils from rural areas worldwide.

- Total toxicity equivalents (TEQs) averaged 1.76 pg/g (SD = 2.47). The PCBs generally were a small fraction of the total TEQs in soil. The mean for total TEQs from this study falls near the center of the range of values across 10 published studies.

- Mercury concentrations averaged 22 ng/g across all sites (SD = 15 ng/g). The mean from this study falls within the range of values from five published studies on mercury levels in soils from rural areas of North America.

This study also evaluated relationships between air concentrations and soil concentrations across sites. A general association between air and soil was observed for the CDDs, based on the significant air-soil correlations observed across sites for most homologue groups and the similarity in air and soil congener profiles observed at most sites. Little association between air and soil could be observed for the CDFs, based on the lack of significant air-soil correlations for homologue groups across sites and the lack of similarity in air and soil congener profiles for many sites. Some association between air and soil was observed for the PCBs. Data limitations restricted the air and soil comparisons to only six PCBs. One of these had a significant air-soil correlation across sites. The air and soil profiles based on these six chemicals were very similar at most sites.

The observations for CDDs and PCBs are consistent with the theory that air transport and deposition are the primary ways that these chemicals are distributed to soils, particularly those in rural areas. The lack of similar observations for the CDFs does not necessarily mean that they are not distributed in a similar manner, but it does suggest that different factors affect the environmental fate of these chemicals.

This study also evaluated relationships between chemical levels in soil and total organic carbon (TOC) levels in soil. The raw data analyses showed significant positive correlations for many of the CDD/CDF homologues and one PCB homologue. However, the correlations were generally not very strong, indicating that other factors, such as grain size, may also be affecting sorption characteristics of the soil.

TEQ levels were estimated both on the basis of applying toxicity equivalence factors (TEFs) to the high-resolution mass spectrometry (HRMS) analyses and on the basis of a bioassay method called Chemical-Activated Luciferase Expression (CALUX). The CALUX results were higher—by varying amounts—than the HRMS total TEQs in almost all of the site composites. Significant positive correlations were found comparing the data on both a raw basis (r = 0.82) and on a log-transformed basis (r = 0.78). The likely reason for the high bias in the CALUX data relative to HRMS data is that CALUX responds to all compounds that activate the aryl hydrocarbon receptors, including a number of compounds other than CDDs, CDFs, and PCBs that may be present in soils.

1. INTRODUCTION

This report provides a national-scale pilot survey of the levels of the following chemicals in rural/remote soils of the United States: chlorinated dibenzo-*p*-dioxins (CDDs), chlorinated dibenzofurans (CDFs), polychlorinated biphenyls (PCBs), and mercury. All samples were collected during 2003 and analyzed at Battelle in Columbus, OH.

The term “dioxins” is used in this study to refer collectively to the 17 2,3,7,8-substituted CDDs and CDFs and the 12 co-planar PCBs (see complete listing in Table 1). Dioxin concentrations are expressed in terms of both total mass and toxicity equivalents (TEQs). TEQs allow concentrations of dioxin mixtures to be expressed as a single value computed by multiplying each congener concentration by a toxicity weight (toxicity equivalence factor [TEF]) and summing across congeners. TEFs are expressed as a fraction equal to or less than 1, with 1 corresponding to the most toxic dioxin congener, 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD). The TEQ data presented here are based on TEFs from the 1998 World Health Organization (WHO) recommendations, as shown in Table 1 (Van den Berg et al., 1998). This report adds subscripts to TEQs when necessary to clarify which chemicals have been included in the TEQ calculation: “D” for CDDs, “F” for CDFs, and “P” for PCBs.

Table 1. Congeners and TEFs used to calculate TEQs

CDDs		**CDFs**		**PCBs**	
Congener	**TEF**	**Congener**	**TEF**	**Congener**	**TEF**
2,3,7,8-TCDD	1.0	2,3,7,8-TCDF	0.1	PCB 77	0.0001
1,2,3,7,8-PeCDD	1.0	1,2,3,7,8-PeCDF	0.05	PCB 81	0.0001
1,2,3,4,7,8-HxCDD	0.1	2,3,4,7,8-PeCDF	0.5	PCB 105	0.0001
1,2,3,6,7,8-HxCDD	0.1	1,2,3,4,7,8-HxCDF	0.1	PCB 114	0.0005
1,2,3,7,8,9-HxCDD	0.1	1,2,3,6,7,8-HxCDF	0.1	PCB 118	0.0001
1,2,3,4,6,7,8-HpCDD	0.01	2,3,4,6,7,8-HxCDF	0.1	PCB 123	0.0001
OCDD	0.0001	1,2,3,7,8,9-HxCDF	0.1	PCB 126	0.1
		1,2,3,4,6,7,8-HpCDF	0.01	PCB 156	0.0005
		1,2,3,4,7,8,9-HpCDF	0.01	PCB 157	0.0005
		OCDF	0.0001	PCB 167	0.00001
				PCB 169	0.01
				PCB 189	0.0001

Source: Van den Berg et al. (1998).

TEF = toxicity equivalence factor
TEQ = toxicity equivalent

Soils can serve as long-term reservoirs for CDDs, CDFs, PCBs, and mercury, with releases to both terrestrial and aquatic systems (U.S. EPA, 2003, 1997; Brzuzy and Hites, 1996). Understanding their distribution in soil is important because they are taken up by plants and animals through soil pathways and bioaccumulate through the human food chain. More than 90% of the general population exposure to dioxin is via food ingestion; human exposure to mercury is similarly dominated by dietary exposure (U.S. EPA, 2003, 1997). The relative importance of soil as a potential source for these chemicals is increasing as their point source emissions are being reduced. The final reason for conducting this study is that relatively few soil surveys of these compounds have been conducted, and none had a national-scale perspective. Many were associated with Superfund sites or other contaminated areas and covered relatively small geographic areas (U.S. EPA, 2003).

1.1. SCOPE OF STUDY

A comprehensive national soil survey would represent all geographic regions of the country, the major land use categories (rural, suburban, urban, agricultural, commercial, and industrial), and a full range of climatic conditions, soil types, terrains, and vegetative covers (e.g., forest, grassland, cropland). As a pilot survey with limited resources, this study could address only a small subset of these lands. It was decided to focus on undisturbed soil in rural/remote areas because this would provide a baseline for evaluating soil levels in other areas. The soil samples were collected at the air monitoring stations of the National Dioxin Air Monitoring Network (NDAMN) (U.S. EPA, 2005a; Cleverly et al., 2006). These stations were located in rural/remote areas, matching the areas of interest for the soil survey. The 35 NDAMN stations were distributed across the continental United States and Alaska (Figure 1), providing the nation-wide perspective desired for this study. Use of these sites provided the opportunity to study air-soil relationships using historical air concentration data available from NDAMN to compare with soil data collected under this study. Finally, NDAMN sites were a practical choice because site operators were already in place, facilitating logistics and reducing the costs of gathering soil samples (see Section 2 for further information about NDAMN and the site selection process).

The original focus of this study was on CDDs, CDFs, and PCBs. Mercury was added to the study after the initial project planning had been completed. Like the CDDs/CDFs and PCBs, mercury is a persistent chemical that accumulates in soil. Also, relatively few soil surveys for mercury have been conducted in the United States and none had a national-scale perspective. Accordingly it was decided that this would be an appropriate addition to the study. Although much of the study design was specific to CDDs/CDFs and PCBs, the procedures were deemed

Figure 1. NDAMN air sampling stations. Circles indicate stations included in soil survey and triangles are excluded stations.

1. Penn Nursery, PA
2/3. Clinton Crops, NC
4. Everglades, FL
5. Lake Dubay, WI
6. Monmouth, IL
7. McNay Farm, IA
8. Lake Scott, KS
9. Keystone State Park, OK
10. Arkadelphia, AR
11. Bennington, VT
12. Jasper, NY
13. Beltsville, MD
14. Caldwell, OH
15. Oxford, OH
16. Dixon Springs, IL
17. Quincy, FL
18. Bay St. Louis, MS
19. Padre Island, TX
20. Fond du Lac, MN
21. North Platte, NE
22. Goodwell, OK
23. Big Bend, TX
24. Grand Canyon, AZ
25. Theodore Roosevelt, ND
26. Craters of the Moon, ID
27. Chiricahua, AZ
28. Rancho Seco, CA
29. Marvel Ranch, OR
30. Ozette Lake, WA
31. Fort Cronkhite, CA
32. Newport, OR
33. Craig, AK
34. Trapper Creek, AK
35. Yaquina Head, OR

reasonable for mercury, and were similar to those used to collect samples for mercury analysis in a Washington State soil survey (Rogowski et al., 1999). Also, as discussed below, a brief investigation indicated that the NDAMN sites appeared unlikely to be impacted by local mercury sources.

Mercury is commonly found in more than 30 minerals, and natural deposits containing these minerals are found in many of the western states (Jasinski, 1994). Some of these deposits may be near the NDAMN sites in Arkansas, Texas, Arizona, California, Oregon, and Alaska. Historically, most mercury mining in the United States has occurred in California. The use of mercury has declined significantly in recent years and recycling is increasing. As a result, demand for new mercury has decreased, and no mining has occurred in the United States since 1990 (Jasinski, 1994). Mercury can be released in the mining and processing of gold ores. About 99% of the gold currently produced in the United States comes from 30 mines (USGS, 2002), and none of them are near the NDAMN sites. Thus, it appears unlikely that the NDAMN sites were impacted by local mining operations.

1.2. STUDY OBJECTIVES

The primary objective of this study was to provide preliminary estimates of the levels of CDDs, CDFs, PCBs, and mercury in rural/remote soils. The data summaries presented here should be interpreted as summaries of the sites sampled and should not be interpreted as being statistically representative of all rural soils. However, the surveyed sites cover a wide range of climates, geographic areas, terrains, and soil types and thus provide a reasonable basis for a preliminary characterization of soil in rural/remote areas.

This study also evaluated relationships between chemical levels in air and soil and relationships between chemical levels in soil and the organic carbon content of soil. As discussed below, it is believed that CDDs/CDFs, PCBs, and mercury are distributed to the environment primarily via air transport and enter soils via deposition from the air (U.S. EPA, 2003, 1997). Understanding air-soil relationships may improve our understanding of these fate processes and ultimately lead to better models for predicting the fate of these chemicals in the environment.

1.3. ENVIRONMENTAL FATE OVERVIEW

CDDs and CDFs are released to the environment primarily as combustion by-products and are widely distributed through the environment via air transport. In the atmosphere, they are present in the vapor phase and sorbed to particles. Wet and dry deposition remove CDDs/CDFs from the atmosphere to soils, plants, or other environmental surfaces. CDDs/CDFs sorbed to plants are transferred to soils during leaf fall or when plants die and subsequently decay. Although these compounds are generally very persistent in soils, losses can occur via run-off, particle resuspension, volatilization, and biological degradation (U.S. EPA, 2003; ATSDR, 1998).

PCBs were produced in large quantities (571,000 metric tons) in this country from 1929 until their ban in 1978. Because PCBs are no longer manufactured or imported in large quantities, significant releases of newly manufactured or imported materials to the environment do not occur. PCBs have become distributed throughout the environment primarily via air transport. Like CDDs/CDFs, PCBs are present in the atmosphere, in both vapor phase and sorbed to particles. Wet and dry deposition remove PCBs from the atmosphere to soil, surface water, plants, and other environmental surfaces. Volatilization is the primary mechanism by which they are released from soils and water back into the atmosphere (ATSDR, 2000).

Mercury occurs naturally as a mineral and in gaseous forms and is distributed throughout the environment by both natural and anthropogenic processes. It is emitted to the atmosphere from multiple types of combustion sources, primarily coal-fired utility boilers, municipal waste combustion, commercial/industrial boilers, and medical waste incinerators (U.S. EPA, 1997). The element has three valence states and is found in the environment in the metallic form and in the form of various inorganic and organic complexes. Most of the soil mercury is thought to be Hg(II). The major features of the bio-geochemical cycle of mercury include degassing of mineral mercury from the lithosphere and hydrosphere, long-range transport in the atmosphere, wet and dry deposition to land and surface water, sorption to soil and sediment particulates, and revolatilization from land and surface water (ATSDR, 1999; U.S. EPA, 1997).

2. SITE SELECTION

As discussed above, all soil samples were collected at NDAMN air sampling stations. The overall purposes of NDAMN were to determine background air concentrations of dioxin-like compounds in rural and remote areas of the United States and to investigate changes that might occur over time. The number of NDAMN sites increased from 9 in 1998 to a peak of 35 in 2002 (Figure 1). Operation was suspended in 2005. The selection of sites for the NDAMN program was not based on a statistical process, but rather judgements on a variety of considerations (U.S. EPA, 2005a; Cleverly et al., 2006):

- *Remoteness*. Sites could not be impacted by nearby industrial and municipal sources. Although no specific criteria were used to determine how far a site could be from an urban area, all were located in rural or remote areas (one exception was the Beltsville, MD, site located in the Washington Metropolitan Area, which was used early in the program for development of sampling protocols; air monitoring data from this site were not included in background estimates). Some sites were located in very remote locations (e.g., the Grand Canyon and Alaska) to explore impacts in pristine areas.

- *Agricultural importance*. Many of the sites were located on farms in agriculturally important areas to help better understand how dioxins enter the human food chain.

- *Air modeling needs*. Some sites were selected to help air modelers verify and calibrate their models.

- *Climate*. Sites were chosen to represent a wide range of climatic conditions.

- *Regions*. Sites were chosen to represent a variety of geographic regions.

- *Practicality*. Sites had to be accessible and secure. Many sites were co-located with existing air monitoring networks to minimize costs.

For purposes of this study, all sites were reviewed for suitability for soil testing. Eight sites were eliminated for the following reasons:

- Clinton Crops, NC (Site 3): a duplicate quality assurance/quality control (QA/QC) monitor at same location as Site 2.

- Beltsville, MD (Site 13): not a regular NDAMN station because it was located within the Washington Metropolitan Area.

- Oxford, OH (Site 15): used in the development of the soil sampling protocol, so not included in the final survey.

- Craters of the Moon, ID (Site 26): sampling not feasible because site was located on a lava bed and operator was not available during testing period.

- Fort Cronkhite, CA (Site 31): soil may have been disturbed by past military operations.

- Newport, OR (Site 32): monitor on roof of building and surrounding soil was likely disturbed.

- Craig, AK (Site 33): sampling not feasible due to rocky soil and steep terrain.

- Yaquina Head, OR (Site 35): sampling not feasible due to rocky soil and steep terrain.

As a result of the review process, a total of 27 sites were included in the soil survey.

3. PROTOCOL DEVELOPMENT

A three-step process was used to develop a protocol for soil sample collection and handling.

Step 1. A literature search was conducted to identify published soil collection guidance. Guidance in *Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies* (U.S. EPA, 1992) was selected as the primary basis for developing the protocol. Other useful reports included soil sampling guidance by the U.S. Department of Housing and Urban Development (HUD, 1995); Rogowski et al. (1999), who conducted a dioxin soil survey in Washington state; and Vikelsoe (2002), who conducted a dioxin soil survey in Denmark.

Step 2. A surface soil sampling protocol and a standard operating procedure (SOP) for collecting soil samples were developed on the basis of information gathered in the literature survey. The surface soil sampling protocol recommends that an initial survey be carried out for each specific application to determine, e.g., an acceptable number of sampling points and sampling depth. For this project, the initial survey was conducted at the NDAMN site in Oxford, OH. The full report on the Oxford survey is presented in Appendix A. Three key issues were evaluated during this initial survey:

- *Grid size and number of samples*. A review of several soil sampling surveys indicated grid squares with sides ranging from 25 ft (7.6 m) to approximately 200 ft (61 m). For this initial survey a 100 ft × 100 ft (30.5 m × 30.5 m) grid was used. Twenty-one samples were collected within the grid. This sample number was selected as a reasonable starting point for making subsequent statistical calculations regarding variability. An additional 4 samples were collected approximately 1,000 ft (305 m) from the grid center. Based on CALUX analysis (described in Section 5.2 and Appendix B), no significant differences were seen between the averages of the grid samples and the distant samples. The CALUX analysis of all samples was used to conduct a statistical analysis that indicated that at least 5 samples were required to derive a mean with less than a 20% standard error. On this basis it was decided to collect 5 samples at each site over a 100 ft ×100 ft (30.5 m × 30.5 m) area (one at each corner and one at the center).

- *Sampling Depth.* EPA indicates that the surface layer of soil (0–15 cm) reflects the deposition of airborne pollutants, especially recently deposited pollutants and pollutants that do not move downward because of attachment to soil particles (U.S. EPA, 1992). Brzuzy and Hites (1995) studied dioxin concentrations in soil as a function of depth and found that more than 80% of total CDDs/CDFs were found in the upper 15 cm. Rogowski et al. (1999) used a sampling depth of 5 cm in a dioxin soil survey in Washington state, and Vikelsoe (2002) used a sampling depth of 10 cm in a dioxin soil survey in Denmark. EPA reports that nearly all mercury in soil is found in the top 20 cm (U.S. EPA, 1997). In the Oxford survey, 10 cm cores were collected and divided into the top 5 cm and bottom 5 cm. The composites for the top and bottom layers were analyzed for CDDs/CDFs, PCBs, and mercury. No significant differences in analyte concentrations were seen. The 5-cm cores were frequently unstable and difficult to handle in the field. On this basis, 10-cm cores were selected for this study.

- *Sample Size.* 600 g of soil were collected at each sampling point. The soil was thoroughly mixed and 10 g subsamples were removed for chemical analysis. Because many of the CDD/CDF congeners were below detection limits, it was decided to double the subsample size to 20 g for the final multisite survey.

Step 3. The SOP was revised on the basis of sampling experience during the Oxford survey and sent to the field operators, who were asked to carefully read it and respond with any questions. The final SOP is presented in Appendix C.

4. SAMPLE COLLECTION, HANDLING, AND STORAGE

The soil samples were collected by the local operators at 26 of the sites. These operators had experience in collecting air samples for the NDAMN program, and at many of the locations they also collected samples for other national air monitoring networks. Some had specific environmental science background and others did not. The operator at the Everglades, FL, site (Site 4) was unable to collect samples because of time constraints, and a Battelle staff member was sent to the site to collect the soil samples. All samples from the 27 sites were collected between August 12, 2003, and October 20, 2003.

The SOP was distributed to the NDAMN operators, who were contacted by e-mail or phone to make sure they received it and understood the sampling procedures. Sampling supplies were purchased and assembled into individual sampling kits and sent to the operators approximately one week before sampling was scheduled to begin. Battelle staff was accessible by phone to the operators during the sampling period to answer any questions that might arise while in the field.

At each NDAMN site, a 100 ft ×100 ft (30.5 m × 30.5 m) sampling area was chosen as near the air monitor as possible and where the terrain was relatively flat and there was no visible evidence of soil disturbance from flooding, erosion, construction, digging, or plowing. Five sampling points were located in an “X” configuration over the area (one at each corner and one at the center). Ground cover and vegetation was removed over a 20 cm × 20 cm area at each sampling point. Core samples of 10 cm each were collected at each point using a metal “tulip bulb”-type planter that had a diameter of approximately 7.5 cm. A total of approximately 600 g of soil was collected at each sampling point, put into three precleaned jars, packed in ice, and shipped to Battelle.

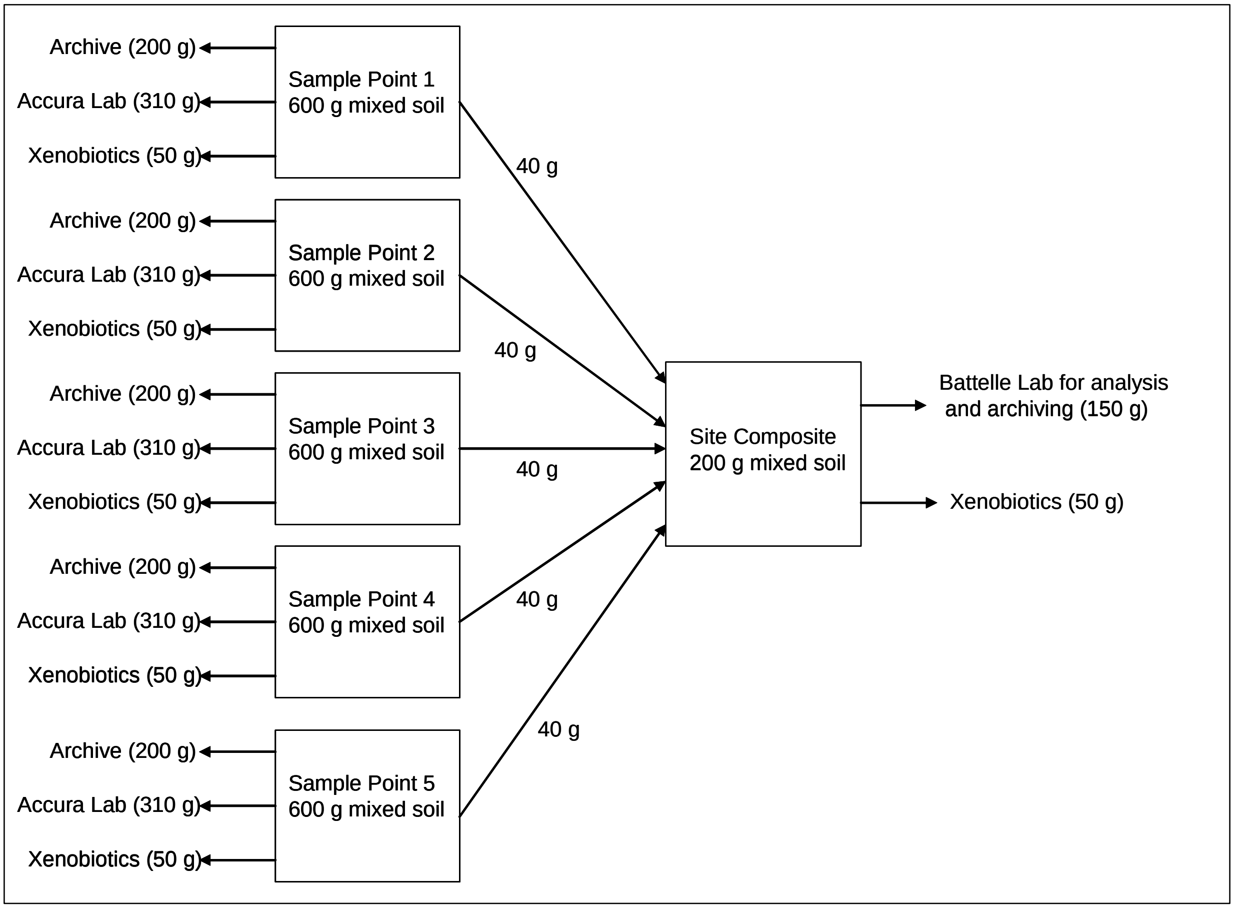

Figure 2. Sample handling diagram.

All of the soil samples were received at Battelle in good condition. As illustrated in Figure 2, the samples from each site were handled as follows:

- The soil from the three sample jars representing one sampling point were thoroughly mixed in a stainless steel bowl with a large stainless steel spoon. Fifty grams of this mixed soil were transferred to a new, pre-cleaned, labeled 4-oz (120-mL) jar. The jar was shipped to Xenobiotic Detection Systems, Inc. (XDS) (Durham, NC), for CALUX analysis. An additional 40 g of mixed soil were transferred to a stainless steel bowl for making a site composite. The remaining mixed soil was put back in the original sampling jars. Sample jar 1 of 3 was set aside for archiving (200 g). Sample jars 2 of 3 and 3 of 3 were sent to Accura Analytical Labs, Inc. (Atlanta, GA), for physical/ chemical parameter testing (containing a total of about 310 g). This process was repeated for the remaining four sampling points.

- The 40 g of soil that had been set aside for the site composite from each of the five sampling points were thoroughly mixed in a stainless steel bowl. Approximately 50 g of this composited soil were placed in a new, pre-cleaned, labeled 4-oz (120-mL) jar. The jar was shipped to XDS (Durham, NC) for CALUX analysis. The remaining 150 g were transferred to a new, precleaned, labeled 8-oz (240-mL) jar. The jar was transferred to Battelle's analytical laboratories for analysis of CDDs, CDFs, PCBs, and mercury. The unused portion was archived.

This process was repeated with the samples from all 27 sites. Sample holding times and temperatures were as follows:

- CDDs/CDFs: all samples were frozen until extraction, extracted within 15 days of receipt, and analyzed within 5 weeks of extraction.

- PCBs: all samples were frozen until extraction, extracted within 15 days of receipt, and analyzed within 10 weeks of extraction.

- Mercury: all samples were refrigerated until extraction, extracted within 28 days of receipt, and analyzed same day (with three exceptions, as noted in Section 6.5).

- CALUX: all samples were held at room temperature, extracted within 30 days of receipt, and analyzed within 3 weeks of extraction.

5. ANALYTICAL METHODS

5.1. PHYSICAL/CHEMICAL PARAMETER TESTS

Physical/chemical parameter testing was performed on soils from 135 individual sampling points (27 sites, 5 sampling points per site). Two 8-oz (240-mL) jars from each of the five sampling points at the 27 sites were sent to Accura Analytical Labs, Inc. (Atlanta, GA), for analysis of pH, total organic carbon (TOC), grain size distribution, and moisture content according to the following methods:

- pH: EPA Method SW 9045C (U.S. EPA, 1995)
- TOC: Walkley-Black Method (Walkley and Black, 1934)
- Grain size distribution: ASTM D422 (ASTM, 2002a)
- Moisture content: ASTM D2216 (ASTM, 2002b)

5.2. CALUX BIOASSAY TEQ

Several biological methods are commercially available for measuring dioxin TEQs, and bids from several companies were received. The CALUX method by XDS was chosen, primarily because it provided the lowest detection limit. XDS has patented a genetically engineered cell line that contains the firefly luciferase gene under trans-activational control of the aryl hydrocarbon receptor. This cell line can be used for the detection and relative quantification of aryl hydrocarbon receptor agonists and is referred to as Chemical-Activated Luciferase

Expression (or CALUX) assay (Denison et al., 1998). The most widely studied class of compounds that activate this system is the polychlorinated diaromatic hydrocarbons (PCDH), which includes 2,3,7,8-TCDD. The CALUX assay can be used to provide a measure of dioxin TEQs in a sample. When the cells are exposed to dioxins and related chemicals, they produce the enzyme luciferase in a time-, dose-, and chemical-specific manner. Luciferase activity is determined by measuring light emitted and is directly proportional to the amount of dioxin-like chemicals within the test samples (see Appendix B for further details of the CALUX method).

CALUX testing was performed on soils from 135 individual sampling points (27 sites, 5 sampling points per site) plus 27 composites representing each site. When XDS received soil samples from Battelle, the samples were logged in and held at room temperature until processing and analysis. Samples were extracted with a bottle sonication method using organic solvents following XDS Method WL-2. Sample extracts were processed through a patent-pending procedure, XDS Method WL-3, which removes commonly interfering substances such as polyaromatic hydrocarbons. All samples were processed in duplicate along with a batch recovery sample for each set of samples in order to provide semi-quantitative results.

5.3. MERCURY ANALYSIS

Total mercury was determined on 27 composites (one from each site) using modified EPA SW846 Method 7471A (U.S. EPA, 1994a). All standards and samples were digested and analyzed by cold vapor atomic absorption. Approximately 2 g of sample were weighed into a biological oxygen demand (BOD) bottle and digested by adding nitric acid, sulfuric acid, and potassium permanganate and heating in a water bath at ~ 95°C. Samples were cooled and the excess permanganate reduced using sodium chloride hydroxylamine hydrochloride. Mercury hydride was generated by the addition of stannous chloride. The hydride was swept into an absorption cell.

5.4. HRMS ANALYSIS OF CDDs/CDFs, AND PCBs

CDD/CDF and PCB congeners were measured in 27 composites (one from each site) using gas chromatography/high-resolution mass spectrometry (referred to as HRMS in the remainder of this document). Soil composites were extracted and analyzed for the 17 2,3,7,8-substituted CDDs/CDFs following general procedures in EPA Method 1613, Revision B (U.S. EPA, 1994b) and Battelle SOPs ASAT.II-001-02 and ASAT.II-002-02. All 209 PCB congeners were determined following general procedures in EPA Method 1668, Revision A (U.S. EPA, 1999) and Battelle SOP ASAT.II-009-00. Specific steps were taken during sample preparation to enhance the detection limit. These steps included extracting nearly twice the standard amount of soil and concentrating the sample to half the standard final extract volume.

Overall, an aliquot of approximately 20 g wet weight of each composite was spiked with isotopically labeled analogs (internal standards) of 15 of the 17 2,3,7,8-substituted CDDs/CDFs and 27 PCBs. The composites were extracted with methylene chloride using accelerated solvent extraction (ASE) techniques. Extracts were processed through a gel permeation chromatography column, spiked with CDD/CDF and PCB cleanup standards, and processed through acid/base silica columns. The extracts were then processed into separate CDD/CDF and PCB fractions using carbon columns. The CDD/CDF fractions were spiked with CDD/CDF recovery standard and concentrated to a final volume of 10 μL. The PCB fractions were spiked with PCB recovery standard and concentrated to a final volume of 25 μL.

Sample extract fractions were analyzed for CDDs/CDFs and PCBs by HRMS in the selected ion monitoring mode at high resolution. Initial analysis for CDDs/CDFs was carried out on a DB-5 or equivalent column. Because 2,3,7,8-tetrachlorodibenzofuran (2,3,7,8-TCDF) is not completely separated from the other TCDF isomers on the DB-5 column, second column confirmation of 2,3,7,8-TCDF levels above the lowest calibration level in the initial analysis was carried out on a DB-225 column or equivalent column. PCBs were determined using an SPB-Octyl column. Approximately 110 of the PCBs were determined as individual congeners, and the remaining congeners adding up to 209 were determined as various sets of co-eluting congeners. All analytes were quantified by isotope dilution or by the internal standards method using the labeled internal standards for quantitation.

5.5. QUALITY ASSURANCE/QUALITY CONTROL

Data quality parameters assessed were accuracy, precision, representativeness, comparability, completeness, and sensitivity. Each of these is discussed below.

The QA/QC measurement quality objectives for accuracy and precision for all analytes are addressed in detail in Appendix D and summarized below:

- *Procedural blanks.* Mercury was nondetect in the blanks, and CDD/CDF blanks were low relative to the field samples. A number of PCBs were detected in the procedural blanks, with up to 16 PCBs exceeding 3 pg/g. In terms of total PCBs, most of the sites exceeded the blanks by a large margin. However, the sites with the lowest total PCBs approached the blank levels, i.e., the lowest site was 248 pg/g, as compared with an average level in blanks of 170 pg/g. Appendix E provides the congener-specific PCB levels in blanks, and these should be considered in interpreting individual congener values.

- *Recoveries.* Lab control spike recoveries and matrix spike recoveries were generally within QC goals.

- *Replicates.* The relative percent differences (RPDs) were generally within QC goals for mercury and CDDs/CDFs. The PCB duplicates had mixed results. Some of the duplicate pairs had excellent agreement, such as those for Theodore Roosevelt, ND (Site 25), where all congeners had an RPD of less than 18% and the RPD for total PCBs was just 2%. Others had poor agreement such as those for Lake Scott, KS (Site 8), where individual congeners had RPDs ranging from 21 to 161% and the RPD for total PCBs was 140%.

Representativeness was addressed through the sampling design and selection of sampling locations to accomplish the project goals. Samples were handled carefully following good laboratory practices to ensure that chain-of-custody and processing were carried out appropriately.

Comparability was addressed by having all samples collected within an approximate 2-month time frame using the same sampling protocol and procedures at each site. Samples were analyzed within holding times, with the exception of three mercury samples. These samples exceeded the specified 28 days and are flagged in Table 2. Due to the holding time exceedance, mercury results for these three samples should be considered minimum concentrations. However, the mean across sites was essentially the same with or without these samples.

The completeness goal for this project was to collect 100% of the planned samples and for 95% of the laboratory data be considered valid. Soil sample collection was planned at 30 NDAMN air sampling stations. Sampling actually took place at 27 stations, resulting in 90% of the planned samples being collected. Details of sample collection are included in Section 4. All laboratory data (100%) from the collected samples are included in the report.

All data were generated following accepted analytical methods, and the reporting units used are consistent with accepted conventions for environmental analyses. Sensitivity was ensured by meeting target reporting limits for each analysis.

6. SOIL MEASUREMENTS

This section presents the measurement results from the study. Averages and other descriptive statistics are generally presented assuming that nondetects were equal to half the detection limit. Wherever this assumption was made, the influence of the nondetects was

Table 2. Soil concentrations (dry weight) by site

Site	Total CDDs (tetras-octas) (pg/g)	Total CDFs (tetras - octas) (pg/g)	Total CDDs and CDFs (tetras - octas) (pg/g)	Total PCBs (pg/g)	Mercury (ng/g)
1. Penn Nursery, PA	6,602	29	6,631	1,366	26[a]
2. Clinton Crops, NC	1,361	5	1,367	475	69[a]
4. Everglades, FL	680	98	778	2,604	22
5. Lake Dubay, WI	96	13	110	15,700	4
6. Monmouth, IL	395	96	491	2,037	25
7. McNay Farm, IA	1,696	47	1,743	358	30
8. Lake Scott, KS	22	2	24	1,115	11
9. Keystone State Park, OK	65	22	87	2,464	9
10. Arkadelphia, AR	685	5	690	4,028	26
11. Bennington, VT	178	28	205	3,023	43
12. Jasper, NY	11,400	77	11,480	1,543	15
14. Caldwell, OH	2,307	16	2,322	1,019	26
16. Dixon Springs, IL	9,574	239	9,813	845	19
17. Quincy, FL	369	10	380	303	13[a]
18. Bay St. Louis, MS	1,686	51	1,738	4,930	24
19. Padre Island, TX	74	5	79	255	5
20. Fond du Lac, MN	130	63	193	1,308	19
21. North Platte, NE	50	14	64	493	13
22. Goodwell, OK	288	43	331	4,954	5
23. Big Bend, TX	22	3	25	24,570	18
24. Grand Canyon, AZ	25	7	32	713	9
25. Theodore Roosevelt, ND	107	21	127	570	0.5
27. Chiricahua, AZ	1,110	8	1,118	509	45
28. Rancho Seco, CA	203	50	253	3,274	37
29. Marvel Ranch, OR	3,534	284	3,817	1,300	24
30. Ozette Lake, WA	105	28	133	2,419	39
34. Trapper Creek, AK	15	4	19	1,224	22
Average	1,585	47	1,632	3,089	22
Standard deviation	2,946	68	2,982	5,241	15
Standard error	567	13	574	1,009	3

[a] Sample exceeded 28 day holding time for mercury analysis and should be considered a minimum value.

evaluated by computing the values assuming both nondetects were equal to zero and nondetects were equal to the full detection limit. For total CDDs/CDFs, total PCBs, and mercury, both calculations gave essentially the same values, and the full results are presented only on the basis of nondetects equal to half the detection limit. For some sites, the TEQ estimates have significant differences, depending on treatment of the nondetects, so these data are presented two ways: TEQ1 assumes nondetects equal zero, and TEQ2 assumes nondetects were equal to half the detection limit. All concentrations are presented on a dry-weight basis.

This section includes comparisons of the soil levels found in the study with those of similar studies found in the published literature. A detailed literature review of levels of CDDs/CDFs, PCBs, and mercury in rural soils is provided in Appendix F. The discussions in this section briefly summarize the key studies from Appendix F. The focus is generally on studies of North America. In the case of total PCBs, however, relatively few data on North America could be found, so studies from other areas are also discussed. It should be noted that the studies included in this review have a wide variety of design features (e.g., detection limits, treatment of nondetects in deriving statistics, congener inclusion, sampling procedures, analytical techniques), which makes it difficult to compare them on a completely equal basis. Information is provided in Appendix F to help readers consider these differences, but no adjustments were made to the values reported in the original studies.

6.1. PHYSICAL/CHEMICAL PARAMETER RESULTS

The complete set of physical/chemical parameter data for samples collected at all sites is presented in Appendix G and briefly summarized here. A wide range of soil types were collected in this study, with grain size distributions (based on criteria from the Unified Soil Classification System, ASTM D2487) as follows (SE is the standard error of the mean and SD is the standard deviation for all samples):

% finer than #4 sieve (4.75 mm): mean of 98.4 (SE = 0.3, SD = 23.9)
% finer than #200 sieve (0.075 mm): mean of 61.3 (SE = 2.2, SD = 25.6)
% finer than 0.005 mm: mean of 18.3 (SE = 0.9, SD = 10.8)

Moisture content averaged 22.2% (SE = 2.1, SD = 23.9). Soil pH averaged 6.0 (SE = 0.1, SD = 1.3). Total organic carbon averaged 34,900 mg/kg (SE = 2,400, SD = 27,800).

6.2. CDD AND CDF RESULTS

CDD/CDF total homologues (tetra and higher) and individual CDD/CDF congener (tetra and higher) results for each site are included in Appendix H. Table 2 shows the CDD and CDF levels (tetra through octa) for the composite samples from each site. CDDs ranged from 15 to 11,400 pg/g, with an average of 1,585 pg/g (SE = 567, SD = 2,945). Total CDFs ranged from 2 to 284 pg/g, with an average of 47 pg/g (SE = 13, SD = 68). Total CDDs/CDFs ranged from 19 to 11,480 pg/g, with an average of 1,632 pg/g (SE = 574, SD = 2,982). These values were calculated assuming that nondetects were equal to half the detection limit. Treatment of the nondetects, however, had a negligible impact on these homologue sums because they were generally very low when compared with the detected levels. For example, at Trapper Creek, AK (site 34), which had the lowest levels, total CDDs/CDFs were 18.71 pg/g assuming that nondetects were equal to zero and 19.03 pg/g assuming that nondetects were equal to the full reporting limit.

Table 3 shows all of the CDD/CDF homologue concentrations for all sites. TCDD homologue concentrations were the lowest, with an average of 0.2 pg/g. The octachlorodibenzo-*p*-dioxin (OCDD) homologue concentrations were the highest, with an average of 1,482 pg/g.

Table 4 summarizes information from four studies on CDD/CDF levels in soils from rural areas of North America. This table shows that the total CDD/CDF concentration ranged from nondetect to 10,000 pg/g, which is similar to the range of 19 to 11,480 pg/g found in the current study.

Total CDDs/CDFs in procedural blanks averaged 2 pg/g. All sites exceeded this value by a wide margin, i.e., the concentration at the lowest site was 19 pg/g. The blank levels are listed in Appendix H along with the homologue levels and should be considered when interpreting these values individually.

6.3. PCB RESULTS

PCB individual congener results for each site are provided in Appendix E. Approximately 110 of the 209 PCB congeners co-eluted with other congeners, preventing resolution of individual levels. In Appendix E, all the congeners that co-eluted are marked with a “C” followed by the PCB compound number with which each one co-eluted. Table 2 provides total PCB levels for the composite samples from each site; concentrations ranged from 255 to 24,570 pg/g, with an average of 3,089 pg/g (SE = 1,009, SD = 5,241). These values were calculated assuming that nondetects were equal to half the detection limit. Treatment of the nondetects, however, had a negligible impact on these sums because they were generally very low when compared with the detected levels. For example, at Dixon Hills, IL (site 19), which had the lowest levels, the total PCBs were 248 pg/g assuming that nondetects were equal to zero and 262 pg/g assuming that nondetects were equal to the full reporting limit.

Table 5 summarizes the PCB homologue concentrations for all sites. Deca-chlorinated biphenyl homologue concentrations were the lowest, with an average of 29 pg/g. Penta-

Table 3. Soil concentrations (pg/g dry weight) of CDD and CDF homologues

Site	Total TCDFs	Total TCDDs	Total PeCDFs	Total PeCDDs	Total HxCDFs	Total HxCDDs	Total HpCDFs	Total HpCDDs	OCDF	OCDD
1. Penn Nursery, PA	1.4	0.2	4.7	1.8	5.9	12.5	7.9	119.3	8.7	6,468
2. Clinton Crops, NC	0.3	0.1	0.8	0.6	0.9	6.7	1.6	55.9	1.3	1,299
4. Everglades, FL	1.3	0.0	19.4	0.2	19.8	12.5	25.8	89.2	31.9	578
5. Lake Dubay, WI	0.9	0.1	1.2	0.5	2.2	2.6	5.0	11.0	3.8	82
6. Monmouth, IL	1.3	0.5	3.0	2.6	9.9	19.6	51.5	64.0	30.1	308
7. McNay Farm, IA	0.5	0.8	1.7	3.1	6.0	17.9	18.4	107.0	20.1	1,568
8. Lake Scott, KS	0.1	0.0	0.4	0.1	0.6	0.6	0.7	2.6	0.6	19
9. Keystone State Park, OK	5.7	0.0	5.2	0.4	3.3	2.3	4.3	11.2	3.3	51
10. Arkadelphia, AR	0.7	0.0	0.6	0.3	0.9	3.7	1.3	31.3	1.1	650
11. Bennington, VT	1.2	0.1	4.4	1.0	5.4	6.1	8.6	30.4	7.9	140
12. Jasper, NY	3.2	0.4	5.4	3.9	11.6	42.3	31.7	442.9	25.5	10,910
14. Caldwell, OH	0.2	0.1	1.0	0.7	2.6	5.4	5.8	48.8	6.2	2,252
16. Dixon Springs, IL	0.5	0.3	3.8	4.3	26.7	41.9	100.1	412.3	108.0	9,116
17. Quincy, FL	0.0	0.0	0.2	0.1	1.1	1.7	4.0	15.5	5.1	352
18. Bay St. Louis, MS	0.6	0.2	1.7	2.0	7.9	17.8	22.4	136.1	18.7	1,530
19. Padre Island, TX	0.0	0.0	0.1	0.1	1.2	1.4	1.2	3.5	2.3	69
20. Fond du Lac, MN	0.5	0.0	1.9	0.6	6.8	4.5	24.7	28.7	28.7	97
21. North Platte, NE	2.3	0.2	2.1	0.1	2.4	2.2	3.5	9.7	3.4	38
22. Goodwell, OK	0.3	0.0	2.6	0.4	7.5	7.0	17.1	80.1	15.5	201
23. Big Bend, TX	0.2	0.0	0.2	0.1	0.6	0.3	0.7	1.5	1.4	20
24. Grand Canyon, AZ	0.0	0.0	0.3	0.0	1.5	0.9	3.2	6.1	2.4	18
25. Theodore Roosevelt, ND	0.4	0.1	4.2	0.4	5.2	2.8	7.0	16.6	4.0	87
27. Chiricahua, AZ	0.3	0.1	1.0	1.5	1.4	10.8	2.4	58.1	2.8	1,040
28. Rancho Seco, CA	0.6	0.0	1.1	0.9	3.9	7.2	21.8	36.2	22.7	159
29. Marvel Ranch, OR	2.1	0.1	4.1	1.2	31.9	77.5	158.4	583.0	87.1	2,872
30. Ozette Lake, WA	5.1	0.6	1.9	13.6	3.5	10.5	11.5	11.8	6.0	69
34. Trapper Creek, AK	1.0	0.0	0.1	0.1	0.3	0.4	1.8	2.3	0.5	12
Mean	1.1	0.2	2.7	1.5	6.3	11.8	20.1	89.4	16.6	1,482
Standard deviation	1.5	0.2	3.7	2.7	7.9	17.1	34.7	147.6	25.6	2,820
Standard error	0.3	0.0	0.7	0.5	1.5	3.3	6.7	28.4	4.9	543

Table 4. Literature summary for CDDs/CDFs in rural soils of North America

Concentration (pg/g)	Location	Site		Reference
		Description	n	
Range: ~990–3100	Elk River, MN	Semi-rural, untilled area	2	Reed et al. (1990)
Range: ND–810 Mean: 73	Ontario, Canada, and U.S. Midwest	Rural	30	Birmingham (1990)
Range: 60–10,000	Michigan and Indiana	Undisturbed	4	Brzuzy and Hites (1995)
Range: 9–258 Mean: 94	Washington	Open, nongrazed	4	Rogowski et al. (1999, 2005)
Range: 79–426 Mean: 267	Washington	Forest, noncommercial	4	Rogowski et al. (1999); Rogowski and Yake (2005)
Range: 19–11,483 Mean: 1,632	United States	Rural/remote	27	Current study

chlorinated biphenyl homologue concentrations were the highest, with an average concentration of 1,013 pg/g.

Only a few studies were found that reported total PCB levels in rural soils (Table 6). The only U.S. study (Vorhees et al., 1999) measured PCBs in residential soils near New Bedford Harbor, MA. The sediments in that area are contaminated with PCBs and may have affected nearby residential soils. The study reported a maximum concentration of 1,800,000 pg/g, which far exceeds the maximums seen in rural areas in other countries and is not likely to be representative of rural areas in the United States. The other three studies summarized in Table 6 show concentrations in rural areas at a variety of locations world-wide, with a range of 26 to 97,000 pg/g. This range is wider but similar to that observed in the current study (255–24,600 pg/g). The average from the large (n = 191) world-wide study by Meijer et al. (2003) was 5,400 pg/g, which is similar to the mean in the current study of 3,087 pg/g.

Total PCBs in procedural blanks averaged 170 pg/g. Although most of the sites exceeded this value by a large margin, the sites with the lowest total PCBs approached this value, i.e., the concentration at the lowest site was 255 pg/g. All congener values and associated blanks are listed in Appendix E. Up to 16 PCBs were detected at >3 pg/g in each blank. The blanks should be considered when interpreting individual congener values, especially those at the low-level sites.

Table 5. PCB homologue concentrations (pg/g dry weight)[a]

Site	Mono-CBs (1–3)	Di-CBs (4–15)	Tri-CBs (16–39)	Tetra-CBs (40–81)	Penta-CBs (82–127)	Hexa-CBs 128–169)	Hepta-CBs (170–193)	Octa-CBs (194–205)	Nona-CBs (206–208)	Deca-CBs (209)
1. Penn Nursery, PA	6	16	33	83	241	391	277	193	82	45
2. Clinton Crops, NC	7	25	26	41	67	100	86	62	39	21
4. Everglades, FL	34	92	164	281	408	596	599	271	100	57
5. Lake Dubay, WI	646	4,472	5,036	3,043	1,229	900	279	55	23	20
6. Monmouth, IL	11	41	115	237	580	646	281	54	32	41
7. McNay Farm, IA	3	10	21	36	78	94	57	32	16	12
8. Lake Scott, KS	12	50	112	155	342	269	126	37	7	4
9. Keystone State Park, OK	121	466	441	341	364	397	234	72	17	10
10. Arkadelphia, AR	24	72	168	371	1,788	1,201	328	59	11	5
11. Bennington, VT	8	38	83	110	277	797	1,068	525	89	29
12. Jasper, NY	7	23	56	118	336	482	307	134	52	28
14. Caldwell, OH	3	12	34	125	451	250	93	41	8	3
16. Dixon Springs, IL	2	10	25	57	132	146	70	45	52	306
17. Quincy, FL	5	11	28	46	71	47	26	19	13	38
18. Bay St. Louis, MS	7	22	155	829	1,756	1,311	566	218	54	12
19. Padre Island, TX	3	9	20	38	88	57	22	11	4	2
20. Fond du Lac, MN	15	29	94	193	390	378	150	36	13	9
21. North Platte, NE	4	10	26	63	144	123	68	30	14	12
22. Goodwell, OK	140	335	460	803	681	1,328	914	245	42	8
23. Big Bend, TX	5	21	52	1,655	15,170	6,755	800	95	13	4
24. Grand Canyon, AZ	6	13	60	149	181	210	76	13	3	2
25. Theodore Roosevelt, ND	3	5	22	47	137	174	125	44	10	4
27. Chiricahua, AZ	3	9	22	49	130	114	81	51	30	21
28. Rancho Seco, CA	9	43	130	518	1,163	996	288	90	30	6
29. Marvel Ranch, OR	14	41	44	149	343	385	183	87	41	13
30. Ozette Lake, WA	56	90	203	390	517	643	303	83	69	64
34. Trapper Creek, AK	14	35	83	233	299	389	141	25	3	2
Mean	43	222	286	376	1,013	710	279	97	32	29
Standard deviation	125	856	956	637	2,868	1,268	277	111	28	58
Standard error	24	165	184	123	552	244	53	21	5	11

[a] Values in parenthesis in the header indicate which PCBs are included in the homologue groups.

Table 6. Literature summary for PCBs in rural soils world-wide

Concentration (pg/g)	Location	Site Description	Site n	Reference
Range: 15,000–1,800,000[a]	New Bedford Harbor, MA	Residential	34	Vorhees et al. (1999)
Range: ND–45,000	Canadian Arctic	Remote, but near radar stations	21	Bright et al. (1995)
Range: 26–97,000 Mean: 5,400	World-wide	Background	191	Meijer et al. (2003)
Mean: 15,000	Poland	Forest soils	4	Masahide et al. (1998)
Range: 255–24,600 Mean: 3,087	United States	Rural/remote	27	Current study

[a] This site was included in this table because it was the only US study found which came close to meeting the criteria of reporting total PCBs in rural areas. However, it should be noted that the site is more suburban than rural and has probably been affected by nearby contaminated sediments.

6.4. TEQ RESULTS

CALUX bioassay TEQ testing was performed on soils from 135 individual sampling points (27 sites, 5 sampling points per site), 27 composites (1 per site), and 16 related field blanks. The complete set of CALUX analysis is presented in Appendix I.

The TEFs listed in Table 1 were used to calculate TEQs for the HRMS data. The TEQ estimates for the composite samples from all sites are shown in Table 7. This table shows both the CALUX-derived and the HRMS-derived estimates and the HRMS estimates on a TEQ1 basis (nondetects equal to zero) and a TEQ2 basis (nondetects equal to one-half the detection limit). The treatment of nondetects made a difference at many of the sites, particularly those with lower concentrations. For the HRMS CDD/CDF concentrations, TEQ2 values exceeded TEQ1 values by more than 50% at 3 sites. For the HRMS PCB concentrations, TEQ2 values exceeded TEQ1 values by more than 50% at 18 sites.

The composite range and averages across sites are summarized below:

HRMS CDD/CDF TEQ2: 0.21 to 11.42 pg/g, average of 1.69 pg/g (SE = 0.48, SD = 2.47)

HRMS PCB TEQ2: 0.017 to 0.38 pg/g, average of 0.072 pg/g (SE = 0.02, SD = 0.082)

HRMS Total TEQ2: 0.24 to 11.49 pg/g, average of 1.76 pg/g (SE = 0.48, SD = 2.47)

CALUX TEQ: 0.62 to 23.01 pg/g, average of 5.11 pg/g (SE = 1.04, SD = 5.38)

Table 7. TEQ soil concentrations by site (pg TEQ/g dry)[a]

	HRMS						
	CDD/CDF		PCB		Total TEQ1	Total TEQ2	CALUX Bioassay
Site	TEQ1	TEQ2	TEQ1	TEQ2			
1. Penn Nursery, PA	2.40	2.40	0.013	0.029	2.41	2.43	9.19±1.84
2. Clinton Crops, NC	0.68	0.69	0.010	0.023	0.69	0.71	2.10±0.33
4. Everglades, FL	1.23	1.48	0.020	0.045	1.25	1.53	2.16±0.89
5. Lake Dubay, WI	0.34	0.35	0.018	0.033	0.36	0.38	3.68±0.21
6. Monmouth, IL	2.42	2.43	0.034	0.049	2.45	2.48	4.97±0.43
7. McNay Farm, IA	2.05	2.05	0.011	0.027	2.06	2.08	11.04±0.30
8. Lake Scott, KS	0.08	0.21	0.013	0.031	0.09	0.24	1.58±0.76
9. Keystone State Park, OK	1.16	1.18	0.009	0.023	1.17	1.20	1.88±0.50
10. Arkadelphia, AR	0.53	0.53	0.047	0.062	0.58	0.59	2.92±0.35
11. Bennington, VT	0.97	0.98	0.210	0.210	1.18	1.19	5.86±0.64
12. Jasper, NY	5.97	5.97	0.012	0.031	5.98	6.00	7.04±1.74
14. Caldwell, OH	0.82	0.84	0.012	0.029	0.83	0.87	4.60±1.81
16. Dixon Springs, IL	6.24	6.24	0.049	0.049	6.29	6.29	12.61±0.00
17. Quincy, FL	0.22	0.41	0.005	0.022	0.23	0.43	1.54±0.11
18. Bay St. Louis, MS	2.08	2.08	0.236	0.236	2.32	2.32	17.06±1.01
19. Padre Island, TX	0.23	0.33	0.004	0.017	0.23	0.35	0.62±0.24
20. Fond du Lac, MN	0.67	0.70	0.015	0.029	0.69	0.73	2.87±0.11
21. North Platte, NE	0.50	0.50	0.083	0.083	0.58	0.58	6.33±0.21
22. Goodwell, OK	0.91	0.94	0.014	0.061	0.92	1.00	3.61±0.46
23. Big Bend, TX	0.13	0.23	0.363	0.379	0.49	0.61	0.62±0.69
24. Grand Canyon, AZ	0.12	0.25	0.004	0.062	0.12	0.31	0.82±0.84
25. Theodore Roosevelt, ND	0.44	0.44	0.004	0.019	0.44	0.46	1.05±0.47
27. Chiricahua, AZ	0.88	0.90	0.006	0.019	0.89	0.92	5.17±1.57
28. Rancho Seco, CA	1.05	1.05	0.046	0.132	1.10	1.18	2.69±0.35
29. Marvel Ranch, OR	11.37	11.42	0.010	0.070	11.38	11.49	23.01±3.19
30. Ozette Lake, WA	0.43	0.63	0.011	0.094	0.44	0.72	1.14±0.59
34. Trapper Creek, AK	0.11	0.29	0.007	0.078	0.12	0.37	1.79±0.58
Mean	1.63	1.69	0.047	0.072	1.68	1.76	5.11
Standard deviation	2.49	2.47	0.09	0.082	2.49	2.47	5.38
Standard error	0.48	0.48	0.02	0.02	0.48	0.48	1.04

[a] TEQ1 values based on nondetects equal to zero. TEQ2 values based on nondetects equal to one-half the detection limit.

HRMS = high-resolution mass spectrometry

EPA's draft report *Exposure and Human Health Reassessment of 2,3,7,8-Tetra-Chlorodibenzo-p-Dioxin (TCDD) and Related Compounds* (U.S. EPA, 2003) presents a preliminary mean CDD/CDF TEQ in North America soil of 2.8 pg TEQ_{DF}/g in rural soils and 9.4 pg TEQ_{DF}/g in urban soils. These estimates were derived setting nondetects equal to zero, so they were compared with the TEQ1 values from this study. This study found an average CDD/CDF TEQ1 of 1.63 pg TEQ/g, which is about 40% lower than the rural soil average reported in EPA's draft report (U.S. EPA, 2003). A summary of CDD/CDF TEQ values from nine studies of rural areas of North America is shown in Table 8. The overall range across the studies is 0 to 22.9 pg TEQ/g, which encompasses the TEQ2 range from the current study (0.21–11.42 pg TEQ/g). The means across studies ranged from 0.4 to 5 pg TEQ/g, which bracket the TEQ2 mean from this study (1.69 pg TEQ/g).

Table 8. Literature summary for CDD/CDF TEQs in rural soils of North America

TEQ Concentration (pg/g)	Location	Site		Reference
		Description	n	
Range: 0.16–2.2 Mean: 0.4	Ontario, Canada, and U.S. Midwest	Rural	30	Birmingham (1990)
Range: 0–57 Mean: 5	British Columbia, Canada	Background	53	BC Environment (1995)
Range: 0.2–0.9	Canadian Arctic	Remote	4	Grundy et al. (1995); Bright et al. (1995)
Range: 0.16–22.9 Mean: 3.1	Southern Mississippi	Rural	36	Rappe et al. (1995); Fiedler et al. (1995)
Mean: 1.4	Columbus, OH	Rural background	3	Lorber et al. (1998)
Range: 0.046–2.4 Mean: 0.71	Washington	Open, nongrazed	4	Rogowski et al. (1999, 2005)
Range: 0.45–5.2 Mean: 3.3	Washington	Forest, noncommercial	4	Rogowski et al. (1999); Rogowski and Yake (2005)
Mean: 5.74	Connecticut	Rural background	34	MRI (1992)
Range: 0.1–9.6 Mean: 1.6	Colorado	Open space background	36	U.S. EPA (2001)
Range: 0.21–11.42 Mean: 1.69	United States	Rural/remote	27	Current study

TEQ = toxicity equivalent

Only one study was found that reported PCB TEQs in rural North America soils (U.S. EPA, 2001). Thirty-six soil samples were collected from areas defined as "open space background" in the Front Range area near Denver, CO. The PCB TEQs averaged 1.2 pg/g, which is higher than the average from the current study of 0.07 pg/g. In fact, this mean exceeds the maximum value of this study (0.379 pg/g). The proximity of these sites to Denver may account for the difference. A rural soil survey was conducted in Poland in 2002 (Wyrzykowska et al., 2005). This study sampled soils in 13 agricultural areas and found a range of 0.054 to 0.42 pg TEQ/g and an average of 0.18 pg TEQ/g. Buckland et al. (1998) evaluated soils collected in New Zealand. The PCB concentrations ranged from 0.067 to 2.3 pg TEQ/g for provincial centers. The PCB TEQ average from the current study falls within the ranges reported for the studies in Poland and New Zealand.

PCBs are generally a small fraction of the total TEQs in soil. The only exception was Big Bend, TX (Site 23), where PCBs contributed 62% of the total TEQ2s.

The distribution of CDD/CDF and PCB TEQ values across sites is displayed graphically in Figures 3 and 4, respectively. Figure 5 compares the total TEQs by CALUX, HRMS TEQ1, and HRMS TEQ2. Figures 6 and 7 show frequency diagrams for levels of CDD/CDF TEQs and PCB TEQs, respectively, across all sites. These diagrams show the number of sites with levels within various ranges. For example, in Figure 6, the first bar shows the number of sites with concentrations between 0 and 1 pg TEQ2/g, the second bar shows the number of sites with concentrations between 1 and 2 pg TEQ2/g, and so on. These results show how the concentrations were distributed across sites. Section 7.3 discusses how the TEQ results derived from CALUX compare with those derived from the HRMS analysis.

CDD/CDF TEQs in procedural blanks had an average TEQ1 of 0.05 and an average TEQ2 of 0.21 pg/g. PCB TEQs in procedural blanks have an average TEQ1 of 0.0056 pg/g and an average TEQ2 of 0.020 pg/g. A few sites had TEQ levels near the blank levels, suggesting that TEQs were very low to nondetect at these sites.

6.5. MERCURY RESULTS

Table 2 provides mercury concentrations for each site composite. The complete set of mercury data is provided in Appendix J. Mercury concentrations ranged from 0.5 to 69 ng/g and averaged 22 ng/g across all sites (SE = 2.9, SD = 15 ng/g). The mean was calculated setting nondetects equal to half the detection limit; however, it was essentially the same whether nondetects were set to zero or to their full detection limit. As indicated in Table 2, three samples had holding times beyond the specified 28 days. Results for these three samples should be considered minimum values based on the holding time exceedance. Recomputing the mean without these samples reduced it to 21 ng/g. A summary of mercury concentrations from rural

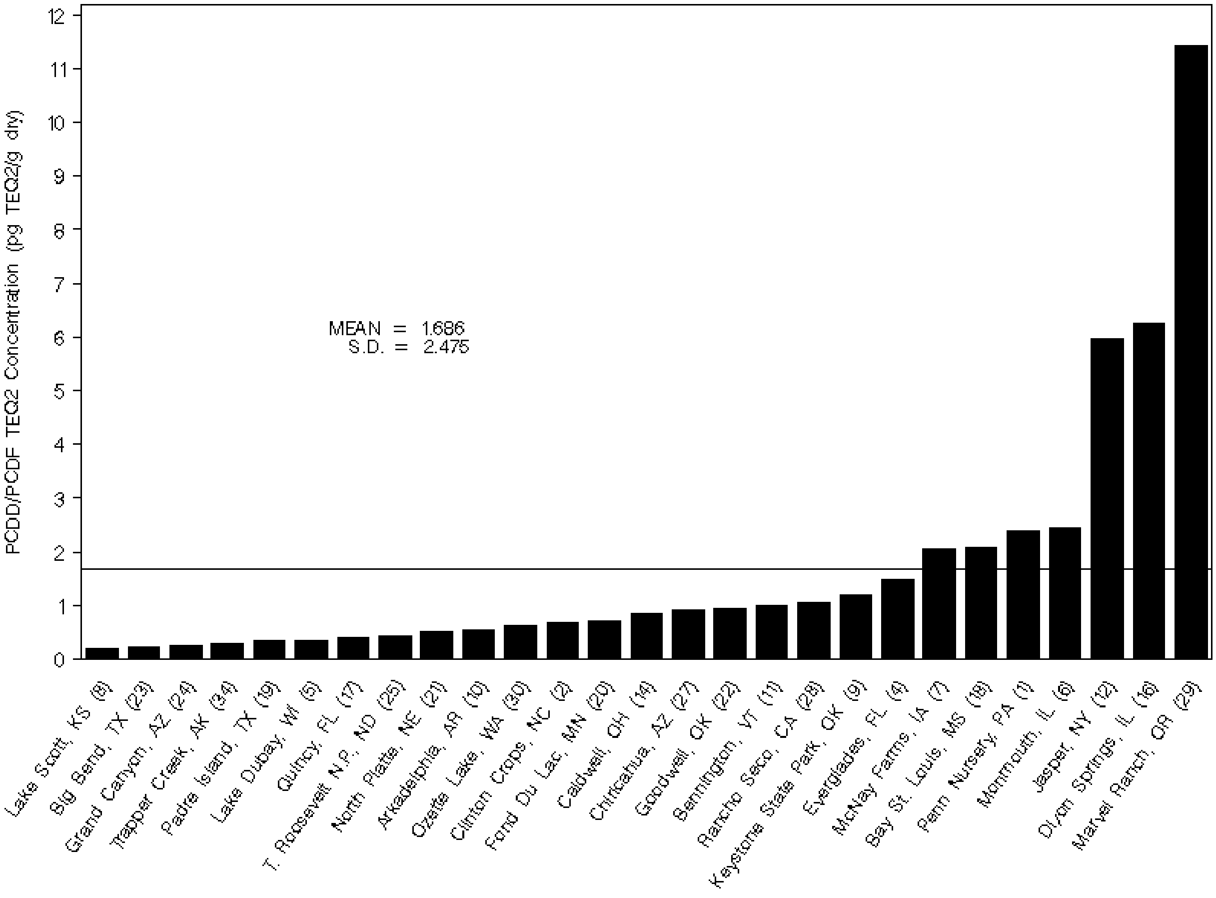

Figure 3. HRMS CDD/CDF TEQ2s for all 27 sites.

areas of North America is shown in Table 9. The five studies from the literature reported concentrations ranging from 10 to 600 ng/g. The mean from the current study (22 ng/g) falls within this range but near the lower end. Mercury was below detection limits in the procedural blanks.

The distribution of mercury concentrations across sites is displayed graphically in Figure 8. Figure 9 shows the frequency diagram for levels of mercury in soil across all sites. This diagram shows the number of sites with concentrations within various ranges. For example, the first bar shows the number of sites with concentrations between 0 and 0.01 mg/kg (0 and 10 ng/g), the second bar shows the number of sites with concentrations between 0.01 and 0.02 mg/kg (10 and 20 ng/g), and so on. These results show how the concentrations were distributed across sites.

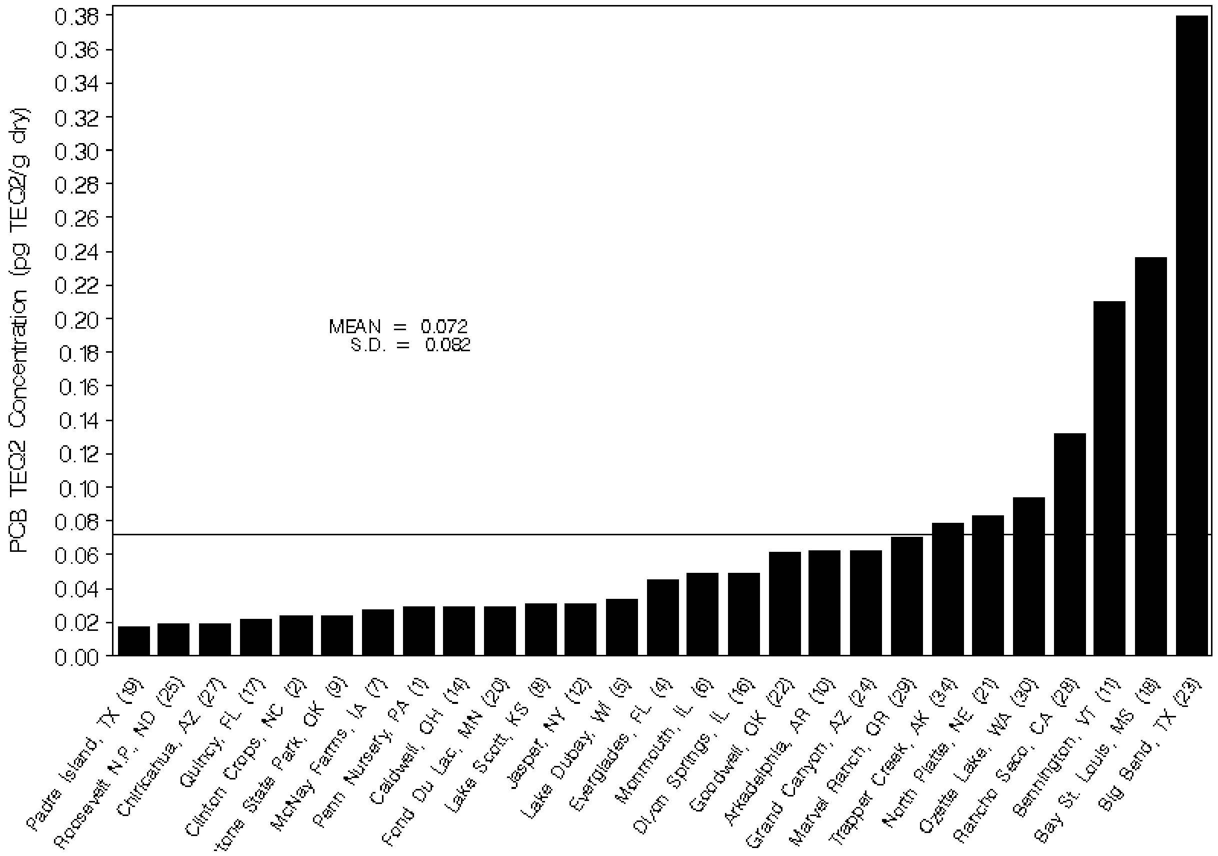

Figure 4. HRMS PCB TEQ2s for all 27 sites.

6.6. CONGENER PROFILES

Appendix K shows four congener profiles for each site: CDDs/CDFs, PCBs, CDD/CDF TEQs, and PCB TEQs. Each of the profiles is discussed below.

- *CDDs/CDFs.* These profiles are very similar across sites, with concentrations of OCDD being the highest at all sites. Heptachlorodibenzo-*p*-dioxins (HpCDDs) were second highest at all sites except three, where octachlorodibenzofuran (OCDF) was second highest. This pattern compares well with the Worldwide Deposition Profile presented by Brzuzy and Hites (1996).

- *PCBs.* These profiles are very similar across sites, with concentrations of PCB 118 being the highest at all sites. PCB 105 was second highest at all sites except one, where PCB 156/157 was second highest.

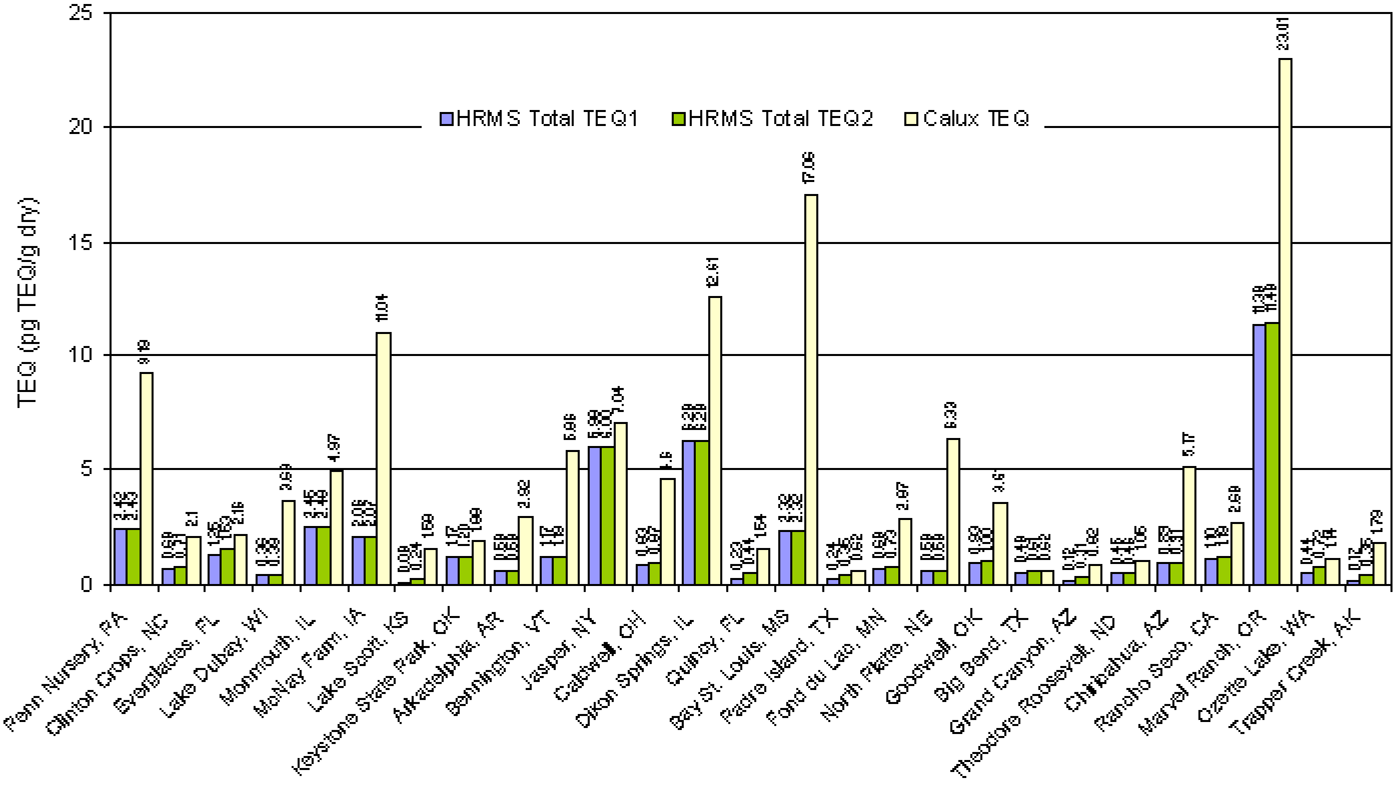

Figure 5. HRMS total TEQ1s, HRMS total TEQ2s, and CALUX TEQs for all 27 sites.

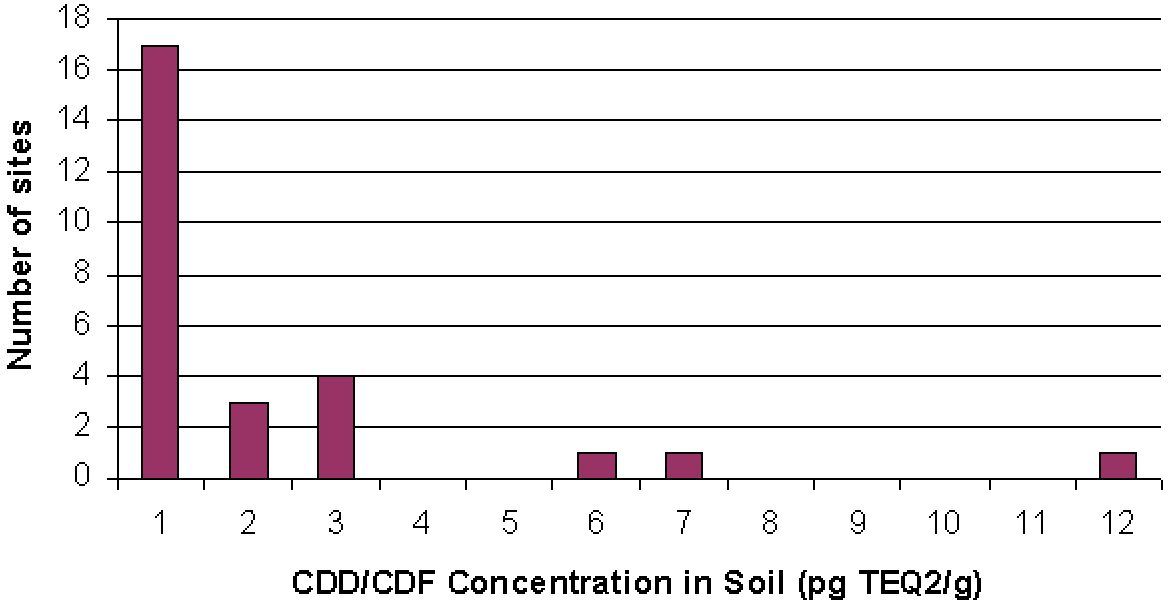

Figure 6. Frequency diagram for CDD/CDF TEQ2 concentrations among 27 sites.

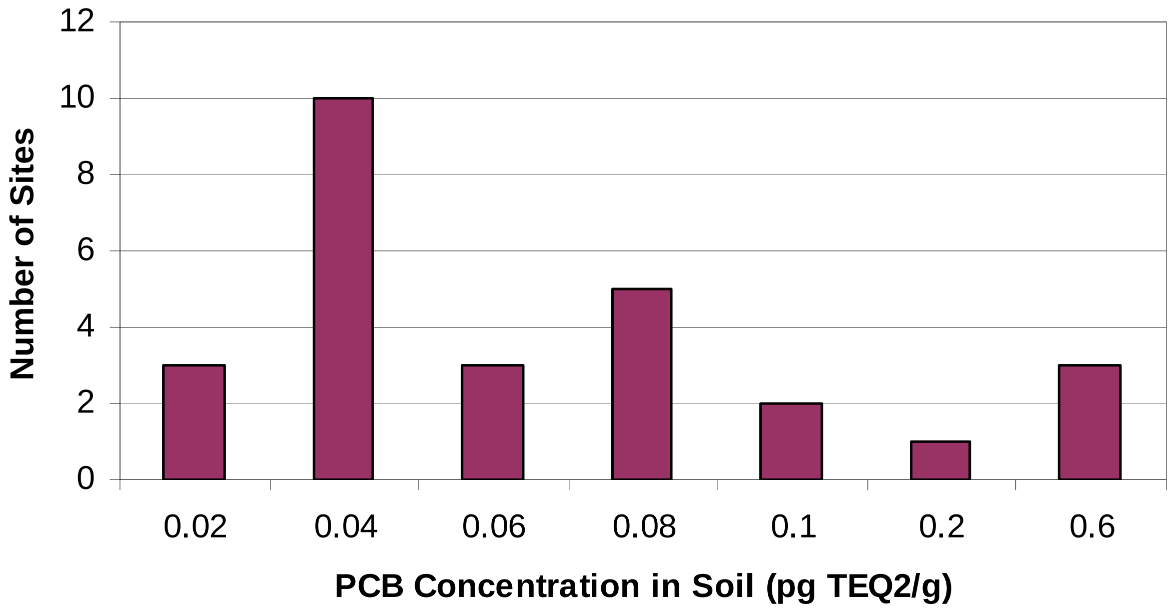

Figure 7. Frequency diagram for PCB TEQ2 concentrations among 27 sites.

Table 9. Literature summary for mercury in rural soils of North America

Concentration (ng/g)	**Location**	**Site**		**Reference**
		Description	**n**	
Range: <10–260	New Jersey	Rural	35	NJDEP (2001)
Range: 10–550	New York	Orchards	13	Merwin et al. (1994)
Range: 12–220	Minnesota	Wilderness	NR	Glass et al. (1990)
Range: <3.2–66 Mean: 11	Washington	Background	13	Rogowski et al. (1999)
Median: <100 Range: <25–600	Michigan	Background	431	MDEQ (2005)
Range: 0.5–69 Mean: 22	United States	Rural/remote	27	Current study

NR = not reported

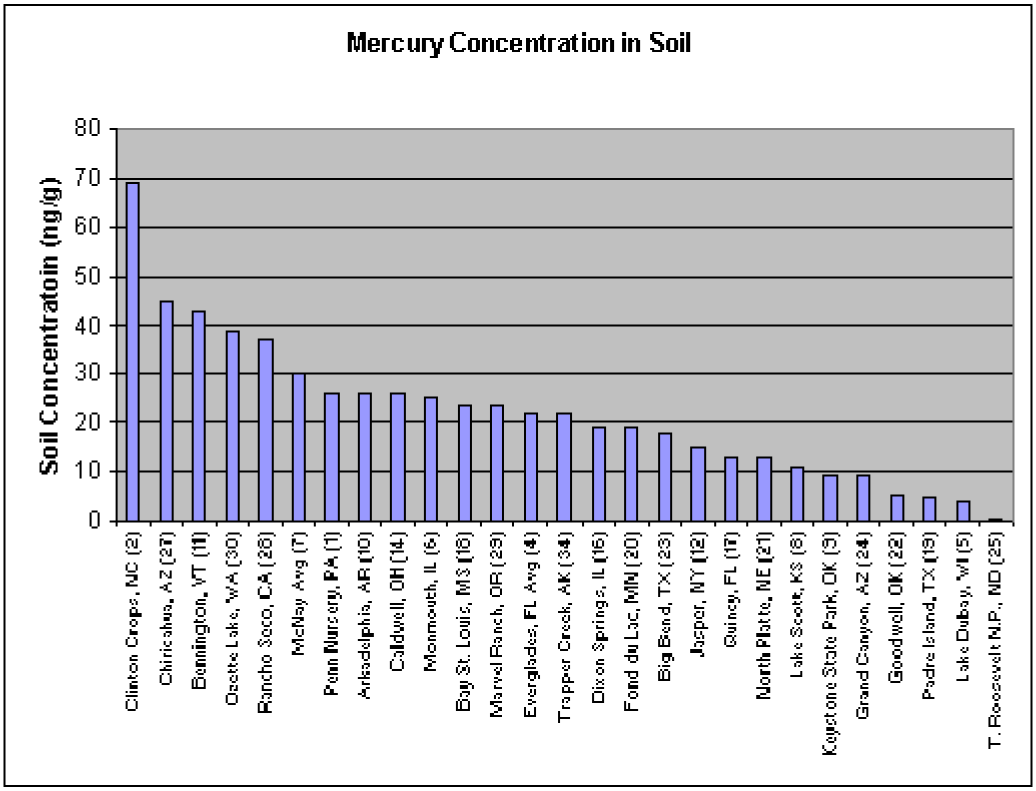

Figure 8. **Mercury concentrations at all 27 sites.**

- *CDD/CDF TEQs*. These profiles vary considerably across sites. The congeners with the highest concentrations were usually OCDD, HpCDDs, pentachlorodibenzo-*p*-dioxins (PeCDDs), or 2,3,4,7,8-pentachlorodibenzofuran (2,3,4,7,8-PeCDF). For TEQ1 and TEQ2 values, in general the HpCDDs and OCDD were significant contributors to TEQ. 1,2,3,7,8-PeCDD and 2,3,4,7,8-PeCDF also contributed significantly in instances where these congeners were detected.

- *PCB TEQs*. These profiles are very similar across sites, with concentrations of PCB 126 (on a TEQ2 basis) being the highest at all sites except two, where PCB 118 was highest. Because of its large TEF, PCB 126 was also a significant contributor to TEQ1 when detected. PCBs 118, 105, 156/157, and 169 were important contributors to TEQ1 and TEQ2 for many sites.

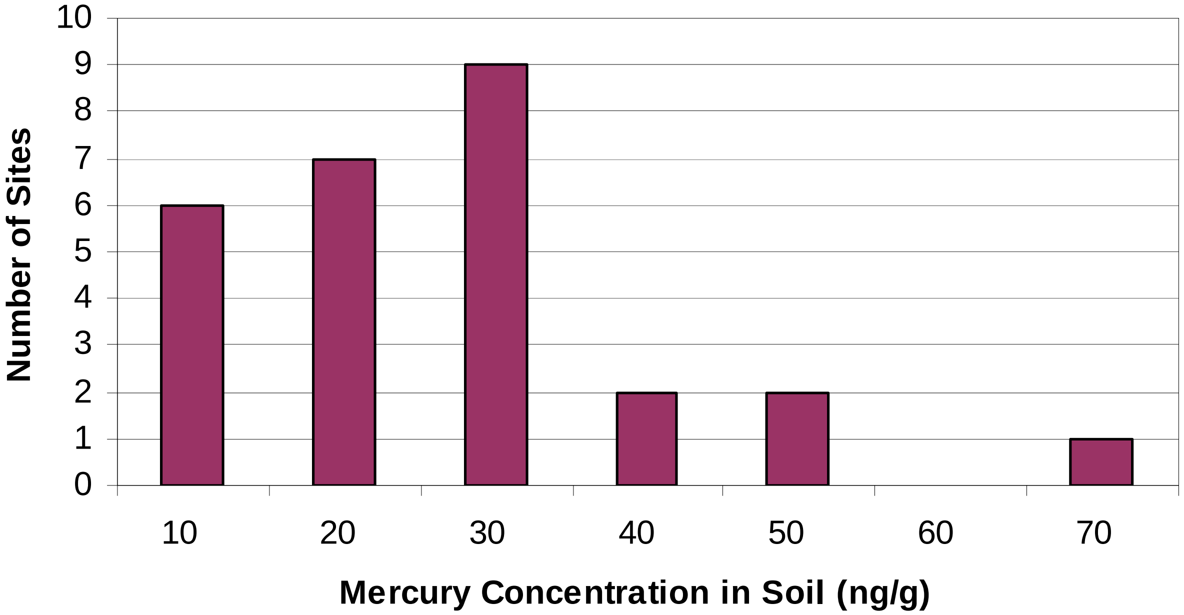

Figure 9. Frequency diagram for mercury concentrations among 27 sites.

7. COMPARATIVE ANALYSES

This chapter presents analyses that compare (1) chemical concentration levels in air with those in soil, (2) chemical concentration levels in soil with those of TOC in soil, and (3) HRMS-derived TEQs with CALUX-derived TEQs. For all analyses, the criteria for statistical significance is $p \leq 0.05$.

7.1. COMPARISON OF AIR AND SOIL CONCENTRATIONS

Air samples have been collected at NDAMN sites across the United States since 1998 and analyzed for concentrations of CDDs/CDFs and certain PCBs. NDAMN used high-volume air samplers that operated for 30 days, four times per year. The NDAMN data for the years 1998 to 2001 are presented in Cleverly et al., 2006 and U.S. EPA, 2005a,b. The comparisons were made using air data that most closely matched the date that the soil samples were collected (2003). Because the peer review of the 2001 air data had not yet been completed when this document was prepared, the 2000 air data were selected for this analysis. NDAMN collected air samples in 2003 so future studies could compare air and soil samples collected in the same year.

Note that Site 29 was located at Hyslop Farms, OR, in 2000 and was moved a few miles to Marvel Ranch, OR, in 2003 because Hyslop Farms is near a major transportation route and

Marvel Ranch is located in a more remote area away from transportation. The air sample was collected at Hyslop Farms and the soil sample at Marvel Ranch. Also, the NDAMN stations Site 28 (Rancho Seco, CA) and Site 34 (Trapper Creek, AK) were not in operation in 2000, so these sites were not included in the analysis.

7.1.1. Air and Soil Concentrations

This section evaluates reationships between the chemical levels in the soil and air across sites. Scatter plots of the raw data generally showed no obvious correlation as many concentrations are clustered close to zero and become more widely dispersed as concentration increases. Often when scatter plots have this form, it is useful to evaluate the correlation based on the natural log of the data. This is illustrated in Figure 10, which shows paired scatter plots for a number of the homologues on the basis of both the raw data and the natural log transform of the data. Table 10 summarizes the natural log linear correlations between air and soil levels across sites for PCBs, CDD/CDFs, and TEQs. The following observations can be made:

- *PCBs.* The PCB air data included only six congeners, limiting the analyses that could be done. Only PCB 77 showed a significant correlation with r = 0.47. The PCB TEQs did not yield a significant correlation.

- *CDDs/CDFs*. The analyses of the CDD/CDF data showed that correlations were significant for four homologues: PeCDDs (r = 0.40), HxCDDs (r = 0.42), HpCDDs (r = 0.48), and OCDD (r = 0.52). Additionally, significant correlations were found for total CDDs (r = 0.51) and total CDDs/CDFs (r = 0.53). None of the furan homologues showed significant correlations.

- *TEQs.* The analyses of the TEQ data showed that the correlations were significant for TEQ_{DF} (r = 0.58) and TEQ_{DFP} (r = 0.54).

The air sample and soil sample TEQs were calculated using the same TEFs; however, the PCB TEQ calculation for soils included additional PCB compounds: PCBs 81, 114, 123, 167, and 189. Figure 11 illustrates the annual average of quarterly air sample TEQ concentrations collected in 2000, and Figure 12 illustrates the soil sample TEQ concentrations collected during one sampling event in 2003. When visually comparing the total TEQ levels in air and soil shown in Figures 11 and 12, some similarities and some differences can be seen:

- Marvel Ranch, OR (Site 29), had the highest air level and the highest soil level. Two other sites had relatively high soil levels: Jasper, NY (Site 12), and Dixon Springs, IL (Site 16). Site 16 had a relatively high air level, but Site 12 did not. Grand Canyon, AZ (Site 24), had the lowest air level and the lowest soil level.

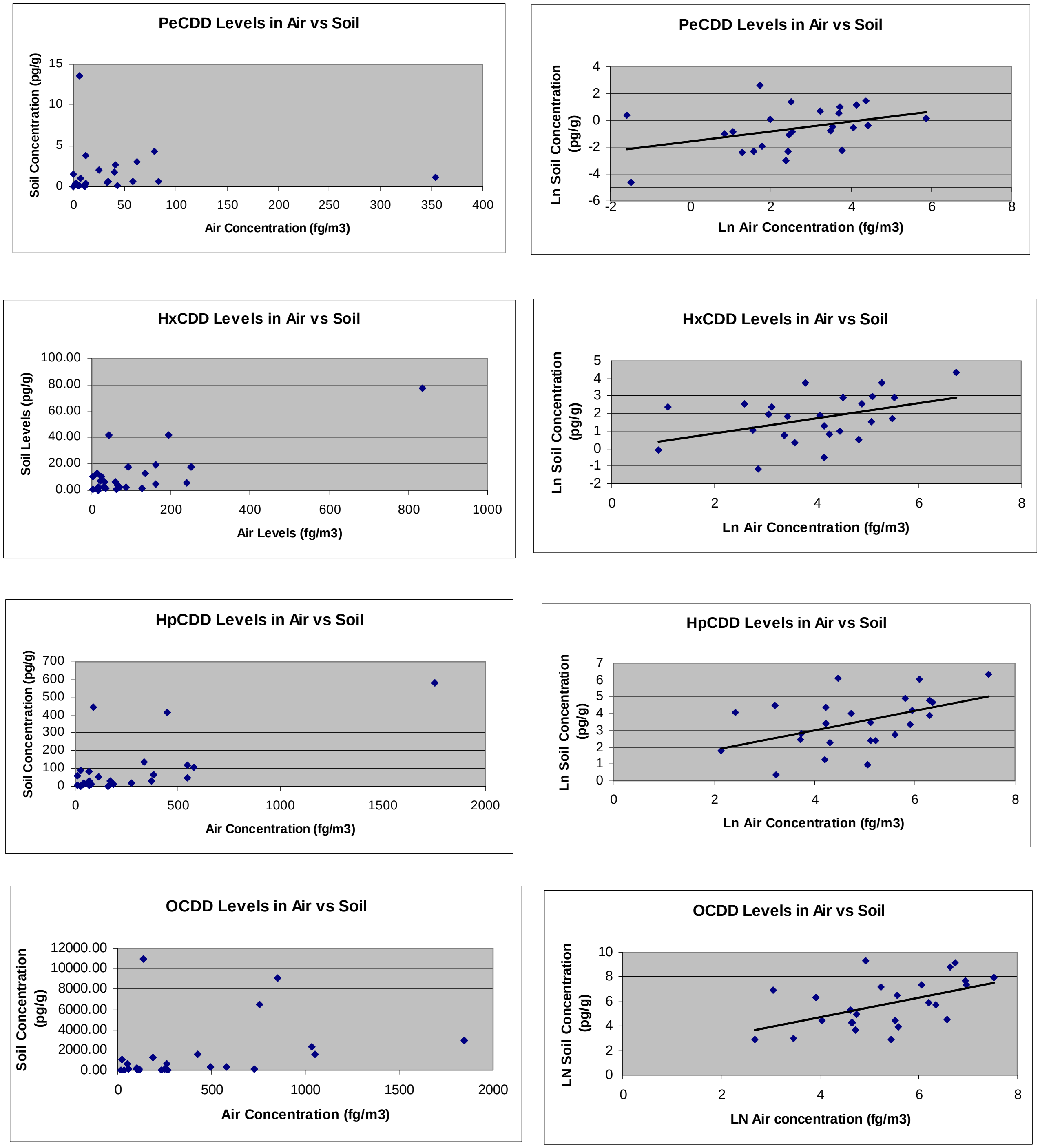

Figure 10. Scatter plots of homologue levels in air versus soil.

Table 10. Correlations across sites between soil concentrations and air concentrations[a,b]

Compound	Correlation coefficient	*p*-value
Total TCDFs	0.11	0.61
Total TCDDs	0.25	0.24
Total PeCDFs	0.15	0.49
Total PeCDDs	**0.40**	**0.05**
Total HxCDFs	0.24	0.26
Total HxCDDs	**0.42**	**0.03**
Total HpCDFs	0.37	0.07
Total HpCDDs	**0.48**	**0.01**
OCDF	0.36	0.07
OCDD	**0.52**	**0.01**
Total CDDs	**0.51**	**0.01**
Total CDFs	0.32	0.11
Total CDDs/CDFs	**0.53**	**0.01**
PCB 77	**0.47**	**0.02**
PCB 105	0.33	0.11
PCB 118	0.31	0.14
PCB126	-0.02	0.94
PCB 156/157	-0.02	0.94
PCB 169	0.16	0.43
TEQ_{DF}	**0.58**	**0.00**
TEQ_{PCB}	0.21	0.32
TEQ_{DFP}	**0.54**	**0.01**

[a] All correlations are based on natural log transforms of the data.

[b] Correlations where $p \leq 0.05$ are shown in bold

- In general, the percent of PCB TEQ contribution to the total TEQ is similar for air samples and soil samples. Notably, Grand Canyon, AZ (Site 24), had a high percentage of PCB relative to the total TEQ in both air and soil. Two sites had a higher percentage of PCB to total TEQ in soil samples than in the air samples: Big Bend, TX (Site 23), and Bennington, VT (Site 11). Bay St. Louis, MS (Site 18), had the highest percentage of PCB TEQs in the air, but a moderate percentage of PCBs in the soil.

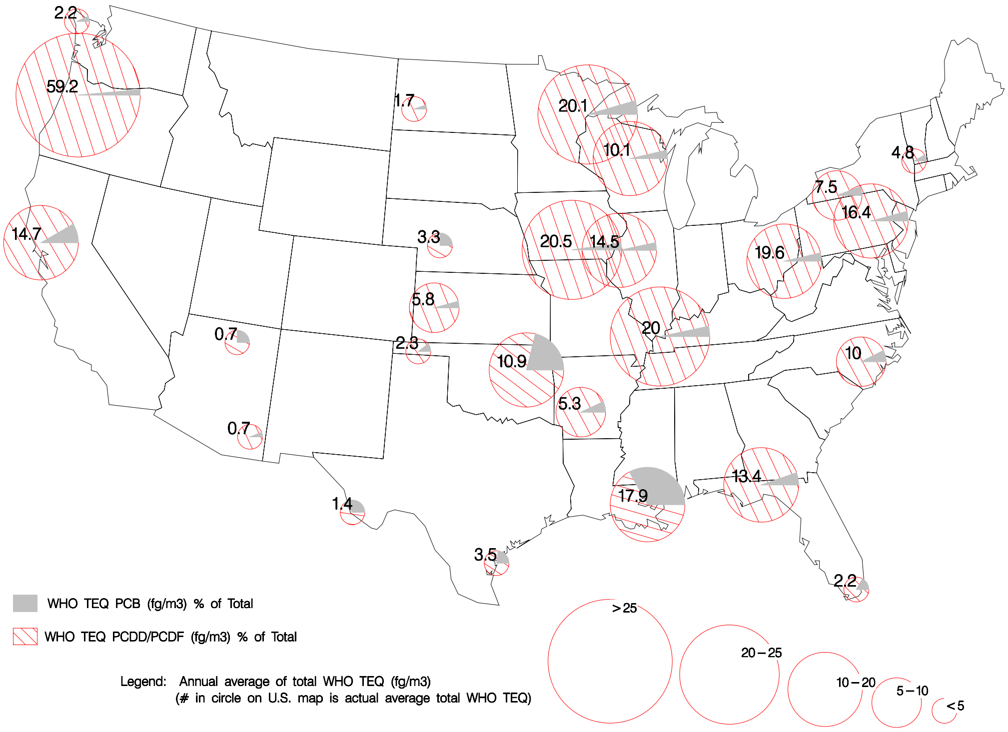

Figure 11. Annual average total TEQs for air samples obtained at NDAMN sites in 2000.

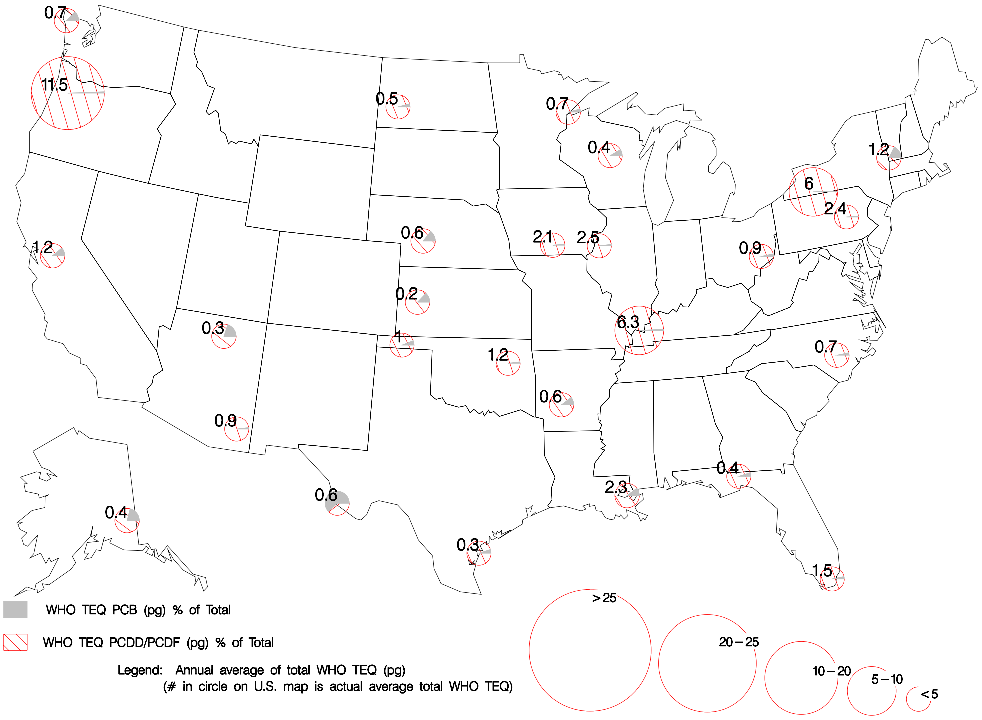

Figure 12. Total TEQ2 for soil samples taken at NDAMN sites in 2003.

Figures 13 and 14 show the scatter plots for the TEQ data on the basis of the raw data and the natural log-transformed data, respectively. The regression line for the log-scale version is:

$$\mathrm{Ln}(\text{soil concentration}) = -0.749 + 0.455\ \mathrm{Ln}(\text{air concentration})$$

The standard error on the intercept is 0.304. The standard error on the slope is 0.144. The closeness of the slope of the regression line to 0.5 indicates a relationship between the soil concentration and the square root of the air concentration. This relationship is not particularly strong (r = 0.54), but it should not be discounted.

Two facts should be considered while evaluating these air-soil correlations. First, dioxin levels in soil result from accumulation over many years, but the air samples used in this comparison were collected over just one year. Second, dioxin levels in the environment have changed over time. Dioxin levels began to rise in the 1930s and peaked in the early 1970s (U.S. EPA, 2003). Also, emissions in the United States decreased approximately 90% from 1987 to 2000 (U.S. EPA, 2006). Accordingly, soil levels may better reflect past air levels than current levels.

7.1.2. Air and Soil Congener Profiles

Appendix L shows paired air and soil congener profiles for all the sites except Rancho Seco, CA (Site 28), and Trapper Creek, AK (Site 34), which had no air data in 2000. These comparisons are presented for the 2,3,7,8-substituted CDD/CDF congeners and six PCB congeners. The PCB congeners included in this analysis were limited to the ones measured in both air and soil (PCBs 77, 118, 106, 126, 156/157, and 169). The air data used to derive these profiles were from samples collected in 2000, but the draft NDAMN report for 2001 suggests that the air congener profiles are similar to those in 2000 (U.S. EPA, 2005b). Observations from these profiles are described below.

- *CDDs.* The two prominent congeners in air and soil were the same at all sites (1,2,3,4,6,7,8-HpCDD and OCDD). The order of congeners in the air and soil was generally the same at most sites: from low to high, tetra, penta, hexas, heptas, and octa. At most sites, the relative proportion of octa to the other congeners was higher in the soil than in the air.

- *CDFs.* The two prominent congeners in air and soil were the same at all sites (1,2,3,4,6,7,8-heptachlorodibenzofuran [1,2,3,4,6,7,8-HpCDF] and OCDF). The order of congeners in the air and soil was somewhat variable across sites. The similarity in relative proportions of the congeners in air and soil was difficult to judge visually.

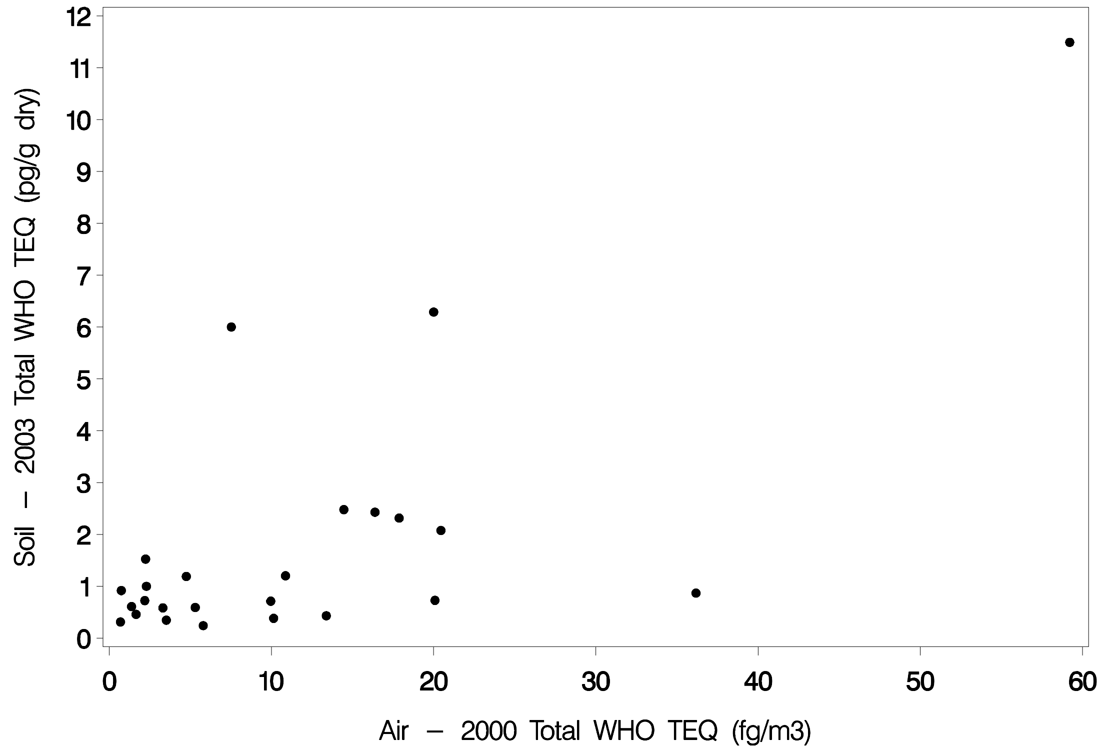

Figure 13. Annual average air total TEQs versus soil total TEQ2s (raw data).

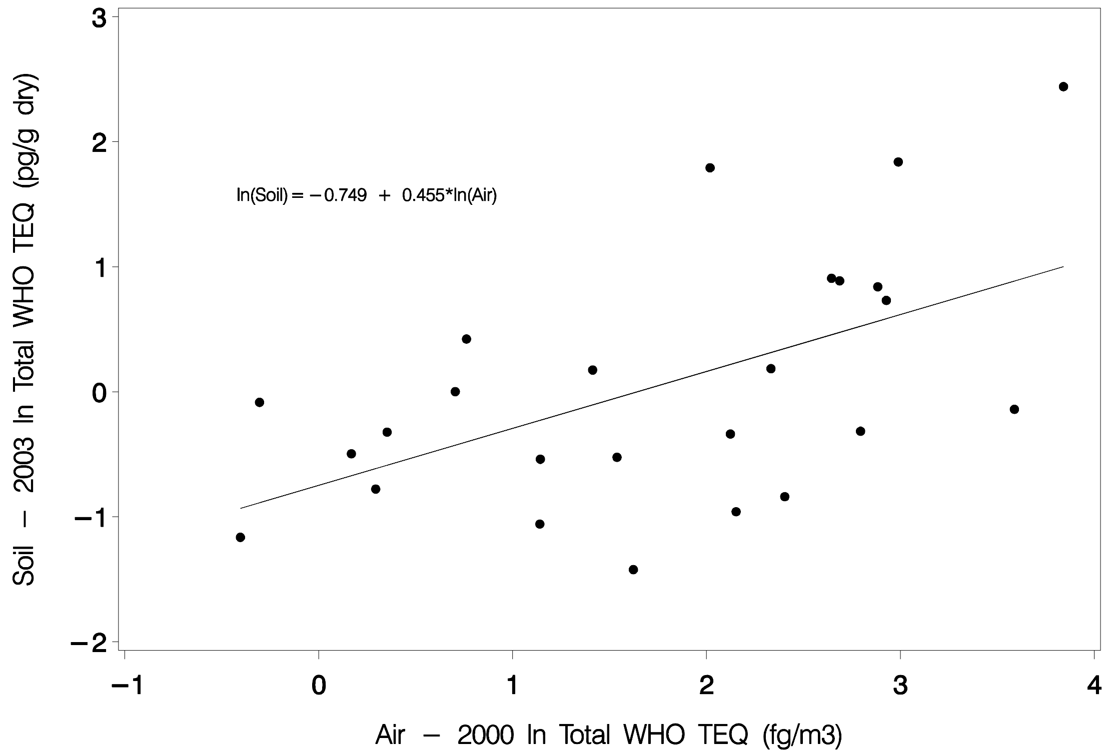

Figure 14. Annual average air total TEQs versus soil total TEQ2s (log transformed data).

- *PCBs.* The two prominent congeners in air and soil were the same at all sites (PCBs 118 and 105). The order of congeners in the air and soil was generally the same at most sites: from low to high, PCBs 169, 126, 77, 156/157, 105, and 118. At most sites, the relative congener proportions in air appeared similar to the proportions in soil.

7.2. COMPARISON OF SOIL CONCENTRATION WITH TOTAL ORGANIC CARBON CONCENTRATION

The tendency for persistent chemicals to bind to organic carbon suggests that CDD/CDF, PCB, and mercury levels in soil may correlate with TOC levels in soil. Other researchers have reported mixed results on this issue. Brzuzy and Hites (1995) observed a strong correlation between organic carbon and total CDDs/CDFs at some sites. At sites where this was not observed, they theorized that deposition exceeded the sorption capacity of the soil. Wilcke and Amelung (2000) studied PCBs in North American grasslands and found no correlation between organic carbon and 14 PCBs. EPA reported that mercury levels in soil are positively correlated with organic matter (U.S. EPA, 1997).

Scatter plots were used to investigate relationships between chemical levels in the soil and TOC levels in the soil across sites. As illustrated in Figure 15, the plots for a number of homologues suggested positive correlations. Correlation analyses were conducted for mercury, TEQs, and all homologue groups (Table 11). Based on these analyses, the following observations can be made:

- *Mercury.* The correlation was not significant.

- *PCBs.* The only significant correlation was for nonachlorobiphenyls (r = 0.47).

- *CDDs/CDFs.* The correlations were significant for seven homologues: TCDFs (r = 0.43), PeCDFs (r = 0.51), PeCDDs (r = 0.44), hexachlorodibenzofurans (HxCDFs) (r = 0.47), HxCDDs (r = 0.46), HpCDFs (r = 0.38), and HpCDDs (r = 0.38). Also, total CDFs had a significant correlation (r = 0.39).

- *TEQs.* The TEQ correlation was significant (r = 0.43). The scatter plot for these data are shown in Figure 16.

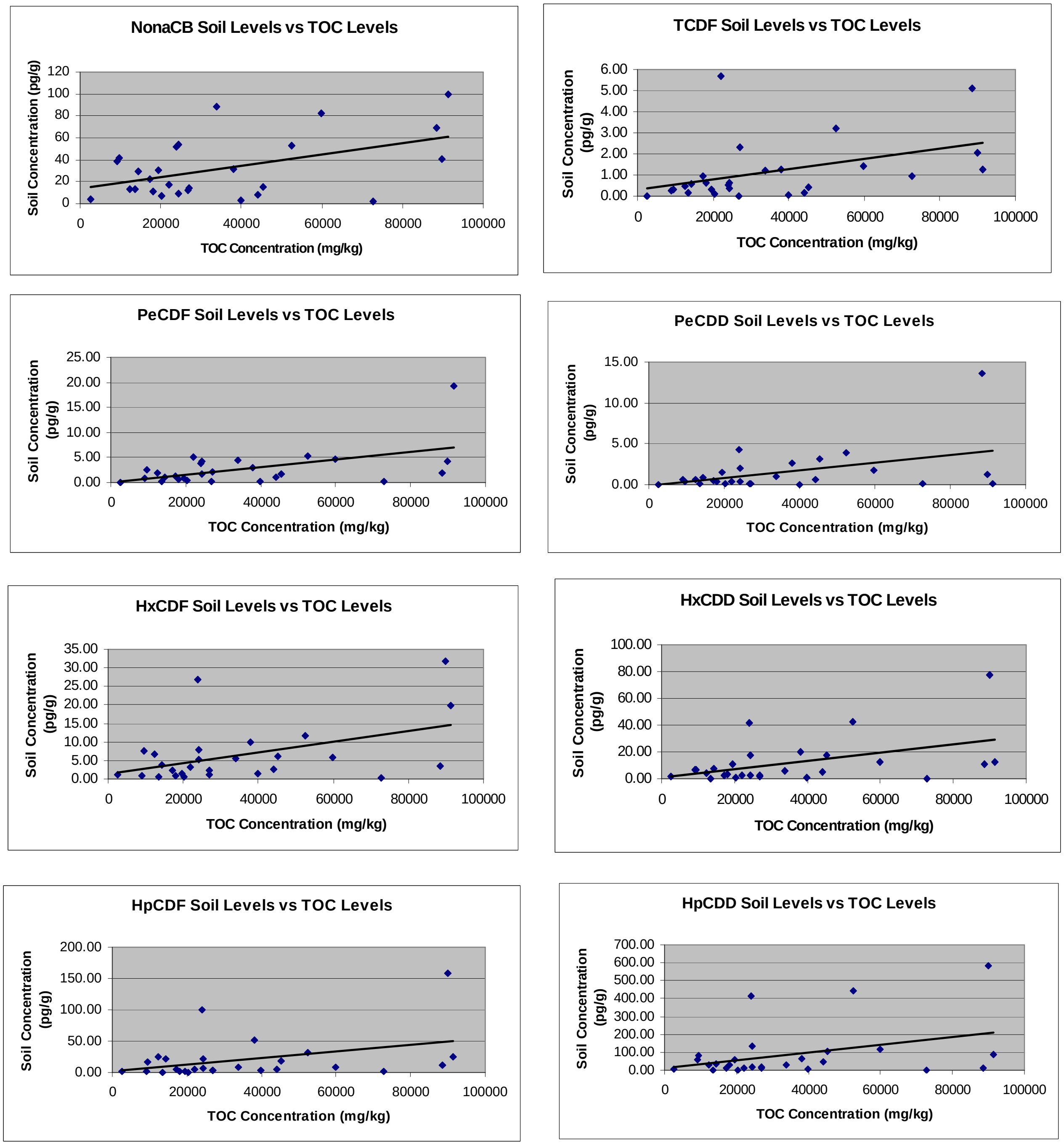

Figure 15. Scatter plots of chemical levels in soil vs TOC levels in soil.

Table 11. Correlation across sites between soil concentration and total organic carbon concentration[a]

Chemical	Correlation coefficient	*p*-value
Mercury	0.11	0.59
Mono-CBs	-0.14	0.49
Di-CBs	-0.15	0.46
Tri-CBs	-0.15	0.47
Tetra-CBs	-0.21	0.28
Penta-CBS	-0.19	0.34
Hexa-CBs	-0.18	0.38
Hepta-CBs	0.01	0.97
Octa-CBs	0.17	0.41
Nona-CBs	**0.47**	**0.01**
Deca-CBs	0.08	0.68
Total PCBs	-0.22	0.27
Total TCDFs	**0.43**	**0.03**
Total TCDDs	0.32	0.10
Total PeCDFs	**0.51**	**0.01**
Total PeCDDs	**0.44**	**0.02**
Total HxCDFs	**0.47**	**0.01**
Total HxCDDs	**0.46**	**0.02**
Total HpCDFs	**0.38**	**0.05**
Total HpCDDs	**0.38**	**0.05**
OCDD	0.21	0.30
OCDF	0.27	0.17
Total CDDs	0.22	0.27
Total CDFs	**0.39**	**0.04**
Total CDDs/CDFs	0.23	0.25
Total TEQ2	**0.43**	**0.02**

[a] Correlations where $p \leq 0.05$ are shown in bold

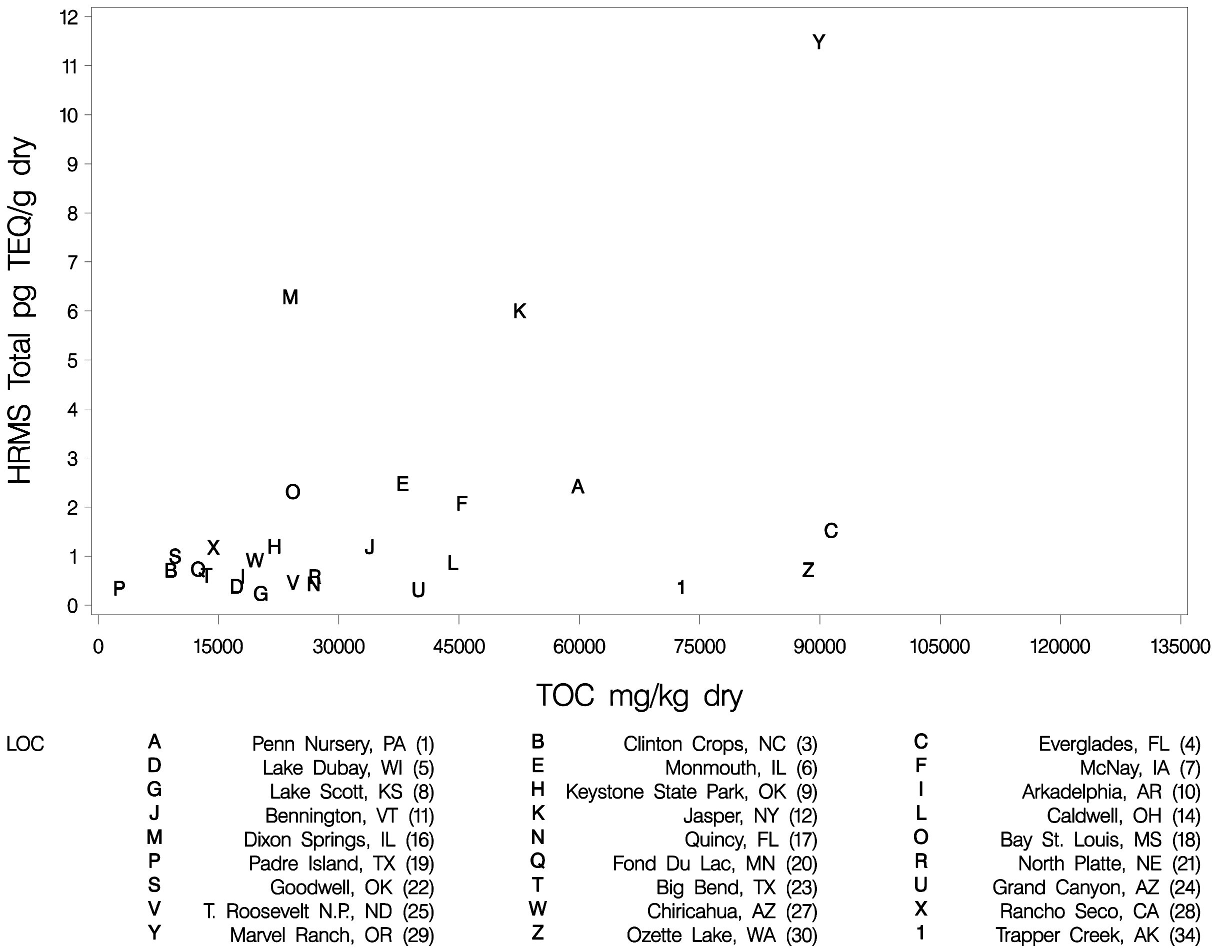

Figure 16. HRMS-based total TEQ2 versus total organic carbon (TOC) concentration of soil.

Although a number of homologue groups showed statistically significant correlations, none were particularly strong (maximum r was 0.51). This could indicate that other factors were also affecting sorption characteristics of the soil. Grain size may also correlate with dioxin levels in soil because organic carbon is sometimes associated with smaller particles and because smaller particles have a higher surface area-to-mass ratio, increasing sorption capacity. The other two soil properties measured in this study, pH and moisture content, do not have clear mechanistic reasons to correlate with dioxin levels in soil.

7.3. COMPARISON OF HRMS TEQs WITH CALUX BIOASSAY TEQs

HRMS and the CALUX bioassay method were used to analyze soils. This study was not intended to provide a detailed evaluation of the CALUX bioassay method; however, as discussed below, a few observations can be made about its performance. For a more detailed analysis of bioassay methods, readers are referred to EPA's Superfund Innovative Technology Evaluation (SITE) Program. Under this program, EPA has evaluated a variety of technologies (including CALUX) for determining the presence of dioxin and dioxin-like compounds in soil and sediment (U.S. EPA, 2005c).

As shown in Table 7, the CALUX results were higher than the HRMS total TEQs in almost all of the site composites. The ratios of CALUX TEQs to HRMS total TEQ2s ranged from 1 to 11. The ratios were about 2 or less at 11 sites and 10 or more at 2 sites. These data are shown as scatter plots in Figures 17 (raw data) and 18 (log transformed data). Both data sets had a significant correlation, with r = 0.82 for the raw data and r = 0.78 for the log-transformed data.

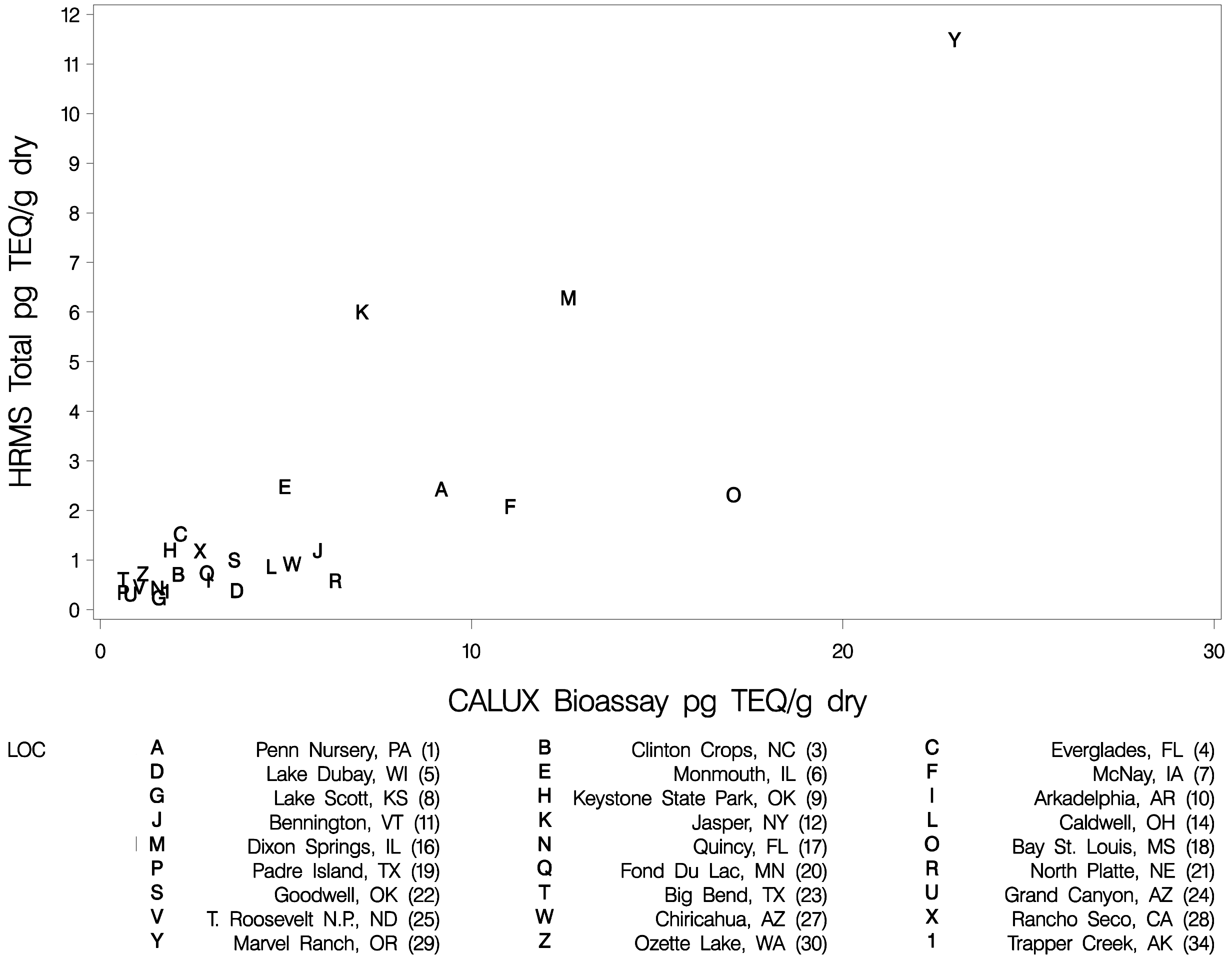

Figure 17. HRMS total TEQ2s versus CALUX bioassay TEQs by site (raw data).

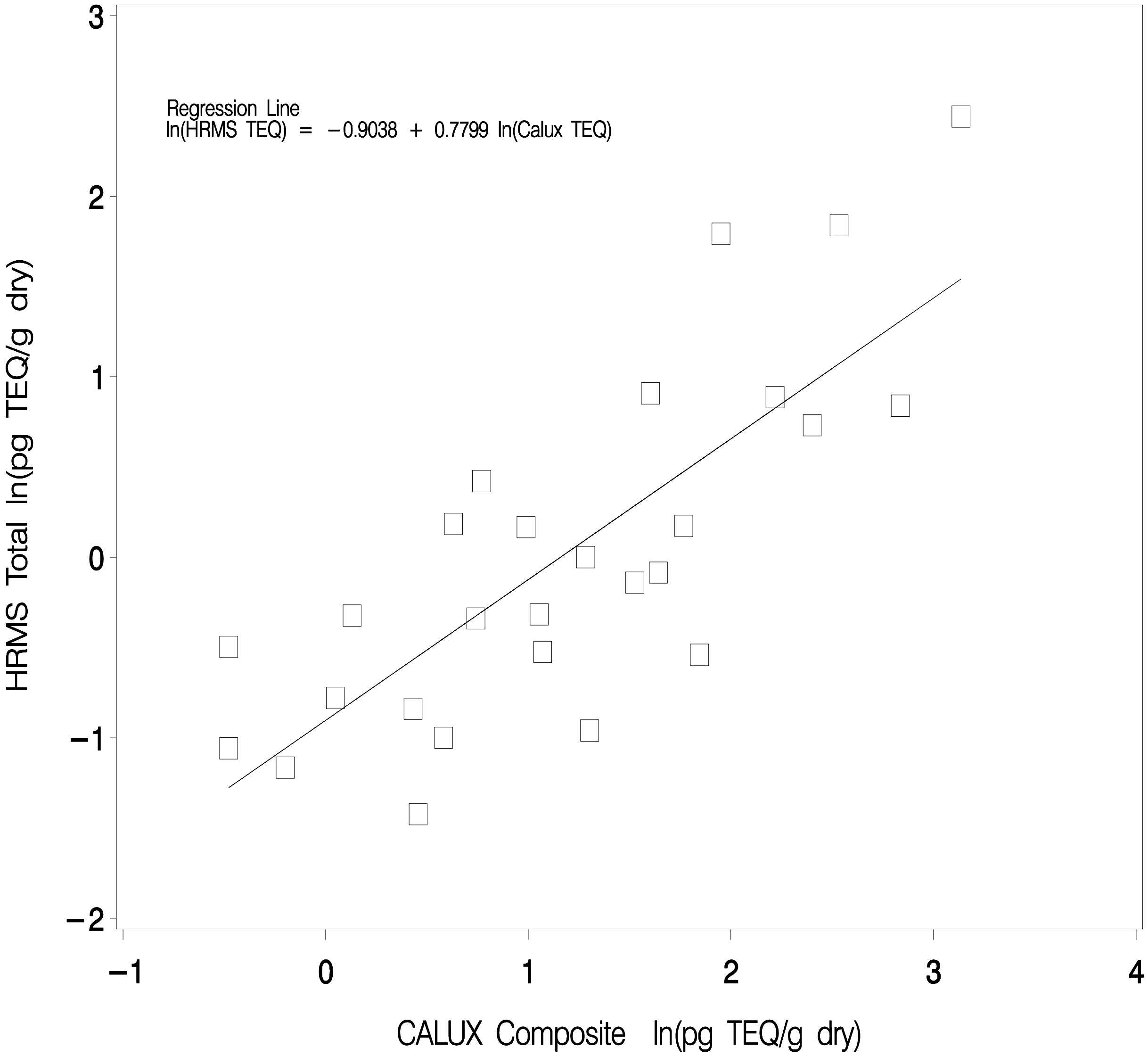

Figure 18. Scatter plot of HRMS total TEQ2 versus Calux bioassay TEQs on natural log-scale with natural log-linear regression line (r = 0.78).

The likely reason for the high bias in the CALUX data relative to the HRMS results is that CALUX responds to all compounds that activate the aryl hydrocarbon receptors, including a number of compounds other than CDDs, CDFs, and PCBs that may be present in soils, such as brominated and fluorinated dibenzo-*p*-dioxins/furans and biphenyls and halogenated napthlenes. Brown et al. (2004) showed that CALUX responds more to the hepta- and octa-chlorinated dioxins/furans and the tetrachlorinated biphenyls than would be expected on the basis of WHO TEF values and that this could lead to overestimation of the TEQ for samples that are contaminated primarily by these compounds. Although the two methods used different sample extraction procedures, it is unlikely that the ASE extraction used for the HRMS analysis was less efficient than the bottle sonication extraction procedures used for the CALUX analysis.

Clark et al. (2003) compared CALUX bioassays with traditional HRMS techniques applied to soil samples. Their data also show a high bias in the CALUX data, but a much stronger correlation (r = 0.98). This stronger correlation may have resulted from the fact that the study was conducted with soil concentrations ranging from 100 to 100,000 pg/g, which are much higher than those measured here. The TEQs in these more highly contaminated samples are probably dominated by the CDDs, CDFs, and PCBs, reducing the influence of the other compounds that activate the aryl hydrocarbon receptors.

Figure 19 shows results of a rank order analysis of the HRMS total TEQ2 versus CALUX TEQ data. This figure illustrates that the CALUX TEQ values are reasonable indicators of the relative TEQs among the sites as the lowest HRMS total TEQ2 values correspond to lowest CALUX TEQ values and as HRMS TEQ values increase CALUX values increase in a similarly corresponding manner.

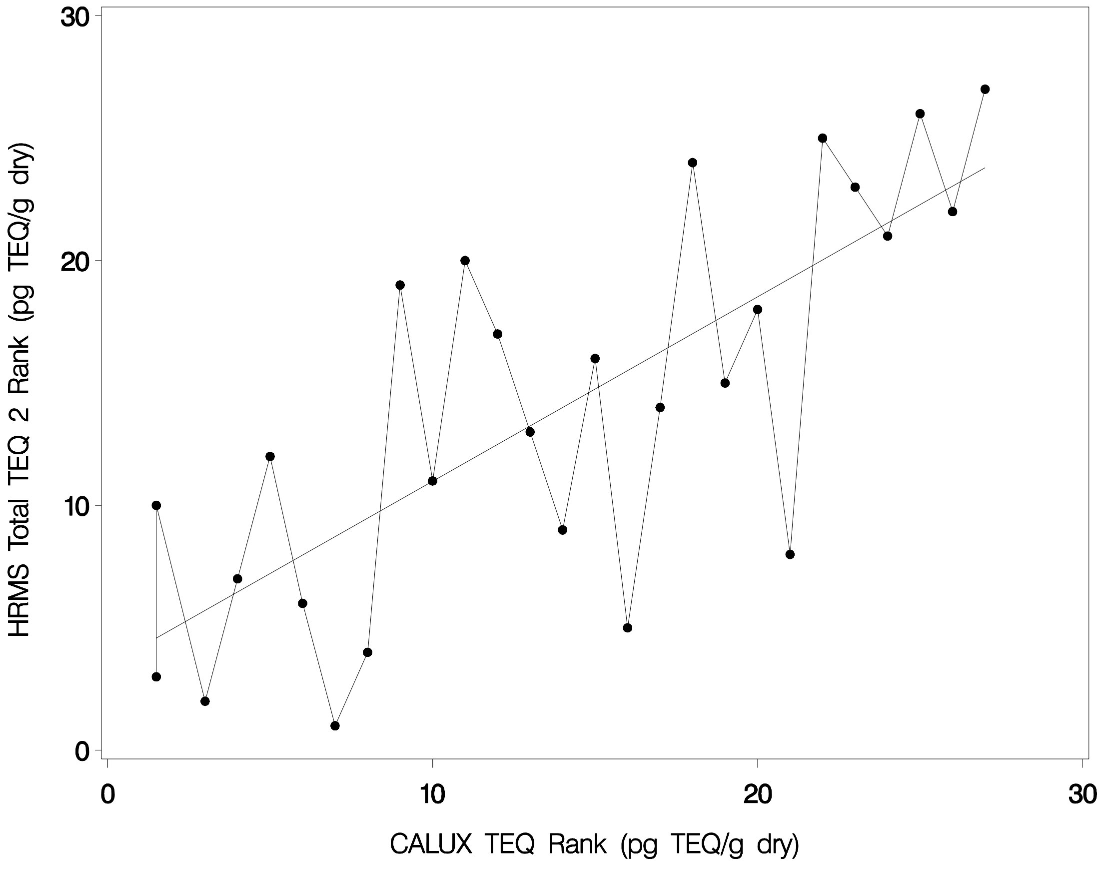

Figure 19. Rank order comparison of CALUX TEQs to HRMS total TEQ2s.

8. UNCERTAINTY

This chapter discusses the factors that contribute to the uncertainty in this study, including site selection, sampling protocol, analytical methods, and treatment of detection limits.

8.1. SITE SELECTION

The NDAMN sites were chosen as sampling locations because they are in rural/remote areas, they are well distributed nationally, they provide an opportunity to examine air-soil relationships, and they offered a cost-effective means for gathering soil samples. A statistically based random sampling design would have reduced bias in the site selection. However, the NDAMN study and the present soil survey were pilot studies and not meant to provide statistically unbiased results. Nonetheless, the results of the present study were consistent with other rural soil surveys.

8.2. SAMPLING PROTOCOL

A key uncertainty issue for a sampling protocol is the number of samples needed to represent a site. The number of samples per site used in this study was based on the coefficient of variation determined from the Oxford, OH, preliminary sample protocol evaluation (Appendix A). This initial study indicated that five samples within a 100 ft × 100 ft area would be sufficient to represent a site. Two aspects of this approach introduce uncertainty. First, it is unknown how representative the Oxford site is of the other sites in terms of spatial variability. Second, the Oxford exercise relied on CALUX analysis, which measures TEQs only. It is uncertain how well these results translate to specific chemicals. This uncertainty is greatest for mercury because it is unrelated to the dioxin-like chemicals detected by CALUX.

This study relied on composite soil samples (made by combining equal portions of five individual soil samples at each site) as the primary basis for chemical analysis. An important uncertainty issue is how well the composites represented the individual samples. The individual samples and the composites were analyzed for TEQs by the CALUX bioassay method, so the resulting data can be used to evaluate representativeness. Figure 20 shows, for each site, the average of the individual samples, the 95% confidence interval on the mean of the individual samples, and the composite sample TEQ value. Two observations from this figure suggest that

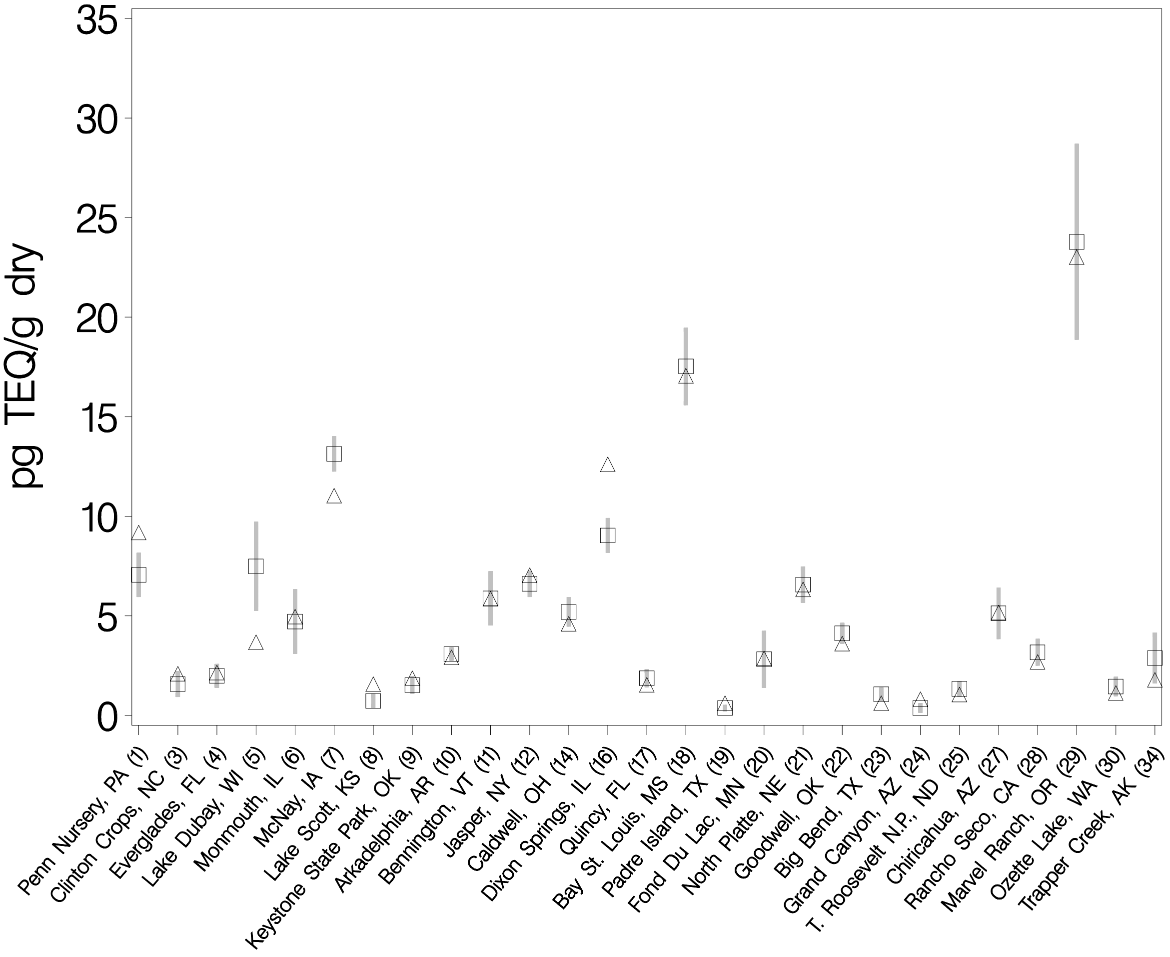

Figure 20. Comparison of five-point mean CALUX bioassay (☐) and assay of composite soil sample (△). Vertical line shows the 95% confidence interval about the mean.

the five-sample composite was adequate for characterizing TEQs at a site. First, most of the individual sampling point concentrations within a single site span a relatively small range, as evidenced by the short vertical bars. This means relatively little information was lost by analyzing the composite only. Second, the average of the individual sampling points was very near the composite concentration at almost all sites. The composite average fell outside the confidence interval at only four sites (1, 5, 7 and 16). A statistical analysis was performed to assess whether there was a statistically significant difference across sites between the average of the individual soil sample CALUX TEQ concentrations and the composite CALUX TEQ concentration. The analysis indicated that there was no statistical difference (at the 0.05 level) between the average and the composite.

Figure 21 shows a scatter plot of the paired values with the y = x line and the results of regressing the composite measurements on the five-point means. Statistical tests (F-tests) showed that the regression intercept was not significantly different from zero and the slope was not significantly different from one. Results from the site composite can therefore be considered to be representative of the five individual samples.

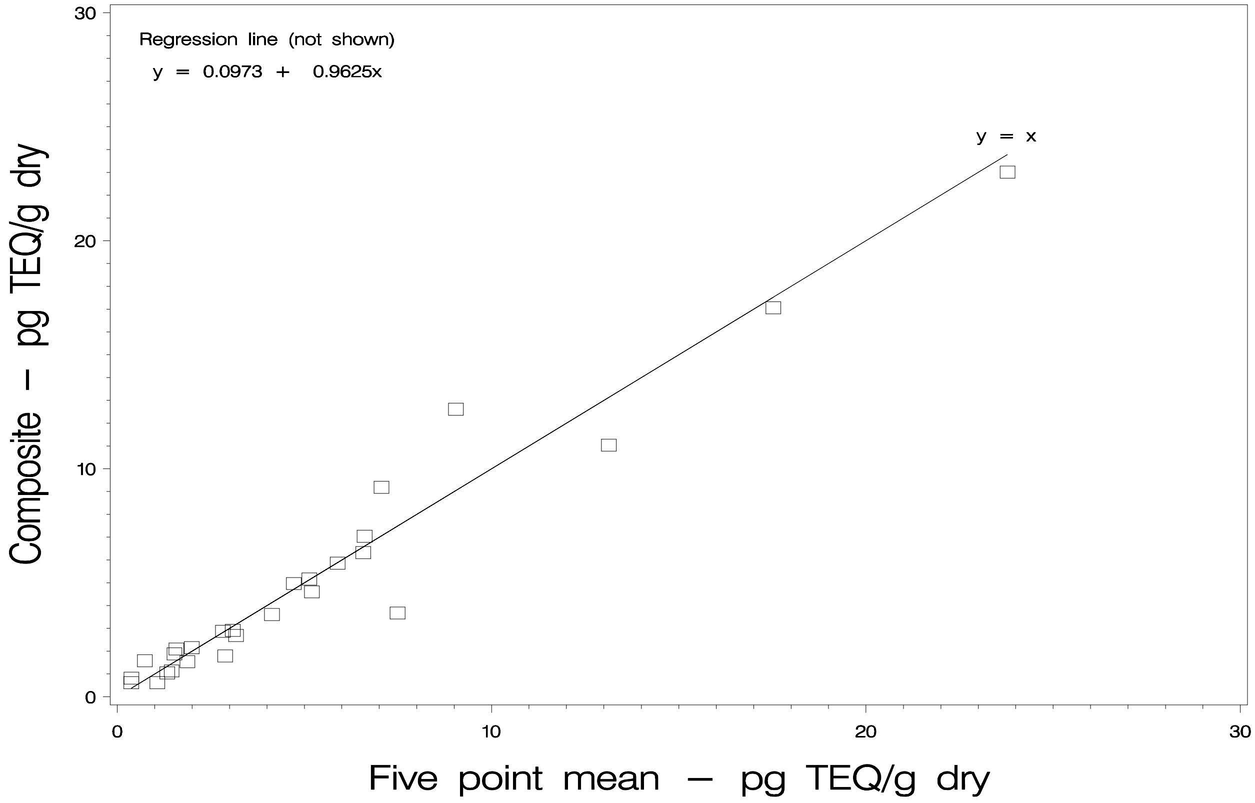

Figure 21. Scatter plot of five-point mean CALUX TEQs versus CALUX TEQs of soil composite (r = 0.97).

The similarity in the composites and averages of the individual samples at each site, as shown in Figure 20, also indicates an internal consistency in sample handling and compositing procedures.

Although more difficult to quantify, some between-site and within-site variation can be attributed to sampling technique because samples were collected by 27 different people with different experience levels. Attempts to control this variable were made by providing training and discussing sampling technique with each sampler, contacting the samplers by phone during collection, conducting quality assurance audits at two sites during sampling, and documenting with photographs the sampling at each site.

Other sources of sampling variability, such as seasonality, temperature, soil conditions, and type and quantity of vegetation, may have contributed to the uncertainty and were not evaluated as part of this study.

8.3. ANALYTICAL METHODS

For a single sampling point, the relative percent difference between duplicate analyses gives some insight into within-sample variation. Within-sample variation is primarily affected by the sample homogenization and the analytical method. Section 5.5 and Appendix D present duplicate analysis results. In general, the relative percent difference between duplicates was within target limits for mercury and CDDs/CDFs, but it was more variable for PCBs.

Analytical protocols and equipment also have inherent uncertainties. Although there is a "true" measure of a concentration, even the best of analytical methods will only approximate the true measure and thereby introduce uncertainty. For this study, HRMS was selected to provide the final congener concentration levels for CDDs/CDFs and PCBs. HRMS analysis methods are the most accurate, sensitive methods currently available for detecting CDDs/CDFs and PCBs. The HRMS data were supported by CALUX analysis to ensure that the site composite analyzed by HRMS was representative of the five individual points that made up the composite. Ideally, all samples would be analyzed by HRMS; however, all studies are constrained by a finite budget and must make use of the best methods and protocols available given that budget.

8.4. TREATMENT OF DATA

In addition to the analytical protocol used, treatment of data can contribute to uncertainty, particularly when the study is concentrating on levels very near the detection limits. Various ways of treating missing data and data below the detection limit can change the mean concentration standard error, introducing uncertainty. In addition, evaluating data on a TEQ basis versus individual congener concentrations can potentially minimize the contribution of a highly variable congener should it have a low TEF, resulting in a small contribution to TEQ, or overestimate the contribution of a highly variable congener with a high TEF, resulting in a large contribution to TEQ.

9. CONCLUSIONS

This study conducted a national-scale pilot survey of levels of CDDs, CDF's, PCB's and mercury in rural/remote soils of the United States. The results presented pertain to the 27 sites sampled and should not be more broadly interpreted as statistically representative of all rural soils in the United States. These results, however, may be a plausible basis for a preliminary characterization of soils in rural/remote areas. The primary measurement results are summarized below.

- Total CDDs averaged 1,585 pg/g (SE = 567, SD = 2945). Total CDFs averaged 47 pg/g (SE = 13, SD = 68). Levels of the TCDD homologues were the lowest, with an average concentration of 0.2 pg/g. Levels of the OCDD homologue were the highest, with an average concentration of 1,482 pg/g. The range of concentrations found here is similar to the range across five published studies on CDD/CDF levels in soils from rural areas of North America.

- Total PCBs averaged 3,089 pg/g (SE = 1,009, SD = 5,241). Levels of the deca-chlorinated biphenyl homologues were the lowest, with an average concentration of 29 pg/g. Levels of the penta-chlorinated biphenyl homologues were the highest, with an average concentration of 1,013 pg/g. The range of concentrations found here is similar to the range across three published studies on PCB levels in soils from rural areas worldwide.

- Total TEQ2s averaged 1.76 pg/g (SE = 0.48, SD = 2.47). The PCBs generally were a small fraction of the total TEQs in soil. The mean for total TEQs from this study falls near the center of the range of values across 10 published studies.

- Mercury concentrations averaged 22 ng/g across all sites (SE = 2.9, SD = 15 ng/g). The mean from this study falls within the range of values from five published studies on mercury levels in soils from rural areas of North America.

Further details on ranges and distributions are provided in Chapter 6.

This study also evaluated relationships between air concentrations and soil concentrations across sites. Based on the log- transformed data, significant positive correlations were observed for TEQs (r = 0.54), PeCDDs (r = 0.40), HxCDDs (r = 0.42), HpCDDs (r = 0.48), OCDD (r = 0.52), PCB 77 (r = 0.47), total CDDs (r = 0.51), and total CDDs/CDFs (r = 0.53). None of the CDFs showed significant correlations. TEQ levels in soil and air were also compared visually using national maps (Figures 11 and 12). Although some correspondence could be seen in the lowest and the highest sites, many sites appeared inconsistent. The congener profiles of the air and soil were compared for the 2,3,7,8-substituted CDDs/CDFs and six PCB congeners. The

CDD and PCB profiles in air and soil were generally similar at all sites. The CDF profiles for air and soil were different at most sites.

The overall conclusions about the air-soil relationships for the three groups of chemicals are as follows:

- *CDDs.* A general association between air and soil was observed, based on the significant air-soil correlations observed across sites for most homologue groups and the similarity in air and soil congener profiles observed at most sites.

- *CDFs.* Little association between air and soil could be observed, based on the lack of significant air-soil correlations for homologue groups across sites and the lack of similarity in air and soil congener profiles for many sites.

- *PCBs.* Some association between air and soil was observed. Data limitations restricted the air and soil comparisons to only six PCBs. One of these had a significant air-soil correlation across sites. The air and soil profiles based on these six chemicals were very similar at most sites.

The observations for CDDs and PCBs are consistent with the theory that air transport and deposition are the primary ways that these chemicals are distributed to soils, particularly those in rural areas. The lack of similar observations for the CDFs does not necessarily mean that they are not distributed in a similar manner, but it does suggest that different factors affect the environmental fate of these chemicals.

This study also evaluated relationships between chemical levels in soil and TOC levels in soil. The raw data analyses showed significant positive correlations for TCDFs ($r = 0.43$), PeCDFs ($r = 0.51$), PeCDDs ($r = 0.44$), HxCDFs ($r = 0.47$), HxCDDs ($r = 0.46$), HpCDFs ($r = 0.38$), HpCDDs ($r = 0.38$), total CDFs ($r = 0.39$), total TEQs ($r = 0.43$), and nonachlorobiphenyls ($r = 0.47$). The correlations were generally not very strong, indicating that other factors, such as grain size, may also be affecting sorption characteristics of the soil.

TEQ levels were estimated both on the basis of applying TEFs to the HRMS analyses and on the basis of the CALUX bioassay method. The CALUX results were higher—by varying amounts—than the HRMS total TEQs in almost all of the site composites. Significant positive correlations were found comparing the data on both a raw basis ($r = 0.82$) and on a log-transformed basis ($r = 0.78$). The likely reason for the high bias in the CALUX data relative to HRMS data is that CALUX responds to all compounds that activate the aryl hydrocarbon receptors, including a number of compounds other than CDDs, CDFs, and PCBs that may be present in soils.

Two observations from this study were unexpected and may warrant further investigation:

- It would be reasonable to expect that PCBs in rural soils should be generally present at greater concentrations than those of CDDs/CDFs. PCBs were produced in large quantities in the United States (571,000 metric tons) from 1929 until their ban in 1978 (ATSDR, 2000). CDDs/CDFs have never been intentionally produced; rather, they are formed in small quantities as by-products of combustion or certain types of chemical manufacturing. Although total PCBs exceeded total CDDs/CDFs at most sites in this study, the opposite was seen at 9 of 27 sites. At two of these sites, the CDD/CDF levels exceeded the PCBs by more than sevenfold.

- The highest total PCBs were found at Big Bend, TX (Site 23). The levels were about eight times higher than the mean across all sites, although total CDD/CDF levels were among the lowest across sites. This is a very remote site and it is unclear why such relatively high PCB levels were found.

Finally, a few thoughts can be offered about the utility of this pilot study to support future surveys. The surface soil sample collection/handling protocol proved to be effective and practical and could be used as a starting point in the design of future studies. Final decisions as to the number of sampling points at each location, sampling depth, and grid size were based on a preliminary single-site survey. This initial survey was used to evaluate the variability in TEQ levels and supported the use of sample compositing as a way to reduce analytical costs. Further TEQ analysis of individual samples and composites at all 27 sites demonstrated that relatively little information was lost by compositing. This experience suggests that future soil surveys with a focus on TEQ levels in rural areas could also reasonably consider analyzing only sample composites. Surveys involving other analytes and other land types should consider preliminary field testing to evaluate the appropriateness of sample compositing.

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APPENDIX A

OXFORD STUDY

Oxford Study

This appendix describes the results from the single-site field test at the Oxford, Ohio NDAMN location. Data are included for the following:

- Physical/chemical parameter testing on soils from 25 individual sampling points (top 0-5 cm).

- CALUX® bioassay TEQ results for soils from 25 individual sampling points (top 0-5 cm) as well as duplicate analysis of one composite made from equivalent portions of the top 0-5 cm from all 25 locations (Oxford-Comp-T) and duplicate analysis of one composite made from equivalent portions of the bottom 5-10 cm from all 25 sampling locations (Oxford- Comp-B), plus results for one field blank, one trip blank, and one equipment blank. The CALUX® bioassay TEQ results from 25 individual samples from the Oxford, Ohio location were evaluated statistically to determine the minimum number of samples that should be collected at an uncontaminated sampling location to ensure a representative sampling for the remainder of the sampling locations planned for a pilot survey of dioxins in soil. Results of this statistical analysis are included with the CALUX® data.

- Mercury determination of duplicate analysis of Oxford-Comp-T and duplicate analysis of Oxford-Comp-B.

- High resolution mass spectrometry (HRMS) determination of individual dioxin/furan and PCB congeners and dioxin/furan and PCB TEQ from duplicate analysis of Oxford-Comp-T and duplicate analysis of Oxford-Comp-B.

The following conclusions have been made from these data:

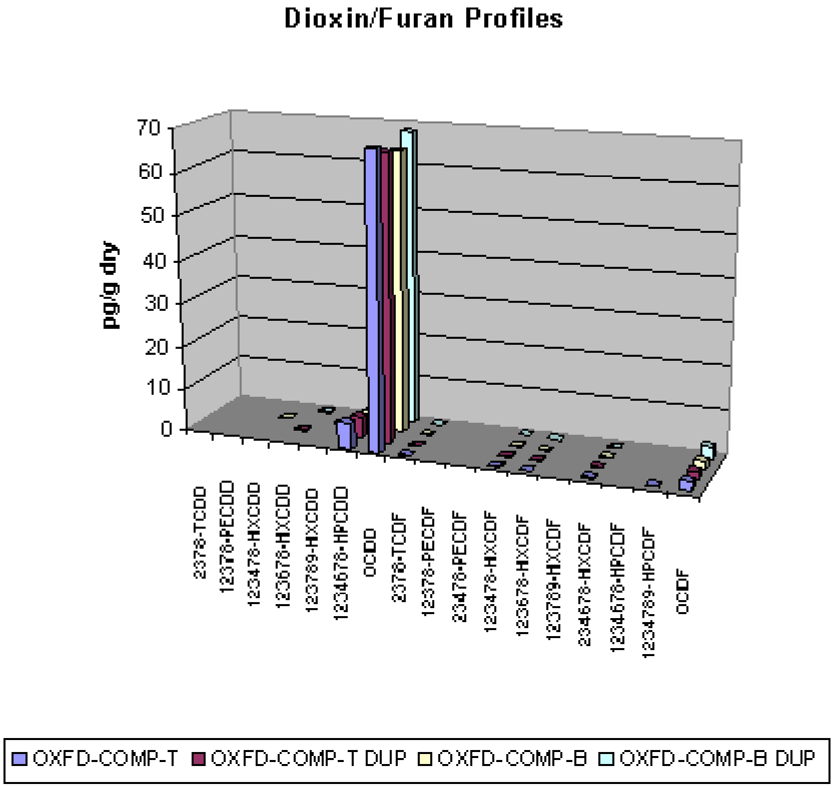
1) A minimum of 5 samples need to be collected at each of the remaining sampling locations based on the statistical analysis of the CALUX® bioassay TEQ results from 25 individual samples from the Oxford, Ohio location.

2) There is no significant difference in the mercury concentration, HRMS dioxin/furan TEQ or CALUX® bioassay TEQ between the composites from the top 0-5 cm (Oxford-Comp-T) and the bottom 5-10 cm (Oxford-Comp-B). The HRMS PCB TEQ using zero for non-detect values (TEQ 1) is lower for Oxford-Comp-T than for Oxford-Comp-B due to detection of PCB 126 in the Oxford-Comp-B samples only. While PCB 126 was detected at a very trace level (below the detection limit) its high toxicity equivalency factor causes it to have a large impact on PCB TEQ. The impact of this single analyte is lessened when one-half the detection limit values are used for non-detects in calculation of TEQ (TEQ 2). The HRMS congener profiles for dioxin/furan and the dioxin-like PCB are also similar between Oxford-Comp-T and Oxford-Comp-B. Based on this, the recommended sampling depth is 10 cm below ground surface depth.

Table 1. Data Summary from Oxford-Comp-T and Oxford-Comp-B

	Hg (mg/Kg dry)	**HRMS Dioxin/Furan (pg TEQ 1/g dry)**	**HRMS Dioxin/Furan (pg TEQ 2/g dry)**	**HRMS PCB (pg TEQ 1 /g dry)**	**HRMS PCB (pg TEQ 2 /g dry)**	**CALUX® Bioassay (pg TEQ/g dry)**
Oxford-Comp-T	<0.050	0.150	3.61	0.017	0.108	1.66
Oxford-Comp-T duplicate	0.077	0.160	4.02	0.011	0.112	2.16
Oxford-Comp-B	0.051	0.155	3.68	0.125	0.134	1.66
Oxford-Comp-B duplicate	0.059	0.170	3.76	0.131	0.140	2.15

TEQ 1 = zero used for non-detects
TEQ 2 = one-half the detection limit used for non-detects


Dioxin/Furan Profiles
pg/g dry
70
60
50
40
30
20
10
0
2378-TCDD
12378-PECDD
123478-HXCDD
123678-HXCDD
123789-HXCDD
1234678-HPCDD
OCDD
2378-TCDF
12378-PECDF
23478-PECDF
123478-HXCDF
123678-HXCDF
123789-HXCDF
234678-HXCDF
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OCDF
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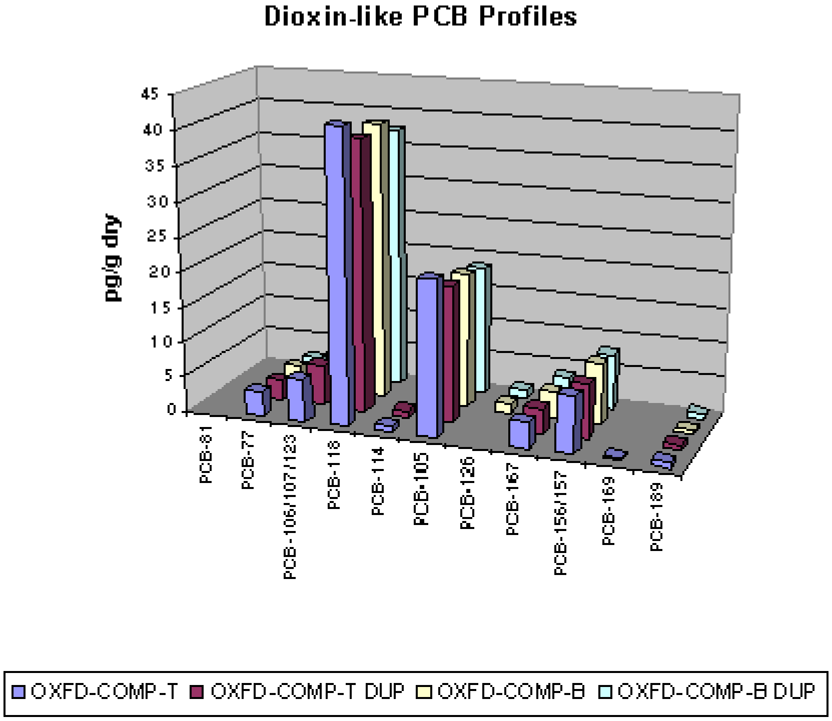
OXFD-COMP-T DUP
OXFD-COMP-B
OXFD-COMP-B DUP


Dioxin-like PCB Profiles
pg/g dry
45
40
35
30
25
20
15
10
5
0
PCB-81
PCB-77
PCB-106/107/123
PCB-118
PCB-114
PCB-105
PCB-126
PCB-167
PCB-156/157
PCB-169
PCB-189
OXFD-COMP-T
OXFD-COMP-T DUP
OXFD-COMP-B
OXFD-COMP-B DUP

3) The average CALUX® TEQ for samples collected within the 100 x 100 ft grid (Oxford -1-T through Oxford-21-T) was 2.39 pg TEQ/g dry versus an average of 2.49 pg TEQ/g dry for the four samples representing a 1000 x 1000 ft grid (Oxford-22-T through Oxford-25-T). Based on this, while it is still recommended for samplers to try to obtain all samples within a 100 x 100 ft grid, this may be expanded to 1000 x 1000 ft if necessary to find undisturbed sampling locations without compromising the samples being representative of the site.

PHYSICAL/CHEMICAL PARAMETER DATA

Sample ID	Moisture Content (%)	Grain Size Distribution			pH	TOC	
		%Finer #4 Sieve	% Finer #200 Sieve	% Finer 0.005mm		Result (mg/kg)	Reporting Limit (mg/kg)
Oxford-1-T	40.9	100.0	86.7	22.9	5.3	22,400	990
Oxford-2-T	41.7	100.0	86.7	24.3	5.3	23,600	990
Oxford-3-T	22.3	98.7	84.9	39.9	7.6	14,600	962
Oxford-4-T	33.4	100.0	84.8	29.6	6.8	24,900	971
Oxford-5-T	30.9	100.0	80.8	23.6	6.3	31,700	988
Oxford-6-T	28.2	99.5	79.4	30.5	7.4	15,400	943
Oxford-7-T	35.7	100.0	84.6	28.2	6.7	21,400	971
Oxford-8-T	38.7	100.0	87.2	25.8	6.2	21,400	962
Oxford-9-T	29.8	98.2	82.8	26.6	6.2	22,600	980
Oxford-10-T	37.0	100.0	86.1	22.6	6.3	24,000	990
Oxford-11-T	41.0	100.0	83.5	22.1	7.1	32,100	1,000
Oxford-12-T	37.5	100.0	79.2	28.4	7.3	25,800	1,000
Oxford-13-T	54.5	100.0	87.0	27.1	5.7	25,200	990
Oxford-14-T	30.5	98.6	75.1	23.6	7.4	26,200	971
Oxford-15-T	41.9	100.0	84.5	22.8	6.7	21,900	952
Oxford-16-T	41.5	100.0	82.3	23.9	7.2	27,800	980
Oxford-17-T	35.8	100.0	87.8	26.7	6.0	22,800	990
Oxford-18-T	42.8	100.0	86.1	26.7	6.7	22,200	990
Oxford-19-T	44.5	100.0	84.8	24.4	6.8	21,800	990
Oxford-20-T	34.9	100.0	89.7	25.5	6.1	27,000	330
Oxford-21-T	24.8	100.0	89.1	24.2	6.2	27,100	1,000
Oxford-22-T	13.3	97.9	68.8	24.3	7.9	11,100	990
Oxford-23-T	27.5	100.0	80.7	25.0	7.3	18,400	971
Oxford-24-T	33.2	100.0	91.1	20.2	7.4	23,300	971
Oxford-25-T	19.2	100.0	85.4	26.3	6.4	10,100	498

CALUX® Bioassay Data

CALUX QC Sample Summaries

Method Blanks

Sample Batch #	pg/g	mean	std dev
B8-82-35	0.311	0.33	0.03
B8-82-36	0.347		
B8-82-37	~~1.219~~	DBQ 0.96 ratio	
B8-82A-32	0.668	0.50	0.11
B8-82A-33	0.502		
B8-82A-34	0.419		
B8-82A-35	0.430		
B8-82B-26	0.490	0.35	0.13
B8-82B-27	0.242		
B8-82B-28	0.333		
B8-82C-6	0.663	0.58	0.05
B8-82C-7	0.572		
B8-82C-8	0.537		
B8-82C-9	0.565		

Surrogate Spike

Sample Batch #	CPM Spiked	CPM Recovered	% Recovery
82-34	1984.5	1313.5	66%
B8-32A-31	2061	1395	68%
B8-82B-25	2002.5	1480.5	74%
B8-82C-5	2384.5	1469	62%

CPM: Counts per minute of ^{14}C TCDD recovered

XDS Solid QC

Sample Batch #	pg/g	mean	std dev
B8-82	16.86	13.28	3.28
B8-82A	10.43		
B8-82B	12.56		

Matrix Spikes

Sample Batch #	pg/g	Spike sample minus sample pg	% Recovery
B8-82-29	3.513	2.523	63%
B8-82-30	0.990		
Matrix spike	4.015		
B8-82A-29	3.304	2.569	69%
B8-82A-30	0.735		
Matrix spike	3.746		
B8-82B-19	3.620	2.540	61%
B8-82B-20	1.080		
B8-82B-21	4.276	3.436	82%
B8-82B-22	0.840		
Matrix spike	4.192		
B8-82C-4	4.985	3.498	93%
B8-82C-1	1.487		
Matrix spike	3.746		

Lab Control Spike

Sample Batch #	pg/g minus blank	% Recovery
B8-82-32	2.309	57%
B8-82-33	2.863	71%
LCS control	4.015	
B8-82A-30	2.905	78%
LCS control	3.746	
B8-82B-24	3.440	82%
LCS control	4.192	

CALUX Sample Summary

XDS ID #	Client ID #	Sample Aliquot	TEQ-ppt (wet weight) PCDD / PCDF	TEQ-ppt (dry weight) PCDD / PCDF	Percent Moisture
A02868	Oxford -1-T	2g	1.92 ± 0.19	2.75 ± 0.28	31%
A02869	Oxford -2-T	2g	1.73 ± 0.45	2.31 ± 0.61	26%
A02870	Oxford -3-T	2g	1.09 ± 0.21	1.31 ± 0.25	18%
A02871	Oxford -4-T	2g	1.28 ± 0.35	1.64 ± 0.45	23%
A02872	Oxford -5-T	2g	2.86 ± 0.86	3.69 ± 1.12	23%
A02873	Oxford -6-T	2g	0.87 ± 0.20	1.11 ± 0.26	23%
A02874	Oxford -7-T	2g	1.40 ± 0.10	1.89 ± 0.14	27%
A02875	Oxford -8-T	2g	1.38 ± 0.05	1.91 ± 0.07	29%
A02876	Oxford -9-T	2g	1.25 ± 0.25	1.58 ± 0.32	22%
A02877	Oxford -10-T	2g	2.67 ± 0.42	3.68 ± 0.59	28%
A02878	Oxford -11(sd)-T	2g	2.07 ± 0.36	3.02 ± 0.51	30%
A02879	Oxford -12-T	2g	1.31 ± 0.28	1.80 ± 0.37	25%
A02880	Oxford -13-T	2g	2.72 ± 0.72	4.20 ± 1.05	33%
A02881	Oxford -14-T	2g	1.29 ± 0.17	1.74 ± 0.23	23%
A02882	Oxford -15-T	2g	1.30 ± 0.21	1.98 ± 0.32	32%
A02883	Oxford -16-T	2g	1.34 ± 0.60	1.97 ± 0.86	30%
A02884	Oxford -17-T	2g	2.36 ± 0.50	3.25 ± 0.67	26%
A02885	Oxford -18-T	2g	1.60 ± 0.19	2.35 ± 0.28	30%
A02886	Oxford -19-T	2g	1.30 ± 0.29	1.82 ± 0.40	26%
A02887	Oxford -20-T	2g	2.81 ± 0.38	3.78 ± 0.49	25%
A02888	Oxford -21-T	2g	1.88 ± 0.26	2.32 ± 0.32	19%
A02889	Oxford -22-T	2g	2.99 ± 0.49	3.35 ± 0.55	11%
A02890	Oxford -23-T	2g	0.83 ± 0.10	1.07 ± 0.13	22%
A02891	Oxford -24-T	2g	3.18 ± 0.10	4.30 ± 0.13	26%
A02892	Oxford -25-T	2g	1.04 ± 0.003	1.24 ± 0.00	16%
A02893	Oxford - TB - 5	2g	ND<0.26	N/A	N/A
A02894	Oxford - FB - 2	2g	ND<0.26	N/A	N/A
A02895	Oxford - ER - 1	356ml	ND <0.05	N/A	N/A
A02896	Oxford - Comp-B	2g	1.33 ± 0.27	1.63 ± 0.34	20%
A02897	Oxford - Comp-B-DUP	2g	1.75 ± 0.37	2.01 ± 0.49	19%
A02898	Oxford - Comp-T-DUP	2g	1.59 ± 0.002	2.01 ± 0.49	26%
A02899	Oxford - Comp-T	2g	1.26 ± 0.26	1.62 ± 0.34	24%
A02900	Oxford - 1MS-T	2g	1.97 ± 0.06	2.80 ± 0.05	30%
A02901	Oxford - 11DUP-T	2g	1.02 ± 0.05	1.42 ± 0.07	28%
A02902	Oxford - 14MS-T	2g	0.95 ± 0.49	1.12 ± 0.74	21%

Statistical Analysis of CALUX® Bioassay TEQ Data

The twenty-five individual top 0-5 cm soil samples had a mean of 2.386 TEQ - ppt (dry weight basis) and a standard deviation of 0.979 TEQ - ppt (dy weight basis). The analysis described below assumes that the variability at the other NDAMN sites will be similar to this site. In particular, it is assumed that soil samples will vary as described by a normal distribution with a site-specific mean and a standard deviation that is proportional to that mean.

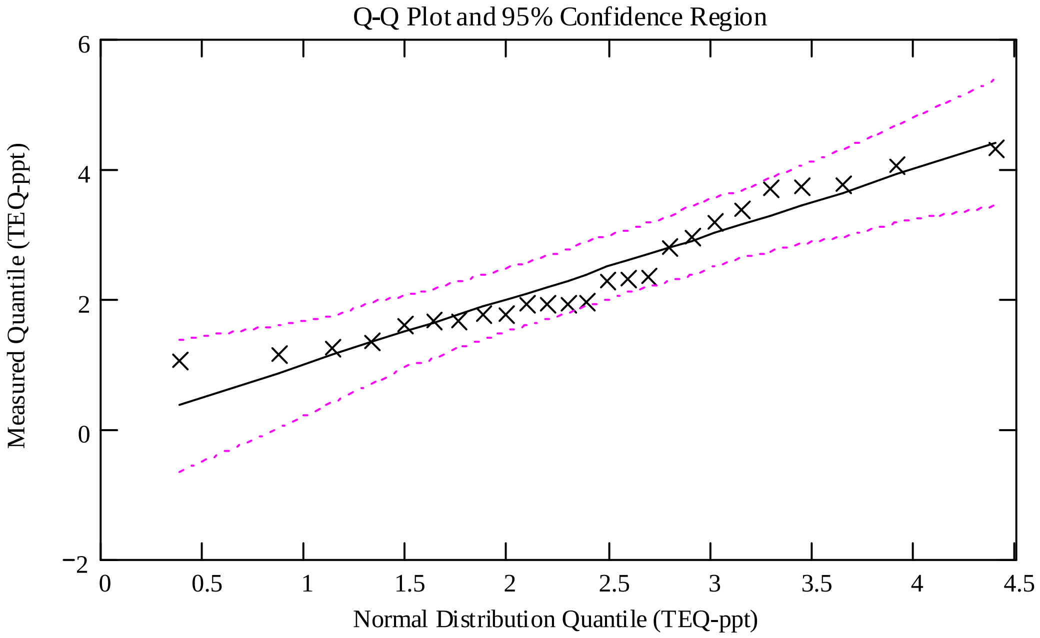


Figure 1. **A Quantile-Quantile Plot of the CALUX ® Bioassay TEQ data with an Approximate 95 Percent Confidence Region.**

While the proportionality assumption cannot be verified with data from one site, it is a reasonable starting assumption. The normality assumption can be checked for this site. A standard check is to plot the quantiles of the observed data against the quantiles of a normal distribution with the same mean and standard deviation. If the data are distributed according to a normal distribution, then the plot should be approximately straight. Figure 1 shows a Quantile-Quantile (Q-Q) plot of the data along with an approximate 95% confidence region for the plot. The approximate confidence region shows the boundaries from generating data sets of 25 points drawn from a normal distribution with the same mean and standard deviation as the soil data. There are no major departures from the normality assumption (a straight line). Hence normal distribution theory can be used.

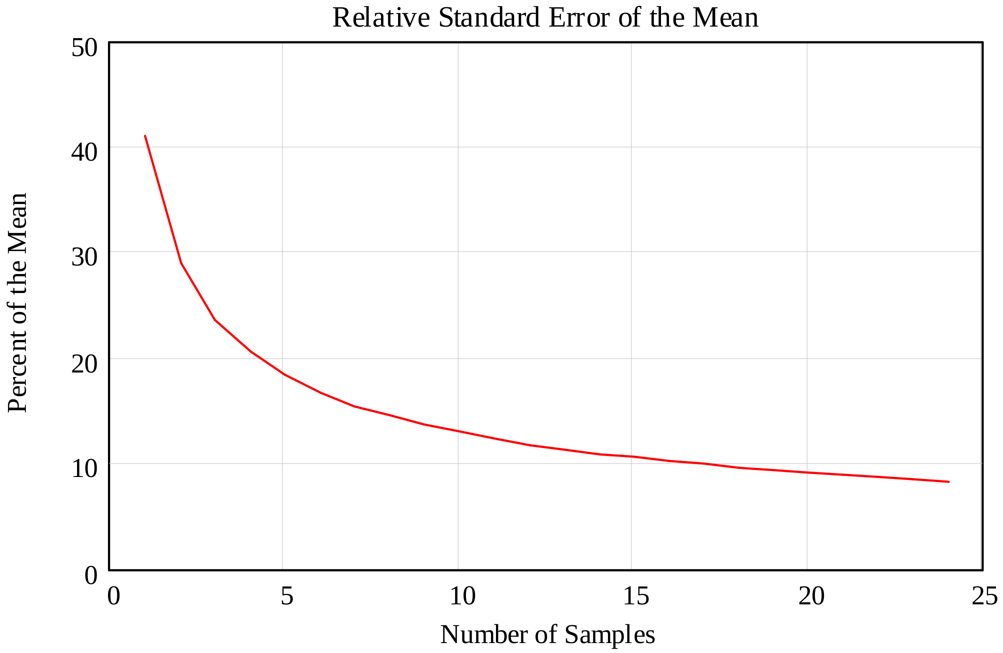


Figure 2. **The Estimated Standard Error of a Site Mean Versus the Number of Sample Values.**

Assuming that the errors are proportional to the mean and that this data is representative of the rest of the network, then individual points should have a coefficient of variation (CV) of approximately 40%. Equation 1 gives the relationship between the number of samples and the expected standard error under the assumptions. Figure 2 shows a plot of that relationship, the approximate standard error for estimating a site mean versus the number of samples taken at the site. Table 1 shows the actual values plotted in Figure 2.

$$CV_{mean} = \frac{Std \cdot 100}{mean \cdot \sqrt{n}} \qquad \text{Eq. 1}$$

Hence, if the individual samples continue to have a CV of 41 percent, the mean of five samples from a site should have a standard error of just under 20 percent. This implies that an interval of the form from 60 percent of the site mean to 140 percent of the mean will be a 95 percent confidence interval for the mean. From Figure 2 and Table 1 it can be seen that 8 samples per site are needed to get a CV of less than 15 percent and 17 data points are needed to get a CV that is less than 10%. (See the graph above and the table below for more details.)

Based on this evaluation, a minimum sampling size of 5 samples per site is recommended to obtain a 20% CV for the site estimate.

Table 1. Estimated Standard Errors of a Site Mean

Sample Size	Estimated Standard Error of the Mean
1	41.0%
2	29.0%
3	23.7%
4	20.5%
5	18.3%
6	16.7%
7	15.5%
8	14.5%
9	13.7%
10	13.0%
11	12.4%
12	11.8%
13	11.4%
14	11.0%
15	10.6%
16	10.3%
17	9.9%
18	9.7%
19	9.4%
20	9.2%

Mercury Data

Mercury Analysis Results

	ug/L	Wet Weight (g)	% Solids	Dry Weight (g)	Final Volume (L)	Hg (mg/Kg dry)
CCB	0.00					
CCV 5.0 ug/L	5.20					
	104%					
Reagent Blank	0.00					
Method Blank	<0.2	0.48	100.00%	0.48	0.1	<0.042
Spiked Method Blank (2.0ug/L)	2.30	0.47	100.00%	0.47	0.1	0.489
Spike Concentration	2.00	0.47	100.00%	0.47	0.1	0.426
Percent Spike Recovery						**115%**
SRM 1944 1	15.70	0.4500	98.75%	0.44	0.1	3.53
Percent Recovery						**104%**
Percent Difference						**3.9%**
SRM 1944 2	9.20	0.2600	98.75%	0.26	0.1	3.58
Percent Recovery						**105%**
Percent Difference						**5.4%**
Comp B	0.20	0.48	81.47%	0.39	0.1	0.051
Comp B Duplicate	0.30	0.62	81.47%	0.51	0.1	0.059
Comp T	0.20	0.52	76.25%	0.40	0.1	<0.050
Comp T Duplicate	0.30	0.51	76.25%	0.39	0.1	0.077
Comp T Spike	2.30	0.51	76.25%	0.39	0.1	0.591
Spike Concentration	2.00	0.51	76.25%	0.39	0.1	0.514
Percent Spike Recovery						**115%**

APPENDIX B

CALUX PAPER

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Analysis of Soil Samples from a Hazardous Waste Site: Comparison of CALUX® Bioassay TEQ Determinations with High Resolution GC/MS

George C. Clark[1], Michael S. Denison[2], Richard W. Morris[3], Michael Chu[1], Andrew Chu[1], and David J. Brown[1]

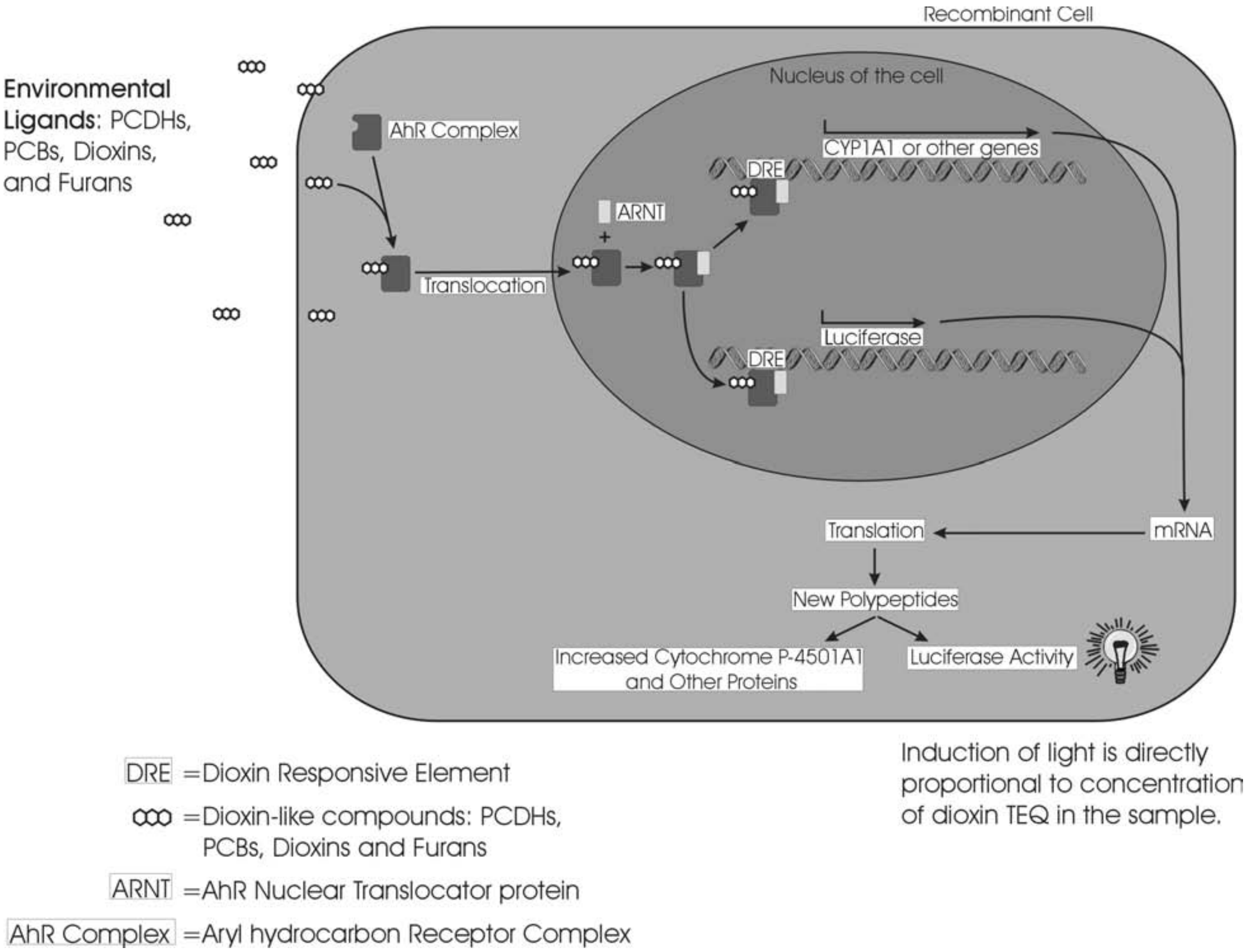
[1] Xenobiotic Detection Systems, 1601 East Geer Street, Suite S, Durham, NC 27704, USA
[2] Department of Environmental Toxicology, Meyer Hall, University of California, Davis, CA
[3] Analytical Sciences Inc., 2605 Meridian Parkway, Suite 200, Durham, NC 27713, USA

Introduction

Remediation of hazardous material contaminated sites requires analysis of levels of dioxin-like chemicals that are potentially important contaminants of these areas. Traditionally high-resolution gas chromatography/mass spectrometry (HRGC/MS) has been used to detect the presence of dioxin-like chemicals. This is a complex, expensive and time consuming method based on measuring the concentrations of 17 individual chlorinated dioxin and furan congeners that are considered toxic. To estimate a Toxic Equivalency (TEQ), the individual concentrations of each toxic congener is multiplied by a Toxic Equivalency Factor (TEF) and these determinations summed to produce a TEQ Determination. This TEQ determination provides an estimate of the potential toxicity of the sample for risk assessment purposes. A rapid, less expensive and more easily performed method of estimating TEQ determinations would aid in remediation efforts of hazardous waste sites.

Xenobiotic Detection Systems, Inc. (XDS) has developed a cellular bioassay based on the mechanism of toxicity of dioxin for estimating TEQ contamination with dioxin-like chemicals. This system has been developed with a rapid method of sample extraction and processing and application of the extract to living cells that respond to dioxin-like chemicals. The cell bioassay is depicted in Figure1; it utilizes a recombinant cell line with a stably integrated AhR-responsive luciferase reporter gene. Exposure of this Chemically Activated Luciferase Expression (CALUX®) bioassay to extracts containing 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) and/or related halogenated aromatic hydrocarbons produces the enzyme luciferase in a time, dose and chemical specific manner. Luciferase activity is determined by measuring light emitted and is directly proportional to the amount of dioxin-like chemicals within the test sample.

Figure 1. Schematic representation of the cellular processes involved in the CALUX® bioassay.



We participated in a double-blind study to compare the results of TEQ determinations for soil samples from a hazardous material remediation area measured by the CALUX® bioassay and HRGC/MS. Here we report the results of this double blind validation study.

Materials and Methods

A corporation under contract from the US Environmental Protection Agency collected soil samples from a hazardous material remediation site. These samples were sent to an independent laboratory for HRGC/MS analysis of TEQ contamination. The laboratory sent an aliquot of each soil to XDS for CALUX® determination. Results of HRGC/MS analysis and CALUX® bioassay results were sent to an independent statistician (Richard W. Morris, Analytical Sciences, Inc.) so that a double-blind format was maintained. After all results were reported comparison of the results was performed.

HRGC/MS. Sediment and ash samples were spiked with $^{13}C_{12}$-labeled PCDD/PCDF standards and analyzed for congener-specific PCDD/PCDFs at the corporation's lab using EPA Method 8290. I-TEQs for PCDDs/PCDFs were calculated using TEF values from the World Health Organization[1].

CALUX® bioassay. XDS has a patented genetically engineered cell line (mouse hepatoma H1L6.1) that contains the gene for firefly luciferase under transactivational control of the aryl hydrocarbon receptor[2]. This cell line can be used for the detection and relative quantificatation of a sample's total dioxin I-TEQ. Using a patent pending sample processing procedure it is also possible to use the CALUX® assay to estimate the I-TEQ contributions of PCDDs/PCDFs or the I-TEQ contributions of the coplanar PCBs[3]. The assay that uses this cell line is called the Chemically-Activated Luciferase Expression or CALUX® assay.

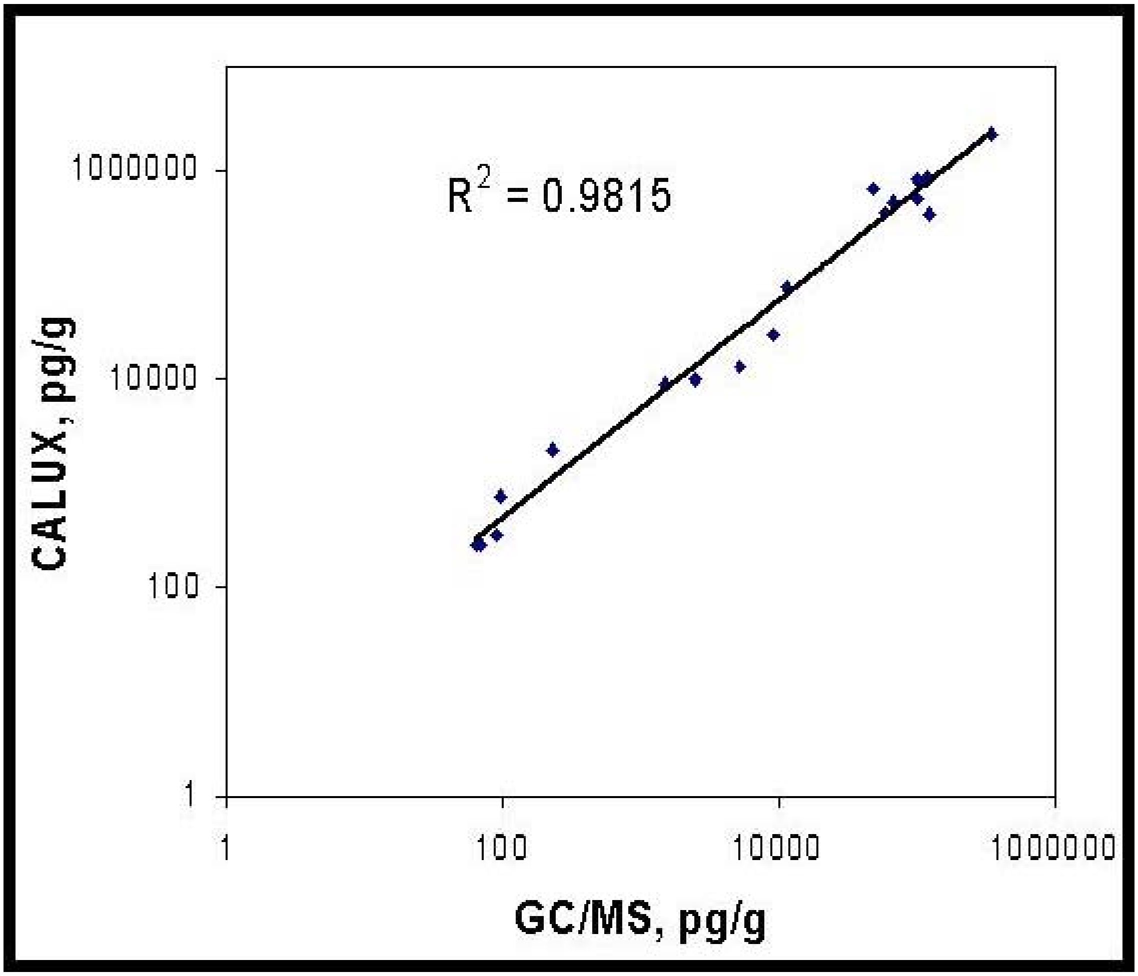
The samples were extracted using a modification of the EPA 8290 extraction method[4]. Briefly, the dried samples were ground and one gram aliquots were placed in solvent cleaned glass vials with PTFE lined caps. The sample was extracted with a 20% solution of methanol in toluene then twice with toluene. During each extraction step the samples were incubated in an ultrasonic water bath. The three extracts from each sample were filtered, pooled and concentrated by vacuum centrifugation. The sample extract was suspended in hexane and prepared for the bioassay by a patent pending clean up method[3]. The eluate from the clean up method was concentrated under vacuum into dimethyl sulfoxide (DMSO). The DMSO solution was used to dose the genetically engineered cells in the CALUX® bioassay.

Prior to dosing cells, the sample extracts in DMSO were suspended in cell culture medium. This medium was then used to expose monolayers of the H1L6.1 cell line grown in 96 well culture plates. In addition to the samples, a standard curve of 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) was assayed (500, 250, 125, 62.5, 31.2, 15.6, 7.8, 3.9, 1.9, and 1.0 parts per trillion (ppt) TCDD). The plates were incubated for a time to produce optimal expression of the luciferase activity in a humidified CO_2 incubator. Following incubation, the medium was removed and the cells were examined microscopically for viability. The induction of luciferase activity was quantified using the luciferase assay kit from Promega.

Results and Discussion

A double-blind format comparison was made of TEQ determinations with dioxin-like chemicals in soil samples with the CALUX® bioassay versus HRGC/MS. The two methods were highly correlated ($R^2 = 0.9815$). Figure 2 depicts a dot plot comparing results from the two assays.

Figure 2. Correlation of CALUX® bioassay determination of TEQ versus HRGC/MS TEQ determinations in soil samples from a hazardous waste site. The study was performed with the corporation by contract with the US Environmental Protection Agency in a double blind format to compare measurements of TEQ contamination in soil samples by the CALUX® bioassay versus HRGC/MS. Results of both analytical procedures correlate highly (R2 = 0.9815).



These data demonstrate that the CALUX® bioassay system provides a sensitive and less expensive system to rapidly evaluate remediation efforts of soils contaminated with dioxin-like chemicals. We are currently investigating if our analysis system can be modified into a kit format in which it could be used in the field to investigate contamination of remediation sites. This would be particularly valuable in that delays in receiving data can be a major cost factor in remediation of these hazardous sites.

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2. Denison, M., Brouwer, A. and Clark, G. (1998). U.S. patent # 5,854,010.
3. Chu, M. and Clark, G. (2000). Patent application submitted.
4. US EPA Method 8290, September 1994.

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The National Institutes of Environmental Health Sciences Grant (R44ES08372-02) supported portions of this research work. The authors would like to acknowledge the contributions of Bill Coakley of the US Environmental Protection Agencies Environmental Response Team in completing this research work.

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APPENDIX C

SAMPLING PROTOCOL AND STANDARD OPERATING PROCEDURE FOR DIOXINS IN SURFACE SOIL

SAMPLING PROTOCOL

for

DIOXINS IN SURFACE SOILS

Contract No.: 68-W-99-033
Work Assignment 5-11

Prepared for:

U.S. EPA
National Center for Environmental Assessment
Office of Research and Development
Washington, DC 20460

Prepared by:

Battelle
505 King Avenue
Columbus, Ohio 43201

August 26, 2003

CONTENTS

FIGURES

TABLES

ATTACHMENTS

ABBREVIATIONS AND ACRONYMS

bgs	below ground surface
DI	deionized
EMS	Emergency Medical Services
EPA	United States Environmental Protection Agency
GPS	global positioning system
HUD	United States Department of Housing and Urban Development
IATA	International Air Transportation Association
ID	identification
MSDS	Material Safety Data Sheets
MS/MSD	matrix spike/matrix spike duplicate
NA	not applicable
PCBs	polychlorinated biphenyls
PCDDs	polychlorinated dibenzo-p-dioxins
PCDFs	polychlorinated dibenzofurans
PPE	personal protective equipment
PVC	polyvinyl chloride
QA	quality assurance
QC	quality control
SRM	standard reference material

1.0 INTRODUCTION

This sampling protocol was prepared to aid in the determination of an initial estimate of background levels of dioxins in soil from non-impacted areas of the United States. This sampling protocol was prepared under Contract Number 68-W-99-033, Work Assignment No. 5-11; Pilot Survey of Dioxins in Soil. This sampling protocol describes the methods for determining sampling locations, number of samples required, and appropriate sampling depth, as well as field methods and procedures for collection of surface soil samples.

For the purposes of this document the term dioxins refers to the broad class of compounds including polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and dioxin-like polychlorinated biphenyls (PCBs).

1.1 Objective

The objective of this sampling protocol is to 1) provide field sampling procedures to be used in the collection of surface soil samples to be analyzed for dioxins and 2) establish sample gathering, handling, and documentation methods that are precise, accurate, representative, complete, and comparable to meet U.S. Environmental Protection Agency (EPA) quality control (QC) requirements.

1.2 Background of Dioxin Source and Fate

Dioxins are formed in trace amounts during almost any type of combustion process. They can also be formed during some chemical processes such as those associated with the manufacture of phenoxy herbicides and bleached wood pulp. These sources lead to the atmospheric transport of dioxins and subsequently the deposition of dioxin in soils and sediment (EPA, 2000). Dioxin compounds in soil tend to have low mobility because of their low water solubilities and vapor pressure. Therefore little vertical migration of dioxins in soils is expected, leaving the majority of atmospherically deposited dioxins in the surface soil (EPA, 2000).

2.0 SAMPLING PROCEDURES

The development of sampling procedures should be based on the objectives of each individual sampling survey. The following are guidelines for sampling soils from non-impacted areas where levels of dioxins should be representative of background and may need to be modified for surveys where analyses other than dioxins are required or if a non-background site is to be studied. The EPA guidance document, *Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies* (EPA, 1992), can be used as an aid in the development of soil sampling protocols for other surveys. Attachment-1 (Standard Operating Procedure [SOP] for Surface Soil Sampling for Dioxins) contains a more detailed description of sample collection and handling procedures, groundcover removal, equipment required, and decontamination procedures.

2.1 Sampling Strategy

Samples can be collected for analysis by two general methods: soil cores (undisturbed samples with no headspace and only in situ voids) and sample container. Soil cores in inert liners can be capped, refrigerated and sent to the laboratory. If the sample container method is used, the collection method, container type, sample size, and preservatives vary according to the analysis to be performed. Soil cores are generally required if an undisturbed sample is needed or if the sample will also be analyzed for volatile compounds. If soil samples will be composited after collection, then soil coring is not necessary since the samples will need to be mixed during compositing. The costs associated with soil coring are generally higher than for the sample container method because of equipment cost and additional sampling time. Also, soil coring may not be feasible in areas that have sandy soil or very fine soil (HUD, 1995). A modified soil coring method using a bulb planter can be used to collect a sample to a predetermined depth. The sample collected with the bulb planter can then be transferred to a sample container.

When conducting sampling care should be taken to avoid the following.

- Disturbed areas (i.e. construction sites, areas around concrete pads or foundations, telephone and electric poles, freshly plowed crop fields, trees, planters, and areas of animal burrowing activity).
- Areas near wooden structures where treated wood may have been used.
- High-traffic areas (i.e. parking lots, roadways, sidewalks).
- Areas with potential for run-on/run-off from rain or snowmelt.
- Areas near known dioxin sources.
- Areas of very dense turf grass.
- Areas which are not level.

The exact number and location of soil samples will depend on the sampling objectives that need to be achieved. If a larger number of samples will be collected to achieve the sampling objectives, then a grid system of sampling is recommended (Figure 2-1) with samples collected at the intersection points. The grid sampling approach is ideal for initial studies at a site or in the development of a survey strategy. The area of interest for the grid will vary from survey to survey. For a background survey of non-impacted sites, it is recommended that samples be taken within a 50 ft. radius of the central sampling location; however, this area can likely be increased to as much as a 500 ft. radius and still be representative of the central sampling point provided that all of the avoidance criteria listed above have been met and that legal access to the area has been obtained.

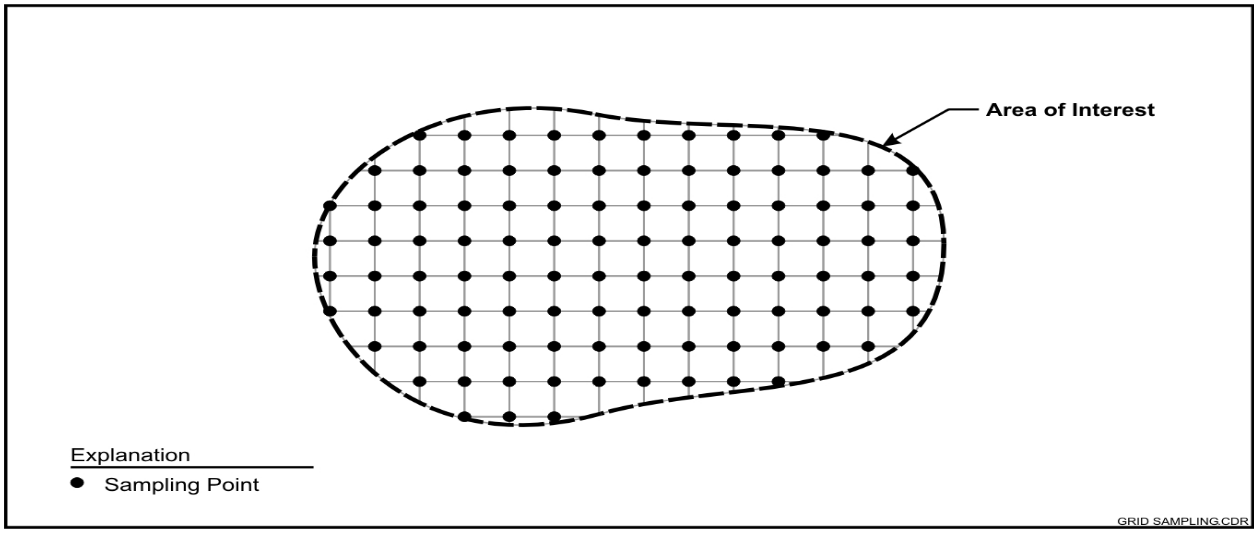

Figure 2-1. Example of a Grid Sampling Technique

If fewer samples will be collected or if an initial survey has been completed to determine the recommended number of sampling points at a site, an “x” sampling pattern can be used (Figure 2-2) (HUD, 1995). Samples can be collected at the intersection of the two lines, at the endpoints, and at any location along the lines if needed to collect the desired number of samples.

The length of the sampling line will depend on the size area to be studied and the placement of buildings, structures, or roadways at the site. The center sampling point should be located approximately in the center of the area to be studied and should allow the remaining sampling points to stay within the area to be studied and away from disturbed areas. The area that each individual sampling point can be moved without relocating the entire sampling grid should be determined in an initial sampling survey and will depend on the number of sampling points and the sampling grid size.

2.1.1 Sampling Location. For the purposes of this sampling protocol, sampling location refers to the general site where a set of samples will be collected. The term sampling point refers to the exact spot within a sampling location where the soil samples will be collected.

In order for a site to be considered for background sampling, there must be some site history available to rule-out any potential for contamination. There should be no known dioxin contamination and the site should not be located near highly populated areas to be considered for background purposes. Possible background sites should be evaluated for dioxin sources utilizing lists of known sources of dioxins such as *Database of Sources of Environmental Releases of Dioxin-Like Compounds in the United States* (EPA, 2001). Maps of the potential sampling location should be studied before the area is selected for a background sample location. Proximity to large urban areas, general site topography, and potential areas of erosion or deposition are some of the information that can be gathered from a review of maps of potential sampling locations. Maps can be found on the internet (topozone.com, mapserver.maptech.com, etc.) or they can be ordered through the United States Geologic Survey (USGS). The USGS has conducted geologically surveys throughout the United States and may have a lot of available data on potential sampling locations. Topographic and aerial maps of many areas of the United States are available through the USGS.

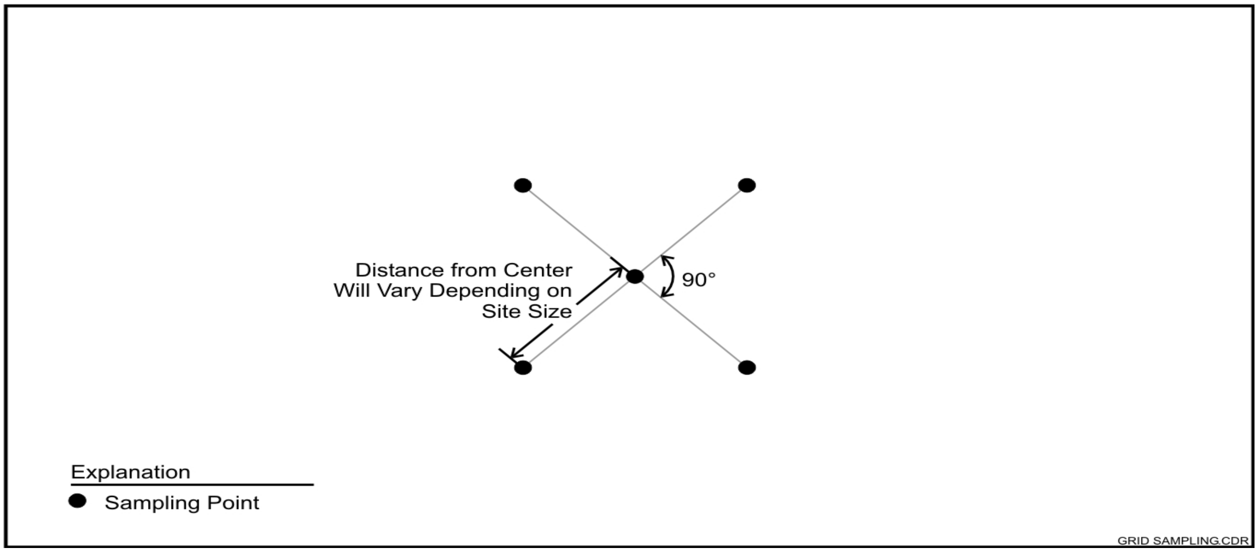

Figure 2-2. Example of “X” Sampling Technique

During field sampling, correct sampling points should be verified using a global positioning system (GPS) or some other survey method to determine latitude and longitude. Since the use of GPS may be impractical for large sampling efforts or where multiple sampling teams will be utilized, sampling points can be identified from a point that has previously been surveyed. In this case the starting point from Figure 2-2 would be located at the surveyed point and the remaining sampling points would be measured from the surveyed point. A compass would be used to determine direction from the surveyed point and a measuring tape would be used to measure the distance away from the surveyed point. Care should be taken to ensure that each sampling point avoids problematic areas as listed in the previous section.

2.1.2 Sampling Depth. Background levels of dioxins, which are deposited across the country via airborne transportation, can be determined by sampling surface soils. Deeper sampling should not be required because dioxins are not considered mobile contaminants, especially when atmospheric deposition is the suspected source (Rogowski et al., 1999).

The definition of surface soil varies throughout literature, but for the purpose of background sampling for dioxins a depth of 0 – 10 cm is generally accepted (Rogowski and Yake, 1999; Vikelsoe, 2002). Where the primary source of dioxins is air deposition, the type of soil will play a role in how deep the dioxins migrate into the soil. Highly organic soils will retain dioxins closer to the surface. The sampling depth should be kept to a minimum to avoid diluting analytes. Ideally an initial survey should be carried out to assess the distribution of dioxins in the soil to determine an adequate sampling depth of 10 cm or less. This can be accomplished by collecting plugs of soil with a bulb planter and segmenting the resulting soil plug into discreet sections (i.e., 0 - 5 cm and 5 -10 cm). The discreet sections of soil can be analyzed to determine how far dioxins have migrated and this information can be used to finalize the sampling depth.

2.1.3 Sample Number. The recommended number of sampling points will be dependent on individual survey objectives. Conducting an initial survey that oversamples a site is recommended. Variability of the data generated from the initial survey can be analyzed to establish the standard error as a function of the number of samples in order to determine an acceptable number of sampling points. Further guidance on sample number can be found in *Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies* (EPA, 1992).

2.1.4 Sample Quantity. The total quantity of sample required will depend on the analyses being performed and if samples will be archived for future analyses. The amount of soil needed for each analysis should be determined and should take into account extra material needed to supply laboratory quality control samples such as matrix spikes and replicates. The amount of soil needed for each analysis should be summed to determine a total amount of soil. Enough additional soil should be collected to allow for compositing and additional or repeat analyses that may be required. As guidance for sampling surface soils for dioxins and chemical/physical parameters, and a few selected additional analytes approximately 600 g of soil should be collected in three 8-oz wide-mouth, amber glass, certified clean sample containers (Environmental Sampling Supply PC class, or equivalent). An additional set of containers can be collected if samples will be archived for future analyses. To fill six 8-oz soil jars with a sampling depth of 0-10 cm an area of approximately 20 cm by 20 cm will need to be sampled. The actual area may vary by site depending on how rocky the soil is and how much vegetation is present.

2.2 Sampling Equipment

Section 2.0 of Standard Operating Procedure [SOP] for Surface Soil Sampling for Dioxins (Attachment 1) contains a detailed list of sampling equipment.

2.3 Sample Collection

Section 3.0 of SOP for Surface Soil Sampling for Dioxins (Attachment 1) contains a detailed information on sampling procedures.

2.4 Long-Term Archiving of Samples

Long-term archiving of soil samples may be required if there is the potential for future analyses (i.e., analysis of individual samples because of composite sample results). For long term storage, EPA Method 1613, Revision B (EPA, 1994) for PCDD/PCDF and EPA Method 1668, Revision A (EPA, 1999) for PCBs state that soil samples can be stored in the dark at a temperature of less than -10°C for up to one year.

If samples are to be archived for potential future analysis of other analytes, it is recommended that they be maintained frozen, at a temperature of less than -10°C. Note that holding times for many other analytes are comparatively short (1 month or less). If the archived samples exceed method recommended holding times, it is recommended that the data be flagged as such.

3.0 FIELD SAMPLING QUALITY ASSURANCE

Quality assurance (QA) is an integrated system of activities in the area of quality planning, assessment, and improvement to provide the survey with a measurable assurance that the established standards of quality are met. Quality control (QC) checks, including both field and laboratory, are specific operational techniques and activities used to fulfill the QA requirements. Project specific field and laboratory QC checks should be specified in a project specific Qaulity Assurance Project Plan.

3.1 Field Quality Control Samples

The field QC samples should be assigned unique sample numbers and submitted as regular (blind) samples to the analytical laboratory. If abnormalities are detected in field QC samples, the data associated with the QC samples should be flagged and appropriate actions should be taken to rectify issues. Field QC samples will be survey specific and should be outlined in the Quality Assurance Project Plan for the survey. Field QC samples may include equipment rinsate blanks, trip blanks, and field blanks. A temperature blank should always be used for sample shipment of samples requiring a temperature preservative.

3.1.1 Equipment Rinsate Blanks. Equipment rinsate blanks are generally collected at a frequency of one per day to ensure that nondedicated sampling devices have been decontaminated effectively. Equipment rinsate blanks consist of the rinsewater used in the final water rinse step of the sampling equipment decontamination procedure before the equipment is sprayed or rinsed with a solvent. Rinsate samples may be collected more frequently if required to meet the survey requirements. Details on collecting an equipment rinsate blank can be found in Section 3.11 of SOP for Surface Soil Sampling for Dioxins (Attachment 1).

3.1.2 Trip Blanks. Trip blanks are generally prepared either by the analytical laboratory or the field sampling crew by filling an amber glass soil jar with clean playsand. Trip blanks are not to be opened in the field. Trip blanks generally only need to be analyzed if contamination is suspected in actual associated site samples. Trip blanks indicate whether the field samples have been contaminated during storage and shipping and are usually collected at a frequency of one per sample cooler.

3.1.3 Field Blanks. Field blanks can indicate contamination of samples during sample collection. Field blanks are usually collected at a frequency of one per site. Field blanks are prepared either by the analytical laboratory or the field sampler(s) by filling an amber glass soil jar with clean playsand. This jar should be opened and placed uncapped on an even surface upwind of the sample location during collection of the soil samples.

3.1.4 Temperature Blanks. Temperature blanks should accompany each cooler containing samples with a temperature preservative requirement. The temperature blank is prepared either by the analytical laboratory or the field sampler(s) by filling an amber glass soil jar with clean playsand. The temperature of the samples is verified upon arrival at the analytical laboratory using the temperature blank.

3.2 Sample Handling and Custody

The following procedures ensure proper handling, custody, and documentation of the samples from field collection through laboratory analysis.

3.2.1 Sample Containers, Preservation and Holding Time. Requirements for sample preservation and holding times are listed in Table 3-1. New, precleaned amber sample containers (Environmental Sampling Supply PC Class, or equivalent) should be used for soil sample collection. Once collected, each containerized sample is labeled and placed into a sample cooler. The sample cooler serves as the shipping container and should be packed with ice to cool samples to the appropriate temperature for preservation. It is important that wet ice be used to cool and ship samples to maintain proper temperature.

Table 3-1. Sample Holding Times and Preservation Methods[1]

Method	Parameters	Preservation	Holding Time
1613B (USEPA, 1994)	PCDD/PCDF -HRMS	Cool, 4°C	If samples are stored <$^{-}$10°C, samples may be held for one year.
1668A (USEPA, 1999)	PCBs - HRMS	Cool, 4°C	If samples are stored <$^{-}$10°C, samples may be held for one year.
CALUX	Bioassay TEQs	Cool, 4°C	3 months
Walkley-Black (Walkley, 1934)	Total Organic Carbon	Cool, 4°C	28 Days
SW 9045C (EPA, 1995)	pH	Cool, 4°C	NA
ASTM D422 (ASTM, 2002a)	Grain Size Distribution	NA	NA
ASTM D2216 (ASTM, 2002b)	Moisture Content	NA	NA

[1] Note that the information listed in Table 3-1 is for supporting a background dioxin level soil survey. If other methods are required to meet specific survey objectives, then the information in this table should be updated for each method used.

3.2.2 Sample Identification. Each sample should be given a unique sample identification (ID). The sample ID is survey specific and a record of all sample IDs is kept with the field records and recorded on a chain of custody form.

3.2.3 Sample Labeling. Section 3.5 of Standard Operating Procedure [SOP] for Surface Soil Sampling for Dioxins (Attachment 1) contains detailed information on labeling samples.

3.2.4 Sample Packing and Shipment. Section 3.9 of Standard Operating Procedure [SOP] for Surface Soil Sampling for Dioxins (Attachment 1) contains a detailed information on packing and shipping samples.

3.3 Field Documents and Records

3.3.1 Field Logbook. A survey-specific and site-specific field logbook is used to provide daily records of significant events, observations, and measurements during field investigations. The field logbook is also used to document all sampling activities. All logbook entries should be made with indelible ink to provide a permanent record. These logbooks should be maintained as permanent records.

The field logbooks are intended to provide sufficient data and observations to reconstruct events that occurred during field activities. Field logbooks should be bound and prepaginated; the use of designated forms should be used whenever possible to ensure that field records are complete. A site map and area to record sampling locations should be included with the field logbook.

Section 3.6 of Standard Operating Procedure [SOP] for Surface Soil Sampling for Dioxins (Attachment 1) contains a detailed information on completing field logbooks.

3.3.2 Sample Custody. All samples collected must be logged onto a chain-of-custody form in the field prior to shipment to the laboratory. The chain-of-custody form is signed by the individual responsible for custody of the sample containers, and the original accompanies the samples to the laboratory. One copy of the chain-of-custody form should be kept by the field team and included in any survey files.

The laboratory receiving the samples should have a designated sample custodian. This individual is responsible for inspecting and verifying the correctness of the chain-of-custody records upon sample receipt. The sample custodian will accept the samples by signing the chain-of-custody form and noting the condition of the samples in the space provided on the chain-of-custody form or other receipt form.

Immediately after receipt, the samples should be stored in an appropriate secure storage area meeting the holding requirements listed in Table 3-1. The laboratory should maintain custody of the samples as required by the project. The analytical laboratory should keep written records showing the chronology of sample handling during the analysis process by various individuals at the laboratory.

Section 3.7 of Standard Operating Procedure [SOP] for Surface Soil Sampling for Dioxins (Attachment 1) contains a detailed information on completing chain-of-custody.

3.4 Field Corrective Action

Corrective actions may be initiated by any of the participants of the field data generation process (i.e., field technicians, field team leader, etc). It is important to generate corrective actions early in the field sampling process so that the problem has a greater chance of being resolved in a timely and cost-effective manner.

4.0 FIELD SAFETY

4.1 Safety Guidelines for Soil Sampling

Personnel should wear prescribed Personal Protective Equipment (PPE), as appropriate, during sampling activities. Sturdy shoes with good traction (i.e., steel-toed safety shoes) are recommended for use in the field. High levels of dioxins or other contaminants should not be encountered during sampling of background, non-impacted sites; regardless, contamination avoidance should be practiced at all times. Personnel responsible for handling soil samples should wear disposable nitrile gloves (or equivalent) and safety glasses with side shields should be worn during decontamination procedures. Personnel should be advised of the hazard type and potential contaminants present in the samples. Because cigarette smoke is a potential source of dioxins and because flammable materials may be used during equipment decontamination, there should be absolutely no smoking at any time during the sample collection process.

4.1.1 Slip-Trip-Fall Hazards. Although it is difficult to prevent slip-trip-fall hazards, these hazards can be minimized through good housekeeping, proper site control measures and by keeping the work area free of obstructions. Slip, trip, and fall hazards should be addressed through an ongoing proactive housekeeping program that eliminates elements in the work area that have potential for causing substantial loss of footing.

4.1.2 Lifting Hazards. Field operations often require that physical labor tasks be performed. All personnel should employ proper lifting procedures. Additionally, personnel should not attempt to lift bulky or heavy objects (greater than 60 pounds) without assistance.

4.1.3 Decontamination Safety. Exposure to chemical decontamination materials and solutions should be controlled by the use of appropriate personal protective clothing and accessories, which includes safety glasses and nitrile gloves or equivalent. Material Safety Data Sheet (MSDS) can be used to find safety information for the solvent(s) (i.e. methanol, acetone, or hexane) and the non-phosphate detergent.

4.2 Heat and Cold Stress Hazards

4.2.1 Heat Stress. During hot or humid days, or during the performance of strenuous work, extra precautions are necessary to reduce the potential for heat stress. Implementation of worker rotation and rest period schedules and adjustment of the workday to take advantage of the cooler parts of the day may be used to prevent exposure to heat stress hazards. Whenever possible, shade should be utilized or provided to field personnel to help mitigate heat stress hazards. Also, frequent consumption of water or an electrolytic beverage is necessary to prevent dehydration. The levels of heat stress are characterized in Table 4-1.

Table 4-1. Signs and Symptoms of Heat-Related Illnesses and Treatments

Heat Induced Problems			
Problem	**Body Response**	**Signs and Symptoms**	**Treatment**
Heat Cramps	The body loses too much salt from heavy exertion in heat.	Painful spasms of muscles used during work.	Increase fluid intake with electrolytes (Unless otherwise indicated by a doctor). Take frequent breaks, preferably in a cool area.
Heat Exhaustion	The body can't replace fluids and/or salt lost in sweating. Perspiration in heat is important because it cools the body as it evaporates.	Weakness, dizziness, nausea. Pale or flushed appearance. Sweating, moist and clammy skin.	Move to a cool place. Loosen clothes and apply cool compresses. Drink water slowly. Elevate feet 8-12 inches.
Heat Stroke	The body no longer sweats and holds so much heat that body temperature reaches dangerous levels. **Heat stroke is a medical EMERGENCY and can lead to delirium, convulsions, unconsciousness, or death.**	DRY, hot reddish skin, and LACK OF SWEATING! High body temperature and strong, rapid pulse. Chills Confusion	**Treat as a MEDICAL EMERGENCY!** Call for EMS or a doctor immediately! Move to a cool area immediately. Use cool water to soak person's clothes and body. Fan the body. Don't give fluids if victim is unconscious.

EMS = Emergency Medical Services.

4.2.2 Cold Stress. Working under cold conditions can lead to various injuries or health effects, collectively known as cold stress. The hazardous effects of cold on the body may include dehydration, numbness, shivering, frostbite, immersion foot (trench foot), and hypothermia.

The effects of cold stress can be minimized by providing the proper training, controlling temperature and wind whenever possible by using heaters and/or windbreaks. Workers should be rotated if extreme cold conditions are encountered to avoid overexposure. Proper protective clothing, which provides insulation but also allows sweat to evaporate, should be used, including protection for the feet, hands, head, and face. Seek warm locations during breaks and replace lost fluids with warm, sweet, non-caffeine-containing drinks to avoid dehydration.

4.3 Biological Hazards

Biological hazards may include animal bites, insect bites and stings, contact with poisonous plants, and exposure to pathogenic (disease producing) microorganisms. Animal and bird droppings often contain mold, fungus, bacteria or viruses that represent a respiratory hazard. If encountered, personnel should avoid touching droppings.

First aid procedures for biological hazards should follow recommended procedures set by the American Red Cross. Paramedics should be summoned for serious injuries.

5.0 REFERENCES

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Attachment-1

Standard Operating Procedure
for
Surface Soil Sampling for Dioxins

SOP:Surface Soil Sampling for Dioxins
Rev: 1.0
Date: August 26, 2003

STANDARD OPERATING PROCEDURE
for
SURFACE SOIL SAMPLING FOR DIOXINS

Table of Contents

1.0 Scope and Application

The purpose of this Standard Operating Procedure (SOP) is to provide samplers with a step-by-step guide for collecting surface soil samples for dioxin analysis.

For the purposes of this document the term dioxin refers to the broad class of compounds including polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and dioxin-like polychlorinated biphenyls (PCBs).

2.0 Equipment/Materials Required

- Surface Soil Sampling for Dioxins SOP
- Map of the site and potential sampling points
- Field Logbook and Site Map Schematic (See Figures 1 and 2)
- Ballpoint pens
- Permanent markers (Must be used on sample labels, ballpoint pens may run)
- Chain-of-Custody (See Figure 3)
- Cooler(s)
- Nitrile gloves (or equivalent)
- Wooden or metal stakes with reflective plastic ties
- Bulb planter, stainless steel scoops/spades, or coring device
- Trowel for bulb planter method
- Disposable aluminum foil pans or tub/tray lined with aluminum foil
- Hand-held grass clippers
- Sample containers (8-oz amber glass, wide mouth soil jars with lids, Environmental Sampling Supply PC Class, or equivalent)
- Sample Labels
- Clear plastic packing tape (to tape over sample label)
- Strapping or duct tape (to tape up coolers)
- Tape Measure (Length of at least 100 feet)
- Compass or GPS unit
- Ruler (cm)
- Plastic bags (gallon size zip lock for ice and chain-of-custody)
- Plastic bags (quart size zip lock for sample containers)
- Plastic trash bags (lawn and leaf)
- Decontamination supplies (one to two buckets, 2 gallon size or larger), stiff bristle brush, spray bottles, reagent-grade methanol, and non-phosphate detergent)
- Deionized (DI) or distilled water (approximately 4 gallons)
- Container with potable water

- Waste container
- Funnel (to transfer liquid waste to waste container)
- Aluminum foil
- Paper towels
- Bubble wrap
- Federal Express (or other overnight courier service) labels
- Ice (approximately 4 bags)
- Soil/Air thermometer (digital or standard liquid-filled)

Optional equipment includes:

- Safety glasses with side shields, sturdy shoes with good traction [i.e., steel-toed safety shoes], sun screen, and hat
- Sieve (19 millimeter opening)
- Potting soil for use in site restoration
- Disposable or digital camera for site photos
- Custody seals

3.0 Sampling Procedures

Please note that because cigarette smoke is a potential source of dioxins and because flammable materials will be used during equipment decontamination, there should be absolutely no smoking at any time during the sample collection process. Exhaust from vehicles and electrical generators can also be a source of dioxins and therefore sample collection should be performed away from running vehicles or generators.

3.1 Locating Recommended Surface Soil Sampling Points. Recommended sampling points should be determined and placed on a site map prior to beginning field activities. All sampling points must avoid the following problem areas.

- Disturbed areas (i.e. construction sites, areas around concrete pads or foundations, telephone and electric poles, freshly plowed crop fields, trees, planters, and areas of animal burrowing activity).
- Areas near wooden structures where treated wood may have been used.
- High-traffic areas (i.e. parking lots, roadways, sidewalks).
- Areas with potential for run-on/run-off from rain or snowmelt.
- Areas near known dioxin sources.
- Areas of very dense turf grass.
- Areas which are not level.

Once in the field, make sure the center sampling point avoids all problem areas noted above. The sampler should be given instructions as to how far the center point and all other sampling points can be relocated to avoid problem areas without affecting the project goals.

Once at the site, stake out the final sampling points using the wooden or metal stakes with reflective ties or flags and verify the latitude and longitude of each point using global positioning satellite (GPS) or other means of surveying. Sampling points can also be measured from a previously surveyed point using a tape measure and compass. If desired, a disposable or digital camera can be used to take photos documenting each sampling point and the sampling location.

3.2 **Groundcover Removal.** Groundcover may consist of grass, other vegetation, or rocks/pebbles. Areas with dense groundcover, including turf grass, should be avoided.

- Groundcover removal should be performed using gloved hands. The groundcover should only be removed to the point where soil is exposed, being careful not to disturb the soil below. If tall grass or weeds are present they can be cut down using hand-held grass clippers to within 0.25 in. of the soil to the point where exposed soil can be identified.
- If the sampling point does not contain vegetation then any rocks or pebbles can be brushed aside by the sampler(s) using a gloved hand.
- If areas with large or dense vegetation, such as trees, turf grass, or bushes are located at the sampling point the sampling point should be moved (See Section 3.1). Cover from vegetation may affect the deposition of dioxins and therefore may not represent a true background sample.
- An area of approximately 20 cm by 20 cm will need to be uncovered; this can be measured using a ruler. The actual area may vary by site depending on how rocky the soil is and how much vegetation is present.

3.3 Pre-Sample Collection Activities

Picture 1. Grass Removal to within 0.25 in. of the Soil Surface

- Before beginning sampling, take out the field blank and position it upwind of the sampling point making sure that the container is on an even surface in an area where the container will not be knocked over.
- Remove the lid of the field blank container.
- The field blank should be recapped after each sample is collected and moved to the next sampling point where the lid should again be removed.
- After sampling is complete the lid should be secured tightly and the field blank should be handled like the other soil samples following the guidelines in Section 3.9.
- Place bubble wrap on the bottom of the sample cooler.
- Place one garbage bag (lawn and leaf size) in the sample cooler on top of the bubble wrap.
- Divide one bag of ice into several (3 or 4) double bagged gallon-size zip lock bags and place them inside the garbage bag at the bottom of the sample cooler. The garbage bag will help to ensure that water from the ice does not leak out of the cooler during shipping.
- Place the temperature blank and trip blank in the cooler with the ice.
- As samples are collected they should be placed in the cooler with the ice.

3.4 Soil Sampling. Once the vegetation and/or rocks/pebbles have been removed per Section 3.2, a soil sample can be retrieved from 0-10 cm (exact depth should be specified in individual project plans) below ground surface (bgs) using a bulb planter (diameter of approximately 7.5 cm), stainless steel spade/scoop, or coring device. A bulb planter is the recommended method of surface sampling if an intact core is not necessary, but may not be a practical method of sampling in sandy soils. The scoop/spade method is recommended for sampling sandy soils if the bulb planter can not be used.

Picture 2. Sampling Using the Bulb Planter Method

- Put on nitrile gloves (or equivalent).
- Determine the number of sample containers needed at each point to acquire sufficient sample.
- If soil temperature is required, follow the instructions in Section 3.10.
- If a bulb planter is used to collect samples the device should be inserted into the soil to the project specified sampling depth (no more than10 cm bgs) and twisted. A metal trowel should be inserted below the bottom of the bulb planter to ensure that the soil does not fall out when the bulb planter is lifted. Multiple plugs of soil taken next to the first may be needed to fill the required sample containers. Soil from the bulb planter can be placed onto disposable aluminum foil pan or a tub/tray covered with aluminum foil while the remaining sample is collected. Continue collecting plugs until enough sample has been collected to fill all sample containers. Sampling depth can be measured by placing a ruler in the sampling hole.

- Care should be taken to avoid including rocks, pebbles, vegetation, or debris in the sample container. A sieve with a 19 mm opening can be used to remove rocks, pebbles, vegetation, or debris. If a sieve is used the material passing through the sieve should be collected in disposable aluminum foil pan or tub/tray covered with aluminum foil before being transferred to a sampling container.
- If a sieve is not used, the sampler should inspect the sample in the disposable aluminum foil pan or tub/tray covered with aluminum foil and

use a gloved hand to remove rocks, pebbles, vegetation, or debris larger than 19 mm.

- Carefully knead soil to remove roots.

Picture 3. Removal of Rocks, Vegetation, or Debris

- If samples are to be composited in the field, follow compositing instructions in Section 3.12.
- To fill the sample containers, one scoop of soil should be divided equally among the total number of containers, (e.g., for three containers, the first third of the soil on the scoop should go into the first container then the next third in the next container, etc., continuing to fill each container in order until the containers are all full).
- If soil is too sandy or rocky to collect samples using a bulb planter or coring device then a scoop/spade can be used.
- If samples are collected with a coring device, after collecting the sample, the ends of the core should be capped with Teflon caps or Teflon sheets should be placed between the plastic cap and the soil.
- Once containers are full, the rim of the sample container should be wiped using a clean, unused paper towel and the lids should be tightly screwed into place.
- The sample container should be labeled according to Section 3.5 and packed into a cooler according to Section 3.9.
- After collection of the first soil sample the bulb planter and trowel, scoop/spade, or coring device should be decontaminated according to Section 3.8 and the equipment rinsate blank sample should be collected according to Section 3.11. A new disposable aluminum foil pan should be used for each sampling point. If a tub/tray covered in aluminum foil is

used instead of the disposable aluminum pan, the aluminum foil should be changed for each sampling point.

- Sampling equipment should be decontaminated according to Section 3.8 between each sampling point, but the equipment rinsate blank will only need to be collected after the first soil sample is taken each day of sampling.
- Remove and dispose of gloves after sampling and decontamination is complete. Put on a new pair of gloves before collecting a new sample.
- Fill out the field logbook and chain-of-custody per Sections 3.6 and 3.7, respectively.
- Remove stakes once soil samples have been collected and return site to original state as best as possible. Potting soil may be used to fill any holes created by sample removal.

3.5 **Sample Container Labels.** Each sample container should have a sample label affixed to the outside of the container in an obvious location. All information should be recorded using a permanent marker.

- Immediately after sampling record the date and time (military time [i.e., 1330]) of sampling along with the initials of the sampler(s) on the sample label.
- Any other required information should be included (i.e. sample identification number, preservation used). If possible this information should be filled in before sample labels are sent to the field.
- The completed sample label should be placed on the jar in an obvious location, then be completely taped over with clear tape (i.e. packing tape) to prevent the label from getting wet, smudged, or lost during transport. Tape over the label before placing the sample in the cooler because the tape will not stick properly after the sample container is wet or cold.

3.6 **Field Logbooks.** The field logbooks are intended to provide sufficient data and observations to reconstruct events that occurred during field activities. An example field logbook page is included as Figure 1. The following are examples of information to be included by the sampler(s) in a field logbook:

- Project name and location
- Name, date, and time of entry
- Names and responsibilities of field crew members
- Name and titles of any site visitors involved in or actively observing the sampling.

- Weather information including air temperature and recent precipitation; soil temperature if required for a survey.
- Descriptions of deviations or option selections from the sampling SOP procedures and any problems encountered
- Number, amount, and ID of samples taken at each point
- Details of sampling location, including sampling coordinates in latitude and longitude. Actual sampling points should be marked on a map or schematic (See Figure 2).
- Date and time of sample collection
- General observations

3.7 **Chain-of-Custody.** All samples must be logged onto a chain-of-custody form in the field prior to shipment of samples to the laboratory. An example chain-of-custody form is included as Figure 3. As much information as possible should be filled in before sending the chain-of-custody form into the field. The following are examples of information to be recorded using a ballpoint pen by the sampler(s) in the field:

- Sample matrix
- Sample collector's name
- Dates/times of sample collection
- Sample identification numbers
- Number and type of containers for each sample aliquot
- Type of preservation
- Special handling instructions
- Name, date, time, and signature of each individual releasing or receiving the shipping container

The original copy of the chain-of-custody must be included with the samples and a copy should be kept with the field logbook.

3.8 **Decontamination of Sampling Equipment.** Decontamination is a process completed on all reusable or nondedicated field equipment to avoid cross-contamination between samples and to ensure the health and safety of the field sampler(s). The following sequence should be used to clean the bulb planter and trowel, scoop/spade, or coring device prior to taking the first sample and between each use:

1) Nitrile gloves (or equivalent) must be worn during decontamination.

2) Rinse with potable water, collecting rinse water in one of the decontamination buckets.
3) Wash with a spray bottle containing Liquinox™ (or equivalent non-phosphate detergent) and water and clean with the stiff-bristle brush until all evidence of soil or other material has been removed.
4) Rinse with DI or distilled water three times, ensuring that all soap from the previous step has been removed. (After the first sampling point has been completed, the equipment rinsate blank is collected after the third rinse with DI or distilled water before the equipment is sprayed with methanol [See Section 3.11]). For other samples these water rinses should be collected in the second decontamination bucket.
5) Rinse with methanol contained in a spray bottle. Use the spray bottle to completely mist the equipment with methanol.
6) Place the bulb planter and trowel, scoop/spade or coring device on a piece of aluminum foil to keep the equipment clean and air-dry, protected from the environment. The bulb planter, trowel, scoop/spade or coring device must be air-dried before use.
7) A trash bag should be provided for waste paper towels and used nitrile gloves.
8) Decontamination water in the 2-gallon buckets should be disposed of according to applicable regulations.
9) Replace disposable aluminum pans for each sample. If a tub/tray covered in aluminum foil is used instead, replace the foil covering the tub/tray for each sample.

Picture 4. Example Decontamination Set Up for Soil Sampling

3.9 Packing and Shipping Samples. Immediately after sample collection and sample labeling, samples should be packed as follows:

- The cooler should be lined with a garbage bag and filled with wet ice which has been double bagged in gallon-size zip lock bags in order to meet the temperature requirements (4 ± 2˚C).
- The temperature blank and trip blank should accompany the cooler with the soil samples.
- Sample cooler drain spouts (if present) should be taped (using duct tape) from the inside and outside of the cooler to prevent any leakage.
- The sample container should be put in a quart size zip lock bag. The bagged sample container should be wrapped with bubble wrap. Sample containers should then be placed in the sample cooler.
- Once all of the samples have been collected and placed in the cooler the samples should be packed with additional bubble wrap to prevent movement or breakage of the sample jars during transport.
- The completed chain-of-custody form (Section 3.7) should be placed in a resealable bag and taped to the lid of the cooler.
- The cooler should be banded with duct or strapping tape and if required custody seals can be placed along the cooler lid in order to prevent or indicate tampering.
- The cooler containing the environmental samples should be shipped to its destination by Federal Express (or other overnight courier) using the appropriate shipping labels for the courier. The cooler must be scheduled for priority overnight service to ensure that the temperature preservative requirement is not exceeded. Saturday deliveries, if required, should be coordinated with the laboratory.

3.10 Soil and Air Temperature. Soil and air temperature should be measured if required. Soil temperature can be measured with a digital thermometer or a standard liquid-filled thermometer. The digital thermometer can be purchased with the probe that is inserted into the soil to the required sampling depth for the survey. The soil temperature should be measured next to the area where the soil sample is collected, but should not be inserted into the exact location where the sample will be collected. This is to prevent cross-contamination from other sampling locations. The probe should remain in soil during sampling to allow the temperature reading to stabilize, once a stable reading is achieved this temperature should be recorded in the field logbook. A new soil temperature reading should be taken at each new sampling point.

Air temperature can be measured using any thermometer designed for standard temperature readings. The air temperature should be taken away from direct sunlight and sheltered from wind. Allow the temperature reading to stabilize over several minutes. A new temperature reading should be measured at each new sampling point or at least several times throughout a sampling day.

3.11 **Equipment Rinsate Blank Collection.** One equipment rinsate (ER) blank should be collected to ensure that nondedicated sampling devices (bulb planter and trowel, scoop/spade or coring device) have been decontaminated effectively. Equipment rinsate blanks consist of the rinsewater used in the final water rinse step of the sampling equipment decontamination procedure before the equipment is sprayed or rinsed with a solvent.

1) Collect the ER blank after the first sample is collected.
2) Decontaminate the scoop/spade or coring device as described in Section 3.8 steps 1-4.
3) Before the bulb planter and trowel, scoop/spade or coring device is sprayed with methanol, open the ER blank sample containers, rinse the bulb planter and trowel, scoop/spade or coring device with the DI or distilled water into the sample containers. Immediately replace and tighten the lid on the sample container.
4) Write the time and date on the sample label as described in Section 3.5.
5) Continue with step 5 of the decontamination process.

3.12 **Compositing and Sample Processing.** Soil samples can be composited in the field after sample collection is complete or samples can be shipped to the analytical laboratory where compositing under more controlled conditions can be performed.

1) Surface soil samples should be separated by site and by sampling point.
2) If a single sampling point required more than one container to obtain sufficient volume, then the contents of all containers from an individual sampling point should be homogenized into a single sample by emptying the contents of all the containers into an aluminum foil pan or stainless steel bowl and mixing thoroughly. The homogenized sample can then be returned to the original sample containers.
3) If the survey requires compositing samples from multiple sampling points within a single site, composites should be prepared by mixing uniform amounts of soil from each sampling point. Only samples from the same site should be combined to form composite samples. The uniform amount of each soil sample should be placed into an aluminum foil pan or stainless steel bowl and mixed thoroughly. Adequate mixing is achieved by stirring the material in a circular fashion with a stainless steel spoon and occasionally turning the material over.

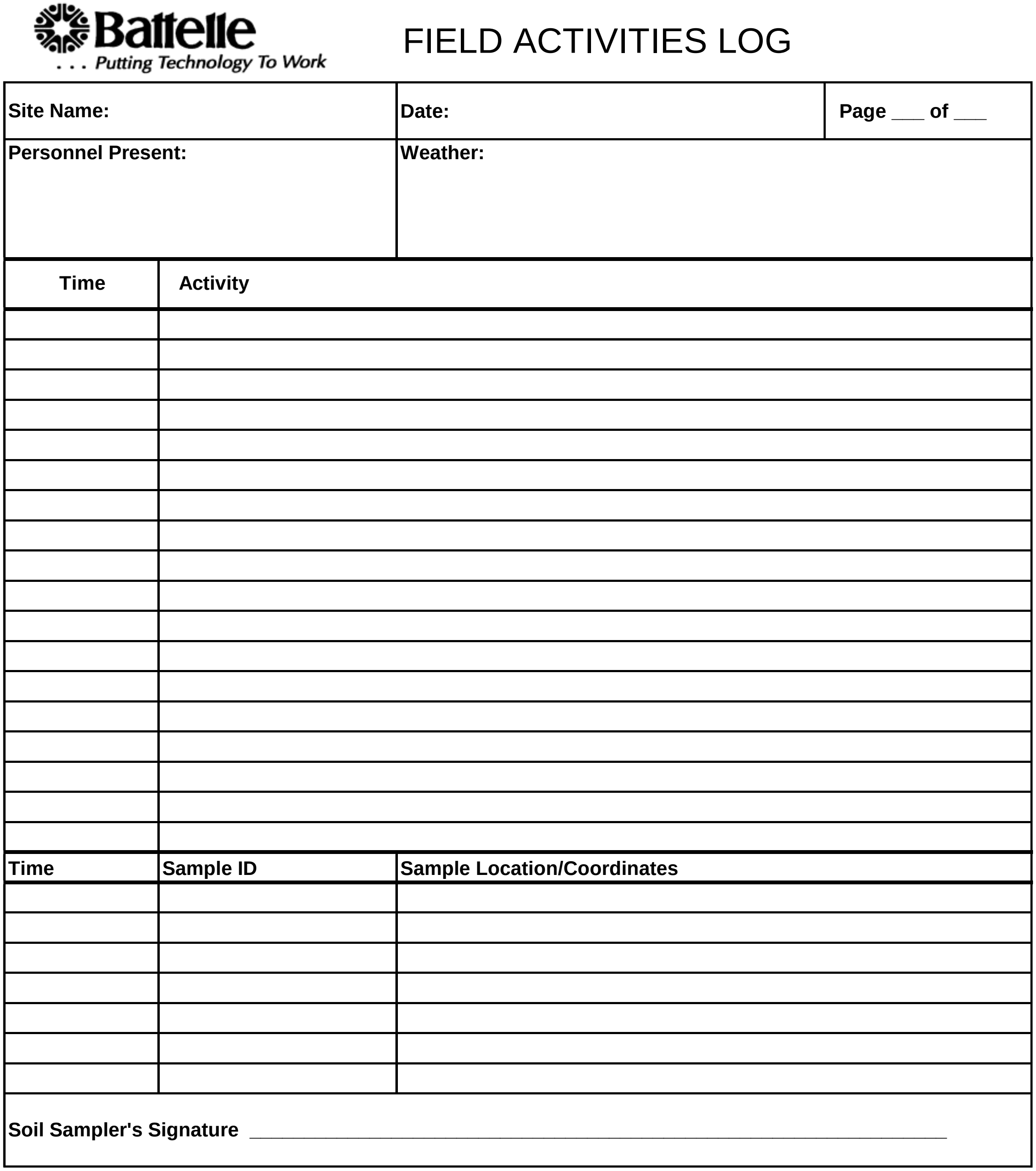
4) Sample identification numbers, which correspond to the sample identification of the individual sampling points that make up the composite, should be assigned to composite samples. The composite should be transferred to a sample container, labeled according to Section 3.5.
5) Individual samples and composites should be prepared for analytical testing or stored following specific survey guidance.

4.0 Health and Safety

Health and safety procedures are discussed in the sampling protocol (Battelle, 2003) and must be observed and implemented prior to any sample collection. The potential for chemical exposure will depend on the nature of the samples being collected and appropriate precautions should be taken. Physical hazards are only those that would be found in any typical outdoor activity. Please note that because cigarette smoke is a potential source of dioxins and because flammable materials will be used during equipment decontamination, there should be absolutely no smoking at any time during the sample collection process.

5.0 References

Battelle. 2003. Sampling Protocol for Dioxins in Soil. Prepared for the Environmental Protection Agency under Contract No. 68-W-99-033, Work Assignment 5-11.



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FIELD ACTIVITIES LOG

Site Name:	**Date:**	**Page ___ of ___**
Personnel Present:	**Weather:**	

Time	**Activity**

Time	**Sample ID**	**Sample Location/Coordinates**

Soil Sampler's Signature ____

Figure 1. Example Field Logbook Page

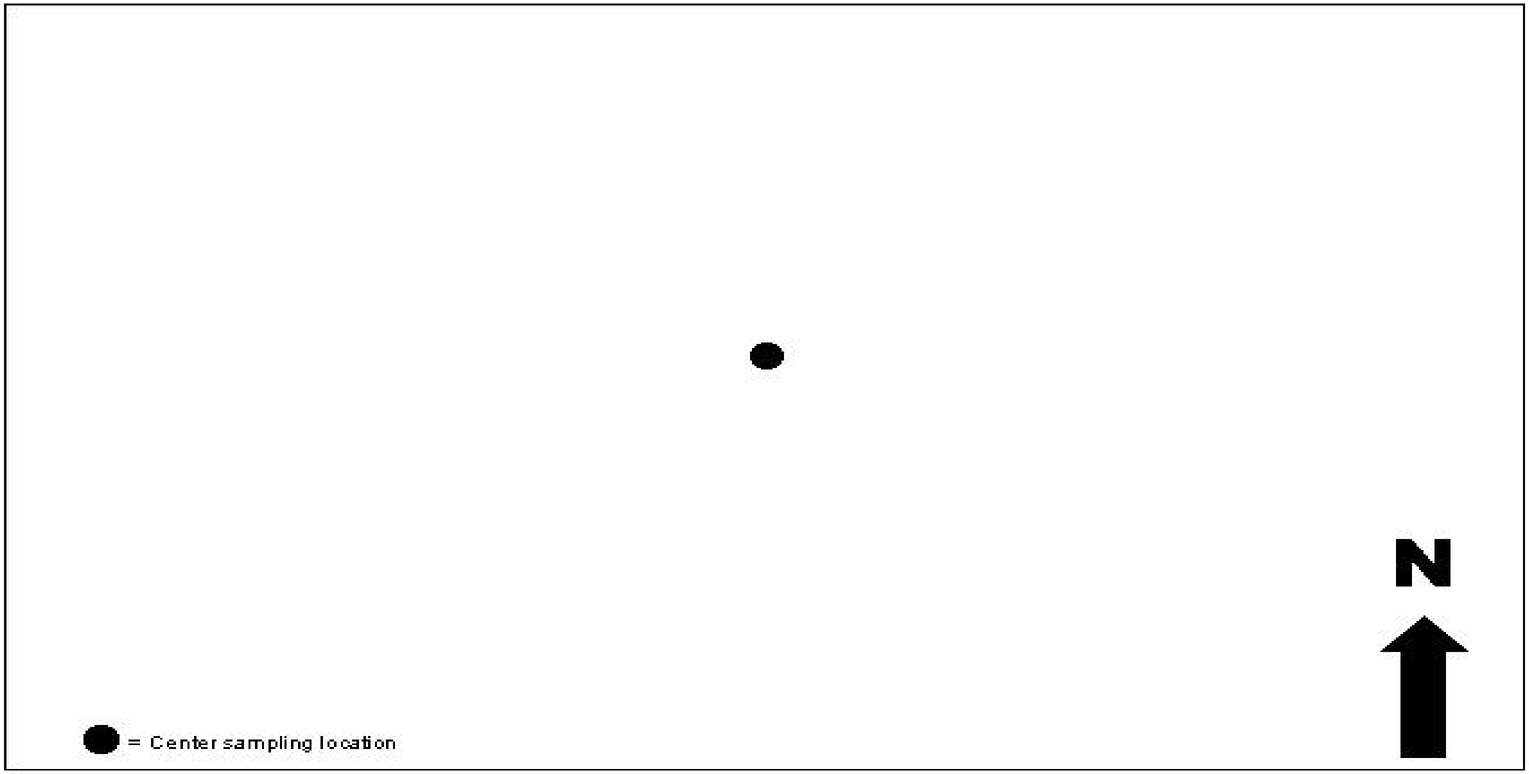
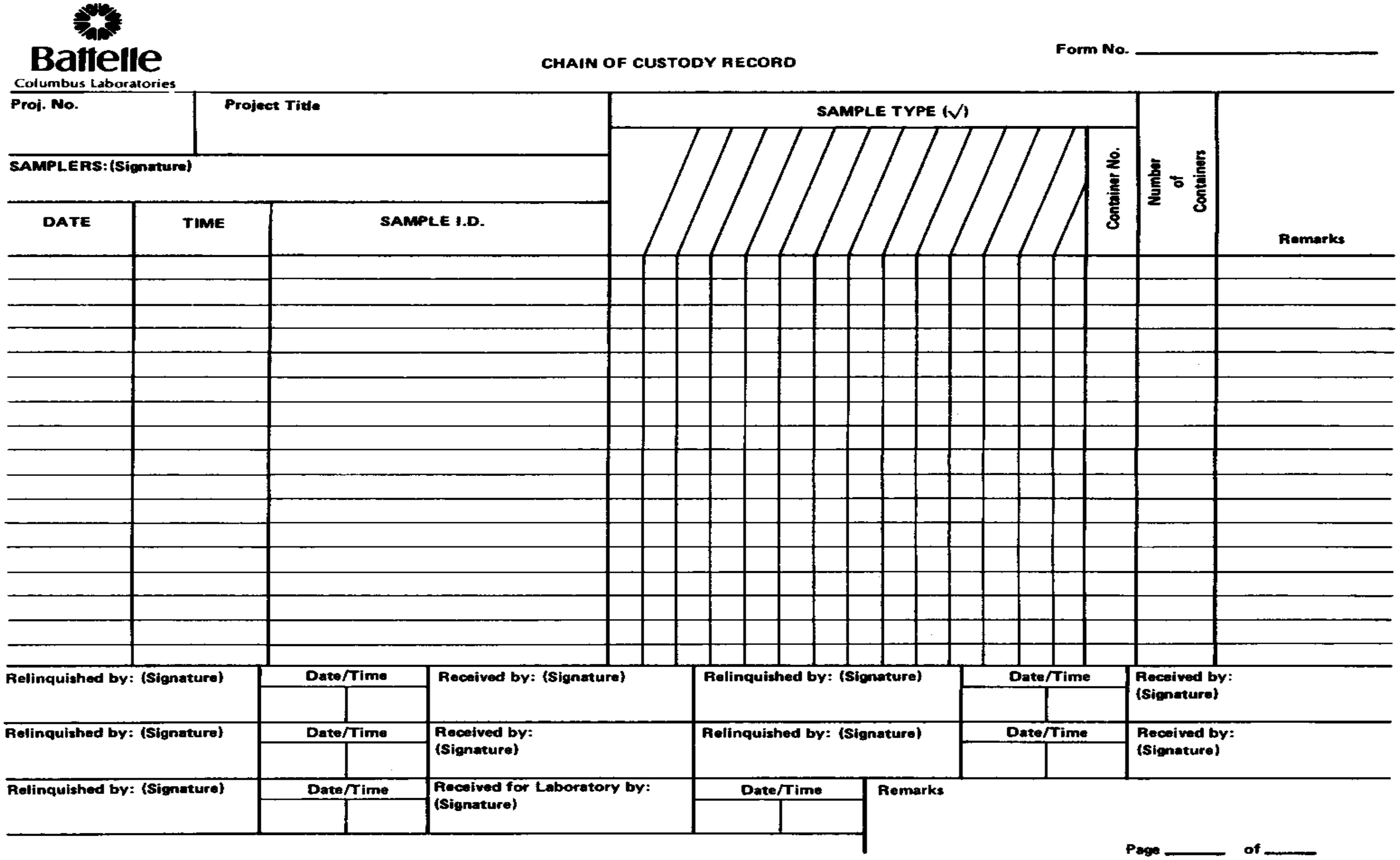


Figure 2. Site Map Schematic



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Columbus Laboratories

CHAIN OF CUSTODY RECORD

Form No. ____________

Proj. No.

Project Title

SAMPLERS: (Signature)

DATE	TIME	SAMPLE I.D.	SAMPLE TYPE (√)													Container No.	Number of Containers	Remarks

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Laboratory by: (Signature)	Date/Time	Remarks	

Page ______ of ______

Figure 3. Example Chain-of-Custody Form

APPENDIX D

QUALITY ASSURANCE/QUALITY CONTROL

WA5-11: CALUX Bioassay

PROJECT:	Pilot Survey of Dioxins in Soil
PARAMETER:	CALUX Bioassay
LABORATORY:	Xenobiotic Detection Systems, Inc. (XDS)
MATRIX:	Soil
SAMPLE CUSTODY:	Samples were received from Battelle on September 25, 2003, and October 30, 2003. All samples were intact and the cooler temperatures were 14°C and 2°C, respectively.

QA/QC MEASUREMENT QUALITY OBJECTIVES:

Parameter	Method Blank	LCS/MS Recovery	SRM % Difference	Replicate Relative Precision
CALUX Bioassay	<3 × RL	>50%	Within 50 PD of certified or consensus values	≤50% RPD

RL: reporting limit; LCS: laboratory control sample; MS: matrix spike; SRM: standard reference material; PD: percent difference; RPD: relative percent difference.

METHOD:

Sample Preparation: Samples were prepared according to XDS Method WL-2 “Extraction” and XDS Method WL-3 “Cleanup.”

Sample Analysis: Samples were analyzed according to XDS Method C-5.

Note: A more detailed description of the methodology is included in final report to Battelle.

HOLDING TIMES:

Samples for CALUX analyses were stored at room temperature until extraction.

Samples were extracted within 30 days of receipt and analyzed within 3 weeks of extraction.

Batch	Extraction Date	Analysis Date
B9-10	10/5/03	10/15/03
B9-10A	10/2/03	10/20/03
B9-10B	10/4/03	10/15/03
B9-10C	10/5/03	10/20/03
B9-10D	10/5/03	10/18/03
B9-10E	10/6/03	10/23/03
B9-10F	10/7/03	10/23/03
B9-10G	10/9/03	10/23/03

B9-10H	10/10/03	10/26/03
B9-10I	10/11/03	10/26/03
B9-10J	10/11/03	10/26/03
B9-10K	10/27/03	11/2/03
B9-10Gr1	11/3/03	11/6/03
B9-10L	11/5/03	11/9/03
B9-27	11/14/03	11/28/03

DETECTION LIMITS:

Reporting Limits (RLs) for CALUX analyses were determined on the basis of the nondetect limit of each bioassay. This level was adjusted for individual sample processing volumes and factors as follows:

RL (pg/g wet weight) = Toxicity equivalent of DMSO blanks
Formula: (2.5 × standard deviation (std dev) DMSO + positive B value [intercept parameter] calculated) corrected for samples size, dilution, and recovery.

Example:
DMSO Relative light units: 439,430,430.
Std dev = 5.10
2 × std dev = 12.76
B value = 39.03
Total = 51.83
Calculated per hill four equation (v*(d^n))/(d^n + k^n) = 0.027 TEQ
Calculated per 2 g sample, 1:4 dilution, and 76% recovery = 0.07 pg/g nondetect limit.

The target RLs of 0.2–0.5 pg/g wet was achieved for all nondetect samples.

BLANKS:

A laboratory method blank (MB) was prepared with each batch. The MB for each assay was less than three times the plate nondetect limit. Six of the method blanks were discarded by Q test.

LABORATORY CONTROL SAMPLE (LCS):

An LCS was prepared with each batch. Analyte recovery was determined to measure data quality in terms of accuracy.

CDD/CDF and PCB mixtures were used for the LCS. One was run on each bioassay.

For samples received on September 25, 2003, and processed in subsequent batches the LCS results were:
CDD/CDF = 77% ± 13%
PCB = 97% ± 15%

For samples received on October 30, 2003, the LCS results were:
CDD/CDF = 113%
PCB = 81%

MATRIX SPIKES (MSs):	Multiple MS samples were prepared and run with these samples. The percent recoveries of analytes in the MSs were calculated to measure data quality in terms of accuracy. All MS recoveries were >50%. MS samples processed with samples received September 25, 2003, were within 80% ± 18%. MS samples processed with the samples received October 30, 2003, were within 66% ± 5%.
REPLICATES:	Each sample was prepared in duplicate. The relative percent difference (RPD) between duplicates was calculated to measure data quality in terms of precision. RPDs ranged from 0% to 49% and were <50% for all samples except as follows: samples A03028, A03052, A03110, A03114, and A03121 were very low level or exhibited nonhomogenous traits that caused std devs higher than 50%.
STANDARD REFERENCE MATERIAL (SRM):	XDS QC sample (A00371) SRM was prepared with each batch. The percent difference (PD) between the measured value and the certified values were calculated to measure data quality in terms of accuracy. SRM PDs were within 50% of certified values.
PROBLEMS/ CORRECTIVE ACTIONS:	Samples with replicate std devs above 50% were re-extracted. With the exception of samples A03028, A03052, A03110, A03114, and A03121, all re-extract std devs were below 50%.

WA5-11: Mercury

PROJECT:	Pilot Survey of Dioxins in Soil
PARAMETER:	Mercury
LABORATORY:	Battelle Columbus, OH
MATRIX:	Soil
SAMPLE CUSTODY:	Sample Sets 1, 2, and 3 were received from Battelle's high-resolution mass spectrometry laboratory on September 9 and 29 and November 3, 2003, respectively. The samples had been stored in a refrigerator until pick-up for mercury analysis.

QA/QC MEASUREMENT QUALITY OBJECTIVES:

Parameter	Method Blank	LCS/MS Recovery	SRM % Difference	Replicate Relative Precision
Mercury	<3 × RL	75–125%	≤25% PD of certified or consensus values	≤20% RPD

RL: reporting limit; LCS: laboratory control sample; MS: matrix spike; SRM: standard reference material; PD: percent difference; RPD: relative percent difference.

METHOD: Sample Preparation and Analysis: The samples were digested in a water bath using sulfuric acid, nitric acid, potassium permanganate, and hydroxyl amine hydrochloride according to EPA SW846 Method 7471A. Approximately 2 g of each soil sample was digested. Samples were analyzed for total mercury using Cold Vapor Atomic Absorption following Method 7471A.

HOLDING TIMES: Samples for mercury analysis were refrigerated until extraction.

Samples were extracted within 28 days of receipt and analyzed the same day as extraction, with the exception of EPA-1 and EPA-17 in Set 2 and EPA-2 in Set 3. Due to a misunderstanding of sample receipt date, EPA-1 and EPA-17 were extracted after the 28-day hold time. EPA-2 was not received at Battelle until after the 28-day hold time.

Batch	Extraction Date	Analysis Date
Set 1	09/09/03	09/09/03
Set 2	09/29/03	09/29/03
Set 3	11/03/03	11/03/03

DETECTION LIMITS: Reporting Limits (RLs) for mercury were based on the detection limit reported in the method (0.2 µg/L). This level was adjusted for individual sample processing volumes and factors as follows:

RL (pg/g dry weight) = (Detection Limit × Digest Volume)/Sample Weight

Where,

Detection limit = 0.2 µg/L
Digest volume = 0.100 L
Sample weight (dry weight basis) = ~2g

The achieved detection limit of ~0.01µg/g met the target RL of 0.04 µg/g.

BLANKS:

A laboratory method blank was prepared with each sample set. No mercury was detected in any of the blanks.

LABORATORY CONTROL SAMPLE (LCS):

An LCS was prepared with each sample set to measure data quality in terms of accuracy.

Mercury was recovered within the 75–125% control limit for the LCS with each sample set.

Set 1 = 110%
Set 2 = 105%
Set 3 = 95%

MATRIX SPIKES (MSs):

An MS sample was prepared with each sample set. The percent recovery of mercury in the MS was calculated to measure data quality in terms of accuracy.

Mercury was recovered within the control limits of 75–125% for the MS in each sample set.

Set 1 = 94%
Set 2 = 85%
Set 3 = 122%

REPLICATES:

One sample was prepared in duplicate with each sample set. The relative percent difference (RPD) between replicate analyses for mercury was calculated to measure data quality in terms of precision.

RPDs were within the limit of 20% except for the duplicates in Set 1. The concentration of mercury in the duplicate sample for Set 1 was very close to the RL, and the absolute difference in the duplicates was less than the RL. Because of the very low levels of mercury in the duplicate sample, this duplicate, with an RPD of 27%, was still considered to show acceptable precision.

Set 1 = 27%
Set 2 = 3%
Set 3 = 15%

STANDARD REFERENCE MATERIAL (SRM):

NIST 1944 NY/NJ Sediment SRM was prepared with each sample set. The percent difference (PD) between the measured value and the certified values was calculated to measure data quality in terms of accuracy. The SRMs were within the 25% PD control limit for each sample set.

Set 1 = 8%
Set 2 = 3%
Set 3 = 17%

PROBLEMS/ CORRECTIVE ACTIONS:

Three samples exceeded the 28-day holding time: EPA-1 and EPA-17 in Set 2 and EPA-2 in Set 3. These samples were analyzed in spite of the holding time exceedence. Data from these three samples are flagged in the report.

The duplicate precision for Set 1 exceeded 20%; however, low mercury levels in this sample contributed to the exceedence, as described in the replicate section above. Because of the low analyte levels, results for the duplicate in Set 1 were still considered to demonstrate acceptable precision.

WA5-11- PCDD/PCDF
QC Batch 49971-13

PROJECT:	Pilot Survey of Dioxins in Soil
PARAMETER:	CDD/CDF
LABORATORY:	Battelle Columbus, OH
MATRIX:	Soil
SAMPLE CUSTODY:	Soil samples for PCDD/PCDF were received at Battelle Columbus between August 14, 2003, and October 22, 2003. Samples processed in QC Batch 49971-13 were homogenized and composited prior to being submitted for PCDD/PCDF extraction on September 10 and 15.

QA/QC MEASUREMENT QUALITY OBJECTIVES:

Parameter	Method Blank	Internal Standard Recovery	LCS/MS Recovery	SRM % Difference	Replicate Relative Precision
CDD/CDF	<3 × RL or associated samples >10X blank concentrations	25–150%	LCS within Method 1613B Table 6 limits for OPR; MS within 50–120% Recovery[a]	≤30% PD of certified or consensus values	≤30% RPD[a]

RL: reporting limit; LCS: laboratory control sample; MS: matrix spike; SRM: standard reference material; PD: percent difference; RPD: relative percent difference.

[a] Analyte concentrations must be >5 × RL.

METHOD: Soil samples were processed and analyzed for 17 2,3,7,8-substituted CDDs and CDFs and total dioixns and furans following general procedures in EPA Method 1613B.

Sample Preparation: Aliquots of each soil sample were weighed into individual jars and mixed with Hydromatrix drying agent. Approximately 20 g wet weight of each soil sample were used. The soil/Hydromatrix mixtures were spiked with $^{13}C_{12}$-labeled CDD/CDF and PCB internal standard solutions. Matrix spike (MS) and the laboratory control sample (LCS) were spiked with native CDDs/CDFs and PCBs at this time. The soil/Hydromatrix mixtures were placed into Accelerated Solvent Extraction (ASE) cells. The samples were ASE extracted using methylene choride. Each extract was processed through gel permeation chromatography cleanup and then spiked with 2,3,7,8-TCDD-$^{37}Cl_4$ and PCB cleanup standards for monitoring recovery of analytes through the cleanup procedures. Each extract was processed through acid/base silica and carbon cleanup columns. Each extract was separated into a CDD/CDF and PCB fraction on the carbon column. The CDD/CDF extracts were spiked with

1,2,3,4-TCDD-$^{13}C_{12}$ and 1,2,3,7,8,9-HxCDD-$^{13}C_{12}$ recovery standard and concentrated to a final sample volume of 10 µL. The PCB extracts were prepared as described in a separate report for PCB analysis of QC Batch 49971-13.

CDD/CDF Analysis: Each extract was analyzed by GC/HRMS in the selected ion-monitoring mode at a resolution of 10,000 or greater. A DB5 column was used for analysis of the 17 2,3,7,8-CDDs/CDFs and a DB225 column was used for second column confirmation of 2,3,7,8-TCDF.

The following revisions to Method 1613 as well as several items to note specifically related to these analyses are summarized below:

1. Quality control samples processed with this batch of samples included one method blank, one LCS, one sediment standard reference material (SRM), one matrix spike, and three samples prepared in duplicate.

2. The GC/HRMS instrumentation was calibrated for CDDs/CDFs at levels specified in Method 1613 with one additional calibration standard at concentrations equivalent to ½ the level of Method 1613's lowest calibration point. The calibration range corresponded to the following levels in the samples, assuming an average sample dry weight of 17.1554 g and a final sample volume of 10 µL: 0.15 to 120 pg/g dry for tetra compounds, 0.73 to 580 pg/g dry for penta through hepta compounds, and 1.50 to 1,200pg/g dry for octa compounds.

Any additional minor revisions to Method 1613 are fully documnented in the analytical record.

HOLDING TIMES:

Samples for CDD/CDF analyses were stored frozen until extraction.

Samples were extracted within 5 days of when the composites were received for CDD/CDF analysis, and initial analysis was completed within 5 weeks of extraction.

SDG Batch	Extraction Date	Analysis Date
49971-13	9/15/2003	10/13–14/2003 10/18/2003 (confirmation)

DETECTION LIMITS:

Reporting Limits (RLs) for CDDs/CDFs were determined on the basis of the lowest reasonably achievable detectable amount, determined as ¼ the lowest calibration standard. This level was adjusted for individual sample processing volumes and factors as follows:

RL (pg/g dry weight) = (0.25 × Concentration in Low Standard × Pre-rejection Volume)/Sample Weight

Where,

Concentration in low standard = 0.25 to 2.5 pg/µL

Pre-injection volume = 10 µL
Sample weight (dry weight basis) = ~ 17 g

The target RLs of 0.13–1.3 pg/g dry were achieved for all samples.

BLANKS:

A laboratory method blank was prepared with the sample delivery group (SDG). Several analytes were found above the detection limit but below the action limit of <3X the RL.

LABORATORY CONTROL SAMPLE:

An LCS was prepared with the SDG. The concentrations found were compared with limits in Method 1613B Table 6 ongoing precision and recovery sample to measure data quality in terms of accuracy.

49971-13: CDDs/CDFs were recovered within the control limits specified in Method 1613B Table 6.

MATRIX SPIKES:

An MS sample was prepared with the SDG. The percent recoveries of CDD/CDF in the MS were calculated to measure data quality in terms of accuracy.

49971-13: All CDDs/CDFs were recovered within the control limits of 50–120% except for 1,2,3,7,8,9-HxCDD, which had a recovery of 125%.

49971-13-17: CDD/CDF recoveries ranged from 94% to 125%**.**

LABELED INTERNAL STANDARDS:

Fifteen labeled internal standards were added to each sample prior to extraction. One labeled internal standard was also added to each sample prior to cleanup. Labeled internal standard recoveries were calculated to measure data quality in terms of accuracy (extraction efficiency).

49971-13: Internal standard recoveries were within the control limits for all analytes in all samples.

REPLICATES:

Three samples were prepared in duplicate with the SDG. The relative percent difference (RPD) between replicate analyses for CDDs/CDFs was calculated to measure data quality in terms of precision.

49971-13: For analytes >5X the RL, the RPDs ranges were

EPA 7: 5–13%
EPA 8: 8–72%
EPA 25: 1–5%

STANDARD REFERENCE MATERIAL (SRM):

NIST 1944 NY/NJ Sediment SRM was prepared with the SDG. Only reference values were available for CDDs/CDFs. The percent difference (PD) between the measured value and the reference values was calculated to measure data quality in terms of accuracy.

49971-13: The SRM was found to have very poor chromatography upon analysis. This sample was diluted and re-analyzed. The diluted analysis still had poor chromatography; however, results were obtained for nine of the analytes.

SRM PDs were within the control limits with the exception of OCDF, which had 35% PD.

PROBLEMS/ CORRECTIVE ACTIONS:

One of the continuing calibrations associated with this set had one native fail the criteria in Method 1613B and another had two natives fail the criteria. The average daily response factor was used to re-calculate the results for these outliers.

WA5-11- PCDD/PCDF
QC Batch 49917-23 and 49971-28

PROJECT:	Pilot Survey of Dioxins in Soil
PARAMETER:	CDD/CDF
LABORATORY:	Battelle Columbus, OH
MATRIX:	Soil
SAMPLE CUSTODY:	Soil samples for CDDs/CDFs were received at Battelle Columbus between August 14, 2003, and October 22, 2003. Samples processed in QC Batch 49971-23 were homogenized and composited prior to being extracted on November 6.

QA/QC MEASUREMENT QUALITY OBJECTIVES:

Parameter	Method Blank	Internal Standard Recovery	LCS/MS Recovery	SRM % Difference	Replicate Relative Precision
CDD/CDF	<3 × RL or associated samples >10X blank concentrations	25–150%	LCS within Method 1613B Table 6 limits for OPR; MS within 50–120% Recovery[a]	≤30% PD of certified or consensus values	≤30% RPD[a]

RL: reporting limit; LCS: laboratory control sample; MS: matrix spike; SRM: standard reference material; PD: percent difference; RPD: relative percent difference.

[a] Analyte concentrations must be >5 × RL.

METHOD: Soil samples were processed and analyzed for seventeen 2,3,7,8-substituted CDDs/CDFs and total dioixns and furans following general procedures in EPA Method 1613B.

Sample Preparation: Aliquots of each soil sample were weighed into individual jars and mixed with Hydromatrix drying agent. Approximately 20 g wet weight of each soil sample were used. The soil/Hydromatrix mixtures were spiked with $^{13}C_{12}$-labeled CDD/CDF and PCB internal standard solutions. Matrix spike (MS) and the laboratory control sample (LCS) were spiked with native CDDs/CDFs and PCBs at this time. The soil/Hydromatrix mixtures were placed into Accelerated Solvent Extraction (ASE) cells. The samples were ASE extracted using methylene choride. Each extract was intended to go through GPC cleanup, but there was a problem with the GPC instrument, and three samples were lost. These three samples (EPA-2 COMP, EPA-4 COMP, and the method blank) were re-extracted in sample delivery group (SDG) 49971-28. Each extract was spiked with 2,3,7,8-TCDD-$^{37}Cl_4$ and PCB cleanup standards for monitoring recovery of analytes through the cleanup procedures. All of the sample extracts from both SDGs were processed through the following cleanup procedures. Each extract was processed through acid/base wash, acid/base silica and carbon cleanup columns. Each extract was separated into a CDD/CDF and PCB fraction on the carbon column. The CDD/CDF fractions were spiked with 1,2,3,4-TCDD-$^{13}C_{12}$ and 1,2,3,7,8,9-HxCDD-$^{13}C_{12}$ recovery standard and concentrated to a final sample volume of 10 µL. The PCB extracts were prepared as described in a separate report for PCB analysis of QC Batches 49971-23 and 49971-28.

CDD/CDF Analysis: Each extract was analyzed by GC/HRMS in the selected ion-monitoring mode at a resolution of 10,000 or greater. A DB5 column was used for analysis of the 17 2,3,7,8-CDDs/CDFs and a DB225 column was used for second column confirmation of 2,3,7,8-TCDF.

The following revisions to Method 1613 as well as several items to note specifically related to these analyses are summarized below:

1. Quality control samples processed with this batch of samples included one method blank, one LCS, one sediment standard reference material (SRM), one matrix spike, and three samples prepared in duplicate.

2. The GC/HRMS instrumentation was calibrated for CDDs/CDFs at levels specified in Method 1613 with one additional calibration standard at concentrations equivalent to ½ the level of Method 1613's lowest calibration point. The calibration range corresponded to the following levels in the samples, assuming an average sample dry weight of 16.599 g and a final sample volume of 10 µL: 0.15 to 120 pg/g dry for tetra compounds, 0.73 to 600 pg/g dry for penta through hepta compounds, and 1.50 to 1,200 pg/g dry for octa compounds.

Any additional minor revisions to Method 1613 are fully documnented in the analytical record.

Samples for CDD/CDF analyses were stored frozen until extraction.

HOLDING TIMES:

Samples were extracted within 15 days of when the last composites were received for CDD/CDF analysis, and initial analysis was completed within 5 weeks of extraction.

SDG Batch	Extraction Date	Analysis Date
49971-23	11/06/2003	11/18–20/2003
49971-28	11/12/2003 (re-extracts)	12/09/2003 (confirmation)

DETECTION LIMITS:

Reporting Limits (RLs) for CDDs/CDFs were determined on the basis of the lowest reasonably achievable detectable amount, determined as ¼ the lowest calibration standard. This level was adjusted for individual sample processing volumes and factors as follows:

RL (pg/g dry weight) = (0.25 × Concentration in Low Standard × Pre-injection Volume)/Sample Weight

Where,

Concentration in low standard = 0.25 to 2.5 pg/µL
Pre-injection volume = 10 µL
Sample weight (dry weight basis) = ~ 16 g

The target RLs of 0.13–1.3 pg/g dry were achieved for all samples.

BLANKS:

A laboratory method blank was prepared with the SDG.

49971-23: Several analytes were found above the detection limit, but below the action limit of <3X the RL.

LABORATORY CONTROL SAMPLE:

An LCS was prepared with the SDG. The concentrations found were compared with limits in Method 1613B Table 6 ongoing precision and recovery sample to measure data quality in terms of accuracy.

49971-23: CDD/CDF were recovered within the control limits specified in Method 1613B Table 6.

MATRIX SPIKES:

An MS sample was prepared with the SDG. The percent recoveries of CDDs/CDFs in the MS were calculated to measure data quality in terms of accuracy.

49971-23: All CDDs/CDFs were recovered within the control limits of 50–120% except for OCDD, which had a recovery of –65%. To be effective, the spike concentration needs to be greater than five times the background concentration of the analyte of interest.[a] For OCDD the spike level was not greater than five times the background concentration.

49971-23-20: CDD/CDF recoveries ranged from –65 to 92%.

LABELED INTERNAL STANDARDS:

Fifteen labeled internal standards were added to each sample prior to extraction. One labeled internal standard was also added to each sample prior to cleanup. Labeled internal standard recoveries were calculated to measure data quality in terms of accuracy (extraction efficiency).

49971-23: Internal standard recoveries were within the control limits for all analytes in all samples except in EPA 4 COMP, EPA 4 COMP DUP, EPA 4 COMP MS, EPA 29 COMP, and EPA 30 COMP. Between one and nine internal standards fell outside the QC limits in these samples.

Poor recovery in EPA 29 COMP appears to be from interferences in the sample rather than from poor extraction efficiency or loss in cleanup. This sample was diluted 1:3 and re-analyzed to minimize the effect of interferences. Dilution results are reported for this sample; however, interference effects were still seen, and recoveries were not significantly improved with the dilution.

REPLICATES:

Three samples were prepared in duplicate with the SDG. The relative percent difference (RPD) between replicate analyses for CDDs/CDFs was calculated to measure data quality in terms of precision.

49971-23: For analytes >5X the RL the RPDs ranged from:

EPA 4: 1–38%
EPA 20: 2–38%
EPA 28: 0–59%

STANDARD REFERENCE MATERIAL (SRM):

NIST 1944 NY/NJ Sediment SRM was prepared with the SDG. Only reference values are available for CDDs/CDFs. The percent difference (PD) between the measured value and the reference values was calculated to measure data quality in terms of accuracy.

49971-23: SRM PDs were within the control limits with the exception of 1,2,3,7,8,9-HxCDD, which had 55% PD.

PROBLEMS/ CORRECTIVE ACTIONS:

All of the continuing calibrations associated with this set had between three and five natives fail the criteria in Method 1613B. The average daily response factor was used to re-calculate the results for these outliers.

[a] Provost, LP; Elder, RS. (1983) Interpretation of percent recovery data. American Laboratory December, pp. 57–62.

APPENDIX E

PCB DATA

The following codes are used in this report:

Codes	Definition
C	indicates that the congener co-elutes, the congener that it co-elutes with is indicated by the number following C
J	reported value < reporting limit
RL	the low calibration level adjusted for sample final volume and weight
U	not detected
&	outside QC limits

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NOTES	QC				
CLIENT_ ID	PROCEDURAL BLANK	Penn Nursery, PA	McNay Farm, IA	Lake Scott, KS	Lake Scott, KS
LAB_SAMP_ ID	Method Blank	EPA-1 COMP	EPA 7 COMP	EPA 8 COMP	EPA 8 COMP DUP
SAMPLE_WGT_VOL	17.3100	15.6769	16.0618	15.5239	15.8020
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT		78.27	81.32	78.17	78.17
COLLECTION_DATE		8/17/2003	9/2/2003	8/19/2003	8/19/2003
RECEIPT_DATE		8/19/2003	9/4/2003	8/23/2003	8/23/2003
COMPOSITE_DATE		9/10/2003	9/10/2003	9/4/2003	9/4/2003
EXTRACT_DATE	9/15/2003	9/15/2003	9/15/2003	9/15/2003	9/15/2003
ANALYSIS_DATE	10/1/2003	10/1/2003	10/1/2003	10/2/2003	10/2/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-20	49971-13-02	49971-13-03	49971-13-04	49971-13-15
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT
REPORTING LIMIT (RL)	0.29	0.32	0.31	0.32	0.32

PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-1	1.02		2.25		1.16		7.79		2.18	
PCB-2		U	1.02		0.55		4.85		0.80	
PCB-3	0.38		2.60		1.16		5.77		1.81	
PCB-4	2.38			U	1.96		24.41			U
PCB-10		U		U		U	1.45			U
PCB-9		U		U		U	4.61			U
PCB-7		U		U		U	2.15			U
PCB-6		U	1.16		1.09		15.44			U
PCB-5		U		U		U	1.69			U
PCB-8	0.92		4.65		2.97		18.02			U
PCB-19	0.68		0.41		0.63		14.30			U
PCB-14		U		U		U	0.38			U
PCB-30		C18		C18		C18		C18		C18
PCB-18	0.72	C	3.40	C	2.94	C	34.38	C	3.72	C
PCB-11		U	5.07		1.62		9.16			U
PCB-17	1.06		1.66		1.48		18.49		2.09	
PCB-13		C12		C12		C12		C12		C12
PCB-27		C16		C16		C16		C16		C16
PCB-12	0.60	C		CU		CU	13.20	C		CU
PCB-24		C16		C16		C16		C16		C16
PCB-16	1.05	C	1.74	C	1.41	C	16.98	C	2.07	C
PCB-15	0.78		4.26		1.46		7.19			U
PCB-54		U		U		U	3.14			U
PCB-32	0.34		1.11		0.92		5.80		1.22	
PCB-34		U		U		U	1.18			U
PCB-23		U		U		U		U		U
PCB-26	0.54	C	1.10	C	0.78	C	16.50	C	0.94	C
PCB-29		C26		C26		C26		C26		C26
PCB-25	0.08	J	0.39		0.34		5.92			U
PCB-50		CU	0.74	C	0.62	C	16.78	C	0.58	C
PCB-53		C50		C50		C50		C50		C50
PCB-31	1.13		6.10		3.27		18.24		4.50	
PCB-28		C20		C20		C20		C20		C20
PCB-20	2.48	C	9.72	C	5.40	C	38.28	C	8.80	C
PCB-45		CU	0.86	C	0.86	C	17.22	C	0.82	C
PCB-21		C20		C20		C20		C20		C20
PCB-51		C45		C45		C45		C45		C45
PCB-33		C20		C20		C20		C20		C20
PCB-46		U		U		U	4.49			U
PCB-22	0.63		2.65		1.45		8.13		2.16	
PCB-52		C43		C43		C43		C43		C43
PCB-73		C43		C43		C43		C43		C43
PCB-43	3.21	C	17.07	C	6.96	C	40.77	C	9.36	C
PCB-36		U		U		U		U		U
PCB-69		C49		C49		C49		C49		C49
PCB-49	1.16	C	7.08	C	2.58	C	18.42	C	2.66	C
PCB-39		U		U		U		U		U
PCB-48		U		U	0.59		11.99		0.72	
PCB-104		U		U		U		U		U
PCB-65		C44		C44		C44		C44		C44
PCB-47		C44		C44		C44		C44		C44
PCB-44	3.30	C	9.06	C	4.35	C	25.89	C	5.97	C
PCB-62		C59		C59		C59		C59		C59
PCB-38		U		U		U		U		U
PCB-75		C59		C59		C59		C59		C59
PCB-59		CU	0.60	C	0.30	CJ	3.96	C	0.27	CJ
PCB-96		U		U		U	7.38			U
PCB-42	0.37		1.29		0.65		6.69		0.93	
PCB-35		U		U		U	7.76			U
PCB-41		C40		C40		C40		C40		C40
PCB-71		C40		C40		C40		C40		C40
PCB-40	1.17	C	2.37	C	1.71	C	16.29	C	2.49	C
PCB-37	1.00		3.96		1.51		8.98		1.42	
PCB-64	1.05		2.97		1.49		6.45		2.17	
PCB-72		U		U		U		U		U
PCB-103		U		U		U	1.29			U
PCB-68		U		U		U		U		U

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NOTES	QC									
CLIENT_ ID	PROCEDURAL BLANK		Penn Nursery, PA		McNay Farm, IA		Lake Scott, KS		Lake Scott, KS	
LAB_SAMP_ ID	Method Blank		EPA-1 COMP		EPA 7 COMP		EPA 8 COMP		EPA 8 COMP DUP	
SAMPLE_WGT_VOL	17.3100		15.6769		16.0618		15.5239		15.8020	
SAMP_WGT_VOL_UNIT	G DRYWT		G DRYWT		G DRYWT		G DRYWT		G DRYWT	
PCT_DRY_WT			78.27		81.32		78.17		78.17	
COLLECTION_DATE			8/17/2003		9/2/2003		8/19/2003		8/19/2003	
RECEIPT_DATE			8/19/2003		9/4/2003		8/23/2003		8/23/2003	
COMPOSITE_DATE			9/10/2003		9/10/2003		9/4/2003		9/4/2003	
EXTRACT_DATE	9/15/2003		9/15/2003		9/15/2003		9/15/2003		9/15/2003	
ANALYSIS_DATE	10/1/2003		10/1/2003		10/1/2003		10/2/2003		10/2/2003	
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-20		49971-13-02		49971-13-03		49971-13-04		49971-13-15	
REPORTING UNIT	PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT	
REPORTING LIMIT (RL)	0.29		0.32		0.31		0.32		0.32	
PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-94		U		U		U	2.45			U
PCB-57	0.23	J	0.75			U		U	0.82	
PCB-95	6.20		24.25		8.09		76.11		14.65	
PCB-58		U		U		U	3.94			U
PCB-100		C93		C93		C93		C93		C93
PCB-93		CU		CU		CU	15.04	C		CU
PCB-67		U		U		U		U		U
PCB-102		C93		C93		C93		C93		C93
PCB-98		C93		C93		C93		C93		C93
PCB-63		U	0.30	J		U		U		U
PCB-88	0.88	C	3.78	C	1.32	C	17.56	C	2.24	C
PCB-61	7.60	C	18.44	C	7.04	C	45.56	C	10.36	C
PCB-70		C61		C61		C61		C61		C61
PCB-76		C61		C61		C61		C61		C61
PCB-91		C88		C88		C88		C88		C88
PCB-74		C61		C61		C61		C61		C61
PCB-84	2.64		6.65		2.27		20.37		5.34	
PCB-66	3.94		9.68		3.16		19.58		3.70	
PCB-55		U		U		U		U		U
PCB-89		U		U		U	3.52			U
PCB-121		U		U		U		U		U
PCB-56	1.72		3.19		1.30		11.48		2.01	
PCB-60	1.01		2.07		0.84		4.10		1.08	
PCB-92	1.75		7.84		2.26		17.66		3.22	
PCB-80		U		U		U		U		U
PCB-155		U		U		U		U		U
PCB-113		C90		C90		C90		C90		C90
PCB-90	10.11	C	46.89	C	11.76	C	100.77	C	17.19	C
PCB-101		C90		C90		C90		C90		C90
PCB-152		U		U		U	0.95			U
PCB-150		U		U		U	0.73			U
PCB-83	4.71	C	28.62	C	7.50	C	37.98	C	7.35	C
PCB-99		C83		C83		C83		C83		C83
PCB-136	1.41		6.06		1.74		23.70		2.79	
PCB-112		C83		C83		C83		C83		C83
PCB-145		U		U		U		U		U
PCB-109		C86		C86		C86		C86		C86
PCB-119		C86		C86		C86		C86		C86
PCB-79		U	1.04			U	2.21			U
PCB-97		C86		C86		C86		C86		C86
PCB-86	7.98	C	23.46	C	6.66	C	62.46	C	11.94	C
PCB-125		C86		C86		C86		C86		C86
PCB-87		C86		C86		C86		C86		C86
PCB-78		U		U		U		U		U
PCB-117		C85		C85		C85		C85		C85
PCB-116		C85		C85		C85		C85		C85
PCB-85	1.59	C	10.11	C	2.91	C	14.04	C	2.88	C
PCB-110	11.58	C	33.42	C	10.94	C	88.40	C	17.72	C
PCB-115		C110		C110		C110		C110		C110
PCB-81		U		U		U		U		U
PCB-148		U		U		U		U		U
PCB-82	1.45		3.33		0.99		10.46		2.19	
PCB-111		U		U		U		U		U
PCB-77		U	3.28		1.25		3.53		0.46	
PCB-151		C135		C135		C135		C135		C135
PCB-135	3.30	C	26.52	C	6.87	C	50.52	C	6.15	C
PCB-154		C135		C135		C135		C135		C135
PCB-120		U		U		U		U		U
PCB-144	0.45		2.72		0.53		7.44		0.83	
PCB-147	5.74	C	45.64	C	9.34	C	89.14	C	12.30	C
PCB-149		C147		C147		C147		C147		C147
PCB-134	0.58	C		CU	0.40	C	6.64	C	1.00	C
PCB-143		C134		C134		C134		C134		C134
PCB-124		C108		C108		C108		C108		C108
PCB-108	0.36	C	2.32	C	0.68	C	3.78	C	0.58	C
PCB-139		CU	1.36	C		CU	1.70	C		CU
PCB-140		C139		C139		C139		C139		C139
PCB-107		C106		C106		C106		C106		C106
PCB-123		C106		C106		C106		C106		C106
PCB-131		U		U		U		U		U
PCB-106		CU	4.42	C	2.17	C	7.53	C	1.21	C
PCB-142		U		U		U		U	0.34	

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NOTES	QC				
CLIENT_ ID	PROCEDURAL BLANK	Penn Nursery, PA	McNay Farm, IA	Lake Scott, KS	Lake Scott, KS
LAB_SAMP_ ID	Method Blank	EPA-1 COMP	EPA 7 COMP	EPA 8 COMP	EPA 8 COMP DUP
SAMPLE_WGT_VOL	17.3100	15.6769	16.0618	15.5239	15.8020
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT		78.27	81.32	78.17	78.17
COLLECTION_DATE		8/17/2003	9/2/2003	8/19/2003	8/19/2003
RECEIPT_DATE		8/19/2003	9/4/2003	8/23/2003	8/23/2003
COMPOSITE_DATE		9/10/2003	9/10/2003	9/4/2003	9/4/2003
EXTRACT_DATE	9/15/2003	9/15/2003	9/15/2003	9/15/2003	9/15/2003
ANALYSIS_DATE	10/1/2003	10/1/2003	10/1/2003	10/2/2003	10/2/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-20	49971-13-02	49971-13-03	49971-13-04	49971-13-15
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT
REPORTING LIMIT (RL)	0.29	0.32	0.31	0.32	0.32

PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-118	9.00		31.73		12.12		71.33		11.47	
PCB-132	2.90		13.62		2.93		29.99		5.55	
PCB-122		U		U		U		U		U
PCB-188		U		U		U		U		U
PCB-114		U		U		U		U		U
PCB-133		U		U		U	1.47			U
PCB-179	0.49		10.24		2.21		12.76		1.84	
PCB-165		U		U		U		U		U
PCB-146	0.90	C	16.92	C	3.82	C	5.28	C	2.24	C
PCB-105	3.28		12.44		6.28		20.66		4.57	
PCB-184		U		U		U		U		U
PCB-161		C146		C146		C146		C146		C146
PCB-176	0.16	J	2.07		0.42		3.81		0.44	
PCB-153	6.02	C	98.42	C	25.08	C	90.32	C	16.74	C
PCB-168		C153		C153		C153		C153		C153
PCB-141	1.13		10.54		2.06		18.88		2.54	
PCB-186		U		U		U		U		U
PCB-130	0.55		4.64		1.27		4.92		1.26	
PCB-127		U		U		U		U		U
PCB-137	0.76	C	9.00	C	2.14	C	9.56	C	1.90	C
PCB-164		C137		C137		C137		C137		C137
PCB-163		C129		C129		C129		C129		C129
PCB-138		C129		C129		C129		C129		C129
PCB-129	6.60	C	109.68	C	25.32	C	87.64	C	19.20	C
PCB-160		C129		C129		C129		C129		C129
PCB-158	0.71		9.26		1.80		8.29		1.53	
PCB-178	0.24	J	10.79		2.50		6.60		1.50	
PCB-175		U	1.56			U		U		U
PCB-126		U		U		U		U		U
PCB-166		C128		C128		C128		C128		C128
PCB-128	1.22	C	18.04	C	3.76	C	7.60	C	3.22	C
PCB-187	1.46		62.64		11.61		34.82		7.24	
PCB-182		U	0.69			U		U		U
PCB-183		C174		C174		C174		C174		C174
PCB-185		C174		C174		C174		C174		C174
PCB-159		U	1.15			U		U		U
PCB-174	1.47	C	48.09	C	9.27	C	46.32	C	7.20	C
PCB-162		U		U		U	0.37			U
PCB-177	0.39		16.40		3.75		12.46		2.65	
PCB-202	0.19	J	16.73		1.96		3.01		1.08	
PCB-167	0.35		6.45		1.35		3.09		0.97	
PCB-181		U		U		U		U		U
PCB-171	0.26	CJ	8.52	C	1.40	C	6.88	C	1.24	C
PCB-173		C171		C171		C171		C171		C171
PCB-201		U	6.61		0.72		2.44			U
PCB-156	0.72	C	8.38	C	2.88	C	7.20	C	1.68	C
PCB-157		C156		C156		C156		C156		C156
PCB-204		U		U		U		U		U
PCB-197	0.10	CJ	6.26	C	0.84	C	2.64	C		CU
PCB-200		C197		C197		C197		C197		C197
PCB-172		U	8.16		1.48		4.53		0.90	
PCB-192		U		U		U		U		U
PCB-193		C180		C180		C180		C180		C180
PCB-180	1.40	C	71.82	C	14.20	C	57.54	C	10.18	C
PCB-191		U		U		U	1.11			U
PCB-170	0.57		27.12		6.84		19.57		4.23	
PCB-190	0.21	J	6.10		1.90		4.65		0.74	
PCB-169	0.32		0.42		0.76			U		U
PCB-198	0.58	C	66.76	C	10.10	C	14.08	C	4.36	C
PCB-199		C198		C198		C198		C198		C198
PCB-196	0.21	J	17.70		2.87		7.02		1.69	
PCB-203	0.49		35.37		5.71		10.06		3.05	
PCB-208	0.23	J	20.84		4.03		2.10		1.11	
PCB-195	0.14	J	9.51		2.10		4.54		1.31	
PCB-189	0.40		1.71		0.50		0.84			U
PCB-207		U	9.39		1.74		1.02			U
PCB-194	0.52		32.18		6.59		13.81		3.09	
PCB-205	0.77		1.73		0.47		0.74			U
PCB-206	1.31		51.71		9.90		6.76		2.94	
PCB-209	1.71		44.74		11.96		4.59		3.73	

J = reported value < Reporting Limit (RL).
U = not detected.
RL = the low calibration level adjusted for sample final volume and weight.
& = outside QC limits.

WA 5-11 Batch 1
BATTELLE
SDG 49971-13
MOD 1668M

NOTES										
CLIENT_ ID	Bennington, VT		Caldwell, OH		Dixon Springs, IL		Quincy, FL		Bay St. Louis, MS	
LAB_SAMP_ ID	EPA 11 COMP		EPA 14 COMP		EPA 16 COMP		EPA 17 COMP		EPA 18 COMP	
SAMPLE_WGT_VOL	15.9883		17.0640		17.6250		14.5225		16.6717	
SAMP_WGT_VOL_UNIT	G DRYWT		G DRYWT		G DRYWT		G DRYWT		G DRYWT	
PCT_DRY_WT	79.62		83.86		86.84		72.84		83.08	
COLLECTION_DATE	8/28/2003		8/21/2003		8/16/2003		8/17/2003		8/19/2003	
RECEIPT_DATE	8/29/2003		8/22/2003		8/18/2003		8/19/2003		8/21/2003	
COMPOSITE_DATE	9/4/2003		9/4/2003		9/4/2003		9/10/2003		9/4/2003	
EXTRACT_DATE	9/15/2003		9/15/2003		9/15/2003		9/15/2003		9/15/2003	
ANALYSIS_DATE	10/2/2003		10/2/2003		10/2/2003		10/2/2003		10/2/2003	
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-05		49971-13-06		49971-13-07		49971-13-08		49971-13-09	
REPORTING UNIT	PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT	
REPORTING LIMIT (RL)	0.31		0.29		0.28		0.34		0.30	
PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-1	3.37		1.26		0.99		2.43		2.95	
PCB-2	1.49		0.51		0.39		0.92		0.87	
PCB-3	3.22		1.37		1.03		1.70		3.37	
PCB-4	6.76		1.74		1.30			U	3.36	
PCB-10		U		U		U		U		U
PCB-9	1.23			U		U		U		U
PCB-7	0.83			U		U		U		U
PCB-6	4.19		1.14		0.83		1.21		1.77	
PCB-5		U		U		U		U		U
PCB-8	11.34		3.40		2.82		4.77		7.41	
PCB-19	2.36		0.55		0.39		0.50		1.21	
PCB-14		U		U		U		U		U
PCB-30		C18		C18		C18		C18		C18
PCB-18	11.26	C	3.30	C	3.02	C	3.32	C	14.46	C
PCB-11	3.04		1.99		1.83		1.78		2.07	
PCB-17	5.86		1.70		1.47		1.70		5.44	
PCB-13		C12		C12		C12		C12		C12
PCB-27		C16		C16		C16		C16		C16
PCB-12	3.34	C	0.76	C	0.38	C		CU	0.94	C
PCB-24		C16		C16		C16		C16		C16
PCB-16	5.73	C	1.83	C	1.59	C	1.95	C	5.01	C
PCB-15	6.49		2.08		1.67		1.96		6.17	
PCB-54	0.44			U		U		U		U
PCB-32	2.78		1.06		1.06		1.24		3.97	
PCB-34		U		U		U		U		U
PCB-23		U		U		U		U		U
PCB-26	4.38	C	1.22	C	0.76	C	1.00	C	3.76	C
PCB-29		C26		C26		C26		C26		C26
PCB-25	1.73		0.46		0.33		0.42		1.21	
PCB-50	3.56	C	0.96	C	0.70	C	0.58	C	5.76	C
PCB-53		C50		C50		C50		C50		C50
PCB-31	12.39		4.91		4.63		4.89		42.67	
PCB-28		C20		C20		C20		C20		C20
PCB-20	22.96	C	9.68	C	7.52	C	8.48	C	45.04	C
PCB-45	4.34	C	1.12	C	0.94	C	0.64	C	6.58	C
PCB-21		C20		C20		C20		C20		C20
PCB-51		C45		C45		C45		C45		C45
PCB-33		C20		C20		C20		C20		C20
PCB-46		U		U		U	0.24	J	1.85	
PCB-22	5.25		2.80		1.91		2.01		12.13	
PCB-52		C43		C43		C43		C43		C43
PCB-73		C43		C43		C43		C43		C43
PCB-43	21.33	C	14.97	C	12.39	C	10.77	C	119.91	C
PCB-36		U		U		U	0.11	J	1.69	
PCB-69		C49		C49		C49		C49		C49
PCB-49	8.38	C	4.32	C	3.72	C	2.80	C	47.64	C
PCB-39		U		U		U		U		U
PCB-48	3.09		1.09		0.74		0.71		8.15	
PCB-104		U		U		U		U		U
PCB-65		C44		C44		C44		C44		C44
PCB-47		C44		C44		C44		C44		C44
PCB-44	11.76	C	9.48	C	6.57	C	5.73	C	64.14	C
PCB-62		C59		C59		C59		C59		C59
PCB-38		U		U		U		U		U
PCB-75		C59		C59		C59		C59		C59
PCB-59	1.26	C	0.75	C	0.48	C	0.30	CJ	3.93	C
PCB-96	1.21		0.26	J	0.16	J		U	1.21	
PCB-42	2.39		1.73		1.06		0.86		11.03	
PCB-35	1.72		0.42		0.12	J		U		U
PCB-41		C40		C40		C40		C40		C40
PCB-71		C40		C40		C40		C40		C40
PCB-40	5.73	C	5.34	C	2.37	C	2.16	C	25.89	C
PCB-37	5.89		5.14		1.84		1.12		18.13	
PCB-64	4.31		4.31		2.55		1.93		39.13	
PCB-72		U		U		U		U		U
PCB-103		U		U		U		U		U
PCB-68		U		U		U		U		U

WA 5-11 Batch 1
BATTELLE
SDG 49971-13
MOD 1668M

NOTES					
CLIENT_ ID	Bennington, VT	Caldwell, OH	Dixon Springs, IL	Quincy, FL	Bay St. Louis, MS
LAB_SAMP_ ID	EPA 11 COMP	EPA 14 COMP	EPA 16 COMP	EPA 17 COMP	EPA 18 COMP
SAMPLE_WGT_VOL	15.9883	17.0640	17.6250	14.5225	16.6717
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	79.62	83.86	86.84	72.84	83.08
COLLECTION_DATE	8/28/2003	8/21/2003	8/16/2003	8/17/2003	8/19/2003
RECEIPT_DATE	8/29/2003	8/22/2003	8/18/2003	8/19/2003	8/21/2003
COMPOSITE_DATE	9/4/2003	9/4/2003	9/4/2003	9/10/2003	9/4/2003
EXTRACT_DATE	9/15/2003	9/15/2003	9/15/2003	9/15/2003	9/15/2003
ANALYSIS_DATE	10/2/2003	10/2/2003	10/2/2003	10/2/2003	10/2/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-05	49971-13-06	49971-13-07	49971-13-08	49971-13-09
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT
REPORTING LIMIT (RL)	0.31	0.29	0.28	0.34	0.30

PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-94		U		U		U		U		U
PCB-57	1.12			U		U	0.46		5.75	
PCB-95	31.60		38.98		16.21		11.30		172.19	
PCB-58	0.94			U		U		U		U
PCB-100		C93		C93		C93		C93		C93
PCB-93	2.16	C		CU		CU	0.28	CJ	4.32	C
PCB-67		U		U	0.17	J		U		U
PCB-102		C93		C93		C93		C93		C93
PCB-98		C93		C93		C93		C93		C93
PCB-63		U		U	0.21	J	0.18	J	5.08	
PCB-88	5.52	C	5.88	C	2.30	C	1.60	C	24.08	C
PCB-61	21.56	C	40.56	C	13.00	C	9.64	C	235.20	C
PCB-70		C61		C61		C61		C61		C61
PCB-76		C61		C61		C61		C61		C61
PCB-91		C88		C88		C88		C88		C88
PCB-74		C61		C61		C61		C61		C61
PCB-84	7.87		15.40		4.99		3.70		49.09	
PCB-66	8.12		18.96		5.43		3.89		140.96	
PCB-55		U		U		U		U		U
PCB-89		U		U		U		U		U
PCB-121		U		U		U		U		U
PCB-56	4.08		9.38		2.41		1.58		42.98	
PCB-60	2.18		5.09		1.44		1.12		33.42	
PCB-92	7.61		11.96		3.83		1.89		37.22	
PCB-80		U		U		U		U		U
PCB-155		U		U		U		U		U
PCB-113		C90		C90		C90		C90		C90
PCB-90	42.09	C	74.73	C	21.09	C	11.13	C	209.67	C
PCB-101		C90		C90		C90		C90		C90
PCB-152		U		U		U		U		U
PCB-150		U		U		U		U		U
PCB-83	21.06	C	30.60	C	11.10	C	4.44	C	118.44	C
PCB-99		C83		C83		C83		C83		C83
PCB-136	18.19		9.10		3.06		1.45		29.22	
PCB-112		C83		C83		C83		C83		C83
PCB-145		U		U		U		U		U
PCB-109		C86		C86		C86		C86		C86
PCB-119		C86		C86		C86		C86		C86
PCB-79	1.27		2.08		0.48			U	5.30	
PCB-97		C86		C86		C86		C86		C86
PCB-86	21.54	C	58.14	C	12.96	C	7.32	C	156.00	C
PCB-125		C86		C86		C86		C86		C86
PCB-87		C86		C86		C86		C86		C86
PCB-78		U		U		U		U		U
PCB-117		C85		C85		C85		C85		C85
PCB-116		C85		C85		C85		C85		C85
PCB-85	11.64	C	12.75	C	5.46	C	1.56	C	79.95	C
PCB-110	42.82	C	89.52	C	20.04	C	10.96	C	236.70	C
PCB-115		C110		C110		C110		C110		C110
PCB-81		U	1.43			U	0.26	J		U
PCB-148		U		U		U		U		U
PCB-82	3.27		10.20		1.99		1.28		25.21	
PCB-111		U		U		U		U		U
PCB-77	2.79		2.24		0.82		0.34	J	25.05	
PCB-151		C135		C135		C135		C135		C135
PCB-135	70.26	C	23.88	C	9.21	C	3.12	C	74.43	C
PCB-154		C135		C135		C135		C135		C135
PCB-120	0.46			U		U		U		U
PCB-144	6.93		3.38		1.04		0.43		11.16	
PCB-147	103.14	C	43.96	C	13.68	C	4.86	C	124.76	C
PCB-149		C147		C147		C147		C147		C147
PCB-134	2.94	C	3.58	C	0.82	C	0.40	C	8.80	C
PCB-143		C134		C134		C134		C134		C134
PCB-124		C108		C108		C108		C108		C108
PCB-108	3.56	C	3.22	C	1.26	C	0.36	C	12.82	C
PCB-139		CU	1.20	C		CU		CU		CU
PCB-140		C139		C139		C139		C139		C139
PCB-107		C106		C106		C106		C106		C106
PCB-123		C106		C106		C106		C106		C106
PCB-131		U		U		U		U		U
PCB-106	9.03	C	6.21	C	3.11	C	0.94	C	38.38	C
PCB-142		U		U		U		U		U

WA 5-11 Batch 1
BATTELLE
SDG 49971-13
MOD 1668M

NOTES					
CLIENT_ ID	Bennington, VT	Caldwell, OH	Dixon Springs, IL	Quincy, FL	Bay St. Louis, MS
LAB_SAMP_ ID	EPA 11 COMP	EPA 14 COMP	EPA 16 COMP	EPA 17 COMP	EPA 18 COMP
SAMPLE_WGT_VOL	15.9883	17.0640	17.6250	14.5225	16.6717
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	79.62	83.86	86.84	72.84	83.08
COLLECTION_DATE	8/28/2003	8/21/2003	8/16/2003	8/17/2003	8/19/2003
RECEIPT_DATE	8/29/2003	8/22/2003	8/18/2003	8/19/2003	8/21/2003
COMPOSITE_DATE	9/4/2003	9/4/2003	9/4/2003	9/10/2003	9/4/2003
EXTRACT_DATE	9/15/2003	9/15/2003	9/15/2003	9/15/2003	9/15/2003
ANALYSIS_DATE	10/2/2003	10/2/2003	10/2/2003	10/2/2003	10/2/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-05	49971-13-06	49971-13-07	49971-13-08	49971-13-09
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT
REPORTING LIMIT (RL)	0.31	0.29	0.28	0.34	0.30

PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-118	37.97		68.14		17.38		8.34		347.87	
PCB-132	24.08		19.75		4.71		2.41		64.97	
PCB-122		U		U		U		U		U
PCB-188		U		U		U		U		U
PCB-114		U	1.46			U	0.19	J	7.35	
PCB-133	3.03		0.98		0.72			U		U
PCB-179	54.08		5.06		3.24		0.87		21.91	
PCB-165		U		U		U		U		U
PCB-146	28.04	C		CU	5.96	C	1.40	C	39.16	C
PCB-105	24.27		21.75		8.17		3.86		232.74	
PCB-184		U		U		U		U		U
PCB-161		C146		C146		C146		C146		C146
PCB-176	13.26		1.52		0.54			U	5.29	
PCB-153	214.40	C	49.70	C	40.02	C	9.48	C	290.46	C
PCB-168		C153		C153		C153		C153		C153
PCB-141	30.32		9.62		2.92		0.87		33.47	
PCB-186		U		U		U		U		U
PCB-130	7.57		3.47		1.57		0.50		16.64	
PCB-127		U		U		U		U		U
PCB-137	16.36	C	6.48	C	3.00	C	0.94	C	28.68	C
PCB-164		C137		C137		C137		C137		C137
PCB-163		C129		C129		C129		C129		C129
PCB-138		C129		C129		C129		C129		C129
PCB-129	198.80	C	53.88	C	41.80	C	12.88	C	391.36	C
PCB-160		C129		C129		C129		C129		C129
PCB-158	12.19		5.52		2.69		0.88		33.07	
PCB-178	34.96		3.06		3.98		1.19		19.01	
PCB-175	4.21		0.56		0.19	J	0.11	J	1.76	
PCB-126	1.80			U	0.41			U	1.28	
PCB-166		C128		C128		C128		C128		C128
PCB-128	25.78	C	7.44	C	6.14	C	2.40	C	81.54	C
PCB-187	195.24		17.21		17.03		4.91		115.92	
PCB-182		U		U		U		U	0.38	
PCB-183		C174		C174		C174		C174		C174
PCB-185		C174		C174		C174		C174		C174
PCB-159	5.45			U		U		U		U
PCB-174	226.17	C	19.65	C	10.38	C	3.24	C	87.42	C
PCB-162	0.95			U	0.25	J		U	1.51	
PCB-177	68.81		4.43		4.92		1.80		34.79	
PCB-202	27.02		2.22		6.46		2.16		13.72	
PCB-167	8.38		1.92		2.31		0.80		20.47	
PCB-181	U	U		U	U	U				
PCB-171	26.06	C	3.08	C	1.76	C	0.60	C	18.32	C
PCB-173		C171		C171		C171		C171		C171
PCB-201	14.61		1.30		1.25		0.31	J	4.19	
PCB-156	17.36	C	4.72	C	4.58	C	1.78	C	57.78	C
PCB-157		C156		C156		C156		C156		C156
PCB-204	U	U		U	U	U				
PCB-197	18.68	C	1.56	C	1.26	C	0.42	C	6.38	C
PCB-200		C197		C197		C197		C197		C197
PCB-172	20.92		1.95		1.58		0.52		12.87	
PCB-192	U	U		U	U	U				
PCB-193		C180		C180		C180		C180		C180
PCB-180	290.80	C	23.20	C	14.98	C	6.32	C	140.90	C
PCB-191	3.47		0.39		0.30			U	2.45	
PCB-170	100.02		9.16		7.61		3.62		79.68	
PCB-190	25.23		2.11		2.51		0.97		20.15	
PCB-169	1.42			U	0.29		0.22	J	1.39	
PCB-198	143.44	C	11.16	C	13.90	C	5.74	C	66.64	C
PCB-199		C198		C198		C198		C198		C198
PCB-196	55.74		4.40		2.74		0.91		15.87	
PCB-203	88.03		6.93		8.40		3.41		39.37	
PCB-208	17.27		1.63		20.87		3.62		11.41	
PCB-195	46.01		3.55		2.91		1.29		19.81	
PCB-189	3.61		0.26	J	0.52		0.28	J	3.92	
PCB-207	6.97		0.73		8.05		1.25		2.76	
PCB-194	124.96		8.73		6.85		3.75		47.35	
PCB-205	6.14		0.54		0.66		0.50		4.07	
PCB-206	64.32		5.41		23.17		7.76		39.63	
PCB-209	29.44		2.92		305.80		38.27		12.46	

J = reported value < Reporting Limit (RL).
U = not detected.
RL = the low calibration level adjusted for s
& = outside QC limits.

WA 5-11 Batch 1
BATTELLE
SDG 49971-13
MOD 1668M

NOTES										
CLIENT_ ID	Padre Island, TX		North Platte, NE		Theodore Roosevelt, ND		Theodore Roosevelt, ND		Chiricahua, AZ	
LAB_SAMP_ ID	EPA 19 COMP		EPA 21 COMP		EPA 25 COMP		EPA 25 COMP DUP		EPA 27 COMP	
SAMPLE_WGT_VOL	20.2499		18.5140		18.6793		18.9976		19.5077	
SAMP_WGT_VOL_UNIT	G DRYWT		G DRYWT		G DRYWT		G DRYWT		G DRYWT	
PCT_DRY_WT	99.15		91.61		93.59		93.59		96.20	
COLLECTION_DATE	8/19/2003		8/13/2003		8/12/2003		8/12/2003		8/18/2003	
RECEIPT_DATE	8/20/2003		8/14/2003		8/19/2003		8/19/2003		8/20/2003	
COMPOSITE_DATE	9/4/2003		9/4/2003		9/4/2003		9/4/2003		9/4/2003	
EXTRACT_DATE	9/15/2003		9/15/2003		9/15/2003		9/15/2003		9/15/2003	
ANALYSIS_DATE	10/2/2003		10/2/2003		10/2/2003		10/2/2003		10/2/2003	
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-10		49971-13-11		49971-13-12		49971-13-16		49971-13-13	
REPORTING UNIT	PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT	
REPORTING LIMIT (RL)	0.25		0.27		0.27		0.26		0.26	
PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-1	1.03		1.39		1.09		0.93		0.97	
PCB-2	0.40		0.84		0.62		0.59		0.45	
PCB-3	1.07		1.72		1.10		0.99		1.28	
PCB-4	1.68		1.63		1.62			U	1.28	
PCB-10		U		U		U		U		U
PCB-9		U		U		U		U		U
PCB-7		U		U		U		U		U
PCB-6	1.05			U	0.79			U	0.70	
PCB-5		U		U		U		U		U
PCB-8	3.10		3.10		2.71			U	3.04	
PCB-19	0.43		0.49		0.44		0.48		0.37	
PCB-14		U		U		U		U		U
PCB-30		C18		C18		C18		C18		C18
PCB-18	2.66	C	2.88	C	2.66	C	2.82	C	2.52	C
PCB-11	1.12		1.92		1.45			U	1.47	
PCB-17	1.45		1.63		1.44		1.55		1.19	
PCB-13		C12		C12		C12		C12		C12
PCB-27		C16		C16		C16		C16		C16
PCB-12		CU		CU		CU		CU		CU
PCB-24		C16		C16		C16		C16		C16
PCB-16	1.29	C	1.50	C	1.38	C	1.26	C	1.38	C
PCB-15	1.62		2.27		1.36			U	1.59	
PCB-54		U	0.12	J		U		U		U
PCB-32	0.87		1.03		0.85		0.95		0.96	
PCB-34		U		U		U		U		U
PCB-23		U		U		U		U		U
PCB-26	0.74	C	0.84	C	0.66	C	0.78	C	0.68	C
PCB-29		C26		C26		C26		C26		C26
PCB-25	0.26		0.33		0.28			U	0.29	
PCB-50	0.66	C	0.70	C	0.58	C	0.70	C	0.48	C
PCB-53		C50		C50		C50		C50		C50
PCB-31	3.30		4.61		3.51		3.66		3.81	
PCB-28		C20		C20		C20		C20		C20
PCB-20	6.00	C	7.76	C	5.88	C	6.32	C	6.64	C
PCB-45	0.74	C	0.94	C	0.74	C	0.78	C	0.74	C
PCB-21		C20		C20		C20		C20		C20
PCB-51		C45		C45		C45		C45		C45
PCB-33		C20		C20		C20		C20		C20
PCB-46		U	0.18	J	0.24	J	0.21	J		U
PCB-22	1.53		2.16		1.70		1.74		1.80	
PCB-52		C43		C43		C43		C43		C43
PCB-73		C43		C43		C43		C43		C43
PCB-43	9.60	C	11.73	C	9.93	C	8.64	C	9.57	C
PCB-36	0.08	J		U		U	0.10	J	0.11	J
PCB-69		C49		C49		C49		C49		C49
PCB-49	2.54	C	3.96	C	2.60	C	2.44	C	2.82	C
PCB-39		U		U	0.09	J		U		U
PCB-48	0.59		0.81		0.70		0.54		0.60	
PCB-104		U	0.08	J		U		U		U
PCB-65		C44		C44		C44		C44		C44
PCB-47		C44		C44		C44		C44		C44
PCB-44	4.92	C	7.11	C	5.22	C	5.31	C	5.70	C
PCB-62		C59		C59		C59		C59		C59
PCB-38		U	0.08	J	0.12	J		U		U
PCB-75		C59		C59		C59		C59		C59
PCB-59	0.18	CJ	0.42	C		CU		CU	0.30	C
PCB-96	0.12	J	0.13	J	0.13	J	0.25	J	0.08	J
PCB-42	0.77		1.01		0.75		0.61		0.74	
PCB-35		U		U	0.09	J		U		U
PCB-41		C40		C40		C40		C40		C40
PCB-71		C40		C40		C40		C40		C40
PCB-40	1.83	C	2.64	C	1.89	C	2.04	C	2.22	C
PCB-37	1.04		2.07		1.54		1.72		1.36	
PCB-64	1.68		2.52		2.15		2.01		1.98	
PCB-72		U		U		U		U		U
PCB-103		U		U		U		U		U
PCB-68		U		U		U		U	0.09	J

WA 5-11 Batch 1
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NOTES										
CLIENT_ ID	Padre Island, TX		North Platte, NE		Theodore Roosevelt, ND		Theodore Roosevelt, ND		Chiricahua, AZ	
LAB_SAMP_ ID	EPA 19 COMP		EPA 21 COMP		EPA 25 COMP		EPA 25 COMP DUP		EPA 27 COMP	
SAMPLE_WGT_VOL	20.2499		18.5140		18.6793		18.9976		19.5077	
SAMP_WGT_VOL_UNIT	G DRYWT		G DRYWT		G DRYWT		G DRYWT		G DRYWT	
PCT_DRY_WT	99.15		91.61		93.59		93.59		96.20	
COLLECTION_DATE	8/19/2003		8/13/2003		8/12/2003		8/12/2003		8/18/2003	
RECEIPT_DATE	8/20/2003		8/14/2003		8/19/2003		8/19/2003		8/20/2003	
COMPOSITE_DATE	9/4/2003		9/4/2003		9/4/2003		9/4/2003		9/4/2003	
EXTRACT_DATE	9/15/2003		9/15/2003		9/15/2003		9/15/2003		9/15/2003	
ANALYSIS_DATE	10/2/2003		10/2/2003		10/2/2003		10/2/2003		10/2/2003	
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-10		49971-13-11		49971-13-12		49971-13-16		49971-13-13	
REPORTING UNIT	PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT	
REPORTING LIMIT (RL)	0.25		0.27		0.27		0.26		0.26	
PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-94		U		U		U		U		U
PCB-57		U	0.59		0.59		0.73		0.56	
PCB-95	13.38		17.55		18.72		19.07		14.43	
PCB-58		U		U		U		U		U
PCB-100		C93		C93		C93		C93		C93
PCB-93		CU		CU		CU	0.44	C		CU
PCB-67		U		U		U		U		U
PCB-102		C93		C93		C93		C93		C93
PCB-98		C93		C93		C93		C93		C93
PCB-63		U		U	0.15	J		U		U
PCB-88	1.80	C	2.54	C	3.08	C	3.22	C	2.62	C
PCB-61	8.44	C	16.12	C	11.84	C	11.32	C	12.32	C
PCB-70		C61		C61		C61		C61		C61
PCB-76		C61		C61		C61		C61		C61
PCB-91		C88		C88		C88		C88		C88
PCB-74		C61		C61		C61		C61		C61
PCB-84	4.17		5.97		5.74		5.85		5.01	
PCB-66	3.14		5.55		4.82		4.68		4.89	
PCB-55		U		U		U	0.16	J		U
PCB-89		U		U		U		U		U
PCB-121		U		U		U		U		U
PCB-56		U	2.92		2.20		2.13		2.37	
PCB-60	0.78		1.36		1.09		0.99		1.22	
PCB-92	2.60		4.42		3.47		3.75		3.50	
PCB-80		U		U		U		U		U
PCB-155		U		U		U		U		U
PCB-113		C90		C90		C90		C90		C90
PCB-90	14.40	C	23.10	C	17.40	C	19.08	C	19.65	C
PCB-101		C90		C90		C90		C90		C90
PCB-152		U		U		U		U		U
PCB-150		U		U		U		U		U
PCB-83	5.82	C	11.70	C	9.24	C	9.39	C	10.29	C
PCB-99		C83		C83		C83		C83		C83
PCB-136	2.38		3.25		5.40		5.78		3.28	
PCB-112		C83		C83		C83		C83		C83
PCB-145		U		U		U		U		U
PCB-109		C86		C86		C86		C86		C86
PCB-119		C86		C86		C86		C86		C86
PCB-79	0.40		0.65		0.58		0.72		0.65	
PCB-97		C86		C86		C86		C86		C86
PCB-86	9.84	C	14.58	C	13.08	C	13.74	C	14.40	C
PCB-125		C86		C86		C86		C86		C86
PCB-87		C86		C86		C86		C86		C86
PCB-78		U		U		U		U		U
PCB-117		C85		C85		C85		C85		C85
PCB-116		C85		C85		C85		C85		C85
PCB-85	2.10	C	4.59	C	3.48	C	3.21	C	4.02	C
PCB-110	14.88	C	23.34	C	30.38	C	31.92	C	24.78	C
PCB-115		C110		C110		C110		C110		C110
PCB-81		U		U		U		U		U
PCB-148		U		U		U		U	0.12	J
PCB-82	1.54		2.50		2.16		2.20		2.65	
PCB-111		U		U		U		U		U
PCB-77	0.50		2.00		1.04		1.04		0.76	
PCB-151		C135		C135		C135		C135		C135
PCB-135	4.89	C	10.11	C	17.79	C	17.88	C	9.15	C
PCB-154		C135		C135		C135		C135		C135
PCB-120		U		U		U		U		U
PCB-144	0.71		1.28		2.16		2.06		1.21	
PCB-147	8.26	C	16.68	C	30.88	C	33.98	C	17.42	C
PCB-149		C147		C147		C147		C147		C147
PCB-134	0.60	C	0.84	C	1.72	C	2.04	C	0.96	C
PCB-143		C134		C134		C134		C134		C134
PCB-124		C108		C108		C108		C108		C108
PCB-108	0.56	C	1.12	C	0.52	C	0.86	C	1.00	C
PCB-139	0.24	CJ		CU	0.52	C	0.48	C		CU
PCB-140		C139		C139		C139		C139		C139
PCB-107		C106		C106		C106		C106		C106
PCB-123		C106		C106		C106		C106		C106
PCB-131		U		U		U		U		U
PCB-106	1.10	C	2.18	C	1.40	C	1.66	C	2.07	C
PCB-142	0.15	J		U		U		U	0.29	

WA 5-11 Batch 1
BATTELLE
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NOTES					
CLIENT_ ID	Padre Island, TX	North Platte, NE	Theodore Roosevelt, ND	Theodore Roosevelt, ND	Chiricahua, AZ
LAB_SAMP_ ID	EPA 19 COMP	EPA 21 COMP	EPA 25 COMP	EPA 25 COMP DUP	EPA 27 COMP
SAMPLE_WGT_VOL	20.2499	18.5140	18.6793	18.9976	19.5077
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	99.15	91.61	93.59	93.59	96.20
COLLECTION_DATE	8/19/2003	8/13/2003	8/12/2003	8/12/2003	8/18/2003
RECEIPT_DATE	8/20/2003	8/14/2003	8/19/2003	8/19/2003	8/20/2003
COMPOSITE_DATE	9/4/2003	9/4/2003	9/4/2003	9/4/2003	9/4/2003
EXTRACT_DATE	9/15/2003	9/15/2003	9/15/2003	9/15/2003	9/15/2003
ANALYSIS_DATE	10/2/2003	10/2/2003	10/2/2003	10/2/2003	10/2/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-10	49971-13-11	49971-13-12	49971-13-16	49971-13-13
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT
REPORTING LIMIT (RL)	0.25	0.27	0.27	0.26	0.26

PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-118	9.57		18.62		14.99		17.09		16.63	
PCB-132	4.21		5.64		11.85		12.86		6.93	
PCB-122		U		U		U		U		U
PCB-188		U		U		U		U	0.33	
PCB-114	0.20	J		U		U	0.19	J		U
PCB-133		U	0.60			U	0.71		0.57	
PCB-179	0.99		2.70		6.42		6.64		3.52	
PCB-165		U		U		U		U		U
PCB-146	1.64	C	4.62	C	5.58	C	5.70	C	4.52	C
PCB-105	4.28		9.08		7.23		7.72		7.13	
PCB-184		U		U		U		U		U
PCB-161		C146		C146		C146		C146		C146
PCB-176	0.32		0.63		1.59		1.88		0.90	
PCB-153	9.44	C	27.86	C	24.78	C	26.44	C	22.02	C
PCB-168		C153		C153		C153		C153		C153
PCB-141	1.58		3.20		5.10		5.26		3.25	
PCB-186		U		U		U		U		U
PCB-130	0.88		1.93		2.73		2.83		1.87	
PCB-127		U		U		U		U		U
PCB-137	1.34	C	2.90	C	4.70	C	5.00	C	2.78	C
PCB-164		C137		C137		C137		C137		C137
PCB-163		C129		C129		C129		C129		C129
PCB-138		C129		C129		C129		C129		C129
PCB-129	13.16	C	30.12	C	37.60	C	40.80	C	25.88	C
PCB-160		C129		C129		C129		C129		C129
PCB-158	1.30		2.11		3.75		4.02		2.37	
PCB-178	0.69		3.06		3.81		3.75		3.65	
PCB-175		U	0.29		0.53		0.53		0.71	
PCB-126		U	0.71			U		U		U
PCB-166		C128		C128		C128		C128		C128
PCB-128	2.40	C	4.70	C	7.24	C	7.64	C	4.70	C
PCB-187	3.04		13.54		21.62		21.20		16.65	
PCB-182		U	0.12	J		U		U		U
PCB-183		C174		C174		C174		C174		C174
PCB-185		C174		C174		C174		C174		C174
PCB-159		U		U	0.59		0.83		0.62	
PCB-174	3.78	C	11.55	C	25.80	C	25.44	C	15.33	C
PCB-162		U		U		U		U		U
PCB-177	1.49		4.80		9.55		9.60		4.97	
PCB-202	0.46		2.45		2.69		2.69		3.82	
PCB-167	0.63		1.64		1.46		1.25		1.62	
PCB-181	U	U		U	U	U				
PCB-171	0.66	C	1.82	C	4.00	C	4.36	C	2.20	C
PCB-173		C171		C171		C171		C171		C171
PCB-201	0.35		1.05		1.74		1.73		2.29	
PCB-156	2.04	C	3.68	C	3.04	C	3.64	C	3.04	C
PCB-157		C156		C156		C156		C156		C156
PCB-204	U	U		U	U	U				
PCB-197	0.30	C	0.98	C	1.82	C	1.72	C	1.96	C
PCB-200		C197		C197		C197		C197		C197
PCB-172	0.50		1.80		2.70		2.85		2.48	
PCB-192	U	U		U	U	U				
PCB-193		C180		C180		C180		C180		C180
PCB-180	5.42	C	15.74	C	29.50	C	30.30	C	19.18	C
PCB-191		U	0.29			U	0.70			U
PCB-170	3.17		7.92		13.98		13.85		7.77	
PCB-190	0.73		2.06		2.83		2.88		1.74	
PCB-169	0.17	J	0.67			U		U	0.22	J
PCB-198	2.66	C	8.92	C	11.36	C	11.58	C	15.64	C
PCB-199		C198		C198		C198		C198		C198
PCB-196	0.85		2.38		4.88		4.43		5.03	
PCB-203	1.63		5.11		7.93		7.99		9.54	
PCB-208	0.79		3.96		1.97		1.95		8.00	
PCB-195	1.00		2.31		4.03		3.50		2.82	
PCB-189	0.37		0.56		0.59		0.62		0.65	
PCB-207	0.47		1.45		0.96		0.80		3.06	
PCB-194	3.02		6.31		10.14		9.07		9.33	
PCB-205	0.68		0.68		0.70			U	0.59	
PCB-206	3.19		8.91		6.78		6.57		19.19	
PCB-209	1.93		11.81		3.88		3.94		20.91	

J = reported value < Reporting Limit (RL).
U = not detected.
RL = the low calibration level adjusted for s
& = outside QC limits.

WA5-11 Batch 2
BATTELLE
SDG 49971-23&28
MOD 1668M

NOTES	QC				
CLIENT_ ID	PROCEDURAL BLANK	Clinton Crops, NC	Everglades, FL	Everglades, FL	Lake Dubay, WI
LAB_SAMP_ ID	Method Blank	EPA-2 COMP	EPA-4 COMP	EPA-4 COMP Duplicate	EPA-5 COMP
SAMPLE_WGT_VOL	16.6259	18.2470	11.0188	11.0058	16.7103
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT		90.91	54.96	54.96	84.78
COLLECTION_DATE		9/6/2003	10/20/2003	10/20/2003	8/12/2003
RECEIPT_DATE		10/21/2003	10/22/2003	10/22/2003	8/18/2003
COMPOSITE_DATE		10/27/2003	10/27/2003	10/27/2003	9/4/2003
EXTRACT_DATE	11/12/2003	11/12/2003	11/12/2003	11/6/2003	11/6/2003
ANALYSIS_DATE	12/14/2003	12/14/2003	12/14/2003	12/18/2003	12/14/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-28-04	49971-28-02	49971-28-03	49971-23-17	49971-23-04
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT
REPORTING LIMIT (RL)	0.30	0.27	0.45	1.82	0.30

PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-1	1.22		6.02		20.49		3.97		406.36	
PCB-2	0.68		0.29		18.40		3.34		119.20	
PCB-3	0.90		0.85		17.20		4.59		119.97	
PCB-4	1.56		14.27		33.38			U	2199.20	
PCB-10		U	2.05		2.46			U	395.02	
PCB-9		U		U	10.99			U	46.02	
PCB-7		U		U	4.81			U		U
PCB-6	1.70		0.88		27.05			U	55.73	
PCB-5		U		U	3.35			U		U
PCB-8	2.54		2.30		28.81		7.93		62.58	
PCB-19	0.85		3.75		17.99		3.90		1394.84	
PCB-14		U		U		U		U		U
PCB-30		C18		C18		C18		C18		C18
PCB-18	2.85	C	2.04	C	49.66	C	10.74	C	55.68	C
PCB-11	1.40		0.87		16.47		7.12		85.66	
PCB-17	1.86		4.31		29.90		5.92		1899.27	
PCB-13		C12		C12		C12		C12		C12
PCB-27		C16		C16		C16		C16		C16
PCB-12	1.27	C	1.84	C	21.16	C		CU	827.58	C
PCB-24		C16		C16		C16		C16		C16
PCB-16	1.49	C	2.79	C	21.78	C	6.36	C	1059.72	C
PCB-15	1.28		2.64		12.29			U	799.53	
PCB-54		U		U	2.81			U	24.17	
PCB-32	0.69		0.89		4.64		3.39		152.56	
PCB-34		U		U		U		U		U
PCB-23		U		U	1.07			U	22.54	
PCB-26	1.56	C	1.08	C	19.32	C	3.66	C	282.28	C
PCB-29		C26		C26		C26		C26		C26
PCB-25	0.53		0.31		7.11		1.35	J	19.71	
PCB-50	1.41	C	0.96	C	23.40	C	5.98	C	204.58	C
PCB-53		C50		C50		C50		C50		C50
PCB-31	2.43		2.69		22.43		9.78		72.08	
PCB-28		C20		C20		C20		C20		C20
PCB-20	5.79	C	5.12	C	45.20	C	20.80	C	38.72	C
PCB-45	1.39	C	1.36	C	25.46	C	6.66	C	390.14	C
PCB-21		C20		C20		C20		C20		C20
PCB-51		C45		C45		C45		C45		C45
PCB-33		C20		C20		C20		C20		C20
PCB-46		U	0.25	J	6.80			U	8.71	
PCB-22	1.26		1.16		7.74		4.52			U
PCB-52		C43		C43		C43		C43		C43
PCB-73		C43		C43		C43		C43		C43
PCB-43	5.97	C	7.77	C	62.79	C	42.30	C	390.27	C
PCB-36		U		U		U		U	20.25	
PCB-69		C49		C49		C49		C49		C49
PCB-49	2.93	C	2.96	C	34.96	C	17.58	C	368.98	C
PCB-39		U		U		U		U		U
PCB-48	1.26		0.60		16.37		6.17			U
PCB-104		U		U		U		U	1.54	
PCB-65		C44		C44		C44		C44		C44
PCB-47		C44		C44		C44		C44		C44
PCB-44	4.25	C	6.27	C	38.55	C	25.20	C	1449.63	C
PCB-62		C59		C59		C59		C59		C59
PCB-38		U		U		U		U		U
PCB-75		C59		C59		C59		C59		C59
PCB-59	0.46	C	0.27	CJ	2.55	C	2.04	C		CU
PCB-96	0.30	J		U	5.74			U	13.39	
PCB-42	1.16		0.96		6.48		6.28			U
PCB-35	0.47		0.15	J	6.35		1.83			U
PCB-41		C40		C40		C40		C40		C40
PCB-71		C40		C40		C40		C40		C40
PCB-40	2.16	C	1.74	C	11.04	C	11.55	C		CU
PCB-37	1.08		1.13		11.23		6.23		17.80	
PCB-64	1.21		1.45		4.94		9.57			U
PCB-72		U	0.09	J		U		U	56.72	
PCB-103		U		U		U		U	30.86	
PCB-68		U		U		U		U	53.91	

WA5-11 Batch 2
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NOTES	QC									
CLIENT_ ID	PROCEDURAL BLANK		Clinton Crops, NC		Everglades, FL		Everglades, FL		Lake Dubay, WI	
LAB_SAMP_ ID	Method Blank		EPA-2 COMP		EPA-4 COMP		EPA-4 COMP Duplicate		EPA-5 COMP	
SAMPLE_WGT_VOL	16.6259		18.2470		11.0188		11.0058		16.7103	
SAMP_WGT_VOL_UNIT	G DRYWT		G DRYWT		G DRYWT		G DRYWT		G DRYWT	
PCT_DRY_WT			90.91		54.96		54.96		84.78	
COLLECTION_DATE			9/6/2003		10/20/2003		10/20/2003		8/12/2003	
RECEIPT_DATE			10/21/2003		10/22/2003		10/22/2003		8/18/2003	
COMPOSITE_DATE			10/27/2003		10/27/2003		10/27/2003		9/4/2003	
EXTRACT_DATE	11/12/2003		11/12/2003		11/12/2003		11/6/2003		11/6/2003	
ANALYSIS_DATE	12/14/2003		12/14/2003		12/14/2003		12/18/2003		12/14/2003	
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-28-04		49971-28-02		49971-28-03		49971-23-17		49971-23-04	
REPORTING UNIT	PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT	
REPORTING LIMIT (RL)	0.30		0.27		0.45		1.82		0.30	
PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-94	0.14	J		U		U		U	24.15	
PCB-57	0.39		0.41			U		U	14.41	
PCB-95	7.15		9.44		67.32		38.16		350.66	
PCB-58		U		U		U		U		U
PCB-100		C93		C93		C93		C93		C93
PCB-93	1.02	C	0.56	C	18.12	C	6.80	C	93.48	C
PCB-67	0.33			U	1.79		1.38	J	4.44	
PCB-102		C93		C93		C93		C93		C93
PCB-98		C93		C93		C93		C93		C93
PCB-63		U		U		U		U	2.45	
PCB-88	1.56	C	1.62	C	17.20	C	8.26	C	195.48	C
PCB-61	6.96	C	7.92	C	55.76	C	35.80	C	45.68	C
PCB-70		C61		C61		C61		C61		C61
PCB-76		C61		C61		C61		C61		C61
PCB-91		C88		C88		C88		C88		C88
PCB-74		C61		C61		C61		C61		C61
PCB-84	2.23		2.92		17.42		11.94		62.55	
PCB-66	3.59		3.31		25.92		16.09		16.92	
PCB-55		U		U		U		U		U
PCB-89	0.28	J		U	2.13			U		U
PCB-121		U		U		U		U		U
PCB-56	1.75		1.72		11.88		10.61		4.43	
PCB-60	0.76		0.90		4.90		4.85		1.92	
PCB-92	1.70		2.15		17.90		13.36		93.47	
PCB-80		U		U		U		U		U
PCB-155		U		U		U		U		U
PCB-113		C90		C90		C90		C90		C90
PCB-90	8.74	C	10.74	C	85.80	C	53.37	C	131.64	C
PCB-101		C90		C90		C90		C90		C90
PCB-152		U		U		U		U		U
PCB-150		U		U		U		U	4.94	
PCB-83	4.39	C	5.28	C	33.12	C	22.62	C	63.60	C
PCB-99		C83		C83		C83		C83		C83
PCB-136	1.31		2.27		16.10		8.33		82.15	
PCB-112		C83		C83		C83		C83		C83
PCB-145		U		U		U		U		U
PCB-109		C86		C86		C86		C86		C86
PCB-119		C86		C86		C86		C86		C86
PCB-79		U		U		U		U	2.51	
PCB-97		C86		C86		C86		C86		C86
PCB-86	5.75	C	6.90	C	42.96	C	31.68	C	42.90	C
PCB-125		C86		C86		C86		C86		C86
PCB-87		C86		C86		C86		C86		C86
PCB-78		U		U		U		U		U
PCB-117		C85		C85		C85		C85		C85
PCB-116		C85		C85		C85		C85		C85
PCB-85	0.99	C	1.95	C	6.36	C	8.01	C	7.38	C
PCB-110	8.20	C	10.18	C	49.40	C	48.38	C	48.76	C
PCB-115		C110		C110		C110		C110		C110
PCB-81		U		U		U		U		U
PCB-148		U		U		U		U	3.73	
PCB-82	1.09		1.16		8.02		4.61		2.69	
PCB-111		U		U		U		U		U
PCB-77	0.50		0.69		4.88		5.38		1.89	
PCB-151		C135		C135		C135		C135		C135
PCB-135	3.34	C	6.60	C	33.84	C	22.89	C	117.18	C
PCB-154		C135		C135		C135		C135		C135
PCB-120		U		U		U		U		U
PCB-144	0.39			U	0.70		3.28			U
PCB-147	7.36	C	14.92	C	94.58	C	69.34	C	332.14	C
PCB-149		C147		C147		C147		C147		C147
PCB-134	0.61	C	0.62	C		CU	3.48	C	26.82	C
PCB-143		C134		C134		C134		C134		C134
PCB-124		C108		C108		C108		C108		C108
PCB-108	0.35	C	0.46	C	2.84	C	1.98	C		CU
PCB-139	0.20	CJ		CU		CU		CU		CU
PCB-140		C139		C139		C139		C139		C139
PCB-107		C106		C106		C106		C106		C106
PCB-123		C106		C106		C106		C106		C106
PCB-131		U		U		U		U		U
PCB-106	0.67	C	0.95	C	10.78	C	9.23	C	6.18	C
PCB-142		U		U		U		U		U

WA5-11 Batch 2
BATTELLE
SDG 49971-23&28
MOD 1668M

NOTES	QC									
CLIENT_ ID	PROCEDURAL BLANK		Clinton Crops, NC		Everglades, FL		Everglades, FL		Lake Dubay, WI	
LAB_SAMP_ ID	Method Blank		EPA-2 COMP		EPA-4 COMP		EPA-4 COMP Duplicate		EPA-5 COMP	
SAMPLE_WGT_VOL	16.6259		18.2470		11.0188		11.0058		16.7103	
SAMP_WGT_VOL_UNIT	G DRYWT		G DRYWT		G DRYWT		G DRYWT		G DRYWT	
PCT_DRY_WT			90.91		54.96		54.96		84.78	
COLLECTION_DATE			9/6/2003		10/20/2003		10/20/2003		8/12/2003	
RECEIPT_DATE			10/21/2003		10/22/2003		10/22/2003		8/18/2003	
COMPOSITE_DATE			10/27/2003		10/27/2003		10/27/2003		9/4/2003	
EXTRACT_DATE	11/12/2003		11/12/2003		11/12/2003		11/6/2003		11/6/2003	
ANALYSIS_DATE	12/14/2003		12/14/2003		12/14/2003		12/18/2003		12/14/2003	
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-28-04		49971-28-02		49971-28-03		49971-23-17		49971-23-04	
REPORTING UNIT	PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT	
REPORTING LIMIT (RL)	0.30		0.27		0.45		1.82		0.30	
PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-118	6.10		7.55		43.03		45.52		45.40	
PCB-132	3.05		4.42		28.17		27.96		24.51	
PCB-122		U		U		U		U		U
PCB-188		U		U		U		U		U
PCB-114		U		U		U	1.69	J		U
PCB-133		U	0.45			U		U	10.50	
PCB-179	0.83		4.47		13.28		8.20		29.72	
PCB-165		U		U		U		U		U
PCB-146	1.59	C	3.80	C	24.24	C	29.18	C	40.40	C
PCB-105	2.38		3.40		32.76		37.48		13.85	
PCB-184		U		U		U		U		U
PCB-161		C146		C146		C146		C146		C146
PCB-176	0.25	J	1.08		3.76			U	3.52	
PCB-153	8.04	C	25.52	C	138.14	C	134.04	C	94.58	C
PCB-168		C153		C153		C153		C153		C153
PCB-141	1.58		3.61		8.09		19.10		8.52	
PCB-186		U		U		U		U		U
PCB-130	0.51		0.88			U	7.16			U
PCB-127		U		U		U		U		U
PCB-137	0.84	C	2.02	C	5.40	C	14.42	C		CU
PCB-164		C137		C137		C137		C137		C137
PCB-163		C129		C129		C129		C129		C129
PCB-138		C129		C129		C129		C129		C129
PCB-129	7.71	C	23.60	C	147.08	C	182.40	C	128.32	C
PCB-160		C129		C129		C129		C129		C129
PCB-158	0.74		1.54		3.30		9.31			U
PCB-178	0.37		3.42		16.50		17.97		16.51	
PCB-175	0.10	J	0.28			U		U		U
PCB-126		U		U		U		U		U
PCB-166		C128		C128		C128		C128		C128
PCB-128	1.21	C	3.62	C	32.96	C	47.18	C	7.70	C
PCB-187	2.52		18.88		111.44		118.87		59.66	
PCB-182	0.05	J		U		U		U		U
PCB-183		C174		C174		C174		C174		C174
PCB-185		C174		C174		C174		C174		C174
PCB-159		U		U		U		U		U
PCB-174	2.43	C	16.02	C	17.43	C	38.52	C	31.62	C
PCB-162		U	0.26	J	2.17		1.25	J	1.90	
PCB-177	0.90		5.23		25.02		24.32		29.11	
PCB-202	0.18	J	8.34		28.68		32.21		5.22	
PCB-167	0.35		1.33		11.74		10.42		4.73	
PCB-181		U		U		U		U		U
PCB-171	0.37	C	1.82	C	13.86	C	13.76	C	8.30	C
PCB-173		C171		C171		C171		C171		C171
PCB-201	0.07	J	1.63		4.75		3.87		2.14	
PCB-156	0.86	C	2.38	C	22.98	C	19.42	C	9.48	C
PCB-157		C156		C156		C156		C156		C156
PCB-204		U		U		U		U		U
PCB-197	0.18	CJ	1.34	C	2.58	C	3.20	C	1.78	C
PCB-200		C197		C197		C197		C197		C197
PCB-172	0.19	J	1.84		14.79		18.59		6.46	
PCB-192		U		U		U		U		U
PCB-193		C180		C180		C180		C180		C180
PCB-180	2.98	C	21.68	C	221.02	C	228.66	C	56.96	C
PCB-191	0.10	J		U	1.17			U		U
PCB-170	1.44		7.47		112.62		127.13		27.00	
PCB-190	0.24	J	2.14		18.15		16.16		6.71	
PCB-169	0.51		0.73			U		U	0.71	
PCB-198	0.83	C	21.64	C	101.86	C	94.70	C	18.26	C
PCB-199		C198		C198		C198		C198		C198
PCB-196	0.39		4.05		7.30		11.98		1.65	
PCB-203	0.53		10.57		31.97		26.71		4.29	
PCB-208	0.67		11.07		32.18		35.00		6.04	
PCB-195	0.65		3.27		16.97		17.32		5.59	
PCB-189	0.84		1.04		4.21		3.66		1.96	
PCB-207	0.51		2.36		3.80		4.44		2.22	
PCB-194	1.91		9.06		69.57		77.76		15.08	
PCB-205	1.96		1.85		5.38		4.99		1.06	
PCB-206	3.29		25.55		60.10		64.74		14.31	
PCB-209	3.16		21.49		54.62		59.18		20.42	

J = reported value < Reporting Limit (RL).
U = not detected.
RL = the low calibration level adjusted for sample final volume and weight.
& = outside QC limits.

WA5-11 Batch 2
BATTELLE
SDG 49971-23&28
MOD 1668M

NOTES				
CLIENT_ ID	Monmouth, IL	Keystone State Park, OK	Arkadelphia, AK	Jasper, NY
LAB_SAMP_ ID	EPA-6 COMP	EPA-9 COMP	EPA-10 COMP	EPA-12 COMP
SAMPLE_WGT_VOL	17.3948	17.8582	17.5451	14.1085
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	87.36	88.71	88.91	70.95
COLLECTION_DATE	8/15/2003	8/18/2003	9/10/2003	8/20/2003
RECEIPT_DATE	8/18/2003	8/19/2003	9/12/2003	8/22/2003
COMPOSITE_DATE	9/4/2003	9/4/2003	9/18/2003	9/4/2003
EXTRACT_DATE	11/6/2003	11/6/2003	11/6/2003	11/6/2003
ANALYSIS_DATE	12/15/2003	12/15/2003	12/15/2003	12/15/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-05	49971-23-06	49971-23-07	49971-23-08
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT
REPORTING LIMIT (RL)	0.29	0.28	0.28	0.35

PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-1	3.64		91.84		8.92		2.60	
PCB-2	3.68		8.39		7.79		2.00	
PCB-3	3.73		20.57		7.31		2.44	
PCB-4	7.55		247.17		18.05		4.37	
PCB-10	0.58		43.54		1.16			U
PCB-9		U	6.95			U		U
PCB-7	U		U	U		U		
PCB-6	6.00		13.48		14.04		2.89	
PCB-5	U		U	U		U		
PCB-8	8.42		17.00		13.52		5.31	
PCB-19	4.67		98.82		10.11		2.50	
PCB-14	U		U	U		U		
PCB-30		C18		C18		C18		C18
PCB-18	18.02	C	28.88	C	31.94	C	8.20	C
PCB-11	5.22			U	6.69		2.90	
PCB-17	11.00		131.16		19.95		5.48	
PCB-13		C12		C12		C12		C12
PCB-27		C16		C16		C16		C16
PCB-12	6.68	C	68.68	C	12.00	C	2.88	C
PCB-24		C16		C16		C16		C16
PCB-16	9.00	C	76.74	C	13.83	C	4.29	C
PCB-15	5.49		69.01		6.45		3.48	
PCB-54	0.83		2.60		2.02		0.33	J
PCB-32	3.78		12.94		5.35		2.12	
PCB-34		U	2.09		1.14			U
PCB-23	0.50		0.26	J	0.23	J		U
PCB-26	8.96	C	24.40	C	13.50	C	3.14	C
PCB-29		C26		C26		C26		C26
PCB-25	3.66		5.49		5.50		1.34	
PCB-50	10.04	C	22.34	C	15.96	C	4.30	C
PCB-53		C50		C50		C50		C50
PCB-31	12.23		16.62		14.76		6.70	
PCB-28		C20		C20		C20		C20
PCB-20	23.60	C	25.84	C	30.72	C	13.08	C
PCB-45	10.16	C	33.36	C	15.26	C	4.62	C
PCB-21		C20		C20		C20		C20
PCB-51		C45		C45		C45		C45
PCB-33		C20		C20		C20		C20
PCB-46	2.83		3.37		3.90		1.25	
PCB-22	4.93		4.33		5.45		2.79	
PCB-52		C43		C43		C43		C43
PCB-73		C43		C43		C43		C43
PCB-43	40.44	C	54.27	C	75.75	C	24.93	C
PCB-36	1.62		2.67		2.23		0.50	
PCB-69		C49		C49		C49		C49
PCB-49	26.36	C	37.64	C	31.76	C	11.70	C
PCB-39	0.84		0.91			U		U
PCB-48	7.96		9.42		12.76		3.80	
PCB-104	U		U	U		U		
PCB-65		C44		C44		C44		C44
PCB-47		C44		C44		C44		C44
PCB-44	24.54	C	100.71	C	34.23	C	14.70	C
PCB-62		C59		C59		C59		C59
PCB-38	U		U	U		U		
PCB-75		C59		C59		C59		C59
PCB-59	1.29	C	2.49	C	3.24	C	0.75	C
PCB-96	3.05		4.35		5.36		1.52	
PCB-42	3.30		4.74		7.35		2.26	
PCB-35	3.31		3.64		6.03		1.40	
PCB-41		C40		C40		C40		C40
PCB-71		C40		C40		C40		C40
PCB-40	5.94	C	7.68	C	13.41	C	4.08	C
PCB-37	8.41		6.54		6.68		3.79	
PCB-64	3.56		3.44		10.47		3.15	
PCB-72	1.81		3.64		1.85			U
PCB-103	1.41		2.56		2.16			U
PCB-68	1.08		2.51		0.75			U

WA5-11 Batch 2
BATTELLE
SDG 49971-23&28
MOD 1668M

NOTES								
CLIENT_ ID	Monmouth, IL		Keystone State Park, OK		Arkadelphia, AK		Jasper, NY	
LAB_SAMP_ ID	EPA-6 COMP		EPA-9 COMP		EPA-10 COMP		EPA-12 COMP	
SAMPLE_WGT_VOL	17.3948		17.8582		17.5451		14.1085	
SAMP_WGT_VOL_UNIT	G DRYWT		G DRYWT		G DRYWT		G DRYWT	
PCT_DRY_WT	87.36		88.71		88.91		70.95	
COLLECTION_DATE	8/15/2003		8/18/2003		9/10/2003		8/20/2003	
RECEIPT_DATE	8/18/2003		8/19/2003		9/12/2003		8/22/2003	
COMPOSITE_DATE	9/4/2003		9/4/2003		9/18/2003		9/4/2003	
EXTRACT_DATE	11/6/2003		11/6/2003		11/6/2003		11/6/2003	
ANALYSIS_DATE	12/15/2003		12/15/2003		12/15/2003		12/15/2003	
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-05		49971-23-06		49971-23-07		49971-23-08	
REPORTING UNIT	PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT	
REPORTING LIMIT (RL)	0.29		0.28		0.28		0.35	
PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-94	2.03		2.73		3.00			U
PCB-57	4.09		4.31		13.26		1.46	
PCB-95	59.59		60.35		216.00		37.08	
PCB-58	U	U	U	U				
PCB-100		C93		C93		C93		C93
PCB-93	11.68	C	14.84	C	15.12	C	4.60	C
PCB-67	3.37		2.72		3.51			U
PCB-102		C93		C93		C93		C93
PCB-98		C93		C93		C93		C93
PCB-63	0.99			U	1.00			U
PCB-88	16.06	C	18.80	C	37.28	C	7.58	C
PCB-61	45.60	C	23.40	C	69.28	C	20.68	C
PCB-70		C61		C61		C61		C61
PCB-76		C61		C61		C61		C61
PCB-91		C88		C88		C88		C88
PCB-74		C61		C61		C61		C61
PCB-84	11.71		12.89		66.43		8.76	
PCB-66	16.66		10.00		26.64		7.62	
PCB-55	U	U	U	U				
PCB-89	2.23		1.70			U	0.72	
PCB-121	U	U	U	U				
PCB-56	11.17		5.36		10.84		4.01	
PCB-60	3.89		1.92		2.84		1.77	
PCB-92	19.08		16.74		46.19		11.35	
PCB-80	U	U	U	U				
PCB-155	U	U	U	U				
PCB-113		C90		C90		C90		C90
PCB-90	93.33	C	68.94	C	311.31	C	58.62	C
PCB-101		C90		C90		C90		C90
PCB-152	0.41		0.50		0.67			U
PCB-150	0.44		0.75		0.63			U
PCB-83	74.91	C	32.52	C	140.85	C	36.57	C
PCB-99		C83		C83		C83		C83
PCB-136	12.98		13.93		30.99		8.40	
PCB-112		C83		C83		C83		C83
PCB-145	U	U	U	U				
PCB-109		C86		C86		C86		C86
PCB-119		C86		C86		C86		C86
PCB-79	3.12		2.21		10.89		1.55	
PCB-97		C86		C86		C86		C86
PCB-86	42.42	C	29.46	C	198.66	C	28.14	C
PCB-125		C86		C86		C86		C86
PCB-87		C86		C86		C86		C86
PCB-78	U	U	U	U				
PCB-117		C85		C85		C85		C85
PCB-116		C85		C85		C85		C85
PCB-85	16.83	C	7.14	C	44.46	C	10.41	C
PCB-110	71.54	C	37.22	C	339.92	C	54.18	C
PCB-115		C110		C110		C110		C110
PCB-81	U	U	U	U				
PCB-148	U	U	U	U				
PCB-82	8.49		3.59		32.35		3.91	
PCB-111	U	U	U	U				
PCB-77	7.33		2.30		3.79		3.72	
PCB-151		C135		C135		C135		C135
PCB-135	28.38	C	27.93	C	65.55	C	24.66	C
PCB-154		C135		C135		C135		C135
PCB-120	U	U	U	U				
PCB-144	U	U	U	U				
PCB-147	100.84	C	71.28	C	208.26	C	76.48	C
PCB-149		C147		C147		C147		C147
PCB-134		CU	7.16	C	20.64	C		CU
PCB-143		C134		C134		C134		C134
PCB-124		C108		C108		C108		C108
PCB-108	3.96	C	2.30	C	10.70	C	2.84	C
PCB-139	2.78	C	0.76	C		CU		CU
PCB-140		C139		C139		C139		C139
PCB-107		C106		C106		C106		C106
PCB-123		C106		C106		C106		C106
PCB-131	U	U	U	U				
PCB-106	11.24	C	4.93	C	20.74	C	7.71	C
PCB-142	U	U	U	U				

WA5-11 Batch 2
BATTELLE
SDG 49971-23&28
MOD 1668M

NOTES								
CLIENT_ ID	Monmouth, IL		Keystone State Park, OK		Arkadelphia, AK		Jasper, NY	
LAB_SAMP_ ID	EPA-6 COMP		EPA-9 COMP		EPA-10 COMP		EPA-12 COMP	
SAMPLE_WGT_VOL	17.3948		17.8582		17.5451		14.1085	
SAMP_WGT_VOL_UNIT	G DRYWT		G DRYWT		G DRYWT		G DRYWT	
PCT_DRY_WT	87.36		88.71		88.91		70.95	
COLLECTION_DATE	8/15/2003		8/18/2003		9/10/2003		8/20/2003	
RECEIPT_DATE	8/18/2003		8/19/2003		9/12/2003		8/22/2003	
COMPOSITE_DATE	9/4/2003		9/4/2003		9/18/2003		9/4/2003	
EXTRACT_DATE	11/6/2003		11/6/2003		11/6/2003		11/6/2003	
ANALYSIS_DATE	12/15/2003		12/15/2003		12/15/2003		12/15/2003	
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-05		49971-23-06		49971-23-07		49971-23-08	
REPORTING UNIT	PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT	
REPORTING LIMIT (RL)	0.29		0.28		0.28		0.35	
PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-118	93.48		31.11		218.07		39.01	
PCB-132	29.63		19.57		100.58		21.44	
PCB-122	U	U	U	U				
PCB-188	U	U	U	U				
PCB-114	U	U	U	U				
PCB-133	2.68			U		U		U
PCB-179	17.64		12.11		18.71		17.48	
PCB-165	U	U	U	U				
PCB-146	22.38	C	14.80	C	30.08	C	16.06	C
PCB-105	36.22		10.60		78.28		21.23	
PCB-184	U	U	U	U				
PCB-161		C146		C146		C146		C146
PCB-176	4.55		3.14		6.26		3.44	
PCB-153	197.40	C	94.00	C	219.88	C	131.70	C
PCB-168		C153		C153		C153		C153
PCB-141		U	16.62		57.14		14.81	
PCB-186	U	U	U	U				
PCB-130	4.95		4.05		18.67		3.64	
PCB-127	U	U	U	U				
PCB-137	7.82	C	7.38	C	35.96	C	8.12	C
PCB-164		C137		C137		C137		C137
PCB-163		C129		C129		C129		C129
PCB-138		C129		C129		C129		C129
PCB-129	174.28	C	85.72	C	286.16	C	133.28	C
PCB-160		C129		C129		C129		C129
PCB-158	8.68		5.88		26.22		6.19	
PCB-178	11.24		9.28		10.36		14.94	
PCB-175	1.47		1.20		1.94		1.08	
PCB-126	U	U	U	U				
PCB-166		C128		C128		C128		C128
PCB-128	22.14	C	11.06	C	50.48	C	16.88	C
PCB-187	49.39		45.35		50.11		60.84	
PCB-182	U	U	U	U				
PCB-183		C174		C174		C174		C174
PCB-185		C174		C174		C174		C174
PCB-159	U	U	U	U				
PCB-174	40.80	C	35.76	C	60.57	C	46.38	C
PCB-162	1.56		1.41		2.23		1.51	
PCB-177	21.12		16.18		23.77		21.79	
PCB-202	6.22		4.97		4.18		14.04	
PCB-167	7.66		4.55		11.82		5.06	
PCB-181	U	U	U	U				
PCB-171	9.94	C	7.44	C	14.02	C	8.36	C
PCB-173		C171		C171		C171		C171
PCB-201	2.95		1.93		1.92		3.96	
PCB-156	18.84	C	8.36	C	33.62	C	11.12	C
PCB-157		C156		C156		C156		C156
PCB-204	U	U	U	U				
PCB-197	1.82	C	1.64	C	1.72	C	2.84	C
PCB-200		C197		C197		C197		C197
PCB-172	6.82		6.05		7.91		7.99	
PCB-192	U	U	U	U				
PCB-193		C180		C180		C180		C180
PCB-180	74.70	C	62.42	C	80.90	C	82.08	C
PCB-191	1.10			U	1.56			U
PCB-170	32.32		25.82		41.16		30.50	
PCB-190	7.35		6.26		7.80		9.22	
PCB-169	1.18			U		U		U
PCB-198	18.20	C	21.62	C	17.20	C	48.86	C
PCB-199		C198		C198		C198		C198
PCB-196	0.69		6.17		6.23		9.27	
PCB-203	4.06		12.22		9.76		22.91	
PCB-208	9.77		3.70		2.29		13.49	
PCB-195	5.24		5.85		4.70		7.49	
PCB-189	1.66		1.73		1.66		1.37	
PCB-207	4.20		1.72		0.90		5.04	
PCB-194	13.02		14.99		12.44		23.03	
PCB-205	1.28		1.97		0.75		1.42	
PCB-206	17.76		11.87		7.59		33.88	
PCB-209	40.70		10.15		5.36		28.13	

J = reported value < Reporting Limit (RL
U = not detected.
RL = the low calibration level adjusted f
& = outside QC limits.

WA5-11 Batch 2
BATTELLE
SDG 49971-23&28
MOD 1668M

NOTES										
CLIENT_ ID	Fond du Lac, MN		Fond du Lac, MN		Goodwell, OK		Big Bend, TX		Grand Canyon, AZ	
LAB_SAMP_ ID	EPA-20 COMP		EPA-20 COMP Duplicate		EPA-22 COMP		EPA-23 COMP		EPA-24 COMP	
SAMPLE_WGT_VOL	19.0782		19.6037		17.5068		18.1705		19.0697	
SAMP_WGT_VOL_UNIT	G DRYWT		G DRYWT		G DRYWT		G DRYWT		G DRYWT	
PCT_DRY_WT	97.1		97.1		88.38		91.6		95.74	
COLLECTION_DATE	8/22/2003		8/22/2003		8/20/2003		9/8/2003		8/26/2003	
RECEIPT_DATE	8/26/2003		8/26/2003		8/22/2003		9/10/2003		8/29/2003	
COMPOSITE_DATE	9/4/2003		9/4/2003		9/4/2003		9/18/2003		9/4/2003	
EXTRACT_DATE	11/6/2003		11/6/2003		11/6/2003		11/6/2003		11/6/2003	
ANALYSIS_DATE	12/15/2003		12/16/2003		12/18/2003		12/15/2003		12/18/2003	
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-09		49971-23-18		49971-23-10		49971-23-11		49971-23-12	
REPORTING UNIT	PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT	
REPORTING LIMIT (RL)	0.26		0.26		0.86		0.28		1.05	
PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-1	4.22		5.57		52.02		1.95		2.55	
PCB-2	4.70		5.10		46.92		1.51		1.45	
PCB-3	5.03		4.89		40.56		1.86		1.87	
PCB-4	9.95		10.51		89.02		3.91			U
PCB-10		U	0.89		6.46			U		U
PCB-9		U	2.78		21.33		0.87			U
PCB-7		U	1.30		10.15		0.49			U
PCB-6		U	7.16		58.06		2.78		3.50	
PCB-5		U	0.63		6.75		0.29			U
PCB-8		U	7.39		52.85		4.15		4.83	
PCB-19	6.11		5.08		35.74		2.17		2.36	
PCB-14	U			U U		U				U
PCB-30		C18		C18		C18		C18		C18
PCB-18	18.24	C	18.22	C	103.68	C	6.10	C	9.36	C
PCB-11		U	5.28		24.72		2.23			U
PCB-17	12.01		11.95		65.72		4.11		5.94	
PCB-13		C12		C12		C12		C12		C12
PCB-27		C16		C16		C16		C16		C16
PCB-12		CU	7.28	C	47.00	C	3.12	C		CU
PCB-24		C16		C16		C16		C16		C16
PCB-16	8.34	C	8.01	C	48.96	C	3.06	C	5.37	C
PCB-15		U	3.91		17.77		2.85			U
PCB-54	1.05		0.89		14.59		0.52			U
PCB-32	3.13		3.09		17.00		1.75		2.15	
PCB-34	0.41		0.53		2.73			U		U
PCB-23		U		U	0.76	J		U		U
PCB-26	7.18	C	6.44	C	30.16	C	3.28	C	3.72	C
PCB-29		C26		C26		C26		C26		C26
PCB-25	3.04		2.33		9.48		1.62		1.20	
PCB-50	9.04	C	11.10	C	64.24	C	17.28	C	8.18	C
PCB-53		C50		C50		C50		C50		C50
PCB-31	9.35		7.19		34.79		5.42		4.88	
PCB-28		C20		C20		C20		C20		C20
PCB-20	20.16	C	15.44	C	70.36	C	14.56	C	8.84	C
PCB-45	9.38	C	10.32	C	67.92	C	6.84	C	6.78	C
PCB-21		C20		C20		C20		C20		C20
PCB-51		C45		C45		C45		C45		C45
PCB-33		C20		C20		C20		C20		C20
PCB-46	2.05		2.79		15.84		1.66			U
PCB-22	3.76		3.18		10.71		2.21		1.76	
PCB-52		C43		C43		C43		C43		C43
PCB-73		C43		C43		C43		C43		C43
PCB-43	38.67	C	39.18	C	135.36	C	330.60	C	33.84	C
PCB-36		U		U	1.58			U		U
PCB-69		C49		C49		C49		C49		C49
PCB-49	17.08	C	21.12	C	84.94	C	72.40	C	15.68	C
PCB-39		U	0.49		0.83	J		U		U
PCB-48	7.30		9.62		52.18		5.19		6.16	
PCB-104	U			U U		U				U
PCB-65		C44		C44		C44		C44		C44
PCB-47		C44		C44		C44		C44		C44
PCB-44	20.31	C	22.44	C	81.42	C	105.03	C	19.11	C
PCB-62		C59		C59		C59		C59		C59
PCB-38		U	0.27			U		U	9.01	
PCB-75		C59		C59		C59		C59		C59
PCB-59	2.31	C	1.74	C	14.79	C	3.30	C	1.74	C
PCB-96	2.55			U	9.94		7.02			U
PCB-42	4.75		4.58		28.76		14.58		5.03	
PCB-35	2.88		2.71		13.89		1.55		1.51	
PCB-41		C40		C40		C40		C40		C40
PCB-71		C40		C40		C40		C40		C40
PCB-40	9.00	C	8.16	C	52.05	C	37.77	C	11.46	C
PCB-37	3.72		4.41		13.07		5.49		1.77	
PCB-64	6.66		3.68		18.50		109.27		4.72	
PCB-72	0.94		1.20		6.55			U		U
PCB-103		U		U	3.52			U		U
PCB-68		U	0.47		1.93			U		U

WA5-11 Batch 2
BATTELLE
SDG 49971-23&28
MOD 1668M

NOTES										
CLIENT_ ID	Fond du Lac, MN		Fond du Lac, MN		Goodwell, OK		Big Bend, TX		Grand Canyon, AZ	
LAB_SAMP_ ID	EPA-20 COMP		EPA-20 COMP Duplicate		EPA-22 COMP		EPA-23 COMP		EPA-24 COMP	
SAMPLE_WGT_VOL	19.0782		19.6037		17.5068		18.1705		19.0697	
SAMP_WGT_VOL_UNIT	G DRYWT		G DRYWT		G DRYWT		G DRYWT		G DRYWT	
PCT_DRY_WT	97.1		97.1		88.38		91.6		95.74	
COLLECTION_DATE	8/22/2003		8/22/2003		8/20/2003		9/8/2003		8/26/2003	
RECEIPT_DATE	8/26/2003		8/26/2003		8/22/2003		9/10/2003		8/29/2003	
COMPOSITE_DATE	9/4/2003		9/4/2003		9/4/2003		9/18/2003		9/4/2003	
EXTRACT_DATE	11/6/2003		11/6/2003		11/6/2003		11/6/2003		11/6/2003	
ANALYSIS_DATE	12/15/2003		12/16/2003		12/18/2003		12/15/2003		12/18/2003	
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-09		49971-23-18		49971-23-10		49971-23-11		49971-23-12	
REPORTING UNIT	PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT		PG/G DRYWT	
REPORTING LIMIT (RL)	0.26		0.26		0.86		0.28		1.05	
PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-94		U		U	5.30			U		U
PCB-57	4.74		2.67		3.80		46.04			U
PCB-95	77.62		41.57		105.88		1941.21		23.95	
PCB-58	U	U	U	U		U				
PCB-100		C93		C93		C93		C93		C93
PCB-93	8.40	C		CU	35.24	C	39.68	C	4.40	C
PCB-67	1.72		2.23		11.01			U		U
PCB-102		C93		C93		C93		C93		C93
PCB-98		C93		C93		C93		C93		C93
PCB-63		U	0.47		3.20		4.84			U
PCB-88	13.30	C		CU	30.42	C	285.72	C	5.42	C
PCB-61	30.60	C	28.68	C	77.96	C	499.92	C	18.92	C
PCB-70		C61		C61		C61		C61		C61
PCB-76		C61		C61		C61		C61		C61
PCB-91		C88		C88		C88		C88		C88
PCB-74		C61		C61		C61		C61		C61
PCB-84	18.90			U	19.52		553.82		5.93	
PCB-66	13.04		10.12		30.72		255.45		5.68	
PCB-55		U		U	1.74			U		U
PCB-89		U		U	5.65			U		U
PCB-121	U	U	U	U		U				
PCB-56	6.20		5.06		18.43		60.49		3.64	
PCB-60	1.98		1.61		3.89		15.94			U
PCB-92	18.93		14.72		33.33		373.64		7.98	
PCB-80	U	U	U	U		U				
PCB-155	U	U	U	U		U				
PCB-113		C90		C90		C90		C90		C90
PCB-90	108.24	C	58.05	C	149.82	C	2644.59	C	31.98	C
PCB-101		C90		C90		C90		C90		C90
PCB-152	0.33			U	1.80			U		U
PCB-150	0.32			U	1.36			U		U
PCB-83	51.36	C	22.26	C	64.74	C	1249.20	C	15.87	C
PCB-99		C83		C83		C83		C83		C83
PCB-136	11.36		7.15		30.60		193.71		4.95	
PCB-112		C83		C83		C83		C83		C83
PCB-145	U	U	U	U		U				
PCB-109		C86		C86		C86		C86		C86
PCB-119		C86		C86		C86		C86		C86
PCB-79	4.03		1.62		3.51		45.17			U
PCB-97		C86		C86		C86		C86		C86
PCB-86	58.08	C	31.26	C	53.88	C	1724.10	C	17.52	C
PCB-125		C86		C86		C86		C86		C86
PCB-87		C86		C86		C86		C86		C86
PCB-78	U	U	U	U		U				
PCB-117		C85		C85		C85		C85		C85
PCB-116		C85		C85		C85		C85		C85
PCB-85	13.59	C		CU	10.56	C	369.15	C	3.09	C
PCB-110	94.18	C	32.42	C	64.06	C	2897.68	C	25.66	C
PCB-115		C110		C110		C110		C110		C110
PCB-81	U	U	U	U		U				
PCB-148		U		U	0.47	J		U		U
PCB-82	8.14			U	6.23		260.15		2.59	
PCB-111	U	U	U	U		U				
PCB-77	2.33		2.39		7.89		21.22			U
PCB-151		C135		C135		C135		C135		C135
PCB-135	23.61	C	9.69	C	81.54	C	371.64	C	14.61	C
PCB-154		C135		C135		C135		C135		C135
PCB-120		U		U	3.16			U		U
PCB-144		U	0.56		11.07		57.29		1.43	
PCB-147	76.06	C	40.10	C	263.00	C	1071.80	C	38.64	C
PCB-149		C147		C147		C147		C147		C147
PCB-134	8.28	C		CU	8.84	C	95.32	C	2.20	C
PCB-143		C134		C134		C134		C134		C134
PCB-124		C108		C108		C108		C108		C108
PCB-108	3.26	C		CU	5.52	C	78.52	C	1.28	C
PCB-139		CU		CU	4.60	C	24.98	C		CU
PCB-140		C139		C139		C139		C139		C139
PCB-107		C106		C106		C106		C106		C106
PCB-123		C106		C106		C106		C106		C106
PCB-131		U		U		U	21.74			U
PCB-106	6.67	C		CU	11.06	C	142.65	C	2.44	C
PCB-142	U	U	U	U		U				

WA5-11 Batch 2
BATTELLE
SDG 49971-23&28
MOD 1668M

NOTES

CLIENT_ ID	Fond du Lac, MN	Fond du Lac, MN	Goodwell, OK	Big Bend, TX	Grand Canyon, AZ
LAB_SAMP_ ID	EPA-20 COMP	EPA-20 COMP Duplicate	EPA-22 COMP	EPA-23 COMP	EPA-24 COMP
SAMPLE_WGT_VOL	19.0782	19.6037	17.5068	18.1705	19.0697
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	97.1	97.1	88.38	91.6	95.74
COLLECTION_DATE	8/22/2003	8/22/2003	8/20/2003	9/8/2003	8/26/2003
RECEIPT_DATE	8/26/2003	8/26/2003	8/22/2003	9/10/2003	8/29/2003
COMPOSITE_DATE	9/4/2003	9/4/2003	9/4/2003	9/18/2003	9/4/2003
EXTRACT_DATE	11/6/2003	11/6/2003	11/6/2003	11/6/2003	11/6/2003
ANALYSIS_DATE	12/15/2003	12/16/2003	12/18/2003	12/15/2003	12/18/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-09	49971-23-18	49971-23-10	49971-23-11	49971-23-12
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT
REPORTING LIMIT (RL)	0.26	0.26	0.86	0.28	1.05

PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-118	66.92			U	44.51		1917.27		19.96	
PCB-132	33.16		23.17		66.48		609.59		13.77	
PCB-122		U		U	1.34			U		U
PCB-188	U	U		U U		U				
PCB-114		U		U		U	24.47			U
PCB-133		U		U	5.30			U		U
PCB-179	9.01		8.94		49.91		39.86		4.62	
PCB-165	U	U		U U		U				
PCB-146	12.96	C	10.52	C	56.22	C	159.06	C	9.92	C
PCB-105	24.47			U	12.74		657.10		6.73	
PCB-184	U	U		U U		U				
PCB-161		C146		C146		C146		C146		C146
PCB-176	2.66		2.61		10.49		14.54		1.37	
PCB-153	88.32	C	82.22	C	323.64	C	1183.70	C	46.30	C
PCB-168		C153		C153		C153		C153		C153
PCB-141	18.59		12.70		43.90		241.27		9.36	
PCB-186	U	U		U U		U				
PCB-130	6.61		2.83		14.47		105.42			U
PCB-127	U	U		U U		U				
PCB-137	13.36	C	7.20	C	31.04	C	201.12	C	5.22	C
PCB-164		C137		C137		C137		C137		C137
PCB-163		C129		C129		C129		C129		C129
PCB-138		C129		C129		C129		C129		C129
PCB-129	106.36	C	84.00	C	295.16	C	1692.12	C	43.48	C
PCB-160		C129		C129		C129		C129		C129
PCB-158	9.68		4.95		22.01		169.24		3.69	
PCB-178	4.63		4.81		34.11		18.70		2.03	
PCB-175	0.84		1.06		3.81		4.30			U
PCB-126		U		U	2.15			U		U
PCB-166		C128		C128		C128		C128		C128
PCB-128	17.20	C	12.18	C	31.48	C	306.66	C	4.32	C
PCB-187	23.34		23.48		176.26		99.95		11.56	
PCB-182		U	0.20	J		U	0.79			U
PCB-183		C174		C174		C174		C174		C174
PCB-185		C174		C174		C174		C174		C174
PCB-159		U		U	3.56			U		U
PCB-174	28.41	C	27.48	C	154.98	C	167.82	C	15.03	C
PCB-162	1.07		0.50		2.81		6.50			U
PCB-177	11.02		10.43		85.76		57.52		5.07	
PCB-202	1.98		2.24		14.88		4.47		0.67	J
PCB-167	4.69		3.45		10.91		56.94		1.65	
PCB-181		U		U		U	2.19			U
PCB-171	6.08	C	5.26	C	32.18	C	39.32	C	2.20	C
PCB-173		C171		C171		C171		C171		C171
PCB-201	1.13		1.31		6.29		3.25		0.43	J
PCB-156	11.34	C	7.82	C	15.14	C	185.24	C	2.84	C
PCB-157		C156		C156		C156		C156		C156
PCB-204		U		U		U	0.32			U
PCB-197	0.98	C	0.86	C	8.90	C	3.94	C		CU
PCB-200		C197		C197		C197		C197		C197
PCB-172	3.85		3.55		23.48		17.92		1.73	
PCB-192	U	U		U U		U				
PCB-193		C180		C180		C180		C180		C180
PCB-180	39.30	C	41.10	C	225.46	C	184.20	C	18.64	C
PCB-191	0.53			U	3.39		4.38			U
PCB-170	18.28		14.61		103.51		120.72		7.92	
PCB-190	3.75		1.91		3.29		21.22		1.19	
PCB-169		U	0.48			U		U		U
PCB-198	10.10	C	11.58	C	65.58	C	24.82	C	2.86	C
PCB-199		C198		C198		C198		C198		C198
PCB-196	3.68		4.23		19.60		11.25		1.91	
PCB-203	5.51		5.35		44.21		15.42		1.86	
PCB-208	2.85		3.25		7.88		3.14		0.29	J
PCB-195	2.44		2.92		28.03		8.99		1.10	
PCB-189	0.95		1.22		4.35		5.91			U
PCB-207	1.61		1.74		3.04		1.78			U
PCB-194	7.61		8.29		53.93		19.49		3.07	
PCB-205	1.15		1.31		3.18		2.90			U
PCB-206	7.78		8.51		30.71		7.97		2.30	
PCB-209	8.43		9.34		8.48		4.18		1.81	

J = reported value < Reporting Limit (RL
U = not detected.
RL = the low calibration level adjusted f
& = outside QC limits.

WA5-11 Batch 2
BATTELLE
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MOD 1668M

NOTES					
CLIENT_ ID	Rancho Seco, CA	Rancho Seco, CA	Marvel Ranch, OR	Ozette Lake, WA	Trapper Creek, AK
LAB_SAMP_ ID	EPA-28 COMP	EPA-28 COMP Duplicate	EPA-29 COMP	EPA-30 COMP	EPA-34 COMP
SAMPLE_WGT_VOL	19.1987	19.2911	18.2966	13.4569	11.7051
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	98.76	98.76	90.88	68.71	58.27
COLLECTION_DATE	8/14/2003	8/14/2003	8/20/2003	8/20/2003	9/1/2003
RECEIPT_DATE	8/18/2003	8/18/2003	8/21/2003	8/22/2003	9/3/2003
COMPOSITE_DATE	9/4/2003	9/4/2003	9/4/2003	9/4/2003	9/10/2003
EXTRACT_DATE	11/6/2003	11/6/2003	11/6/2003	11/6/2003	11/6/2003
ANALYSIS_DATE	12/19/2003	12/16/2003	12/18/2003	12/18/2003	12/18/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-13	49971-23-19	49971-23-14	49971-23-15	49971-23-16
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT
REPORTING LIMIT (RL)	1.56	0.26	1.09	1.49	1.28

PARAM_NAME	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-1	4.91		3.12		3.68		18.56		5.84	
PCB-2	1.81		1.57		5.79		19.77		3.90	
PCB-3	4.60		2.61		4.84		17.74		4.40	
PCB-4		U	6.11			U		U		U
PCB-10		U	0.35			U		U		U
PCB-9		U	1.22			U	7.23			U
PCB-7		U	0.71			U		U		U
PCB-6	6.85		3.73		2.44		19.32		6.75	
PCB-5	U			U	U	U				U
PCB-8	16.28		5.70		3.56		22.98		10.12	
PCB-19	10.67		2.85		1.94		13.22		5.75	
PCB-14	U			U	U	U				U
PCB-30		C18		C18		C18		C18		C18
PCB-18	18.70	C	10.34	C	8.46	C	39.08	C	15.68	C
PCB-11	8.72		3.56		26.92		11.93		4.45	
PCB-17	23.54		6.53		4.36		22.88		13.23	
PCB-13		C12		C12		C12		C12		C12
PCB-27		C16		C16		C16		C16		C16
PCB-12	6.80	C	2.58	C	2.86	C	16.56	C	5.32	C
PCB-24		C16		C16		C16		C16		C16
PCB-16	22.65	C	5.37	C	4.02	C	18.90	C	11.28	C
PCB-15	13.86		4.30		2.34		8.47		4.55	
PCB-54		U	0.50			U		U		U
PCB-32	11.08		2.66		1.96		8.27		3.88	
PCB-34		U	0.24	J		U		U		U
PCB-23	U			U	U	U				U
PCB-26	17.56	C	4.12	C	2.48	C	13.80	C	7.40	C
PCB-29		C26		C26		C26		C26		C26
PCB-25	9.73		1.64		0.64	J	4.99		2.55	
PCB-50	29.22	C	4.74	C	4.38	C	20.62	C	11.92	C
PCB-53		C50		C50		C50		C50		C50
PCB-31	17.94		8.15		4.73		20.04		9.78	
PCB-28		C20		C20		C20		C20		C20
PCB-20	41.12	C	16.00	C	9.40	C	37.04	C		CU
PCB-45	21.94	C	4.90	C	4.96	C	20.38	C	11.44	C
PCB-21		C20		C20		C20		C20		C20
PCB-51		C45		C45		C45		C45		C45
PCB-33		C20		C20		C20		C20		C20
PCB-46	8.72		1.24			U	4.49		2.85	
PCB-22	5.58		3.31		2.21		6.94		3.60	
PCB-52		C43		C43		C43		C43		C43
PCB-73		C43		C43		C43		C43		C43
PCB-43	192.75	C	40.17	C	27.24	C	79.35	C	40.80	C
PCB-36	U			U	U	U				U
PCB-69		C49		C49		C49		C49		C49
PCB-49	109.68	C	14.08	C	14.22	C	40.04	C	22.96	C
PCB-39		U	0.61			U		U		U
PCB-48	7.86		4.76		3.67		17.27		8.68	
PCB-104	U			U	U	U				U
PCB-65		C44		C44		C44		C44		C44
PCB-47		C44		C44		C44		C44		C44
PCB-44	133.62	C	21.54	C	17.40	C	45.78	C	32.28	C
PCB-62		C59		C59		C59		C59		C59
PCB-38	U			U	U	U				U
PCB-75		C59		C59		C59		C59		C59
PCB-59		CU	1.35	C		CU	5.67	C	2.04	C
PCB-96	2.17		1.73			U	4.15			U
PCB-42	24.23		3.52		3.54		11.59		6.62	
PCB-35	1.78		1.43			U	5.38		2.43	
PCB-41		C40		C40		C40		C40		C40
PCB-71		C40		C40		C40		C40		C40
PCB-40	57.45	C	6.87	C	6.84	C	21.39	C	13.32	C
PCB-37	7.58		4.96			U	8.36		3.32	
PCB-64	27.52		5.66		5.80		12.82		7.01	
PCB-72	4.44		0.77			U	2.46			U
PCB-103 U	U U							U		U
PCB-68	2.40		0.46			U		U		U

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NOTES					
CLIENT_ ID	Rancho Seco, CA	Rancho Seco, CA	Marvel Ranch, OR	Ozette Lake, WA	Trapper Creek, AK
LAB_SAMP_ ID	EPA-28 COMP	EPA-28 COMP Duplicate	EPA-29 COMP	EPA-30 COMP	EPA-34 COMP
SAMPLE_WGT_VOL	19.1987	19.2911	18.2966	13.4569	11.7051
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	98.76	98.76	90.88	68.71	58.27
COLLECTION_DATE	8/14/2003	8/14/2003	8/20/2003	8/20/2003	9/1/2003
RECEIPT_DATE	8/18/2003	8/18/2003	8/21/2003	8/22/2003	9/3/2003
COMPOSITE_DATE	9/4/2003	9/4/2003	9/4/2003	9/4/2003	9/10/2003
EXTRACT_DATE	11/6/2003	11/6/2003	11/6/2003	11/6/2003	11/6/2003
ANALYSIS_DATE	12/19/2003	12/16/2003	12/18/2003	12/18/2003	12/18/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-13	49971-23-19	49971-23-14	49971-23-15	49971-23-16
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT
REPORTING LIMIT (RL)	1.56	0.26	1.09	1.49	1.28

PARAM_NAME		RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-94	U	U U							U		U
PCB-57			U	3.38			U		U		U
PCB-95		195.72		91.64		35.60		78.21		35.79	
PCB-58	U	U U							U		U
PCB-100			C93		C93		C93		C93		C93
PCB-93		16.92	C	5.64	C	3.40	C	14.00	C	5.56	C
PCB-67		5.41		1.09			U	4.65		2.14	
PCB-102			C93		C93		C93		C93		C93
PCB-98			C93		C93		C93		C93		C93
PCB-63		2.74		0.52			U		U		U
PCB-88		39.38	C	15.56	C	6.94	C	15.92	C	8.36	C
PCB-61		129.68	C	29.44	C	28.92	C	51.76	C	35.00	C
PCB-70			C61		C61		C61		C61		C61
PCB-76			C61		C61		C61		C61		C61
PCB-91			C88		C88		C88		C88		C88
PCB-74			C61		C61		C61		C61		C61
PCB-84		66.11		26.94		11.35		19.83		10.34	
PCB-66		67.88		11.76		12.15		21.98		15.70	
PCB-55	U	U U							U		U
PCB-89	U	U U							U		U
PCB-121	U	U U							U		U
PCB-56		18.56		5.96		6.53		12.38		6.81	
PCB-60		5.15		2.09		3.49		5.59		3.34	
PCB-92		66.42		24.48		12.46		21.93		13.70	
PCB-80	U	U U							U		U
PCB-155	U	U U							U		U
PCB-113			C90		C90		C90		C90		C90
PCB-90		271.05	C	116.22	C	55.05	C	104.61	C	55.89	C
PCB-101			C90		C90		C90		C90		C90
PCB-152	U	U U							U		U
PCB-150	U	U U							U		U
PCB-83		139.89	C	63.12	C	30.78	C	47.88	C	26.58	C
PCB-99			C83		C83		C83		C83		C83
PCB-136		26.72		12.63		6.40		14.63		8.94	
PCB-112			C83		C83		C83		C83		C83
PCB-145	U	U U							U		U
PCB-109			C86		C86		C86		C86		C86
PCB-119			C86		C86		C86		C86		C86
PCB-79			U	2.74			U		U		U
PCB-97			C86		C86		C86		C86		C86
PCB-86		175.44	C	68.88	C	34.62	C	44.28	C	27.30	C
PCB-125			C86		C86		C86		C86		C86
PCB-87			C86		C86		C86		C86		C86
PCB-78			U	0.30			U		U		U
PCB-117			C85		C85		C85		C85		C85
PCB-116			C85		C85		C85		C85		C85
PCB-85		35.31	C	15.21	C	10.29	C	11.70	C	7.23	C
PCB-110		294.68	C	112.76	C	58.82	C	60.12	C	43.10	C
PCB-115			C110		C110		C110		C110		C110
PCB-81	U	U U							U		U
PCB-148	U	U U							U		U
PCB-82		26.40		10.76		6.32		7.54		3.52	
PCB-111	U	U U							U		U
PCB-77		7.44		4.59		2.37		4.82		2.84	
PCB-151			C135		C135		C135		C135		C135
PCB-135		59.28	C	27.51	C	16.65	C	39.09	C	22.68	C
PCB-154			C135		C135		C135		C135		C135
PCB-120	U	U U							U		U
PCB-144		7.64			U	2.84		5.87		2.98	
PCB-147		239.58	C	90.24	C	65.54	C	125.58	C	77.48	C
PCB-149			C147		C147		C147		C147		C147
PCB-134		19.04	C	8.22	C	3.46	C	6.26	C	4.90	C
PCB-143			C134		C134		C134		C134		C134
PCB-124			C108		C108		C108		C108		C108
PCB-108		9.00	C	3.40	C	2.32	C	4.06	C	2.00	C
PCB-139			CU	1.32	C		CU		CU		CU
PCB-140			C139		C139		C139		C139		C139
PCB-107			C106		C106		C106		C106		C106
PCB-123			C106		C106		C106		C106		C106
PCB-131	U	U U							U		U
PCB-106		14.91	C	8.03	C	4.85	C	6.54	C	5.61	C
PCB-142	U	U U							U		U

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LAB_SAMP_ ID	EPA-28 COMP	EPA-28 COMP Duplicate	EPA-29 COMP	EPA-30 COMP	EPA-34 COMP
SAMPLE_WGT_VOL	19.1987	19.2911	18.2966	13.4569	11.7051
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	98.76	98.76	90.88	68.71	58.27
COLLECTION_DATE	8/14/2003	8/14/2003	8/20/2003	8/20/2003	9/1/2003
RECEIPT_DATE	8/18/2003	8/18/2003	8/21/2003	8/22/2003	9/3/2003
COMPOSITE_DATE	9/4/2003	9/4/2003	9/4/2003	9/4/2003	9/10/2003
EXTRACT_DATE	11/6/2003	11/6/2003	11/6/2003	11/6/2003	11/6/2003
ANALYSIS_DATE	12/19/2003	12/16/2003	12/18/2003	12/18/2003	12/18/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-13	49971-23-19	49971-23-14	49971-23-15	49971-23-16
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT
REPORTING LIMIT (RL)	1.56	0.26	1.09	1.49	1.28

PARAM_NAME		RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL	RESULT	LAB_QUAL
PCB-118		224.99		70.78		42.39		49.92		34.05	
PCB-132		114.32		34.45		26.06		39.45		23.66	
PCB-122	U	U U							U		U
PCB-188	U	U U							U		U
PCB-114	U	U U							U		U
PCB-133	U	U U							U		U
PCB-179		17.12		10.94		10.06		17.13		7.84	
PCB-165	U	U U							U		U
PCB-146		50.46	C	16.90	C	12.32	C	27.98	C	15.60	C
PCB-105		73.62		28.99		20.80		18.45		12.32	
PCB-184	U	U U							U		U
PCB-161			C146		C146		C146		C146		C146
PCB-176		4.71		2.60		2.66		4.76		3.26	
PCB-153		291.96	C	137.92	C	85.12	C	154.06	C	89.56	C
PCB-168			C153		C153		C153		C153		C153
PCB-141		50.69		21.31		14.12		26.33		14.92	
PCB-186	U	U U							U		U
PCB-130		21.66		8.26		5.48		8.28		4.62	
PCB-127	U	U U							U		U
PCB-137		40.90	C	14.30	C	10.02	C	13.30	C	10.02	C
PCB-164			C137		C137		C137		C137		C137
PCB-163			C129		C129		C129		C129		C129
PCB-138			C129		C129		C129		C129		C129
PCB-129		347.96	C	149.92	C	95.16	C	133.48	C	79.88	C
PCB-160			C129		C129		C129		C129		C129
PCB-158		33.44		10.88		9.25		10.77		6.49	
PCB-178		11.77		8.24		5.08		8.98		3.83	
PCB-175		1.44	J	0.97		0.96	J	1.23	J		U
PCB-126	U	U U							U		U
PCB-166			C128		C128		C128		C128		C128
PCB-128		56.42	C	22.28	C	15.54	C	15.84	C	10.50	C
PCB-187		47.84		38.32		29.79		50.93		22.78	
PCB-182		0.60	J		U	0.41	J	0.60	J		U
PCB-183			C174		C174		C174		C174		C174
PCB-185			C174		C174		C174		C174		C174
PCB-159	U	U U							U		U
PCB-174		63.33	C	38.16	C	34.05	C	56.85	C	29.07	C
PCB-162			U	1.03			U		U		U
PCB-177		24.67		13.68		11.50		21.44		10.31	
PCB-202		8.44		5.81		7.05		7.41		1.78	
PCB-167		10.06		6.22		3.18		4.60		2.97	
PCB-181	U	U U							U		U
PCB-171		14.00	C	6.88	C	6.46	C	11.56	C	4.66	C
PCB-173			C171		C171		C171		C171		C171
PCB-201		3.10		2.37		3.12		2.97		1.04	J
PCB-156		30.86	C	15.64	C	7.14	C	8.28	C	5.66	C
PCB-157			C156		C156		C156		C156		C156
PCB-204	U	U U							U		U
PCB-197		3.54	C	1.72	C	2.04	C	2.74	C		CU
PCB-200			C197		C197		C197		C197		C197
PCB-172		13.56		6.03		4.18		6.95		3.18	
PCB-192	U	U U							U		U
PCB-193			C180		C180		C180		C180		C180
PCB-180		91.98	C	59.42	C	48.84	C	78.82	C	34.04	C
PCB-191		1.60		1.13		1.10			U		U
PCB-170		44.39		26.21		19.89		30.73		14.03	
PCB-190		10.63		7.31		4.07		7.22		2.20	
PCB-169			U	0.86			U		U		U
PCB-198		27.72	C	25.50	C	27.00	C	23.98	C	6.78	C
PCB-199			C198		C198		C198		C198		C198
PCB-196		9.78		7.16		7.84		7.22		3.17	
PCB-203		17.07		13.82		17.42		13.45		3.48	
PCB-208		7.37		6.32		8.53		18.13			U
PCB-195		7.60		5.16		4.69		5.84		1.61	
PCB-189		1.79		1.94		1.05	J	1.34	J		U
PCB-207		3.59		3.27		3.37		3.73			U
PCB-194		21.34		15.26		17.02		17.73		5.58	
PCB-205		1.05	J	2.19		0.65	J	0.78	J		U
PCB-206		21.93		17.50		28.76		47.16		2.14	
PCB-209			U	12.10		12.72		63.96		1.70	

J = reported value < Reporting Limit (RL
U = not detected.
RL = the low calibration level adjusted f
& = outside QC limits.

APPENDIX F

LITERATURE REVIEW OF CDD, CDF, PCB, AND MERCURY LEVELS IN SOIL

Appendix F: Literature Review of CDD, CDF, PCB, and Mercury Levels in Soil

This appendix provides a literature review of studies reporting CDD, CDF, PCB, and mercury levels in soil. The review focuses mainly on rural soils in the U.S., but it includes some information on studies from other countries as well. Section 1 (CDDs and CDFs) was excerpted (with minor editing/updating) from the U.S. Environmental Protection Agency's (EPA's) draft dioxin reassessment (U.S. EPA, 2000). It should be noted that the studies included in this review have a wide variety of design features (e.g., detection limits, treatment of nondetects in deriving statistics, congener inclusion, sampling procedures, analytical techniques) that make them difficult to compare on a completely equal basis. Information is provided to help readers consider these differences, but no adjustments were made to the values reported in the original studies.

1. CDDs and CDFs

1.1. North American Data

Soil sampled in 1987 from the vicinity of a sewage sludge incinerator was compared with soil from rural and urban sites in Ontario, Canada, by Pearson et al. (1990). Soil in the vicinity of the incinerator showed a general increase in CDD concentration with increasing degree of chlorination (Table 1). Of the CDFs, only OCDF was detected (mean concentration, 43 ppt). Rural wood lot soil samples contained only OCDD (mean concentration, 30 ppt). Soil samples from undisturbed urban parkland settings revealed only HpCDDs and OCDD, but all CDF congener groups (Cl4 to Cl8) were present. Those samples showed an increase in concentration from the HpCDDs to OCDD and PeCDFs to OCDF. TCDFs had the highest mean value (29 ppt) of all the CDF congener groups. Resampling of one urban site in 1988, however, showed high variability in the concentrations of CDDs and CDFs.

Reed et al. (1990) analyzed background soil samples from a semi-rural location in Elk River, MN, as part of a baseline assessment prior to the operation of a refuse-derived fuel-powered electric generation station. Four soil samples (two from an untilled site and two from a tilled site) were collected and analyzed for CDD/CDFs. Of the CDD/CDF congeners, OCDD concentrations were the highest, ranging from 340 ppt to 3,300 ppt. OCDF concentrations ranged from nondetect (ND) to 270 ppt. The 2,3,7,8-tetra- and-penta-chlorinated congeners were not detected in any of the samples analyzed (Table 2).

Data were collected on CDD and CDF levels in soil samples from industrial, urban, and rural sites in Ontario and some U.S. midwestern states (Birmingham, 1990). CDD/CDF levels in rural soils were primarily ND, although the HpCDDs and OCDD were found in a few samples. In urban soils, the tetra through octa homologue groups were measured for both CDDs and CDFs. The HpCDDs and OCDD dominated and were two orders of magnitude greater than in the rural soils. These soils also contained measurable quantities of the TCDDs and PeCDDs. Industrial soils did not contain any TCDDs or PeCDDs, but they contained the highest levels of the TCDFs, HpCDFs, and OCDF. Total CDD/CDF concentrations averaged 73 ± 50 ppt in rural soils (n = 30), 2,075 ± 3,608 ppt in urban soils (n = 47), and 8,314 ± 9,955 ppt in industrial soils (n = 20) when NDs were assumed to be zero. I-TEQ_{DF}s were also calculated for these three types of sites by Birmingham (1990) by assuming that the 2,3,7,8-substituted CDD/CDF congeners

Table 1. Mean CDD and CDF concentrations (ppt) in Canadian soil in 1987[a]

Homolog group	Soil near sludge incinerator (n = 12)	Urban background (n = 11)	Rural background (n = 26)
TCDDs	69 (ND–430)	ND	ND
PeCDDs	81 (ND–540)	ND	ND
HxCDDs	9 (ND–70)	ND	ND
HpCDDs	43 (ND–300)	31 (ND–140)	ND
OCDD	570 (ND–1,500)	1,461 (ND–11,000)	30 (ND–100)
Total CDDs	772 (ND–2,770)	1,492 (ND–11,140)	30 (ND–100)
TCDFs	ND	29 (ND–120)	ND
PeCDFs	ND	1 (ND–10)	ND
HxCDFs	ND	7 (ND–35)	ND
HpCDFs	ND	9 (ND–60)	ND
OCDF	43 (ND–230)	16 (ND–160)	ND
Total CDFs	43 (ND–230)	65 (ND–262)	ND

[a] Data collected in 1987 in Ontario Canada; range presented in parentheses.

ND = not detected

Source: Pearson et al. (1990).

represent specified proportions of the homologue group concentrations and by applying I-TEF$_{DF}$s. Birmingham estimated the I-TEQ$_{DF}$s to be 0.4 ± 0.6 ppt for rural soil, 11.3 ± 21.8 ppt for urban soils, and 40.8 ± 33.1 for industrial soils.

Nine background soil samples were collected from the Yarmouth Pole Yard site located in Yarmouth, ME (Tewhey Associates, 1997). One of these samples, collected from soil near the base of a utility pole, yielded an I-TEQ$_{DF}$ concentration of 57,000 pg/g. The I-TEQ$_{DF}$ concentrations for the other eight samples ranged from 0.73 pg/g to 5.9 pg/g when NDs were assumed to be zero and 1.46 pg/g to 6.07 pg/g when NDs were assumed to be one-half the detection limit. These samples are from rural background locations. The mean I-TEQ$_{DF}$ for these eight samples was 3.58 pg/g (TEQ$_{DF}$WHO98 was 2.89 pg/g) when NDs were set to zero and 3.93 pg/g when NDs were set to one-half the detection limit. The sample collected near the utility pole was not included in these mean TEQ values because its results were not considered to be representative of typical rural background concentrations.

In an effort to determine whether incineration of municipal waste influenced CDD/CDF levels in the immediate area of waste incineration facilities, soil samples were collected from cities with and without operating incinerators throughout Connecticut. Between the years of 1987 and 1990, 34 soil samples were collected from eight different Connecticut cities where no municipal waste incinerators were operating (MRI, 1992). These pre-operational samples were considered to be representative of rural background concentrations. The total I-TEQ$_{DF}$ reported for these samples was 6.07 pg/g, with NDs assumed to be one-half the detection limit. When the

Table 2. Dioxin/furan levels (ppt) in four background soil samples from Elk River, Minnesota[a]

Congener	Tilled (n = 2)	Untilled (n = 2)
2,3,7,8-TCDD	ND	ND
Total TCDD	ND	ND
1,2,3,7,8-PeCDD	ND	ND
Total PeCDD	ND–38	ND
1,2,3,4,7,8-HxCDD	ND	ND
1,2,3,6,7,8-HxCDD	ND	ND–14
1,2,3,7,8,9-HxCDD	ND–8.7	ND–9.9
Total HxCDD	12–99	29–53
1,2,3,4,6,7,8-HpCDD	37–360	78–300
Total HpCDD	62–640	150–530
OCDD	340–3,300	680–2,300
2,3,7,8-TCDF	ND	ND
Total TCDF	ND–1.2	ND
1,2,3,7,8-PeCDF	ND	ND
2,3,4,7,8-PeCDF	ND	ND
Total PeCDF	ND–41	18–45
1,2,3,4,7,8-HxCDF	ND	ND
1,2,3,6,7,8-HxCDF	ND	ND
2,3,4,6,7,8-HxCDF	ND	ND–7.1
1,2,3,7,8,9-HxCDF	ND	ND
Total HxCDF	6.7–86	20–150
1,2,3,4,6,7,8-HpCDF	11–80	26–72
1,2,3,4,7,8,9-HpCDF	ND	ND
Total HpCDF	30–260	30–82
OCDF	ND–270	60–120

[a] Detection limits varied from 0.75 to 2.9 ppt on a congener-specific basis.

ND = Not detected.

Source: Reed et al. (1990).

total TEQ was recalculated in units of TEQ_{DF}-WHO98, the total TEQ for these samples was 5.74 pg TEQ_{DF}-WHO98/g. The proportion of NDs ranged from 3 to 11% of samples for each analyte, with the exception of 2,3,7,8-TCDD and 1,2,3,7,8,9-HxCDF, which had 56 and 49% NDs, respectively (MRI, 1992).

The Ministry of Environment in British Columbia conducted a 2-year monitoring study during 1990/1991 and 1991/1992 to evaluate the levels of CDD/CDF contamination in various types of environmental media (BC Environment, 1995). Soil samples were collected from sites close to a source (primary sites) in the receiving environment adjacent to a suspected source (secondary sites) and in areas not expected to be contaminated (background). Primary and secondary sources were identified as chemical or combustion sources. Chemical sources included sites associated with chlorophenol, herbicide, or PCB contamination; oil refineries; pulpmill landfills; or sewage facilities. Combustion sources included biomedical, industrial, municipal, or sewage sludge incineration; PCB or forest fires; pulp mill boilers; salt-laden wood burning, wood waste burners, or slash burning; and scrap iron yards or smelters. The highest mean concentrations of 2,3,7,8-TCDD and 2,3,7,8-TCDF were observed in primary and secondary soils associated with chemical sources (Table 3). For the 53 background samples, 2,3,7,8-TCDD was not detected, and 2,3,7,8-TCDF concentrations ranged from nondetected to 3.2 ppt. For the purposes of calculating I-TEQ_{DF} values for this study, nondetects were set to zero. I-TEQ_{DF}s were highest among samples associated with primary and secondary chemical sources (Table 3). The mean I-TEQ_{DF} for the background soil samples was 5.0 ppt (BC Environment, 1995). When the mean TEQ was recalculated in units of TEQ_{DF}-WHO98, the total TEQ for these samples was 4.4 pg TEQ_{DF}-WHO98/g.

Grundy et al. (1995) and Bright et al. (1995) collected soil samples from remote locations in the Canadian Arctic as part of an environmental assessment of abandoned military installations in the Canadian North. Four soil samples from remote pristine areas (i.e., at least 20 km away from any human activity) were analyzed for CDDs/CDFs. The total I-TEQ concentrations for these samples ranged from 0.2 to 0.9 ppt (Grundy et al., 1995). Of the CDD/CDF homologue groups, OCDD and TCDF levels were the highest among these remote soil samples, and the HxCDFs made up the smallest portion of the total CDD/CDF concentrations (Bright et al., 1995).

EPA conducted a 2-year nationwide study to investigate the national extent of 2,3,7,8-TCDD contamination (U.S. EPA, 1987). Results of this large study were summarized broadly in the primary reference (i.e., the number and types of samples per site and range of detection). The method used to analyze samples for five of the seven study “tiers” had a detection limit in soil, sediment, and water of 1 ppb. (Each tier of sites is a grouping of sites with a common past or present use [e.g., industrialized, pristine]). Only Tier 5 (sites where pesticides derived from 2,4,5-trichlorophenol had been or were being used for commercial purposes) and Tier 7 (ambient sampling for fish and soil) had detection limits of 1 ppt.

Seventeen of the 221 urban soil sites and 1 of the 138 rural sites from Tier 7 (background sites not expected to have contamination) had soil concentrations exceeding 1 ppt. The highest concentration detected (11.2 ppt) was found in an urban sample. Results from Tier 7 are consistent with the other studies discussed in this chapter regarding soil concentrations of 2,3,7,8-TCDD in nonindustrial settings.

Rappe et al. (1995) and Fiedler et al. (1995) analyzed soil samples collected from rural

Table 3. Dioxin/furan levels in British Columbia soils

Sample category[a]	Dioxin and furan concentrations (pg/g)[b]		I-TEQ_{DF} (pg/g)[b,c]	
	Range	Mean[d]	Range	Mean[d]
Background soil				
2,3,7,8-TCDD	ND	ND (53)	0.0–57.0	5.0 (53)[e]
2,3,7,8-TCDF	ND–32.0	3.2 (53)		
Primary soil (all sources)				
2,3,7,8-TCDD	ND–85.0	5.2 (31)	0.0–2580.0	252.3 (31)
2,3,7,8-TCDF	ND–520.0	47.9 (31)		
Primary soil (chemical sources)				
2,3,7,8-TCDD	ND–85.0	8.4 (18)	0.0–2580.0	418.5 (18)
2,3,7,8-TCDF	ND–520.0	60.3 (18)		
Primary soil (combustion sources)				
2,3,7,8-TCDD	ND–3.5	0.8 (13)	0.0–125.7	22.3 (13)
2,3,7,8-TCDF	ND–160.0	30.7 (13)		
Secondary soil (all sources)				
2,3,7,8-TCDD	ND–550.0	5.4 (137)	0.0–18721.8	241.7 (137)
2,3,7,8-TCDF	ND–550.0	25.1 (137)		
Secondary soil (chemical sources)				
2,3,7,8-TCDD	ND–550.0	15.4 (47)	0.0–18721.8	668.6 (47)
2,3,7,8-TCDF	ND–550.0	60.7 (47)		
Secondary soil (combustion sources)				
2,3,7,8-TCDD	ND–5.6	0.09 (90)	0.0–472.6	18.7 (90)
2,3,7,8-TCDF	ND–180.0	6.5 (90)		

[a] Background samples were believed to be indicative of ambient levels of dioxins and furans in the environment. Primary samples were collected immediately at a potential source of contamination. Secondary samples were collected from areas directly impacted by the primary source and could be used to indicate movement of contaminants.

[b] Concentrations in picograms/gram (pg/g) dry weight.

[c] I-TEQ_{DF}s are the sum of 17 2,3,7,8-substituted dioxins and furans after the concentration of each individual dioxin or furan is multiplied by its international toxicity equivalency factor (I-TEF_{DF}). For samples with nondetect levels of a dioxin or furan, zero was used as the concentration for the I-TEQ_{DF} calculation.

[d] Number in parenthesis indicates the number of samples (n) used to calculate mean.

[e] When the total TEQ was recalculated using TEF_{DF}-WHO_{98}s, the TEQ_{DF}-WHO_{98} was 4.4 pg/g.

ND = not detected

Source: BC Environment (1995).

sites in southern Mississippi for CDDs and CDFs. Sites not directly impacted by human activities such as heavy traffic or dust were selected. A total of 36 composite soil samples from eight Mississippi counties were analyzed. The I-TEQ_{DF} concentration of CDDs/CDFs in soil ranged from 0.16 to 22.9 ppt dry mass (Fiedler et al., 1995). The mean I-TEQ$_{DF}$ concentration was 3.1 ppt dry mass and the median I-TEQ$_{DF}$ concentration was 0.8 ppt dry mass (Fiedler et al., 1995). CDDs were found at higher concentrations than were CDFs, and OCDD was the most dominant congener.

Soil samples were collected from the National Institutes of Health (NIH) campus in Bethesda, MD, during 1995 in an effort to determine the effect of 30 years of pathological waste incineration on the campus and its surroundings (NIH, 1995). Thirty-seven samples were collected from the soil at a depth of 6 in. The total I-TEQ$_{DF}$ for these samples was 7.83 pg/g when NDs were assumed to be zero and 8.49 pg/g, when NDs were assumed to be one-half the detection limit. OCDD, at a I-TEQ$_{DF}$ concentration of 6.29 pg/g, was the principal contributor to the total I-TEQDF for these samples, regardless of whether NDs were assumed to be zero or one-half the detection limit. It should be noted that using the new TEF$_{DF}$-WHO98s, the TEQ for OCDD would be 10 times lower (i.e., 0.63 pg/g). This reduction would also result in a significant decrease in the total TEQ. The total TEQ$_{DF}$-WHO98 would be 2.21 pg/g when NDs were set to zero. Samples were also collected at depths of 12 and 24 in for comparison with levels found in the shallow (6-in) samples. Although CDD/CDF concentrations found at the surface indicate deposition, strong correlation with I-TEQ$_{DF}$ concentrations at the deeper depths were observed. This seemed to indicate either long-term presence of the source (i.e., greater than 40 years) or soil mixing that occurred either during or after deposition. An expert panel (comprised of toxicologists, chemists, soil scientists, engineers, risk assessors, and public health professionals) concluded that the levels of I-TEQ$_{DF}$ in the samples were low and not significantly different from background. Thus, these samples are assumed to be representative of urban background concentrations. The spatial pattern of I-TEQ$_{DF}$ concentrations showed no particular trends that could be related to the incinerator. Other anthropogenic activities, such as vehicular traffic, other medical waste incinerators not related to NIH, and fireplaces burning in the vicinity, may have contributed to the deposition (NIH, 1995).

Soil samples were collected by EPA (U.S. EPA, 1996) in the vicinity of a municipal waste-to-energy facility in Columbus, OH, to determine whether surface soils around the incinerator contained higher CDD/CDF levels than did soils collected from background sites. The facility is not currently in operation, but CDD/CDF residues may be present in the soil near the facility as a result of past emissions. Samples were collected (1) on site, (2) from urban background locations near the incinerator, and (3) from areas remote from the facility (i.e., rural background sites). The results of the analyses indicated that soil from the rural background sites had the lowest I-TEQ$_{DF}$ concentrations and on-site samples had the highest I-TEQ$_{DF}$ concentrations (Table 4). For rural background soil samples, total I-TEQ$_{DF}$s ranged from 0.9 to 1.3 ppt ($n = 3$) with a mean of 1.1 ppt (TEQ$_{DF}$-WHO98 = 0.9 ppt) when NDs were assumed to be zero and 1.0 to 2.0 ppt with a mean of 1.4 ppt (TEQ$_{DF}$-WHO98 = 1.3 ppt) when NDs were set to one-half the detection limit. Total I-TEQ$_{DF}$s for urban background soils ranged from approximately 3 to 60 ppt ($n = 18$) with a mean of 19 ppt (TEQ$_{DF}$-WHO98 = 21 ppt) when NDs were set to either zero or one-half the detection limit. For on-site samples, all 2,3,7,8-CDD/CDF congeners were detected in all samples ($n = 4$). Total I-TEQ$_{DF}$ concentrations ranged from 50 to 760 ppt with a mean of 356 ppt (TEQ$_{DF}$-WHO98 = 444 ppt). Additional detail and analyses of these data are presented in Lorber et al. (1998a).

Table 4. Number of positive soil samples and CDD/CDF concentrations in background, urban, and impacted sites near a waste-to-energy facility in Ohio

Congener	Background		Urban		Impacted	
	No. of positive samples	**Mean concentration (ppt) (NDs = ½ DL)**	**No. of positive samples**	**Mean concentration (ppt) (NDs = ½ DL)**	**No. of positive samples**	**Mean concentration (ppt) (NDs = ½ DL)**
2,3,7,8-TCDD	2/3	0.39	15/18	2.27	3/3	28.5
1,2,3,7,8-PeCDD	0/3	0.14	18/18	6.58	3/3	180.0
1,2,3,4,7,8-HxCDD	1/3	0.35	18/18	6.14	3/3	142.3
1,2,3,6,7,8-HxCDD	3/3	0.82	18/18	10.9	3/3	137.8
1,2,3,7,8,9-HxCDD	3/3	1.23	18/18	10.8	3/3	201.6
1,2,3,4,6,7,8-HpCDD	3/3	17.7	18/18	190.1	3/3	765.2
OCDD	3/3	160.9	18/18	1,560.2	3/3	1495.4
2,3,7,8-TCDF	0/3	0.45	18/18	4.12	3/3	85.9
1,2,3,7,8-PeCDF	0/3	0.17	17/18	5.50	3/3	139.6
2,3,4,7,8-PeCDF	1/3	0.21	17/18	7.56	3/3	199.9
1,2,3,4,7,8-HxCDF	1/3	0.19	15/18	8.06	3/3	196.8
1,2,3,6,7,8-HxCDF	3/3	0.52	17/18	8.12	3/3	209.1
1,2,3,7,8,9-HxCDF	0/3	0.15	6/18	0.51	3/3	11.6
2,3,4,6,7,8-HxCDF	3/3	0.64	18/18	6.99	3/3	156.7
1,2,3,4,6,7,8-HpCDF	3/3	4.06	18/18	41.7	3/3	641.0
1,2,3,4,7,8,9-HpCDF	1/3	0.27	16/18	3.82	3/3	57.9
OCDF	3/3	10.72	18/18	44.3	3/3	184.5
Mean total I-TEQ$_{DF}$, ppt (ND = ½ DL)	--	1.4	--	19.2	--	356.0
Mean total I-TEQ$_{DF}$, ppt (ND = 0)	--	1.1	--	19.2	--	356.0
Mean total TEQ$_{DF}$-WHO$_{98}$, ppt (NDs = ½ DL)	--	1.3	--	21.0	--	444.5
Mean total TEQ$_{DF}$-WHO$_{98}$, ppt (ND = 0)	--	0.92	--	21.0	--	444.5

DL= detection limit
ND = nondetects

Source: U.S. EPA (1996).

Brzuzy and Hites (1995) examined soil cores from four U.S. locations to evaluate the accuracy of using measurements of CDD/CDF homologue groups in estimating the atmospheric flux of these compounds into the environment. Soil cores were collected from undisturbed areas near Shingleton, Grayling, and Verona, MI, and near Mitchell, IN. CDD/CDF concentrations varied according to depth of the soil samples, with deeper samples having lower CDD/CDF concentrations. Approximately 80% of the CDD/CDF load was contained in the top 15 cm of the cores, and CDD/CDF concentrations were close to the detection limit in samples collected at a depth of 20 to 25 cm. Based on the graphs presented in Brzuzy and Hites (1995), total CDD/CDF concentrations in the uppermost 5 cm of the core ranged from approximately 60 to 200 pg/g for the three Michigan sites. CDDs/CDFs in these soil cores were also found to be highly correlated with the organic carbon content of the soil, indicating that organic carbon is an important factor in the sorption of CDDs/CDFs to soil (Brzuzy and Hites, 1995). Higher concentrations of CDDs/CDFs were observed in two cores taken from the Indiana site. Concentrations in the uppermost layer (i.e., 9 cm) of these cores ranged from approximately 700 pg/g to nearly 10,000 pg/g. CDD/CDF concentrations in these cores peaked at a depth of approximately 40 to 50 cm, with concentrations ranging from approximately 1,000 pg/g to more than 20,000 pg/g. Brzuzy and Hites (1995) used the Michigan data to estimate soil-derived CDD/CDF flux rates ranging from 264 ng/m^2/yr for upper Michigan to 663 ng/m^2/yr for lower Michigan. These soil-derived flux estimates were compared with sediment-derived fluxes from previous studies to determine whether soil samples can also be used to accurately predict atmospheric flux. Good agreement for the fluxes to these two media was observed. In addition, the CDD/CDF homologue profiles for soil and sediment were similar.

Washington State Department of Ecology (Rogowski et al., 1999; Rogowski and Yake, 2005) collected soil samples as part of a study of metals and dioxin-like compounds in agricultural fertilizers and soil amendments. Soils were analyzed to evaluate whether these compounds had accumulated as a result of fertilizer use and to assess typical concentrations of dioxin-like compounds in Washington State soils. Each agricultural sample was a composite of 10 subsamples collected from each sampling location to a depth of 5 cm. Each of the other land use samples was a composite of 10 subsamples collected within a 1-acre sampling unit. The sampling units were selected to represent typical or background locations for each land use. The mean results are summarized below (TEQ values based on one-half the detection limit for NDs):

Forest (n = 8): 3.5 pg/g TEQ, 220 pg/g total CDD/CDFs
Open Areas (n = 8): 1.9 pg/g TEQ, 260 total CDD/CDFs
Urban (n = 14): 5.8 pg/g TEQ, 610 total CDD/CDFs
Agriculture (n = 54): 0.99 pg/g TEQ, 42 total CDD/CDFs

These data were used to derive the following values for nonimpacted lands:

	Total CDD/CDFs (pg/g)	TEQs (pg/g)
Forest, noncommercial (n = 4)		
Range	79–426	0.45–5.2
Mean	267	3.3

Open Area, nongrazed (n = 4)		
Range	9–258	0.046–2.4
Mean	94	0.71

EPA, Region 8, conducted a set of four related studies on dioxin-like compounds in surficial soils along the Denver, CO, Front Range. One of these studies (U.S. EPA, 2001) evaluated regional background soil; other sampling efforts included characterization of the Rocky Mountain arsenal using random samples at the site or from historic use sites. A large number of reference soils were collected and analyzed for CDDs/CDFs and dioxin-like PCBs. These data will be used to assess whether the soil concentrations observed in the Western Tier Parcel of the Rocky Mountain Arsenal, an EPA National Priority List site, are higher than regional background levels. EPA, Region 8, collected and analyzed 162 surface soil samples for investigation into background concentrations of dioxin-like compounds at multiple locations within 1,000 square miles of the Denver, CO, Front Range. The multiland-use areas that were sampled were situated on public lands and were categorized as agricultural (n = 27), commercial (n = 31), industrial (n = 29), open space (n = 36), and residential (i.e., within 200 ft of private land) (n = 39). The fine-soil fractions of samples obtained in the upper 2 in of the soil were analyzed for the 17 dioxins and furans and 12 PCBs (77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, and 189). The mean TEQ_{DFP}-WHO98 ranged from less than 1 ppt TEQ to approximately 100 ppt TEQ (with two outliers of 142 and 155 ppt removed—one from a residential site and one from a commercial site). The mean TEQ_{DFP}-WHO98 values were 1.9 ppt for agricultural sites, 8.5 ppt for commercial sites, 15.4 ppt for industrial sites, 2.8 ppt for open space, and 8.6 ppt for residential locations, with a total mean of 7.5 ppt when NDs were set to one-half the detection limit. PCBs made up approximately 20% of the TEQ_{DFP}-WHO98. The TEQ_{DF} values for open space (n = 36) ranged from 0.1 to 9.1 with a mean of 1.6. The analytical values indicate that open space and agricultural lands had the lowest TEQ_{DFP}-WHO98 concentrations and industrial, commercial, and residential locations had slightly higher concentrations. It should be noted that because sieved samples were analyzed, these results may be higher than whether bulk samples had been analyzed. Further testing is being conducted to identify whether the increased total organic carbon content of agricultural and open space soils have a higher affinity for dioxin-like compounds than do other soil types, thereby skewing the analytical results to produce lower than actual values.

The state of Michigan conducted a soil sampling and assessment program in the Tittabawassee/Saginaw River flood plain to determine the source and extent of dioxin contamination (MDEQ, 2003). Elevated concentrations of dioxin were confirmed within the lower Tittabawassee River flood plain near the river's confluence with the Saginaw River. The upstream area levels were found to be consistent with state background levels. These levels were derived from a compilation of statewide data on CDD/CDF TEQ levels in background soils. This dataset of 68 samples has a range of 0.4 to 35 pg/g TEQ and a mean of 6 pg/g TEQ.

Hilscherova et al. (2003) also studied dioxin levels in the Tittabawassee River flood plain and found similar results. The TEQ levels in the downstream flood plain soils were found to average about 800 pg/g TEQ (n = 7), compared with an average of 4.3 pg/g TEQ (n = 3) in upstream flood plains.

1.2. European Data

Soil samples from rural and semi-urban sites in England, Wales, and lowland Scotland showed a general increase in concentration from the TCDDs to OCDD, whereas CDF levels showed very little variation between the congener groups (Creaser et al., 1989). Concentrations of 2,3,7,8-TCDD at those sites ranged from <0.5 to 2.1 ppt. The median values for the TCDDs to OCDD were 6.0, 4.6, 31, 55, and 143 ppt, respectively. The median values for the TCDFs to OCDF were 16, 17, 32, 15, and 15 ppt. Evaluation of soil data from urban sites in the same geographical area showed that the mean levels for the CDD and CDF congeners were significantly greater ($p<0.01$) than those for rural and semi-urban background soils (Creaser et al., 1990). Concentrations of 2,3,7,8-TCDD at the urban sites ranged from <0.5 to 4.2 ppt. The median values for the TCDDs to OCDD were 40, 63, 141, 256, and 469 ppt, respectively. The median values for the TCDFs to OCDF were 140, 103, 103, 81, and 40 ppt. Significantly elevated levels of the lower congeners, together with higher overall CDD/CDF concentrations, indicate that local sources and short-range transport mechanisms are major contributors of CDDs and CDFs to urban soils. Cox and Creaser (1995) evaluated soils from urban and rural locations in the United Kingdom before the introduction of Integrated Pollution Control in 1991. I-TEQ$_{DF}$s for 11 rural locations ranged from 0.78 to 17.48 ppt with a mean of 5.17 ppt, and the I-TEQ$_{DF}$s for 5 urban samples ranged from 4.88 to 87.34 ppt with a mean of 28.37 ppt.

A soil sampling survey in Salzburg, Austria, also showed that the concentrations of CDDs/CDFs were higher in urban and industrial sites than in rural sites (Boos et al., 1992). The total CDD content of the soils ranged from 33.7 to 1236.7 ppt for urban sites, 92.2 to 455 ppt for industrial sites, and 7.1 to 183.6 ppt for rural sites. The total CDF content of the soils ranged from 45.6 to 260.8 ppt for urban sites, 53.0 to 355.3 ppt for industrial sites, and 12.0 to 77.7 ppt for rural sites. I-TEQ$_{DF}$s ranged from 0.1 to 3.1 ppt for rural sites, 1.0 to 8.3 ppt for urban sites, and 3.5 to 11.5 ppt for industrial sites when NDs were assumed to be zero. When NDs were set to one-half the detection limit, I-TEQ$_{DF}$s ranged from 1.3 to 3.8 ppt for rural sites, 2.0 to 8.6 ppt for urban sites, and 4.1 to 12.5 ppt for industrial sites. Rappe and Kjeller (1987) presented data on CDDs/CDFs in soil collected from rural (n = 3) and industrial (n = 2) sites in various parts of Europe. Concentrations were higher among industrial soils than in rural soils for all of the CDD/CDF homologue groups, and the hepta-chlorinated compounds made up the largest portion of the total CDD/CDF concentrations in both rural and industrial samples. HpCDDs ranged from ND to 17 ppt in rural samples and 370 to 1,600 ppt in industrial samples. HpCDFs ranged from 14 to 22 ppt in rural soils and 260 to 4,500 ppt in industrial soils.

Rotard et al. (1994) measured CDDs/CDFs in soil samples collected from forest, grassland, and plowland sites in western Germany. The highest mean concentrations of CDDs/CDFs were found in the subsoil and topsoil layers of deciduous (38.0 ng I-TEQ$_{DF}$/kg dry matter; n = 9) and coniferous forests (36.9 ng I-TEQ$_{DF}$/kg dry matter; n = 11). Grassland and plowland sites had mean concentrations of 2.3 ng I-TEQ$_{DF}$/kg dry matter (n = 7) and 1.7 ng I-TEQ$_{DF}$/kg dry matter (n = 14), respectively.

Stenhouse and Badsha (1990) collected baseline data for soils around a site proposed for a chemical waste incinerator in Great Britain. All of the 2,3,7,8-substituted CDD/CDF congeners except PeCDD were detected in all samples. Concentrations were highest for the octa-chlorinated CDDs/CDFs. Background I-TEQ_{DF} concentrations ranged from 3 to 20 ppt. The mean I-TEQ_{DF} concentration was 8 ppt (n = 12) with a standard deviation of 4 ppt.

Buckland et al. (1998) evaluated soils collected in New Zealand. Dry weight CDD/CDF concentrations ranged from 0.17 to 1.99 pg I-TEQ_{DF}/g for pristine soils, 0.17 to 0.90 pg I-TEQ_{DF}/g for agricultural soils, and 0.52 to 6.67 pg I-TEQ_{DF}/g for urban soils. The congeners below the detection limit were included in the total TEQ using one-half their detection limits.

2. PCBs

Relatively little data could be found on total PCB levels in rural areas of the U.S. The EPA Region 8 survey (U.S. EPA, 2001) measured the coplanar PCBs in background areas but did not measure total PCBs (see discussion above on TEQ levels).

Wilcke and Amelgung (2000) measured 14 PCBs at 18 grassland sites in the Great Plains of North America. Sites were located in Texas, Kansas, Colorado, Wyoming, Montana, North Dakota, Minnesota, and Saskatchewan (Canada). Samples were collected in the late spring of 1994. Composite samples at a depth of 0 to10 cm were collected at each site. Measured PCB congeners were numbers 1, 8, 20, 28, 35, 52, 101, 118, 138, 153, 180, 199, 206, and 209. The PCB congener sum was 3136 ng/g at one site and at the others ranged from 7.9 to 92.8 ng/g. No correlation was observed between the PCBs and soil organic matter.

Between 1994 and 1995, house dust and yard soil from 34 homes surrounding New Bedford Harbor, MA, were analyzed for PCB concentrations during the dredging of PCB-contaminated sediments (Vorhees et al., 1999). House dust samples were collected from the carpet, and yard soil was collected from the main entryway. The results indicated that concentrations in house dust samples were 10 times higher (260–23,000 ng/g) than yard soil concentrations (15–1,800 ng/g).

Hwang et al. (1999) conducted a PCB soil survey at the Mohawk Nation at Akwesasne, located along the St. Lawrence River in northern New York. Although this is a generally rural area, it is located within 10 miles of several large industrial sources with known PCB releases. All samples were collected in residential yards. Total PCBs averaged 48 ng/g (n = 106, SD = 119 ng/g).

In the Canadian Arctic, a string of 21 radar stations called The Distant Early Warning (DEW) Line stretches along 3,000 km and has been in operation since the 1950s. These radar stations have been associated with former PCB use and contamination (Bright et al. 1995a, b). Site samples from the 21 DEW Line facilities and three additional Arctic radar installations were collected from 1989 to 1992. PCBs were detected in undisturbed soils near the 21 DEW Line sites and as far as 5 km but were not detected in soil 20 km from site. Concentrations ranged from not detected (detection limit, 0.1–5.3 ng/g) to 45 ng/g in soil. These data indicate short-range redistribution of PCBs in a terrestrial environment.

Meijer et al. (2003) presented data from a survey of PCBs and hexachlorobenzene concentrations in 191 global background surface (0–5 cm) soils. Differences of up to four orders of magnitude were found between sites for PCBs. The lowest and highest PCB concentrations (26 and 97,000 pg/g dw) were found in samples from Greenland and mainland Europe (France, Germany, Poland), respectively. The mean total PCB level was 5,410 pg/g. Background soil

PCB concentrations were strongly influenced by proximity to source, region and soil organic matter content.

Masahide et al. (1998) examined soil samples collected at the depth of 0 to 10 cm from various sites located in Poland between 1990 and 1994. The mean dry weight total PCB concentration was 5.4 ng/g for agricultural soils (seven sites) and 15 ng/g for forest soils (four sites), 170 ng/g (n = 31) for urban soils, and 900 ng/g for the soils sampled at a military area. Dry weight PCB concentrations increased from 21 ng/g in Northern Poland to 48–380 ng/g in highly populated and industrialized regions in southern Poland.

Another rural soil survey was conducted in Poland in 2002 (Wyrzykowska et al., 2005). This study sampled soils in 13 agricultural areas and found a range of 0.054 to 0.42 pg TEQ/g and an average of 0.18 pg TEQ/g.

Buckland et al. (1998) evaluated soils collected in New Zealand. The PCB concentrations ranged from 0.067 to 2.3 pg TEQ_P/g (the TEFs used for PCBs were not identified) for provincial centers and 0.087 to 1.33 pg TEQ_P/g for metropolitan centers. The congeners below the detection limit were included in the total TEQ using one-half their detection limits.

3. Mercury

Mercury occurs naturally as a mineral and is distributed throughout the environment by both natural and anthropogenic processes. In a review of the mercury content of virgin and cultivated surface soils from a number of countries, it was found that the average concentrations ranged from 20 to 625 ng/g (ATSDR, 1999). Soil mercury levels are usually less than 200 ng/g in the top soil layer, but values exceeding this level are not uncommon, especially in areas affected by anthropogenic activities (U.S. EPA, 1997). NOAA (1999) reported that background mercury levels in natural soils of the U.S. ranged from ND to 4600 ng/g with a geometric mean of 58 ng/g.

The state of New Jersey formed a mercury task force in 1998 to review current science, inventory sources, estimate impacts, review policies, and recommend emission controls (NJDEP, 2001). This report states that mercury levels in rural New Jersey ranged from <10 to 260 ng/g with a median of <10 (n = 35). The report also provides “background” levels from other states in the range of 1 to 876 ng/g, but it is unclear how many of these are based on true rural areas.

Mercury was detected at soil concentrations ranging from 10 to 550 ng/g in orchard soils in New York State (Merwin et al., 1994) in a study primarily aimed at measuring lead and arsenic. Lead arsenate was used for pest control in fruit orchards for many years in the U.S., and its residues remain in most of these soils. Because arsenic and lead are toxic and only slightly mobile in soils, an analytical survey was conducted in 1993 to determine the concentrations of these elements persisting in soil samples from 13 older and newer orchards in New York State. Mercury levels were found to correlate with both arsenic and lead. Given the activities at these orchards, the mercury in these soils may be elevated over those of other rural areas.

Glass et al. (1990) measured mercury concentrations in precipitation, lake water and sediment, zooplankton, and fish and examined extensive watershed and lake chemistry data for 80 lake watersheds in northeastern Minnesota, including the Superior National Forest, Voyageurs National Park, and Boundary Waters Canoe Area Wilderness. They reported that mercury levels in soils from this region ranged from 12 to 220 ng/g. They also measured mercury in bedrock (gabbros and granites) from this area, finding levels ranging from 5 to 16 ng/g. They also cite other studies indicating that soils away from mercury deposits had concentrations ranging from 20 to 150 ng/g and averaging 70 ng/g.

The state of Michigan has compiled a data set of metal levels in background (unimpacted) soils (MDEQ, 2005). This data set includes 431 samples that were analyzed for mercury. Eighty-three percent of the samples were less than detection limits, the median was <100 ng/g, and the range encompassing 95% of the data points was <25 to 600 ng/g.

Tack et al. (2005) measured mercury levels in baseline soils in Belgium. The soils sampled included agricultural fields, forest, pasture, and fallow land. Sampling depth was 20 cm. The mean concentration was 240 ng/g (n = 316) and the range was 30 to 4,190 ng/g. They also reported background levels from a variety of countries, with levels ranging from 0.8 to 190 ng/g.

Washington State Department of Ecology (Rogowski et al., 1999; Rogowski and Yake, 2005) collected soil samples as part of a study of metals and dioxin-like compounds in agricultural fertilizers and soil amendments (see earlier discussion in Section 1.1). Mercury levels at background sites (n = 13) ranged from <3.2 to 66 ng/g with a mean of 11 ng/g.

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APPENDIX G

PHYSICAL/CHEMICAL PARAMETER DATA

Site	Sample ID	Moisture Content[a] (%)	Grain Size[b] Distribution			pH[c]	Total Organic Content[d]	
			% Finer #4 Sieve	% Finer #200 Sieve	% Finer 0.005mm		Result (mg/kg)	Reporting Limit (mg/kg)
Penn Nursery, PA	EPA-1-1	24.6	81.7	38.5	14.7	3.7	50,500	1,000
	EPA-1-2	26.4	88.0	40.2	15.4	3.8	47,000	1,000
	EPA-1-3	28.3	100.0	48.0	15.7	3.7	68,700	1,000
	EPA-1-4	31.3	68.7	25.7	10.4	3.9	76,200	1,000
	EPA-1-5	25.1	86.6	38.3	15.2	3.5	56,600	1,000
Clinton Crops, NC	EPA-2-1	8.0	100.0	15.1	4.5	6.1	9,100	1,000
	EPA-2-2	9.5	100.0	14.1	4.1	5.2	9,100	1,000
	EPA-2-3	17.1	100.0	21.2	7.3	5.4	12,000	1,000
	EPA-2-4	11.7	100.0	14.4	4.7	5.2	9,700	1,000
	EPA-2-5	8.6	100.0	11.8	3.2	4.3	5,400	1,000
Everglades, FL	EPA-4-1	154.0	100.0	68.3	25.2	7.1	120,000	1,000
	EPA-4-2	85.7	100.0	63.1	22.7	7.7	87,000	1,000
	EPA-4-3	88.4	100.0	46.8	17.5	7.8	82,000	1,000
	EPA-4-4	46.3	100.0	60.7	23.6	7.8	84,000	1,000
	EPA-4-5	107.9	100.0	57.1	19.6	7.7	84,000	1,000
Lake Dubay, WI	EPA-5-1	21.0	100.0	71.1	12.4	5.7	18,500	1,000
	EPA-5-2	19.0	99.2	66.3	10.3	5.8	15,900	1,000
	EPA-5-3	14.9	99.1	68.2	10.5	5.9	14,800	1,000
	EPA-5-4	16.5	98.5	67.0	10.6	5.8	19,300	1,000
	EPA-5-5	19.3	98.9	64.3	10.5	6.0	17,800	1,000
Monmouth, IL	EPA-6-1	12.3	100.0	94.4	23.2	7.1	47,700	1,000
	EPA-6-2	10.1	100.0	95.0	20.7	6.7	47,200	1,000
	EPA-6-3	9.5	100.0	97.3	25.7	6.6	22,700	1,000
	EPA-6-4	6.0	100.0	97.8	31.3	6.6	15,700	1,000
	EPA-6-5	37.2	100.0	95.2	27.6	6.9	56,600	1,000

Site	Sample ID	Moisture Content[a] (%)	Grain Size[b] Distribution			pH[c]	Total Organic Content[d]	
			% Finer #4 Sieve	% Finer #200 Sieve	% Finer 0.005mm		Result (mg/kg)	Reporting Limit (mg/kg)
McNay Farms, IA	EPA-7-1	23.5	100.0	94.7	36.2	6.1	49,900	1,000
	EPA-7-2	28.0	100.0	92.3	35.7	5.9	50,000	1,000
	EPA-7-3	26.1	100.0	95.3	50.5	6.4	50,700	1,000
	EPA-7-4	26.3	100.0	94.1	40.1	6.3	38,700	1,000
	EPA-7-5	18.8	100.0	69.4	35.2	6.8	37,400	1,000
Lake Scott, KS	EPA-8-1	22.3	100.0	77.6	16.0	7.4	19,300	1,000
	EPA-8-2	18.5	100.0	72.9	13.8	7.5	22,800	1,000
	EPA-8-3	29.9	100.0	76.5	16.9	7.3	21,100	1,000
	EPA-8-4	35.2	100.0	68.9	16.0	7.3	19,300	1,000
	EPA-8-5	39.2	100.0	75.3	19.2	6.8	18,900	1,000
Keystone State Park, OK	EPA-9-1	22.9	99.8	70.4	16.7	7.4	14,200	1,000
	EPA-9-2	19.2	100.0	66.5	14.8	6.5	12,800	1,000
	EPA-9-3	7.2	99.0	66.9	12.8	7.2	11,700	1,000
	EPA-9-4	4.8	94.8	42.2	10.5	7.8	43,500	1,000
	EPA-9-5	10.3	96.5	55.9	12.4	7.7	27,700	1,000
Arkadelphia, AR	EPA-10-1	9.2	100.0	58.4	15.4	6.2	15,900	1,000
	EPA-10-2	13.6	100.0	66.4	15.2	5.7	18,700	1,000
	EPA-10-3	9.3	99.4	39.7	12.6	5.6	15,500	1,000
	EPA-10-4	7.1	99.8	55.4	16.4	5.7	15,200	1,000
	EPA-10-5	13.4	100.0	67.4	19.7	5.0	24,900	1,000
Bennington, VT	EPA-11-1	30.7	99.7	63.3	9.8	5.8	33,700	1,000
	EPA-11-2	29.5	98.5	58.5	8.2	5.8	28,300	1,000
	EPA-11-3	23.6	99.6	66.0	9.5	5.6	39,200	1,000
	EPA-11-4	27.4	100.0	53.2	3.5	5.0	32,600	1,000
	EPA-11-5	11.1	92.1	23.2	5.2	7.5	35,500	1,000
Jasper, NY	EPA-12-1	40.3	94.3	74.9	23.3	4.5	49,300	1,000
	EPA-12-2	38.5	100.0	82.2	26.7	4.6	54,700	1,000
	EPA-12-3	42.5	100.0	83.1	22.8	4.5	51,500	1,000
	EPA-12-4	41.7	99.0	82.1	24.9	4.5	51,300	1,000
	EPA-12-5	42.2	100.0	82.0	27.9	4.3	55,700	1,000
Caldwell, OH	EPA-14-1	18.6	100.0	87.6	38.5	4.5	44,700	1,000
	EPA-14-2	28.6	100.0	90.2	56.1	4.3	69,800	1,000

Site	Sample ID	Moisture Content[a] (%)	Grain Size[b] Distribution			pH[c]	Total Organic Content[d]	
			% Finer #4 Sieve	% Finer #200 Sieve	% Finer 0.005mm		Result (mg/kg)	Reporting Limit (mg/kg)
	EPA-14-3	18.8	91.0	79.9	36.9	4.0	36,500	1,000
	EPA-14-4	22.7	100.0	84.5	38.7	4.3	44,600	1,000
	EPA-14-5	16.6	100.0	90.2	39.9	4.7	25,400	1,000
Dixon Springs, IL	EPA-16-1	14.3	100.0	96.7	21.1	5.7	25,400	1,000
	EPA-16-2	11.0	100.0	94.3	14.3	5.7	20,000	1,000
	EPA-16-3	12.7	100.0	94.9	21.2	5.9	21,500	1,000
	EPA-16-4	20.7	100.0	95.6	18.4	5.9	21,800	1,000
	EPA-16-5	22.9	100.0	94.9	16.1	7.1	31,100	1,000
Quincy, FL	EPA-17-1	33.4	100.0	29.4	13.3	5.1	24,200	1,000
	EPA-17-2	45.0	100.0	25.8	10.5	4.9	31,200	1,000
	EPA-17-3	33.3	100.0	28.1	12.1	4.9	17,200	1,000
	EPA-17-4	45.2	100.0	29.0	14.5	4.7	29,100	1,000
	EPA-17-5	41.2	100.0	30.3	13.4	4.8	32,700	1,000
Bay St, Louis, MS	EPA-18-1	33.4	100.0	57.3	13.1	6.5	31,800	1,000
	EPA-18-2	18.2	100.0	52.4	11.9	6.2	25,200	1,000
	EPA-18-3	26.7	100.0	48.0	10.9	6.0	14,300	1,000
	EPA-18-4	19.4	100.0	53.9	13.0	6.2	28,200	1,000
	EPA-18-5	12.2	100.0	62.1	16.5	6.0	21,900	1,000
Padre Island, TX	EPA-19-1	5.0	100.0	1.0	0.0	6.3	2,670	1,000
	EPA-19-2	0.8	100.0	1.3	0.0	5.8	1,730	1,000
	EPA-19-3	0.5	100.0	2.2	0.5	6.0	4,440	1,000
	EPA-19-4	1.3	100.0	1.4	0.0	6.2	2,370	1,000
	EPA-19-5	0.3	100.0	1.2	0.0	6.0	1,860	1,000
Fond du Lac, MN	EPA-20-1	2.4	100.0	17.9	6.4	5.1	19,800	1,000
	EPA-20-2	2.9	100.0	21.7	6.2	5.4	6,980	1,000
	EPA-20-3	1.7	100.0	19.8	5.1	5.5	17,100	1,000
	EPA-20-4	2.4	99.6	12.1	5.1	5.8	9,760	1,000
	EPA-20-5	7.5	100.0	13.7	5.6	5.5	8,630	1,000

Site	Sample ID	Moisture Content[a] (%)	Grain Size[b] Distribution			pH[c]	Total Organic Content[d]	
			% Finer #4 Sieve	% Finer #200 Sieve	% Finer 0.005mm		Result (mg/kg)	Reporting Limit (mg/kg)
North Platte, NE	EPA-21-1	12.1	100.0	89.4	17.7	6.1	23,400	1,000
	EPA-21-2	8.0	100.0	85.4	14.1	5.3	21,100	1,000
	EPA-21-3	10.2	100.0	91.2	20.7	5.6	26,300	1,000
	EPA-21-4	12.4	100.0	89.2	21.7	5.6	31,700	1,000
	EPA-21-5	11.0	100.0	88.4	19.8	5.2	32,600	1,000
Goodwell, OK	EPA-22-1	20.5	100.0	62.3	20.5	7.6	5,450	1,000
	EPA-22-2	14.0	100.0	59.1	22.8	7.7	6,980	1,000
	EPA-22-3	22.4	98.7	52.8	18.3	7.6	21,900	1,000
	EPA-22-4	4.7	100.0	58.4	20.6	7.5	7,620	1,000
	EPA-22-5	3.7	100.0	62.2	20.7	7.5	6,000	1,000
Big Bend, TX	EPA-23-1	8.6	99.9	72.8	32.7	8.4	10,900	1,000
	EPA-23-2	9.0	99.0	74.2	33.7	8.1	14,400	1,000
	EPA-23-3	6.9	99.4	74.1	31.8	8.4	14,800	1,000
	EPA-23-4	10.4	99.4	72.2	31.8	8.0	13,600	1,000
	EPA-23-5	9.1	99.7	68.9	31.1	8.1	14,000	1,000
Grand Canyon, AZ	EPA-24-1	4.5	96.3	59.9	14.8	8.1	36,200	1,000
	EPA-24-2	3.7	96.8	51.9	12.3	8.3	31,600	1,000
	EPA-24-3	5.1	98.4	79.1	26.9	8.4	40,200	1,000
	EPA-24-4	4.2	99.6	77.7	27.4	8.8	49,000	1,000
	EPA-24-5	3.1	97.3	63.3	20.1	8.5	42,800	1,000
Theodore Roosevelt, ND	EPA-25-1	12.2	97.3	76.9	23.7	7.5	27,200	1,000
	EPA-25-2	6.5	96.3	73.1	21.3	7.7	25,000	1,000
	EPA-25-3	6.5	96.4	71.9	23.6	8.0	19,900	1,000
	EPA-25-4	9.5	92.5	60.1	17.1	7.9	25,300	1,000
	EPA-25-5	6.7	98.9	81.5	24.5	7.9	24,000	1,000
Chiricahua, AZ	EPA-27-1	5.1	99.1	56.5	8.5	7.8	29,000	1,000
	EPA-27-2	5.1	99.1	57.4	8.2	7.1	32,600	1,000
	EPA-27-3	5.2	98.7	42.4	5.6	6.8	13,100	1,000
	EPA-27-4	2.2	96.1	48.4	6.1	6.6	13,100	1,000
	EPA-27-5	3.5	93.6	26.7	4.8	6.5	9,930	1,000
Rancho Seco, CA	EPA-28-1	1.1	97.6	57.1	12.4	5.6	13,000	1,000
	EPA-28-2	0.9	91.7	52.6	11.3	5.6	16,500	1,000

Site	Sample ID	Moisture Content[a] (%)	Grain Size[b] Distribution			pH[c]	Total Organic Content[d]	
			% Finer #4 Sieve	% Finer #200 Sieve	% Finer 0.005mm		Result (mg/kg)	Reporting Limit (mg/kg)
	EPA-28-3	0.8	90.2	44.0	8.4	5.7	12,400	1,000
	EPA-28-4	1.5	96.8	60.6	16.7	5.6	15,900	1,000
	EPA-28-5	0.8	91.5	52.0	11.6	5.6	14,100	1,000
Marvel Ranch, OR	EPA-29-1	12.1	98.6	58.3	15.3	5.3	129,000	1,000
	EPA-29-2	10.3	94.6	49.3	18.8	5.6	85,300	1,000
	EPA-29-3	13.1	99.4	60.9	17.1	5.2	132,000	1,000
	EPA-29-4	6.6	100.0	79.1	28.4	4.8	76,200	1,000
	EPA-29-5	5.0	97.3	75.1	38.2	4.6	26,900	1,000
Ozette Lake, WA	EPA-30-1	50.7	100.0	89.3	35.3	3.8	90,200	1,000
	EPA-30-2	51.1	100.0	89.2	36.2	3.9	89,900	1,000
	EPA-30-3	74.7	100.0	82.9	37.5	3.8	113,000	1,000
	EPA-30-4	10.2	89.5	22.3	10.5	4.7	50,400	1,000
	EPA-30-5	44.6	100.0	91.9	46.0	4.9	99,200	1,000
Trapper Creek, AK	EPA-34-1	75.5	100.0	85.5	18.1	4.7	66,200	1,000
	EPA-34-2	67.1	100.0	84.3	15.6	4.8	37,900	1,000
	EPA-34-3	83.3	100.0	82.5	17.2	4.4	100,000	1,000
	EPA-34-4	85.6	100.0	82.6	19.1	4.7	78,400	1,000
	EPA-34-5	81.8	100.0	74.3	16.6	5.0	81,300	1,000

[a] Based on ASTM 2216, which yields the ratio of water mass in sample to mass of dry solids in sample.
[b] Based on ASTM D422.
[c] Based on EPA Method SW 9045C.
[d] Based on Walkey-Black Method.

APPENDIX H

PCDD/PCDF DATA

WA 5-11 Batch 1
BATTELLE
SDG 49971-13
MOD 1613M

NOTES	QC					
CLIENT_ ID	PROCEDURAL BLANK	Penn Nursery, PA	McNay Farm, IA	McNay Farm, IA	Lake Scott, KS	Lake Scott, KS
LAB_SAMP_ ID	METHOD BLANK	EPA 1 COMP	EPA 7 COMP	EPA 7 DUP	EPA 8 COMP	EPA 8 DUP
SAMPLE_WGT_VOL	17.1554	15.6769	16.0618	16.4463	15.5239	15.8020
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT		78.27	81.32	81.32	78.17	78.17
COLLECTION_DATE		8/17/2003	9/2/2003	9/2/2003	8/19/2003	8/19/2003
RECEIPT_DATE		8/19/2003	9/4/2003	9/4/2003	8/23/2003	8/23/2003
COMPOSITE_DATE		9/10/2003	9/10/2003	9/10/2003	9/4/2003	9/4/2003
EXTRACT_DATE	9/15/2003	9/15/2003	9/15/2003	9/15/2003	9/15/2003	9/15/2003
ANALYSIS_DATE	10/13/2003	10/13/2003	10/13/2003	10/14/2003	10/13/2003	10/14/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-20	49971-13-02	49971-13-03	49971-13-14	49971-13-04	49971-13-15
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT

PARAM_NAME	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL
2378-TCDD		U	0.04	0.07		0.04	0.10		0.04	0.07		0.04		U	0.04		U	0.04
12378-PECDD		U	0.18	0.30		0.20	0.48		0.19	0.48		0.19		U	0.20		U	0.20
123478-HXCDD	0.06	J	0.18	0.63		0.20	0.79		0.19	0.87		0.19		U	0.20	0.04	J	0.20
123678-HXCDD	0.08	J	0.18	0.93		0.20	1.47		0.19	1.55		0.19	0.05	J	0.20	0.06	J	0.20
123789-HXCDD	0.09	J	0.18	1.55		0.20	3.11		0.19	2.78		0.19	0.11	J	0.20	0.06	J	0.20
1234678-HPCDD	0.17	J	0.18	53.77		0.20	54.36		0.19	57.36		0.19	1.26		0.20	1.16		0.20
OCDD	0.72		0.36	6468.45		0.40	1516.18		0.39	1619.09		0.38	25.35		0.40	11.98		0.40
2378-TCDF		U	0.04	0.30	#	0.04	0.11		0.04	0.12		0.04	0.04		0.04	0.03	J	0.04
12378-PECDF		U	0.18	0.30		0.20	0.05	J	0.19	0.06	J	0.19	0.02	J	0.20		U	0.20
23478-PECDF		U	0.18	0.43		0.20	0.09	J	0.19	0.10	J	0.19	0.04	J	0.20	0.03	J	0.20
123478-HXCDF	0.07	J	0.18	0.90		0.20	0.40		0.19	0.36		0.19	0.09	J	0.20	0.09	J	0.20
123678-HXCDF	0.05	J	0.18	0.48		0.20	0.24		0.19	0.25		0.19	0.04	J	0.20	0.05	J	0.20
123789-HXCDF	0.08	J	0.18	0.27		0.20	0.02	J	0.19	0.01	J	0.19	0.02	J	0.20		U	0.20
234678-HXCDF	0.08	J	0.18	0.56		0.20	0.24		0.19	0.26		0.19	0.05	J	0.20		U	0.20
1234678-HPCDF	0.11	J	0.18	4.28		0.20	6.44		0.19	5.98		0.19	0.39		0.20	0.33		0.20
1234789-HPCDF	0.12	J	0.18	0.49		0.20	0.38		0.19	0.40		0.19		U	0.20	0.03	J	0.20
OCDF	0.44		0.36	8.70		0.40	18.82		0.39	21.39		0.38	0.58		0.40	0.52		0.40
Total Tetra-Furans		U	0.04	1.44		0.04	0.57		0.04	0.32		0.04	0.08		0.04	0.08		0.04
Total Tetra-Dioxins		U	0.04	0.23		0.04	0.87		0.04	0.75		0.04		U	0.04		U	0.04
Total Penta-Furans		U	0.18	4.66		0.20	1.65		0.19	1.71		0.19	0.40		0.20	0.31		0.20
Total Penta-Dioxins		U	0.18	1.77		0.20	2.86		0.19	3.33		0.19		U	0.20		U	0.20
Total Hexa-Furans	0.28		0.18	5.88		0.20	5.97		0.19	6.08		0.19	0.58		0.20	0.52		0.20
Total Hexa-Dioxins	0.22		0.18	12.52		0.20	17.88		0.19	17.86		0.19	0.64		0.20	0.56		0.20
Total Hepta-Furans	0.24		0.18	7.89		0.20	17.60		0.19	19.18		0.19	0.70		0.20	0.70		0.20
Total Hepta-Dioxins	0.17	J	0.18	119.30		0.20	104.81		0.19	109.19		0.19	2.63		0.20	2.50		0.20

J = reported value < Reporting Limit (RL)
U = not detected
RL = (0.25 * low cal std* final extract volume)/ sample dry weight
# = value from confirmation analysis.

WA 5-11 Batch 1
BATTELLE
SDG 49971-13
MOD 1613M

NOTES

CLIENT_ ID	Bennington, VT	Caldwell, OH	Dixon Springs, IL	Quincy, FL	Bay St. Louis, MS
LAB_SAMP_ ID	EPA 11 COMP	EPA 14 COMP	EPA 16 COMP	EPA 17 COMP	EPA 18 COMP
SAMPLE_WGT_VOL	15.9883	17.0640	17.6250	14.5225	16.6717
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	79.62	83.86	86.84	72.84	83.08
COLLECTION_DATE	8/28/2003	8/21/2003	8/16/2003	8/17/2003	8/19/2003
RECEIPT_DATE	8/29/2003	8/22/2003	8/18/2003	8/19/2003	8/21/2003
COMPOSITE_DATE	9/4/2003	9/4/2003	9/4/2003	9/10/2003	9/4/2003
EXTRACT_DATE	9/15/2003	9/15/2003	9/15/2003	9/15/2003	9/15/2003
ANALYSIS_DATE	10/13/2003	10/13/2003	10/13/2003	10/13/2003	10/14/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-05	49971-13-06	49971-13-07	49971-13-08	49971-13-09
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT

PARAM_NAME	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL
2378-TCDD		U	0.04		U	0.04	0.27		0.04		U	0.04	0.10		0.04
12378-PECDD	0.18	J	0.20	0.11	J	0.18	0.86		0.18		U	0.22	0.40		0.19
123478-HXCDD	0.31		0.20	0.21		0.18	2.12		0.18	0.08	J	0.22	0.55		0.19
123678-HXCDD	0.62		0.20	0.41		0.18	4.99		0.18	0.25		0.22	1.95		0.19
123789-HXCDD	0.90		0.20	0.69		0.18	5.09		0.18	0.18	J	0.22	2.65		0.19
1234678-HPCDD	15.67		0.20	21.47		0.18	213.85		0.18	8.15		0.22	64.87		0.19
OCDD	140.32		0.39	2251.74		0.37	9115.58		0.35	351.98		0.43	1530.37		0.37
2378-TCDF	0.25	#	0.04	0.07		0.04	0.18		0.04		U	0.04	0.14		0.04
12378-PECDF	0.26		0.20	0.06	J	0.18	0.14	J	0.18		U	0.22	0.09	J	0.19
23478-PECDF	0.34		0.20	0.07	J	0.18	0.17	J	0.18		U	0.22	0.11	J	0.19
123478-HXCDF	0.81		0.20	0.26		0.18	1.05		0.18	0.12	J	0.22	0.42		0.19
123678-HXCDF	0.36		0.20	0.14	J	0.18	0.72		0.18	0.05	J	0.22	0.24		0.19
123789-HXCDF	0.04	J	0.20	0.01	J	0.18	0.04	J	0.18	0.07	J	0.22	0.04	J	0.19
234678-HXCDF	0.47		0.20	0.14	J	0.18	0.72		0.18	0.09	J	0.22	0.25		0.19
1234678-HPCDF	4.48		0.20	3.07		0.18	35.50		0.18	1.68		0.22	7.52		0.19
1234789-HPCDF	0.25		0.20	0.14	J	0.18	1.43		0.18	0.15	J	0.22	0.34		0.19
OCDF	7.85		0.39	6.15		0.37	108.01		0.35	5.12		0.43	18.67		0.37
Total Tetra-Furans	1.21		0.04	0.17		0.04	0.51		0.04	0.02	J	0.04	0.64		0.04
Total Tetra-Dioxins	0.05		0.04	0.05		0.04	0.31		0.04		U	0.04	0.20		0.04
Total Penta-Furans	4.44		0.20	1.03		0.18	3.76		0.18	0.15	J	0.22	1.66		0.19
Total Penta-Dioxins	1.04		0.20	0.67		0.18	4.30		0.18		U	0.22	2.00		0.19
Total Hexa-Furans	5.43		0.20	2.58		0.18	26.70		0.18	1.07		0.22	7.85		0.19
Total Hexa-Dioxins	6.08		0.20	5.39		0.18	41.89		0.18	1.68		0.22	17.75		0.19
Total Hepta-Furans	8.61		0.20	5.83		0.18	100.09		0.18	3.97		0.22	22.38		0.19
Total Hepta-Dioxins	30.37		0.20	48.76		0.18	412.30		0.18	15.50		0.22	136.07		0.19

J = reported value < Reporting Limit (RL
U = not detected
RL = (0.25 * low cal std* final extract vol
# = value from confirmation analysis.

WA 5-11 Batch 1
BATTELLE
SDG 49971-13
MOD 1613M

NOTES					
CLIENT_ ID	Padre Island, TX	North Platte, NE	Theodore Roosevelt, ND	Theodore Roosevelt, ND	Chiricahua, AZ
LAB_SAMP_ ID	EPA 19 COMP	EPA 21 COMP	EPA 25 COMP	EPA 25 DUP	EPA 27 COMP
SAMPLE_WGT_VOL	20.2499	18.5140	18.6793	18.9976	19.5077
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	99.15	91.61	93.59	93.59	96.20
COLLECTION_DATE	8/19/2003	8/13/2003	8/12/2003	8/12/2003	8/18/2003
RECEIPT_DATE	8/20/2003	8/14/2003	8/19/2003	8/19/2003	8/20/2003
COMPOSITE_DATE	9/4/2003	9/4/2003	9/4/2003	9/4/2003	9/4/2003
EXTRACT_DATE	9/15/2003	9/15/2003	9/15/2003	9/15/2003	9/15/2003
ANALYSIS_DATE	10/14/2003	10/14/2003	10/14/2003	10/14/2003	10/14/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-13-10	49971-13-11	49971-13-12	49971-13-16	49971-13-13
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT

PARAM_NAME	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL
2378-TCDD		U	0.03	0.08		0.03	0.05		0.03	0.04		0.03		U	0.03
12378-PECDD		U	0.15	0.10	J	0.17	0.09	J	0.17	0.08	J	0.16	0.24		0.16
123478-HXCDD	0.24		0.15	0.12	J	0.17	0.09	J	0.17	0.09	J	0.16	0.40		0.16
123678-HXCDD	0.26		0.15	0.23		0.17	0.37		0.17	0.38		0.16	0.63		0.16
123789-HXCDD	0.35		0.15	0.27		0.17	0.31		0.17	0.27		0.16	0.99		0.16
1234678-HPCDD	1.71		0.15	4.93		0.17	9.23		0.17	9.75		0.16	22.25		0.16
OCDD	69.19		0.31	38.12		0.34	84.53		0.33	88.60		0.33	1039.65		0.32
2378-TCDF	0.02	J	0.03	0.25	#	0.03	0.06		0.03	0.05		0.03	0.10		0.03
12378-PECDF		U	0.15	0.14	J	0.17	0.04	J	0.17	0.04	J	0.16	0.04	J	0.16
23478-PECDF	0.06	J	0.15	0.20		0.17	0.08	J	0.17	0.08	J	0.16	0.09	J	0.16
123478-HXCDF	0.14	J	0.15	0.21		0.17	0.22		0.17	0.22		0.16	0.18		0.16
123678-HXCDF	0.14	J	0.15	0.15	J	0.17	0.16	J	0.17	0.17		0.16	0.10	J	0.16
123789-HXCDF	0.23		0.15	0.04	J	0.17	0.02	J	0.17	0.01	J	0.16	0.01	J	0.16
234678-HXCDF	0.29		0.15	0.13	J	0.17	0.14	J	0.17	0.15	J	0.16	0.12	J	0.16
1234678-HPCDF	0.46		0.15	1.47		0.17	1.83		0.17	2.06		0.16	0.95		0.16
1234789-HPCDF	0.50		0.15	0.11	J	0.17	0.20		0.17	0.17		0.16	0.10	J	0.16
OCDF	2.25		0.31	3.36		0.34	3.97		0.33	4.04		0.33	2.82		0.32
Total Tetra-Furans	0.02	J	0.03	2.30		0.03	0.49		0.03	0.25		0.03	0.33		0.03
Total Tetra-Dioxins		U	0.03	0.17		0.03	0.05		0.03	0.23		0.03	0.05		0.03
Total Penta-Furans	0.06	J	0.15	2.08		0.17	4.18		0.17	4.12		0.16	0.95		0.16
Total Penta-Dioxins	0.05	J	0.15	0.10	J	0.17	0.09	J	0.17	0.77		0.16	1.48		0.16
Total Hexa-Furans	1.17		0.15	2.35		0.17	5.09		0.17	5.26		0.16	1.38		0.16
Total Hexa-Dioxins	1.39		0.15	2.18		0.17	2.74		0.17	2.82		0.16	10.75		0.16
Total Hepta-Furans	1.21		0.15	3.47		0.17	6.89		0.17	7.08		0.16	2.38		0.16
Total Hepta-Dioxins	3.48		0.15	9.69		0.17	16.19		0.17	17.05		0.16	58.13		0.16

J = reported value < Reporting Limit (RL
U = not detected
RL = (0.25 * low cal std* final extract vol
# = value from confirmation analysis.

WA5-11 Batch 2
BATTELLE
SDG 49971-23
MOD 1613M

NOTES	QC				
CLIENT_ ID	PROCEDURAL BLANK	Clinton Crops, NC	Everglades, FL	Everglades, FL	Lake Dubay, WI
LAB_SAMP_ ID	METHOD BLANK	EPA 2 COMP	EPA 4 COMP	EPA 4 COMP DUP	EPA 5 COMP
SAMPLE_WGT_VOL	16.6259	17.8915	10.8883	11.0058	16.7103
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT		90.91	54.96	54.96	84.78
RECEIPT_DATE		10/21/2003	10/22/2003	10/22/2003	8/18/2003
EXTRACT_DATE	11/6/2003	11/6/2003	11/6/2003	11/6/2003	11/6/2003
ANALYSIS_DATE	11/18/2003	11/19/2003	11/19/2003	11/19/2003	11/19/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-28-04	49971-28-02	49971-28-03	49971-23-17	49971-23-04
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT

PARAM_NAME	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL
2378-TCDD		U	0.04	0.03		0.03		U	0.06		U	0.06	0.02	J	0.04
12378-PECDD		U	0.19	0.13	J	0.17		U	0.29		U	0.28	0.06	J	0.19
123478-HXCDD		U	0.19	0.26		0.17	0.37		0.29	0.33		0.28	0.11	J	0.19
123678-HXCDD		U	0.19	0.39		0.17	1.69		0.29	1.92		0.28	0.23		0.19
123789-HXCDD		U	0.19	0.72		0.17	1.49		0.29	1.50		0.28	0.31		0.19
1234678-HPCDD	0.04	J	0.19	17.40		0.17	41.17		0.29	46.71		0.28	5.65		0.19
OCDD	0.61		0.38	1298.51		0.35	651.16		0.57	505.18		0.57	82.19		0.37
2378-TCDF	0.07	#	0.04	0.14		0.03		U#	0.06		U#	0.06	0.16		0.04
12378-PECDF	0.03	J	0.19	0.06	J	0.17		U	0.29	0.30		0.28	0.07	J	0.19
23478-PECDF	0.03	J	0.19	0.07	J	0.17		U	0.29	0.65		0.28	0.09	J	0.19
123478-HXCDF	0.04	J	0.19	0.14	J	0.17	0.62	J	0.29	0.59		0.28	0.23		0.19
123678-HXCDF		U	0.19	0.04	J	0.17	0.77		0.29	0.69		0.28	0.10	J	0.19
123789-HXCDF		U	0.19		U	0.17	0.23	J	0.29		U	0.28		U	0.19
234678-HXCDF		U	0.19	0.07	J	0.17	1.09		0.29	1.25		0.28	0.11	J	0.19
1234678-HPCDF		U	0.19	0.74		0.17	7.58		0.29	8.35		0.28	2.00		0.19
1234789-HPCDF		U	0.19		U	0.17	1.20	J	0.29	0.86		0.28	0.11	J	0.19
OCDF	0.13	J	0.38	1.33		0.35	37.97		0.57	25.85		0.57	3.84		0.37
Total Tetra-Furans	0.10		0.04	0.25		0.03	2.02		0.06	0.56		0.06	0.94		0.04
Total Tetra-Dioxins		U	0.04	0.08		0.03		U	0.06		U	0.06	0.10		0.04
Total Penta-Furans	0.06	J	0.19	0.81		0.17	21.91		0.29	16.81		0.28	1.21		0.19
Total Penta-Dioxins		U	0.19	0.63		0.17		U	0.29		U	0.28	0.48		0.19
Total Hexa-Furans	0.04	J	0.19	0.88		0.17	19.57		0.29	19.93		0.28	2.21		0.19
Total Hexa-Dioxins		U	0.19	6.68		0.17	12.34		0.29	12.68		0.28	2.62		0.19
Total Hepta-Furans		U	0.19	1.59		0.17	24.38		0.29	27.26		0.28	5.03		0.19
Total Hepta-Dioxins	0.08	J	0.19	55.87		0.17	86.55		0.29	91.75		0.28	11.02		0.19

J = reported value < Reporting Limit (RL)
U = not detected
RL = (0.25 * low cal std* final extract volume)/ sample dry weight
# = value from confirmation analysis.

WA5-11 Batch 2
BATTELLE
SDG 49971-23
MOD 1613M

NOTES						
CLIENT_ ID	Monmouth, IL	Keystone State Park, OK	Arkadelphia, AK	Jasper, NY	Fond du Lac, MN	Fond du Lac, MN
LAB_SAMP_ ID	EPA 6 COMP	EPA 9 COMP	EPA 10 COMP	EPA 12 COMP	EPA 20 COMP	EPA 20 COMP DUP
SAMPLE_WGT_VOL	17.3948	17.8582	17.5451	14.1085	19.0782	19.6037
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	87.36	88.71	88.91	70.95	97.10	97.10
RECEIPT_DATE	8/18/2003	8/20/2003	9/12/2003	8/22/2003	8/26/2003	8/26/2003
EXTRACT_DATE	11/6/2003	11/6/2003	11/6/2003	11/6/2003	11/6/2003	11/6/2003
ANALYSIS_DATE	11/19/2003	11/19/2003	11/19/2003	11/19/2003	11/19/2003	11/20/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-05	49971-23-06	49971-23-07	49971-23-08	49971-23-09	49971-23-18
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT

PARAM_NAME	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL
2378-TCDD	0.18		0.04		U	0.03	0.03	J	0.04	0.16		0.04		U	0.03		U	0.03
12378-PECDD	0.75		0.18	0.06	J	0.17	0.08	J	0.18	0.92		0.22	0.10	J	0.16	0.12	J	0.16
123478-HXCDD	0.48	J	0.18	0.09	J	0.17	0.18		0.18	2.13		0.22	0.19		0.16	0.20		0.16
123678-HXCDD	1.58		0.18	0.22		0.17	0.28		0.18	4.45		0.22	0.63		0.16	0.64		0.16
123789-HXCDD	5.18		0.18	0.32		0.17	0.53		0.18	5.17		0.22	0.51		0.16	0.53		0.16
1234678-HPCDD	37.53		0.18	5.07		0.17	12.00		0.18	180.15		0.22	16.78		0.16	16.21		0.16
OCDD	308.18		0.36	51.33		0.35	649.79		0.36	10915.30		0.44	78.02		0.33	115.12		0.32
2378-TCDF		U#	0.04	2.34	#	0.03	0.21	#	0.04	0.99	#	0.04	0.18		0.03	0.12		0.03
12378-PECDF	0.14	J	0.18	1.43		0.17	0.16	J	0.18	0.74		0.22	0.13	J	0.16	0.08	J	0.16
23478-PECDF	0.18		0.18	0.99		0.17	0.12	J	0.18	0.67		0.22	0.11	J	0.16	0.09	J	0.16
123478-HXCDF	0.56		0.18	1.04		0.17	0.21		0.18	1.07		0.22	0.36		0.16	0.37		0.16
123678-HXCDF	0.28		0.18	0.28		0.17	0.07	J	0.18	0.53		0.22	0.24		0.16	0.23		0.16
123789-HXCDF		U	0.18	0.08	J	0.17	0.08	J	0.18	0.13	J	0.22		U	0.16	0.03		0.16
234678-HXCDF	0.48		0.18	0.17		0.17	0.06	J	0.18	0.52		0.22	0.23		0.16	0.26		0.16
1234678-HPCDF	9.39		0.18	1.87		0.17	0.59		0.18	9.59		0.22	6.66		0.16	6.79		0.16
1234789-HPCDF	0.42		0.18	0.15	J	0.17	0.05	J	0.18	0.46	J	0.22	0.41		0.16	0.37		0.16
OCDF	30.10		0.36	3.28		0.35	1.11		0.36	25.48		0.44	30.36		0.33	27.05		0.32
Total Tetra-Furans	1.26		0.04	5.69		0.03	0.65		0.04	3.20		0.04	0.54		0.03	0.36		0.03
Total Tetra-Dioxins	0.53		0.04	0.04		0.03	0.04		0.04	0.36		0.04	0.04		0.03		U	0.03
Total Penta-Furans	3.00		0.18	5.17		0.17	0.57		0.18	5.39		0.22	1.97		0.16	1.76		0.16
Total Penta-Dioxins	2.63		0.18	0.43		0.17	0.33		0.18	3.86		0.22	0.56		0.16	0.61		0.16
Total Hexa-Furans	9.87		0.18	3.30		0.17	0.90		0.18	11.58		0.22	7.20		0.16	6.48		0.16
Total Hexa-Dioxins	19.59		0.18	2.28		0.17	3.69		0.18	42.31		0.22	4.71		0.16	4.33		0.16
Total Hepta-Furans	51.47		0.18	4.30		0.17	1.28		0.18	31.67		0.22	24.46		0.16	24.88		0.16
Total Hepta-Dioxins	64.03		0.18	11.19		0.17	31.28		0.18	442.92		0.22	29.38		0.16	27.94		0.16

J = reported value < Reporting Limit (RL
U = not detected
RL = (0.25 * low cal std* final extract vol
# = value from confirmation analysis.

WA5-11 Batch 2
BATTELLE
SDG 49971-23
MOD 1613M

NOTES															
CLIENT_ ID	Goodwell, OK			Big Bend, TX			Grand Canyon, AZ			Rancho Seco, CA			Rancho Seco, CA		
LAB_SAMP_ ID	EPA 22 COMP			EPA 23 COMP			EPA 24 COMP			EPA 28 COMP			EPA 28 COMP DUP		
SAMPLE_WGT_VOL	17.5068			18.1705			19.0697			19.1987			19.2911		
SAMP_WGT_VOL_UNIT	G DRYWT			G DRYWT			G DRYWT			G DRYWT			G DRYWT		
PCT_DRY_WT	88.38			91.60			95.74			98.76			98.76		
RECEIPT_DATE	8/22/2003			9/10/2003			8/29/2003			8/18/2003			8/18/2003		
EXTRACT_DATE	11/6/2003			11/6/2003			11/6/2003			11/6/2003			11/6/2003		
ANALYSIS_DATE	11/19/2003			11/19/2003			11/19/2003			11/19/2003			11/20/2003		
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-10			49971-23-11			49971-23-12			49971-23-13			49971-23-19		
REPORTING UNIT	PG/G DRYWT			PG/G DRYWT			PG/G DRYWT			PG/G DRYWT			PG/G DRYWT		
PARAM_NAME	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL
2378-TCDD		U	0.04		U	0.03	0.02	J	0.03	0.04		0.03	0.03		0.03
12378-PECDD	0.12	J	0.18		U	0.17		U	0.16	0.27		0.16	0.23		0.16
123478-HXCDD	0.28		0.18	0.10	J	0.17	0.05	J	0.16	0.31		0.16	0.55		0.16
123678-HXCDD	0.90		0.18	0.11	J	0.17	0.12	J	0.16	1.34		0.16	1.43		0.16
123789-HXCDD	0.82		0.18	0.11	J	0.17	0.10	J	0.16	1.36		0.16	1.52		0.16
1234678-HPCDD	32.59		0.18	0.74		0.17	3.36		0.16	21.93		0.16	20.06		0.16
OCDD	200.96		0.36	20.28		0.34	17.80		0.33	111.62		0.33	206.05		0.32
2378-TCDF	0.17		0.04	0.11		0.03	0.03	J	0.03	0.19		0.03	0.16		0.03
12378-PECDF	0.15	J	0.18	0.06	J	0.17		U	0.16	0.12	J	0.16	0.10	J	0.16
23478-PECDF	0.13	J	0.18	0.06	J	0.17		U	0.16	0.12	J	0.16		U	0.16
123478-HXCDF	0.42		0.18	0.14	J	0.17	0.10	J	0.16	0.27		0.16	0.30		0.16
123678-HXCDF	0.22		0.18	0.07	J	0.17	0.04	J	0.16	0.18		0.16	0.24		0.16
123789-HXCDF		U	0.18	0.08	J	0.17		U	0.16	0.03	J	0.16	0.20		0.16
234678-HXCDF	0.19		0.18	0.08	J	0.17	0.03	J	0.16	0.16		0.16	0.36		0.16
1234678-HPCDF	5.56		0.18	0.29		0.17	1.02		0.16	3.95		0.16	3.78		0.16
1234789-HPCDF	0.38		0.18	0.24		0.17	0.07	J	0.16	0.21	J	0.16	0.82		0.16
OCDF	15.53		0.36	1.37		0.34	2.43		0.33	22.64		0.33	22.66		0.32
Total Tetra-Furans	0.34		0.04	0.15		0.03	0.03		0.03	0.71		0.03	0.44		0.03
Total Tetra-Dioxins		U	0.04		U	0.03	0.02	J	0.03	0.04		0.03	0.03		0.03
Total Penta-Furans	2.61		0.18	0.20		0.17	0.27		0.16	1.24		0.16	1.02		0.16
Total Penta-Dioxins	0.37		0.18		U	0.17	0.01	J	0.16	1.05		0.16	0.71		0.16
Total Hexa-Furans	7.47		0.18	0.59		0.17	1.51		0.16	3.62		0.16	4.15		0.16
Total Hexa-Dioxins	7.02		0.18	0.32		0.17	0.92		0.16	7.05		0.16	7.42		0.16
Total Hepta-Furans	17.08		0.18	0.71		0.17	3.16		0.16	23.71		0.16	19.78		0.16
Total Hepta-Dioxins	80.09		0.18	1.47		0.17	6.11		0.16	37.80		0.16	34.50		0.16

J = reported value < Reporting Limit (RL
U = not detected
RL = (0.25 * low cal std* final extract vol
# = value from confirmation analysis.

WA5-11 Batch 2
BATTELLE
SDG 49971-23
MOD 1613M

NOTES			
CLIENT_ ID	Marvel Ranch, OR	Ozette Lake, WA	Trapper Creek, AK
LAB_SAMP_ ID	EPA 29 COMP	EPA 30 COMP	EPA 34 COMP
SAMPLE_WGT_VOL	18.2966	13.4569	11.7051
SAMP_WGT_VOL_UNIT	G DRYWT	G DRYWT	G DRYWT
PCT_DRY_WT	90.88	68.71	58.27
RECEIPT_DATE	8/21/2003	8/22/2003	9/3/2003
EXTRACT_DATE	11/6/2003	11/6/2003	11/6/2003
ANALYSIS_DATE	1/16/2004	11/19/2003	11/20/2003
DIOXIN_ EXTRACT_ LRB_ NUMBER	49971-23-14	49971-23-15	49971-23-16
REPORTING UNIT	PG/G DRYWT	PG/G DRYWT	PG/G DRYWT

PARAM_NAME	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL	RESULT	LAB_QUAL	RL
2378-TCDD		U	0.10		U	0.05		U	0.05
12378-PECDD	1.20		0.51		U	0.23		U	0.27
123478-HXCDD	2.35		0.51	0.11	J	0.23	0.04	J	0.27
123678-HXCDD	20.10		0.51	0.30		0.23	0.10	J	0.27
123789-HXCDD	18.41		0.51	1.85		0.23	0.11	J	0.27
1234678-HPCDD	364.29		0.51	6.13		0.23	1.44		0.27
OCDD	2872.13		1.02	68.79		0.46	12.30		0.53
2378-TCDF	1.23	J#	0.10		U#	0.05	0.11	J	0.05
12378-PECDF	1.19		0.51	0.16	J	0.23	0.07	J	0.27
23478-PECDF	1.10		0.51		U	0.23	0.05	J	0.27
123478-HXCDF	2.78		0.51	0.55		0.23	0.12	J	0.27
123678-HXCDF	2.33		0.51	0.12	J	0.23	0.04	J	0.27
123789-HXCDF	1.01		0.51	0.13	J	0.23	0.01	J	0.27
234678-HXCDF	2.52		0.51	0.16	J	0.23		U	0.27
1234678-HPCDF	45.42		0.51	2.12		0.23	1.35		0.27
1234789-HPCDF	1.63		0.51	0.11	J	0.23		U	0.27
OCDF	87.07		1.02	6.01		0.46	0.52		0.53
Total Tetra-Furans	2.06		0.10	5.11		0.05	0.97		0.05
Total Tetra-Dioxins		U	0.10	0.63		0.05		U	0.05
Total Penta-Furans	4.14		0.51	1.90		0.23	0.12		0.27
Total Penta-Dioxins	1.20		0.51	13.56		0.23		U	0.27
Total Hexa-Furans	31.92		0.51	3.54		0.23	0.33		0.27
Total Hexa-Dioxins	77.46		0.51	10.50		0.23	0.35		0.27
Total Hepta-Furans	158.44		0.51	11.46		0.23	1.78		0.27
Total Hepta-Dioxins	583.01		0.51	11.77		0.23	2.34		0.27

J = reported value < Reporting Limit (RL
U = not detected
RL = (0.25 * low cal std* final extract vol
# = value from confirmation analysis.

APPENDIX I

CALUX DATA

CALUX Bioassay Data- Field Samples

Site	Sample ID	XDS ID	Percent Moisture	TEQ (pg/g dry weight)
Penn Nursery, PA	EPA-1-1	A03011	21	6.03 ± 0.79
	EPA-1-2	A03012	21	7.08 ± 0.84
	EPA-1-3	A03013	19	8.31 ± 1.74
	EPA-1-4	A03014	24	4.66 ± 1.40
	EPA-1-5	A03015	23	9.22 ± 2.99
	EPA-1-COMP	A03016	21	9.19 ± 1.84
Clinton Crops, NC	EPA-2-1	A03278	8	0.97 ± 0.23
	EPA-2-2	A03279	10	0.92 ± 0.18
	EPA-2-3	A03280	14	2.99 ± 0.15
	EPA-2-4	A03281	10	2.23 ± 0.04
	EPA-2-5	A03282	8	0.79 ± 0.36
	EPA-2-COMP	A03283	10	2.10 ± 0.33
Everglades, FL	EPA-4-1	A03272	65	3.37 ± 0.10
	EPA-4-2	A03273	50	1.79 ± 0.46
	EPA-4-3	A03274	45	1.84 ± 0.36
	EPA-4-4	A03275	32	2.07 ± 0.08
	EPA-4-5	A03276	52	0.88 ± 0.32
	EPA-4-COMP	A03277	47	2.16 ± 0.89
Lake Dubay, WI	EPA-5-1	A03059	17	10.83 ± 0.13
	EPA-5-2	A03060	14	9.68 ± 0.19
	EPA-5-3	A03061	12	10.31 ± 2.47
	EPA-5-4	A03062	13	3.69 ± 0.59
	EPA-5-5	A03063	16	2.94 ± 0.01
	EPA-5-COMP	A03064	16	3.68 ± 0.21
Monmouth, IL	EPA-6-1	A02987	10	9.18 ± 1.47
	EPA-6-2	A02988	8	4.84 ± 0.10
	EPA-6-3	A02989	7	3.38 ± 0.36
	EPA-6-4	A02990	3	1.95 ± 0.55
	EPA-6-5	A02991	26	4.23 ± 0.79
	EPA-6-COMP	A02992	11	4.97 ± 0.43

Site	Sample ID	XDS ID	Percent Moisture	TEQ (pg/g dry weight)
McNay Farms, IL	EPA-7-1	A03017	18	15.59 ± 0.39
	EPA-7-2	A03018	24	13.01 ± 0.83
	EPA-7-3	A03019	22	12.41 ± 2.38
	EPA-7-4	A03020	21	12.23 ± 1.89
	EPA-7-5	A03021	16	12.43 ± 1.78
	EPA-7-COMP	A03022	18	11.04 ± 0.30
Lake Scott, KS	EPA-8-1	A03129	19	ND<0.50
	EPA-8-2	A03130	16	1.22 ± 0.54
	EPA-8-3	A03131	22	1.12 ± 0.36
	EPA-8-4	A03132	25	ND<0.56
	EPA-8-5	A03133	31	0.82 ± 0.33
	EPA-8-COMP	A03134	23	1.58 ± 0.76
Keystone State Park, OK	EPA-9-1	A02993	17	0.87 ± 0.30
	EPA-9-2	A02994	15	2.00 ± 0.41
	EPA-9-3	A02995	7	2.34 ± 0.07
	EPA-9-4	A02996	3	1.20 ± 0.17
	EPA-9-5	A02997	7	1.23 ± 0.14
	EPA-9-COMP	A02998	9	1.88 ± 0.50
Arkadelphia, AR	EPA-10-1	A03123	8	3.17 ± 0.04
	EPA-10-2	A03124	11	3.10 ± 1.02
	EPA-10-3	A03125	9	2.59 ± 0.19
	EPA-10-4	A03126	8	2.86 ± 0.02
	EPA-10-5	A03127	12	3.72 ± 0.21
	EPA-10-COMP	A03128	6	2.92 ± 0.35
Bennington, VT	EPA-11-1	A03035	24	5.89 ± 0.23
	EPA-11-2	A03036	23	5.67 ± 0.42
	EPA-11-3	A03037	19	8.27 ± 0.53
	EPA-11-4	A03038	21	7.30 ± 1.16
	EPA-11-5	A03039	10	2.30 ± 0.34
	EPA-11-COMP	A03040	15	5.86 ± 0.64
Jasper, NY	EPA-12-1	A03083	32	7.57 ± 0.17
	EPA-12-2	A03084	29	7.23 ± 0.53

Site	Sample ID	XDS ID	Percent Moisture	TEQ (pg/g dry weight)
	EPA-12-3	A03085	30	4.98 ± 0.81
	EPA-12-4	A03086	20	6.58 ± 0.20
	EPA-12-5	A03087	30	6.69 ± 0.93
	EPA-12-COMP	A03088	30	7.04 ± 1.74
Caldwell, OH	EPA-14-1	A03089	16	4.20 ± 0.12
	EPA-14-2	A03090	22	5.66 ± 0.79
	EPA-14-3	A03091	15	3.81 ± 0.05
	EPA-14-4	A03092	21	5.87 ± 0.13
	EPA-14-5	A03093	14	6.46 ± 0.81
	EPA-14-COMP	A03094	16	4.60 ± 1.81
Dixon Springs, IL	EPA-16-1	A02999	11	8.00 ± 0.63
	EPA-16-2	A03000	9	7.16 ± 0.11
	EPA-16-3	A03001	10	10.25 ± 1.37
	EPA-16-4	A03002	15	10.08 ± 1.28
	EPA-16-5	A03003	17	9.74 ± 0.76
	EPA-16-COMP	A03004	13	12.61 ± 0.00
Quincy, FL	EPA-17-1	A03041	24	2.48 ± 0.35
	EPA-17-2	A03042	35	1.55 ± 0.57
	EPA-17-3	A03043	29	2.52 ± 0.60
	EPA-17-4	A03044	29	1.80 ± 0.65
	EPA-17-5	A03045	27	1.03 ± 0.33
	EPA-17-COMP	A03046	29	1.54 ± 0.11
Bay St, Louis, MS	EPA-18-1	A03095	25	14.68 ± 0.63
	EPA-18-2	A03096	17	16.60 ± 1.02
	EPA-18-3	A03097	21	15.22 ± 2.58
	EPA-18-4	A03098	17	18.24 ± 1.40
	EPA-18-5	A03099	12	22.89 ± 2.63
	EPA-18-COMP	A03100	18	17.06 ± 1.01
Padre Island, TX	EPA-19-1	A03101	5	0.53 ± 0.19
	EPA-19-2	A03102	1	0.25 ± 0.01
	EPA-19-3	A03103	1	0.40 ± 0.07
	EPA-19-4	A03104	2	0.34 ± 0.07

Site	Sample ID	XDS ID	Percent Moisture	TEQ (pg/g dry weight)
	EPA-19-5	A03105	0	0.36 ± 0.05
	EPA-19-COMP	A03106	2	0.62 ± 0.24
Fond du Lac, MN	EPA-20-1	A03135	2	2.89 ± 1.29
	EPA-20-2	A03136	3	1.85 ± 0.37
	EPA-20-3	A03137	2	6.91 ± 0.99
	EPA-20-4	A03138	2	1.00 ± 0.49
	EPA-20-5	A03139	7	1.47 ± 0.02
	EPA-20-COMP	A03140	2	2.87 ± 0.11
North Platte, NE	EPA-21-1	A03065	9	5.71 ± 0.53
	EPA-21-2	A03066	7	4.88 ± 1.10
	EPA-21-3	A03067	8	6.72 ± 1.60
	EPA-21-4	A03068	11	8.74 ± 1.33
	EPA-21-5	A03069	9	6.80 ± 0.34
	EPA-21-COMP	A03070	16	6.33 ± 0.21
Goodwell, OK	EPA-22-1	A03141	18	4.93 ± 2.42
	EPA-22-2	A03142	11	4.72 ± 0.89
	EPA-22-3	A03143	18	4.27 ± 0.88
	EPA-22-4	A03144	5	3.14 ± 0.41
	EPA-22-5	A03145	3	3.60 ± 0.30
	EPA-22-COMP	A03146	12	3.61 ± 0.46
Big Bend, TX	EPA-23-1	A03023	6	0.98 ± 0.31
	EPA-23-2	A03024	7	1.54 ± 0.22
	EPA-23-3	A03025	5	0.63 ± 0.07
	EPA-23-4	A03026	9	1.52 ± 0.44
	EPA-23-5	A03027	7	0.69 ± 0.01
	EPA-23-COMP	A03028	7	0.62 ± 0.69

Site	Sample ID	XDS ID	Percent Moisture	TEQ (pg/g dry weight)
Grand Canyon, AZ	EPA-24-1	A03047	5	0.70 ± 0.26
	EPA-24-2	A03048	4	0.60 ± 0.25
	EPA-24-3	A03049	5	ND<0.37
	EPA-24-4	A03050	4	ND<0.41
	EPA-24-5	A03051	3	ND<0.41
	EPA-24-COMP	A03052	5	0.82 ± 0.84
Theodore Roosevelt, ND	EPA-25-1	A03071	22	1.97 ± 0.24
	EPA-25-2	A03072	0	1.68 ± 0.36
	EPA-25-3	A03073	5	1.41 ± 0.52
	EPA-25-4	A03074	10	0.84 ± 0.25
	EPA-25-5	A03075	6	0.78 ± 0.22
	EPA-25-COMP	A03076	6	1.05 ± 0.47
Chiricahua, AZ	EPA-27-1	A03005	4	6.19 ± 0.48
	EPA-27-2	A03006	5	7.44 ± 0.40
	EPA-27-3	A03007	4	6.21 ± 0.27
	EPA-27-4	A03008	4	3.53 ± 0.86
	EPA-27-5	A03009	5	2.28 ± 0.03
	EPA-27-COMP	A03010	5	5.17 ± 1.57
Rancho Seco, CA	EPA-28-1	A03077	0	2.19 ± 0.20
	EPA-28-2	A03078	0	2.20 ± 0.04
	EPA-28-3	A03079	1	3.32 ± 1.27
	EPA-28-4	A03080	1	4.61 ± 1.30
	EPA-28-5	A03081	1	3.55 ± 0.20
	EPA-27-COMP	A03082	0	2.69 ± 0.35
Marvel Ranch, OR	EPA-29-1	A03053	9	22.74 ± 5.99
	EPA-29-2	A03054	8	20.73 ± 0.55
	EPA-29-3	A03055	11	33.62 ± 0.53
	EPA-29-4	A03056	5	30.30 ± 1.33
	EPA-29-5	A03057	4	11.52 ± 0.40
	EPA-29-COMP	A03058	6	23.01 ± 3.19
Ozette Lake, WA	EPA-30-1	A03147	31	1.54 ± 0.22
	EPA-30-2	A03148	33	1.25 ± 0.43
	EPA-30-3	A03149	42	0.56 ± 0.06
	EPA-30-4	A03150	6	1.44 ± 0.34
	EPA-30-5	A03151	30	2.48 ± 0.32
	EPA-30-COMP	A03152	27	1.14 ± 0.59

Site	Sample ID	XDS ID	Percent Moisture	TEQ (pg/g dry weight)
Trapper Creek, AK	EPA-34-1	A03029	40	1.12 ± 0.10
	EPA-34-2	A03030	41	2.00 ± 0.47
	EPA-34-3	A03031	40	2.22 ± 0.54
	EPA-34-4	A03032	42	2.57 ± 0.98
	EPA-34-5	A03033	43	6.53 ± 2.73
	EPA-34-COMP	A03034	44	1.79 ± 0.58

CALUX Bioassay Data- Field Blanks

	Sample ID	XDS ID	Percent Moisture (%)	TEQ (pg/g dry weight)
Lake Dubay, WI	EPA-5-FB	A03107	0	0.29 ± 0.09
McNay Farm, IL	EPA-7-FB	A03108	1	0.76 ± 0.01
Lake Scott, KS	EPA-8-FB	A03109	2	0.46 ± 0.11
Arkadelphia, AR	EPA-10-FB	A03110	3	0.66 ± 0.50
Bennington, VT	EPA-11-FB	A03111	0	ND<0.23
Caldwell, OH	EPA-14-FB	A03112	0	0.44 ± 0.15
Dixon Springs, IL	EPA-16-FB	A03113	1	0.69 ± 0.09
Bay St. Louis, MS	EPA-18-FB	A03114	0	0.66 ± 0.62
Padre Island, TX	EPA-19-FB	A03115	0	0.27 ± 0.10
Big Bend, TX	EPA-23-FB	A03116	4	ND<0.50
Grand Canyon, AZ	EPA-24-FB	A03117	0	ND<0.50
Theodore Roosevelt, ND	EPA-25-FB	A03118	0	0.84 ± 0.31
Chiricahua, AZ	EPA-27-FB	A03119	0	ND<0.23
Rancho Seco, CA	EPA-28-FB	A03120	0	ND<0.50
Ozette Lake, WA	EPA-30-FB	A03121	0	0.63 ± 0.44
Trapper Creek, AK	EPA-34-FB	A03122	0	ND<0.50

APENDIX J

MERCURY DATA

Mercury Analysis Results: Set 1

Sample Id	Concentration (µg/L)	Digest Volume (L)	Digest Weight (g)	Concentration (µg/g wet)	Percent Dry	Final Concentration (µg/g)
CCV	4.9	0.1				
Percent Recovery	**98%**					
Blk	<0.2	0.1				
LCS Blk	<0.2	0.1	2.04		100	0.000
LCS	2.2	0.1	2.08	0.106	100	0.106
spike concentration	2	0.1	2.08	0.096		
Percent Recovery				**110%**		
Nist 1944 (TV 3.4)	11.1	0.1	0.35	3.171	98.75	3.132
Percent Recovery						**92%**
Relative Percent Difference						**8%**
EPA-25COMP	0.01	0.1	1.88	0.0005	93.59	0.0005
EPA-5COMP	<0.2	0.1	2.16	<0.009	84.78	<0.008
EPA-28COMP	0.8	0.1	2.14	0.037	98.76	0.037
EPA-21COMP	0.3	0.1	2.12	0.014	91.61	0.013
EPA-21COMP DUP	0.7	0.1	3.78	0.019	91.61	0.017
Relative Percent Difference						**27%**
EPA-16COMP	0.4	0.1	1.80	0.022	86.84	0.019
EPA-19COMP	<0.2	0.1	2.25	<.009	99.15	<.009
EPA-27COMP	0.8	0.1	1.72	0.047	96.20	0.045
EPA-6COMP	0.5	0.1	1.74	0.029	87.36	0.025
EPA-9COMP	0.2	0.1	1.90	0.011	88.71	0.009
EPA-14COMP	0.6	0.1	1.91	0.031	83.86	0.026
EPA-12COMP	0.4	0.1	1.86	0.022	70.95	0.015
EPA-24COMP	0.2	0.1	2.10	0.010	95.74	0.009
EPA-8COMP	0.4	0.1	2.79	0.014	78.17	0.011
EPA-18COMP	0.6	0.1	2.05	0.029	83.08	0.024
EPA-11COMP	0.9	0.1	1.67	0.054	79.62	0.043
EPA-22COMP	0.1	0.1	1.81	0.006	88.38	0.005
EPA-30COMP	1	0.1	1.76	0.057	68.71	0.039
EPA-20COMP	0.4	0.1	2.02	0.020	97.10	0.019
EPA-29COMP	0.4	0.1	1.50	0.027	90.88	0.024
EPA-29 SPK	2.3	0.1	1.55	0.148	90.88	0.135
spike concentration	2	0.1	1.55	0.129	90.88	0.117
Percent Recovery						**94%**
CCV	4.8	0.1				
Percent Recovery	**96%**					

Mercury Analysis Results: Set 2

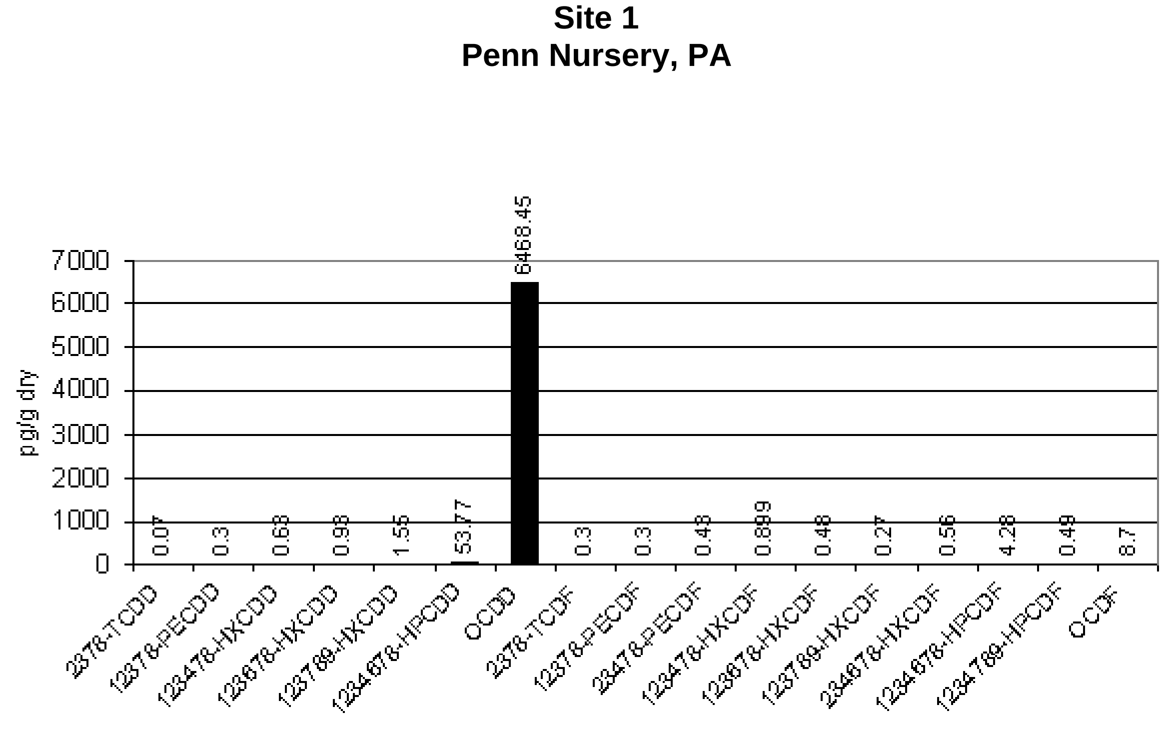
Sample Id	Concentration (µg/L)	Digest Volume (L)	Digest Weight (g)	Concentration (µg/g wet)	Percent Dry	Final Concentration (µg/g)
CCV	5.2	0.1				
Percent Recovery	**104%**					
Blk	<0.2	0.1				
LCS Blk	<0.2	0.1	2.12			
LCS	2.1	0.1	2.11	0.100		
spike concentration	2	0.1	2.11	0.095		
Percent Recovery				**105%**		
Nist 1944 (TV 3.4)	10.7	0.1	0.32	3.344	98.75	3.302
Percent Recovery						**97%**
Relative Percent Difference						**3%**
EPA-10COMP	0.9	0.1	3.12	0.029	88.91	0.026
EPA-10COMP DUP	0.6	0.1	2.15	0.028	88.91	0.025
Relative Percent Difference						**3%**
EPA-1COMP	0.7	0.1	2.14	0.033	78.27	0.026
EPA-7COMP	0.8	0.1	2.17	0.037	81.32	0.030
EPA-17COMP	0.7	0.1	3.78	0.019	72.84	0.013
EPA-34COMP	0.8	0.1	2.08	0.038	58.27	0.022
CCV	5					
EPA-23COMP	0.5	0.1	2.50	0.020	91.6	0.018
EPA-23COMP SPK	2.2	0.1	2.54	0.087	91.6	0.079
spike concentration	2	0.1	2.54	0.079	91.6	0.072
Percent Recovery						**85%**
CCV	5.1	0.1				
Percent Recovery	**102%**					

Mercury Analysis Results: Set 3

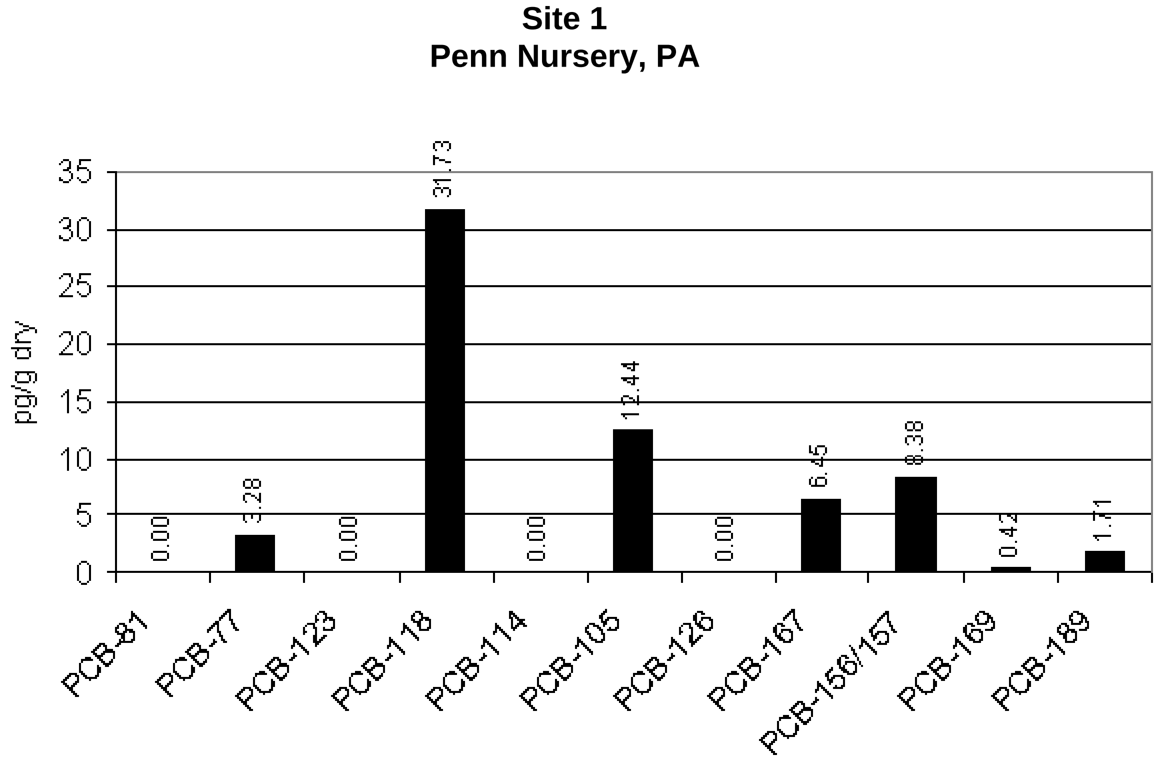
Sample Id	Concentration (µg/L)	Digest Volume (L)	Digest Weight (g)	Concentration (µg/g wet)	Percent Dry	Final Concentration (µg/g)
CCV	5	0.1				
Percent Recovery	**100%**					
Blk	<0.2	0.1				
LCS Blk	<0.2	0.1	2.12			
LCS	1.9	0.1	2.11	0.090		
spike concentration	2	0.1	2.11	0.095		
Percent Recovery				**95%**		
Nist 1944 (TV 3.4)	13	0.1	0.32	4.063	98.75	4.012
Percent Recovery						**118%**
Relative Percent Difference						**17%**
EPA-4COMP	0.9	0.1	2.3	0.039	54.96	0.022
EPA-4COMP DUP	0.7	0.1	2.07	0.034	54.96	0.019
Relative Percent Difference						**15%**
EPA-2COMP	1.5	0.1	1.99	0.075	90.91	0.069
EPA-2COMP SPK	4.3	0.1	2.48	0.173	90.91	0.158
spike concentration	2	0.1	2.48	0.081	90.91	0.073
Percent Recovery						**122%**
CCV	4.8	0.1				
Percent Recovery	**96%**					

APPENDIX K

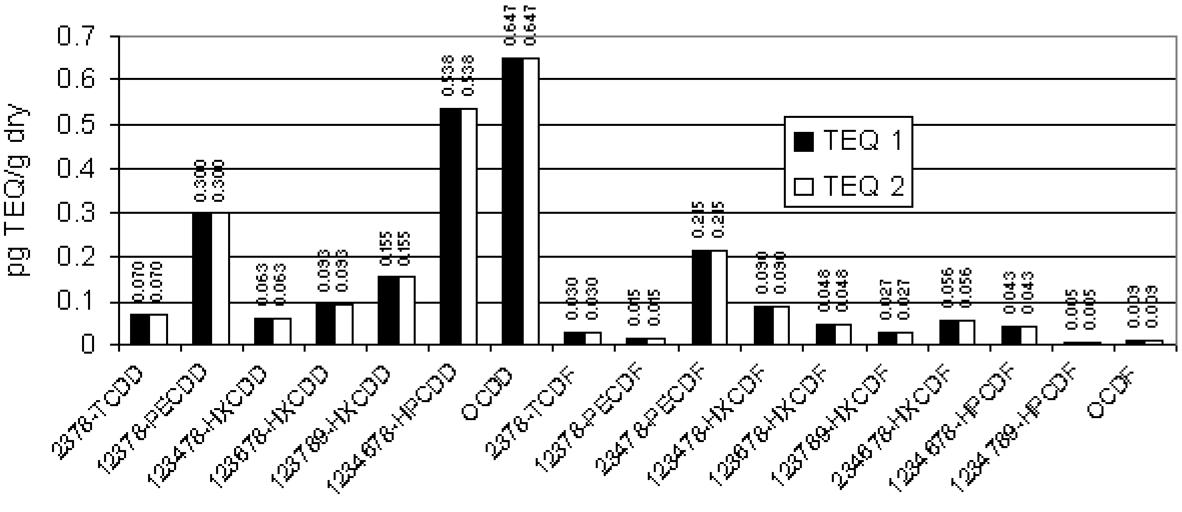
DIOXIN/FURAN AND PCB PROFILES



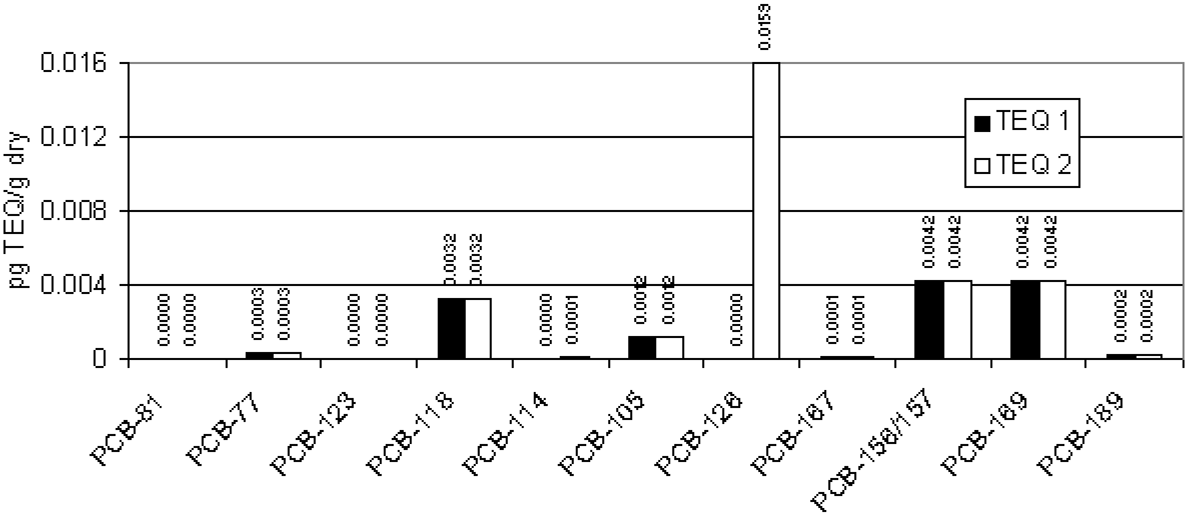
PCDD/PCDF Concentration Profiles for Site 1



Dioxin-like PCB Concentration Profiles for Site 1

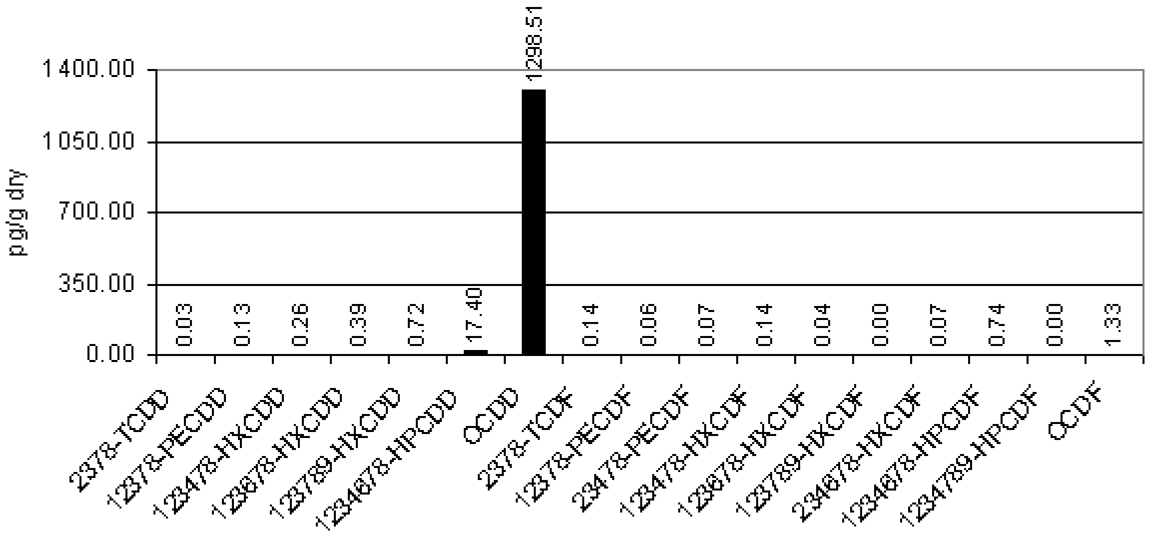


PCDD/PCDF TEQ Profiles for Site 1



Dioxin-like PCB TEQ Profiles for Site 1

Site 2
Clinton Crops, NC



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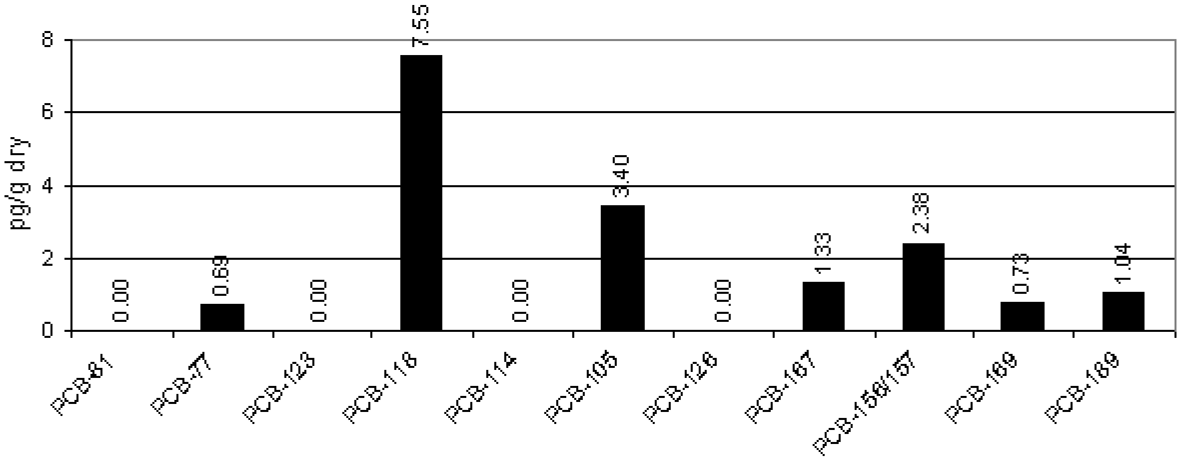
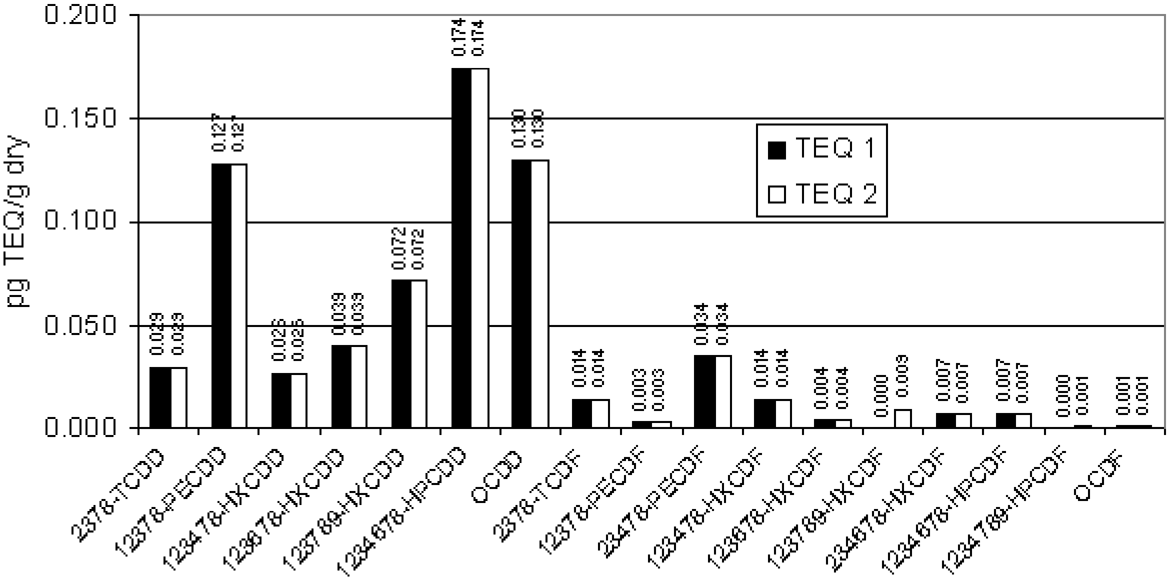
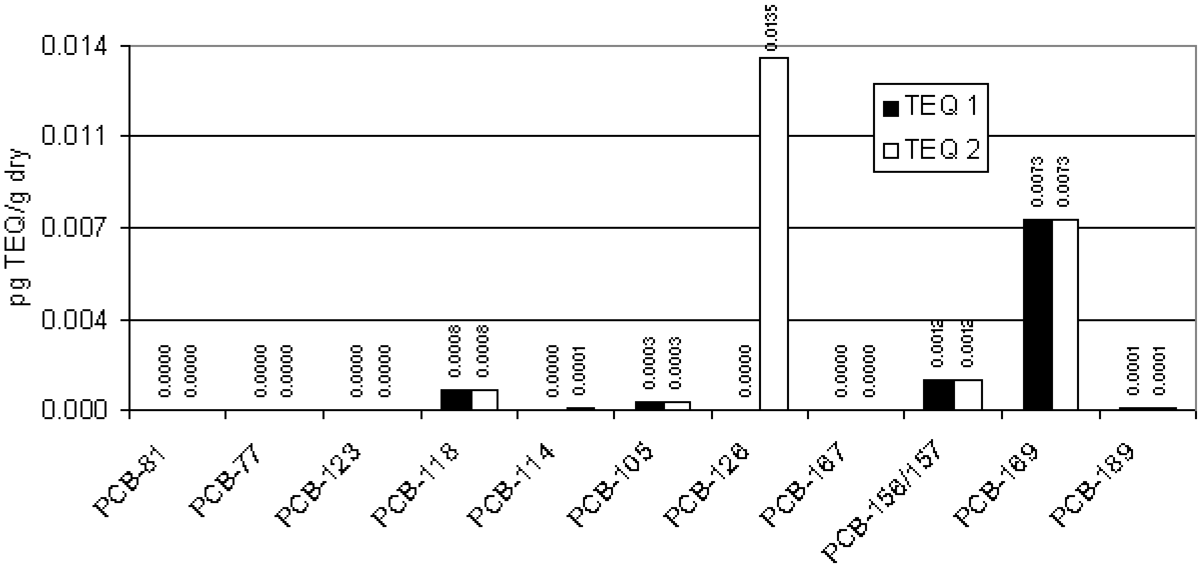


Dioxin-like PCB Concentration Profiles for Site 2

Site 2
Clinton Crops, NC

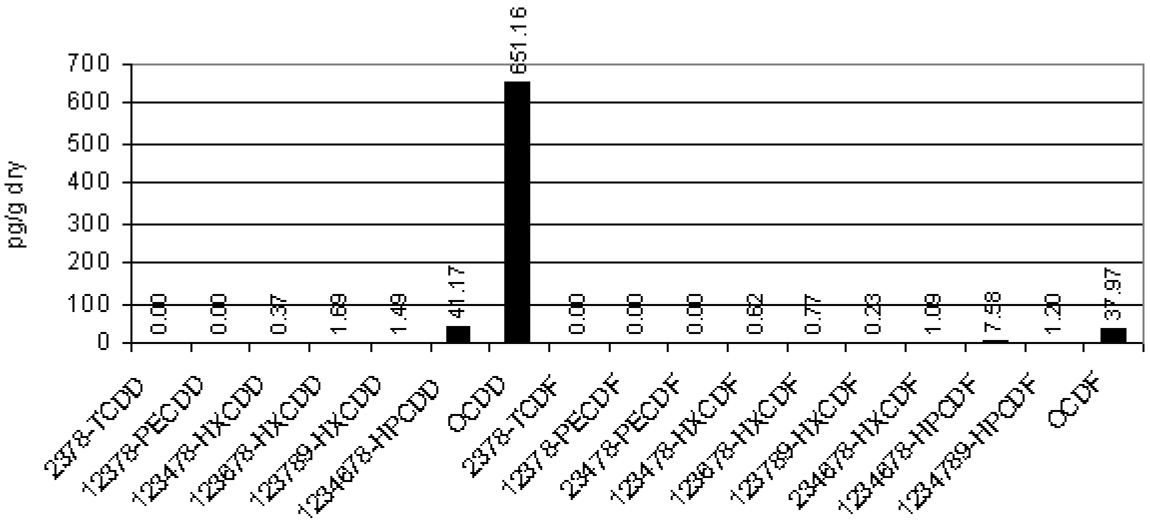


PCDD/PCDF TEQ Profiles for Site 2

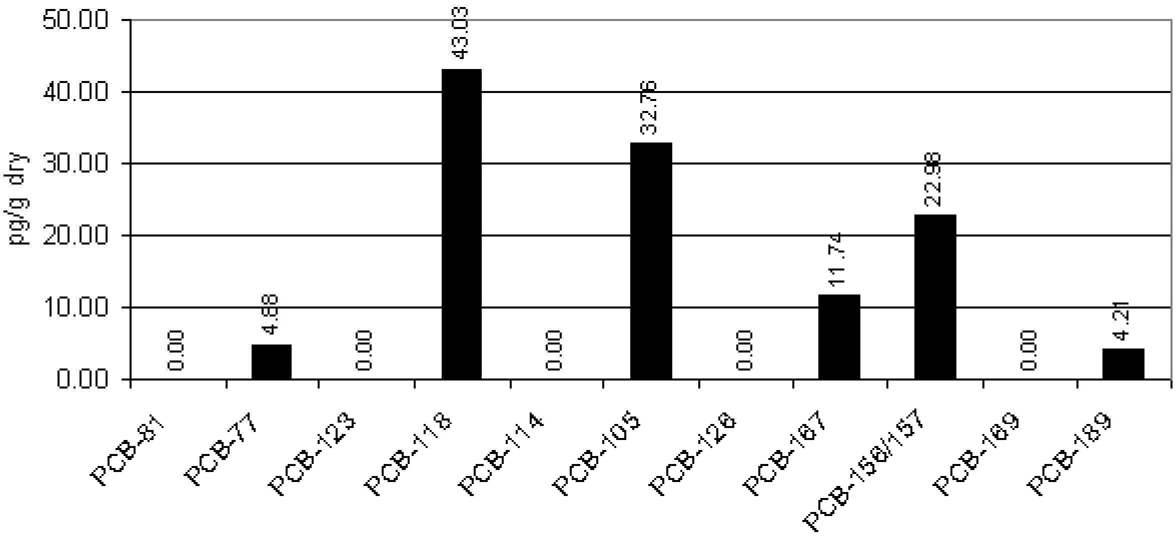


Dioxin-like PCB TEQ Profiles for Site 2

Site 4
Everglades, FL

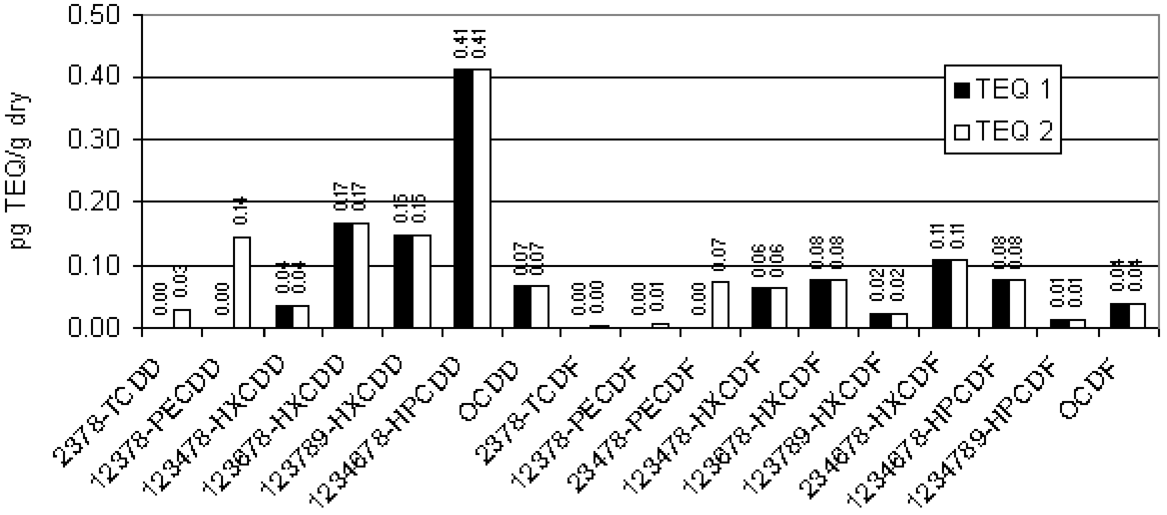


PCDD/PCDF Concentration Profiles for Site 4

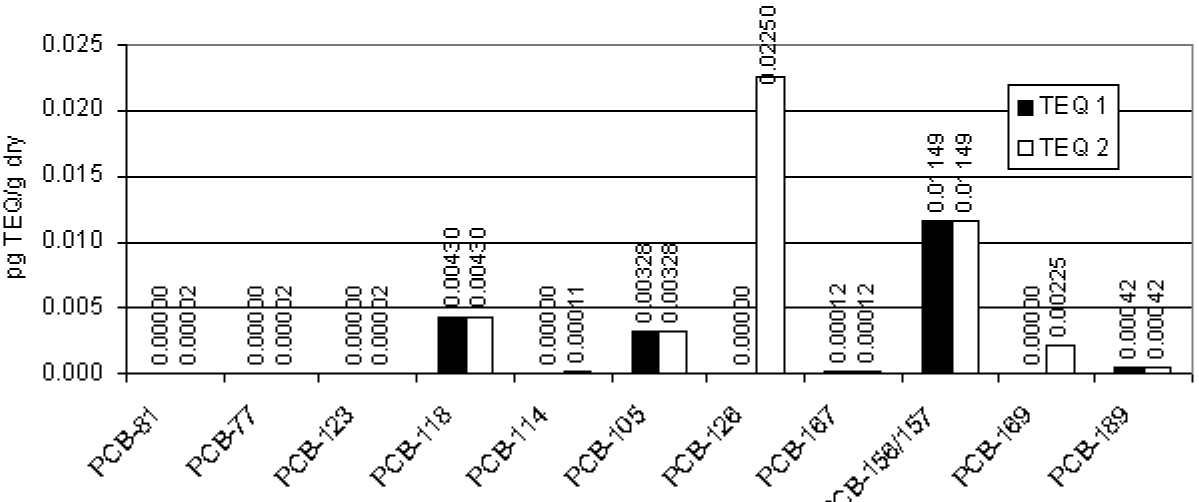


Dioxin-like PCB Concentration Profiles for Site 4

Site 4
Everglades, FL

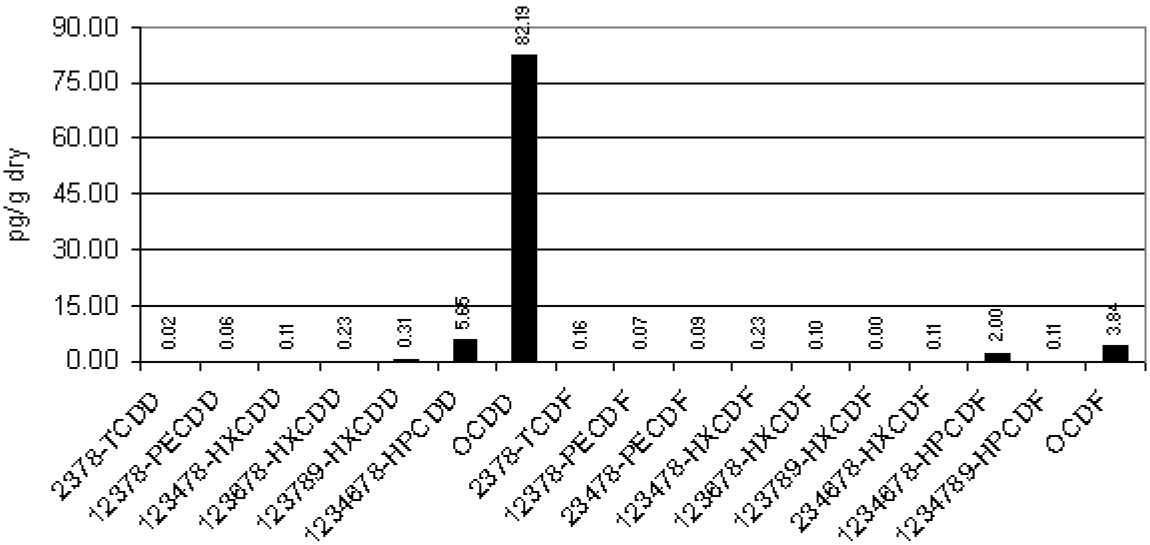


PCDD/PCDF TEQ Profiles for Site 4

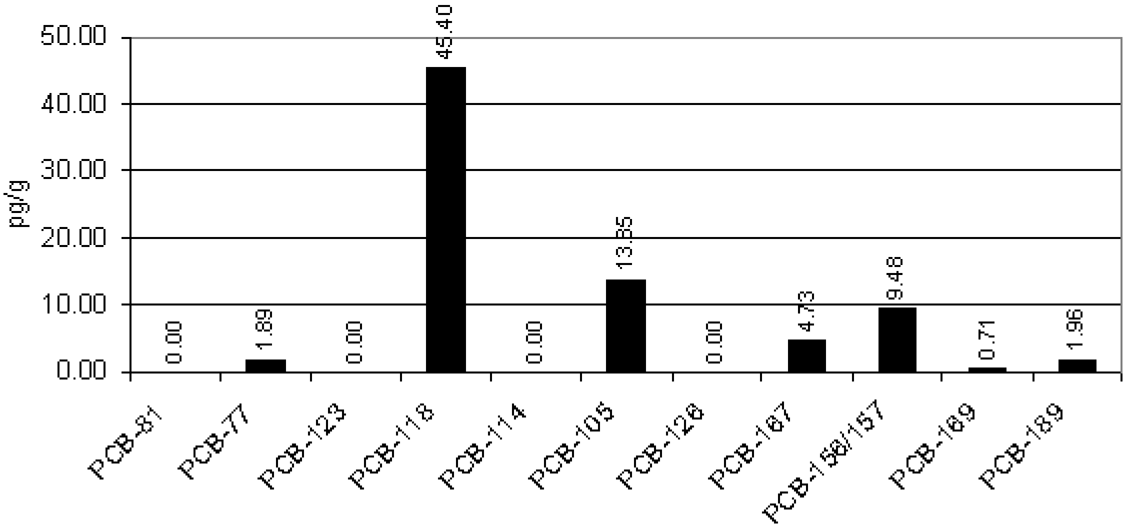


Dioxin-like PCB TEQ Profiles for Site 4

Site 5
Lake Dubay, WI

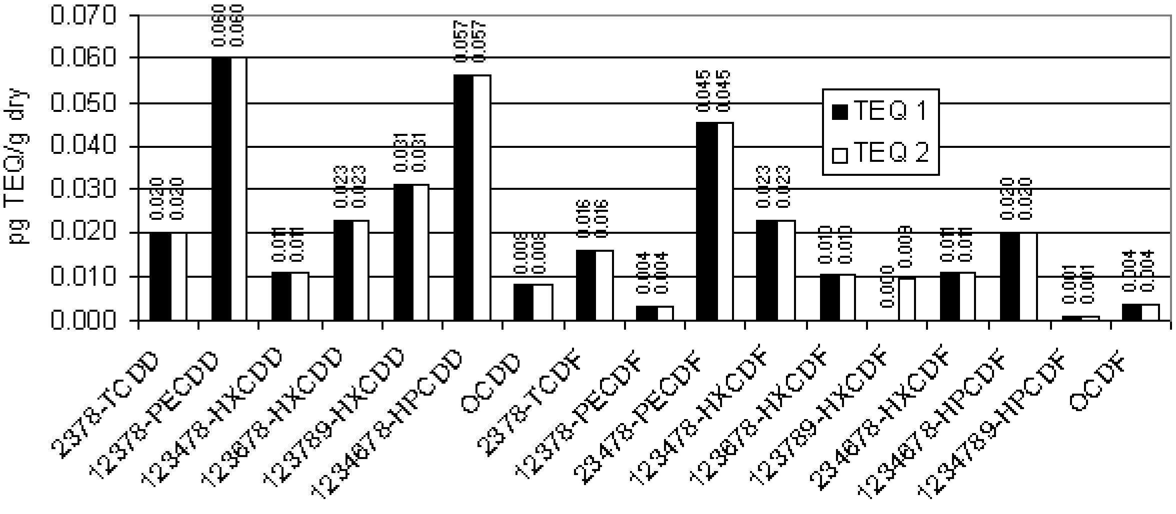


PCDD/PCDF Concentration Profiles for Site 5

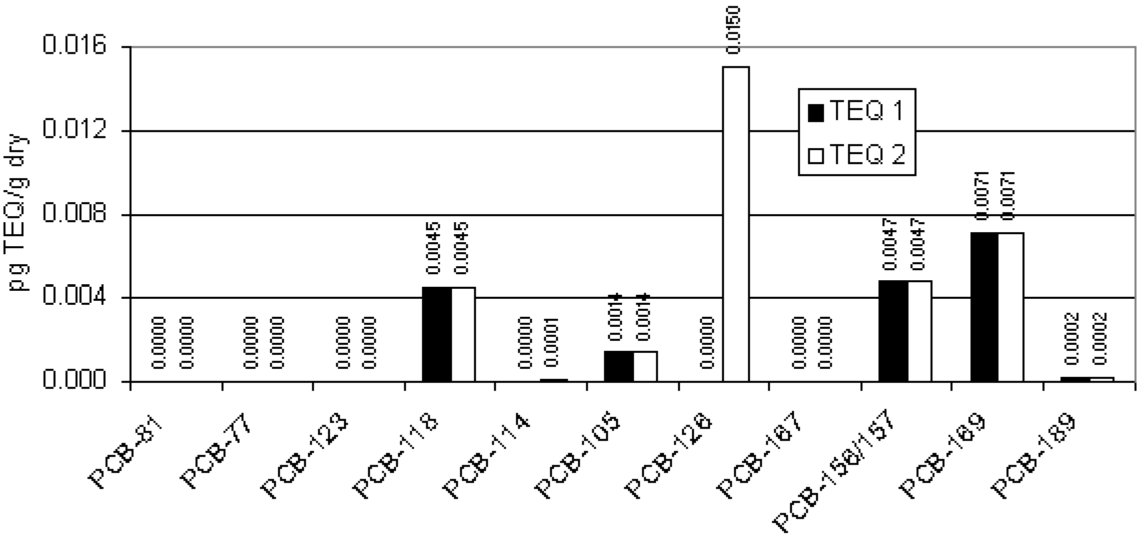


Dioxin-like PCB Concentration Profiles for Site 5

Site 5
Lake Dubay, WI

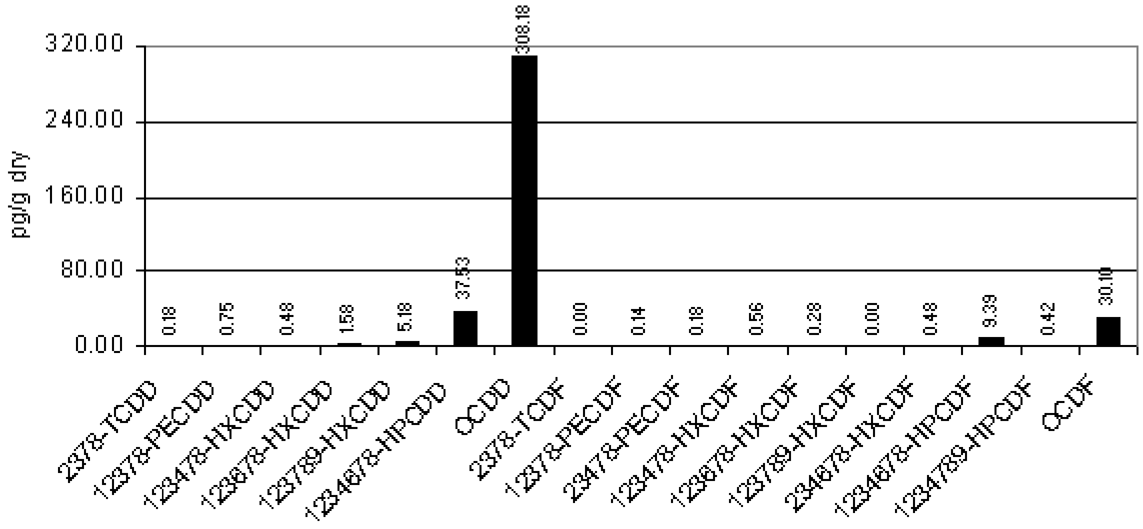


PCDD/PCDF TEQ Profiles for Site 5

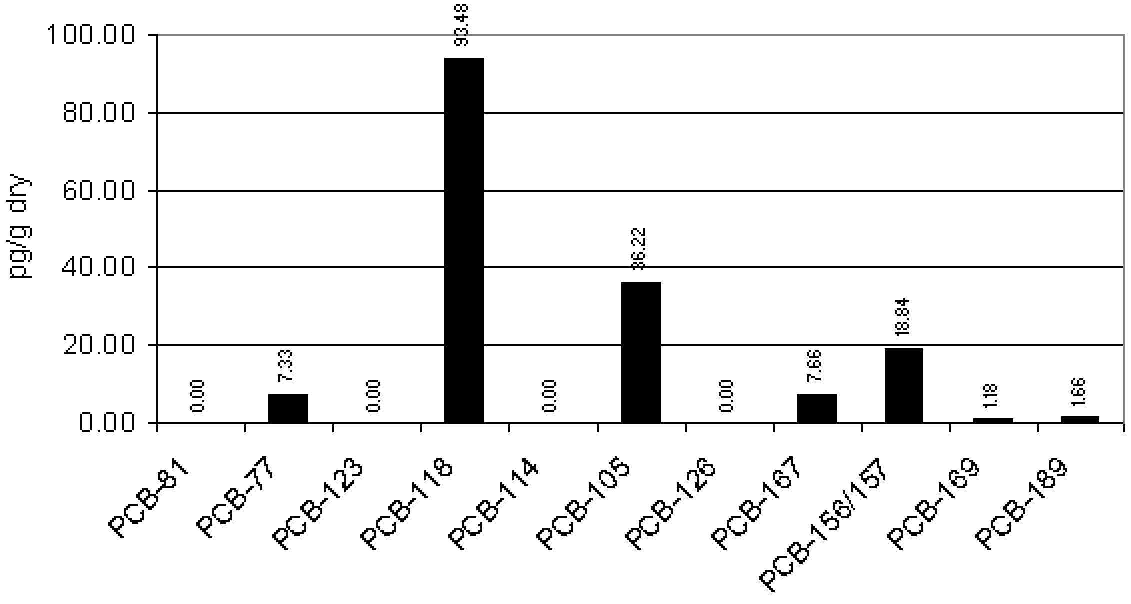


Dioxin-like PCB TEQ Profiles for Site 5

Site 6
Monmouth, IL

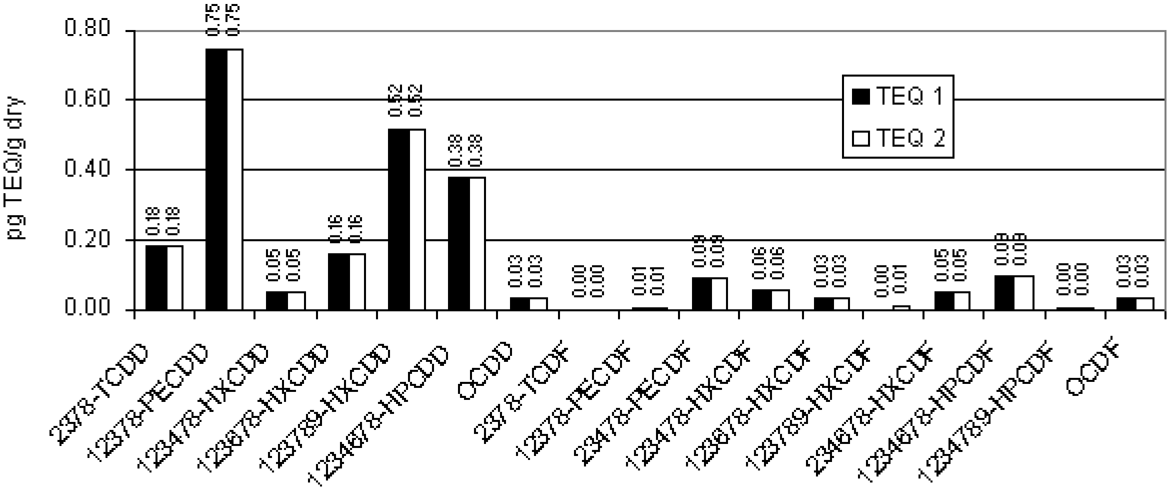


PCDD/PCDF Concentration Profiles for Site 6

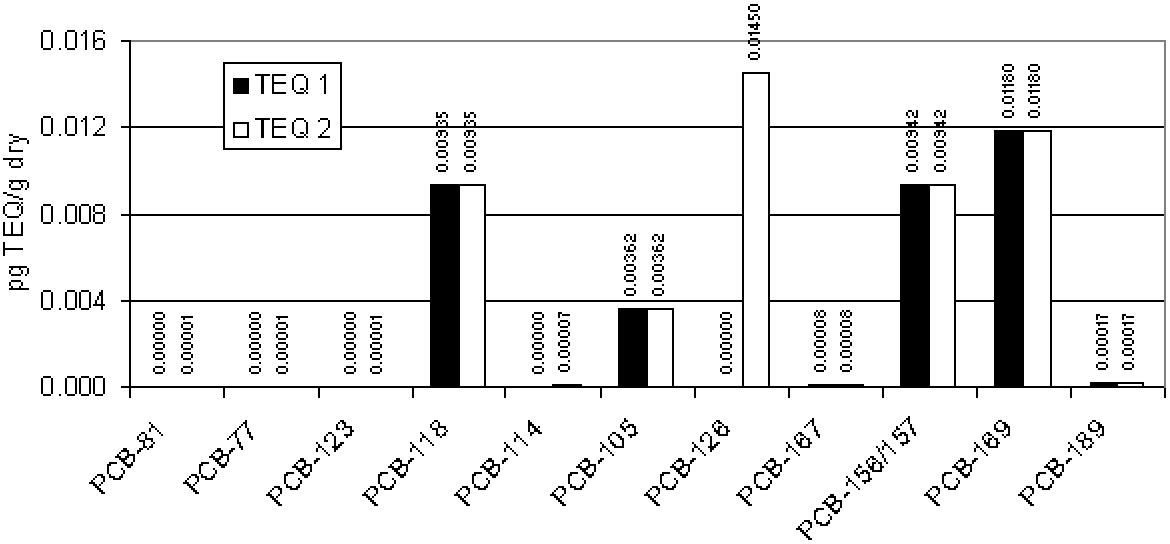


Dioxin-like PCB Concentration Profiles for Site 6

Site 6
Monmouth, IL

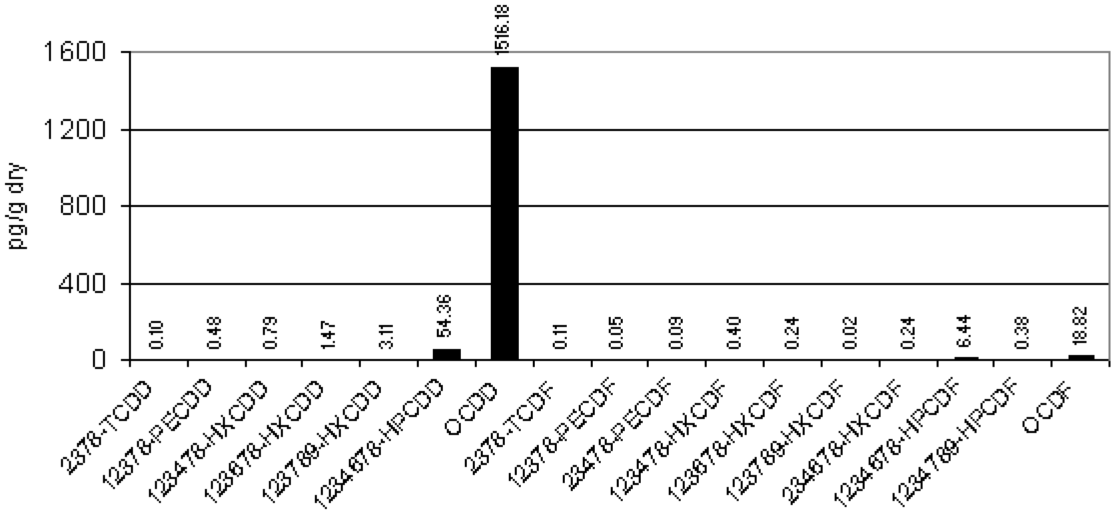


PCDD/PCDF TEQ Profiles for Site 6

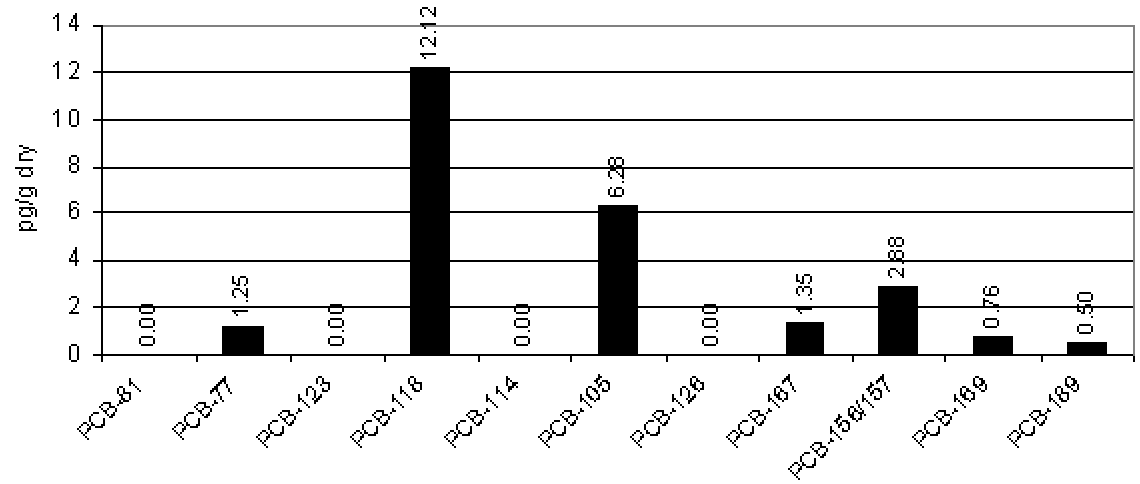


Dioxin-like PCB TEQ Profiles for Site 6

Site 7
McNay Farms, IA

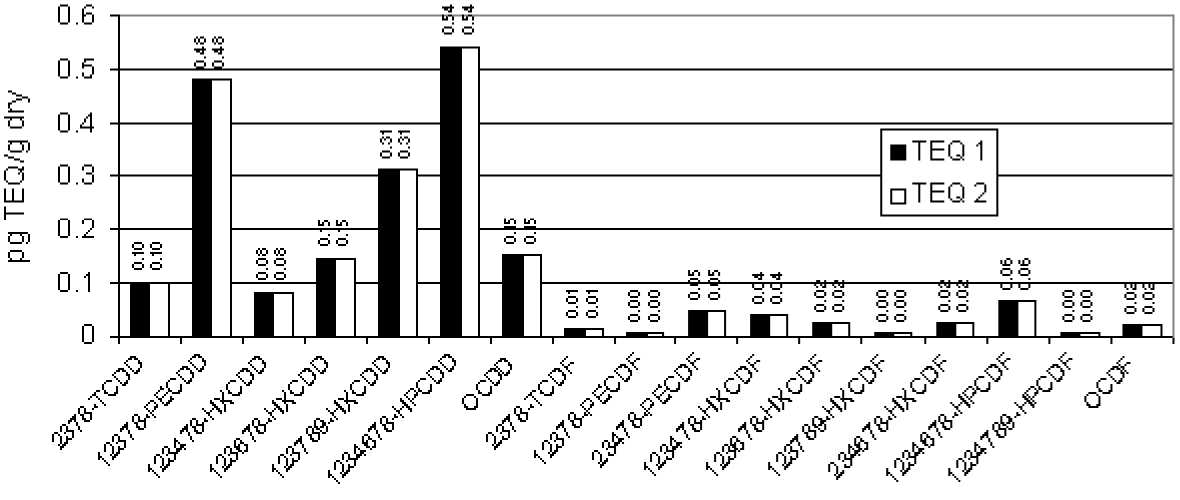


PCDD/PCDF Concentration Profiles for Site 7

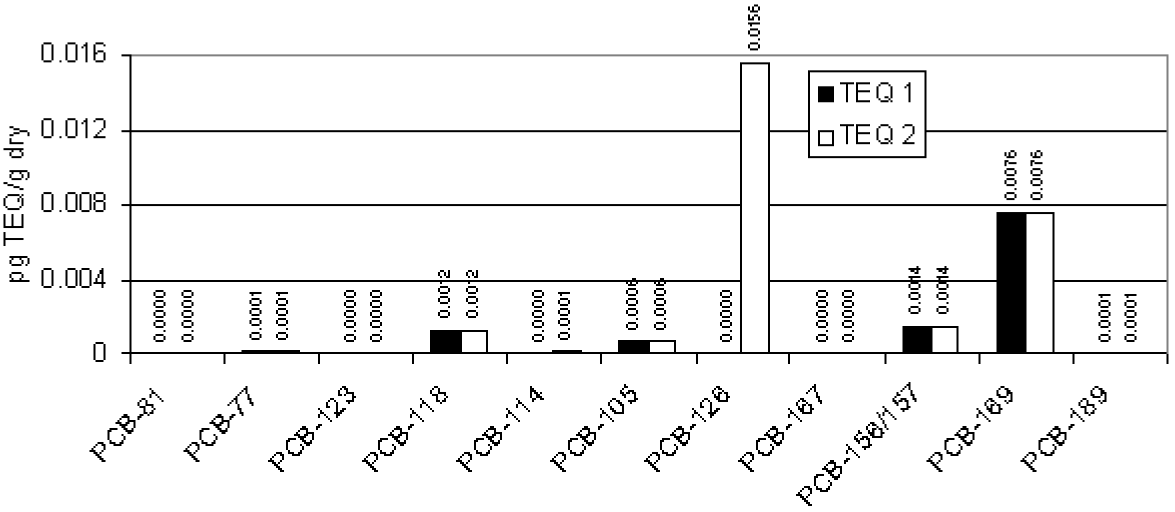


Dioxin-like PCB Concentration Profiles for Site 7

Site 7
McNay Farms, IA

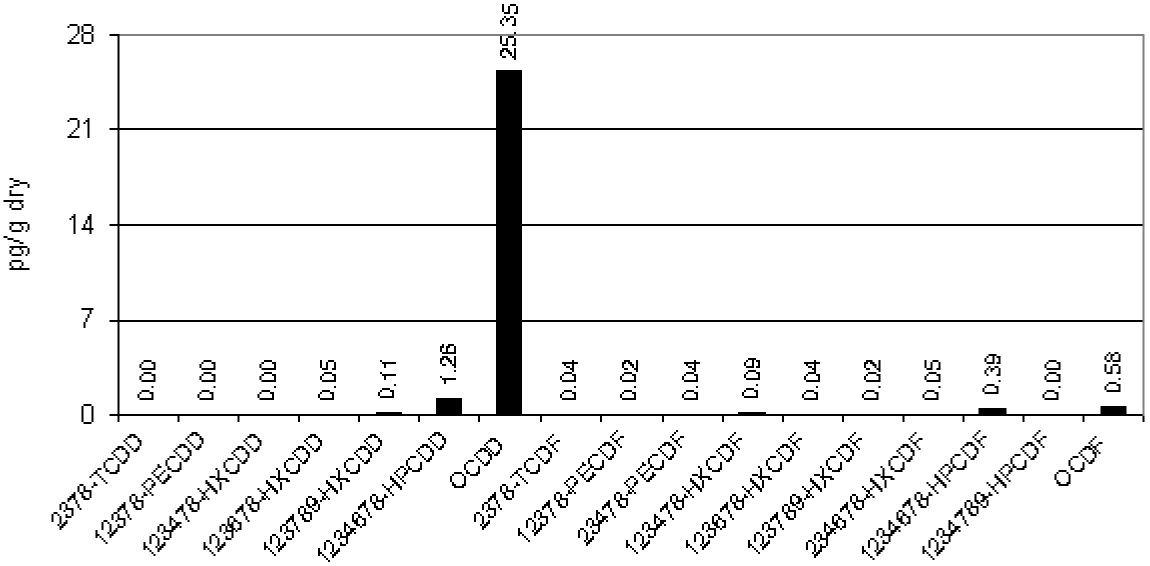


PCDD/PCDF TEQ Profiles for Site 7

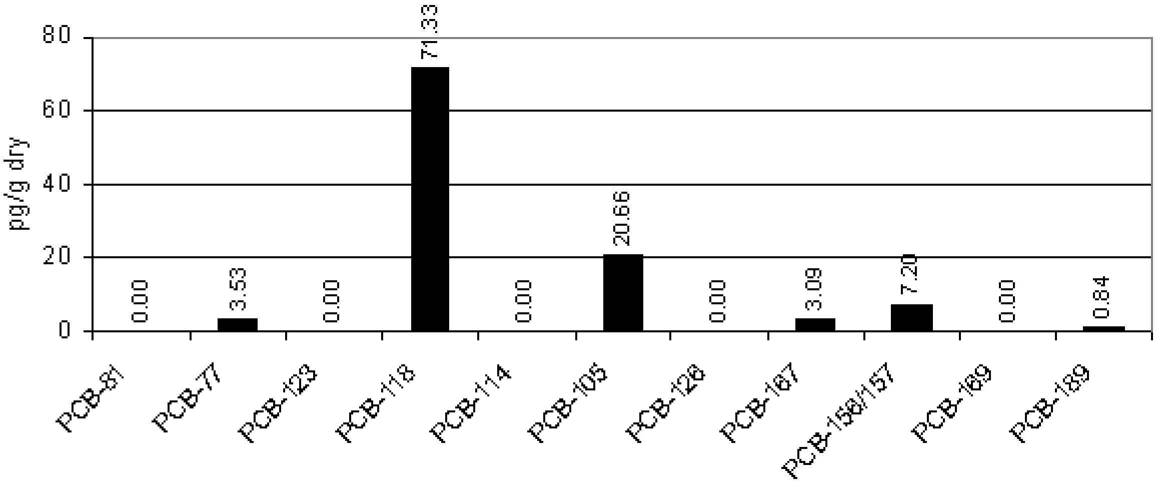


Dioxin-like PCB TEQ Profiles for Site 7

Site 8
Lake Scott, KS

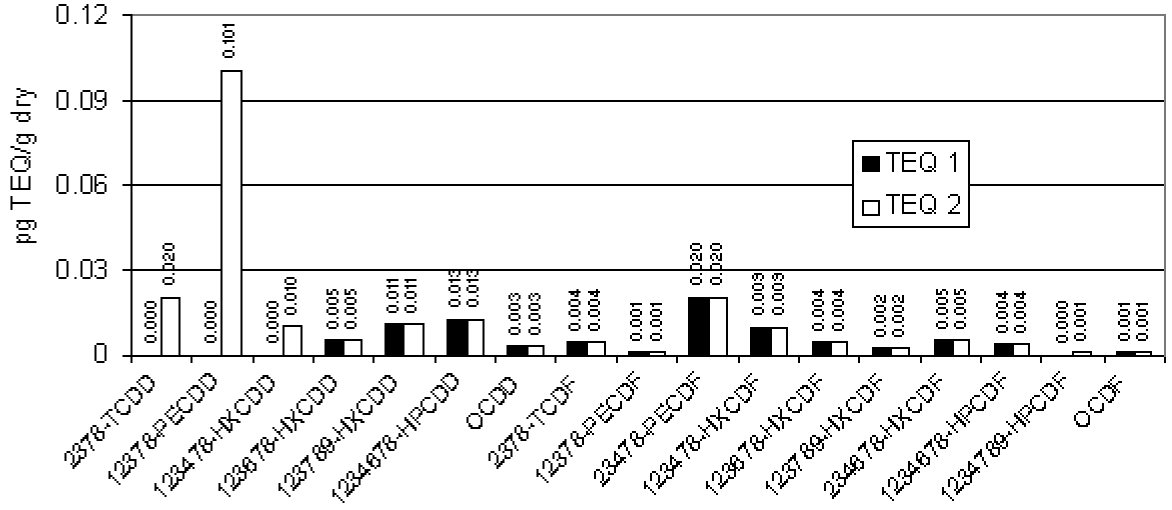


PCDD/PCDF Concentration Profiles for Site 8

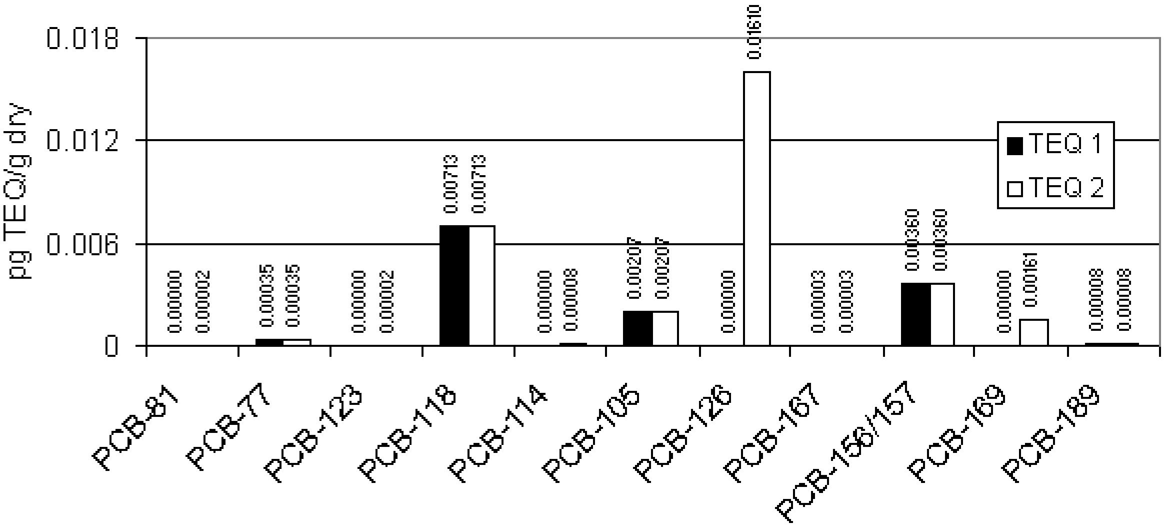


Dioxin-like PCB Concentration Profiles for Site 8

Site 8
Lake Scott, KS

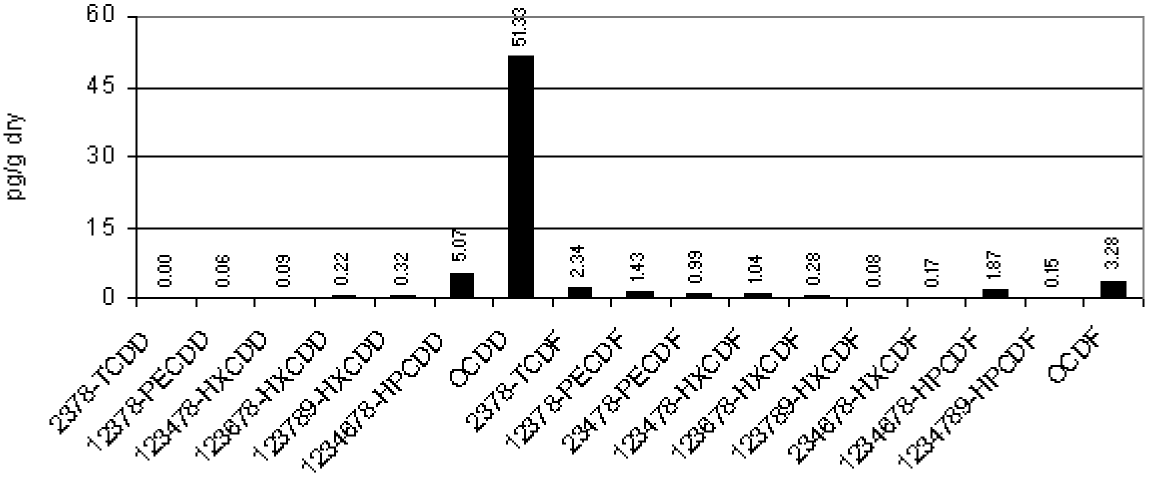


PCDD/PCDF TEQ Profiles for Site 8

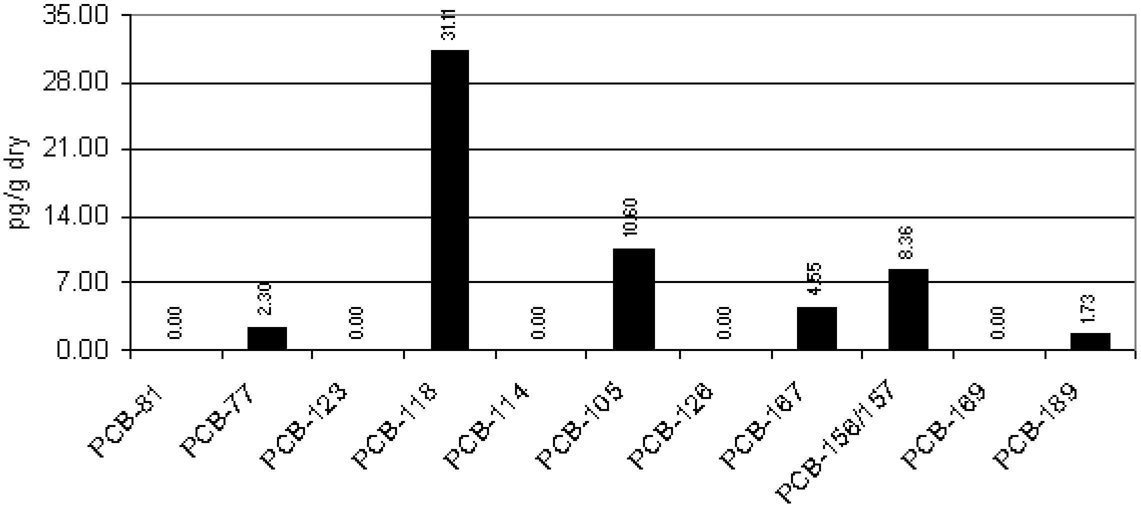


Dioxin-like PCB TEQ Profiles for Site 8

Site 9
Keystone State Park, OK

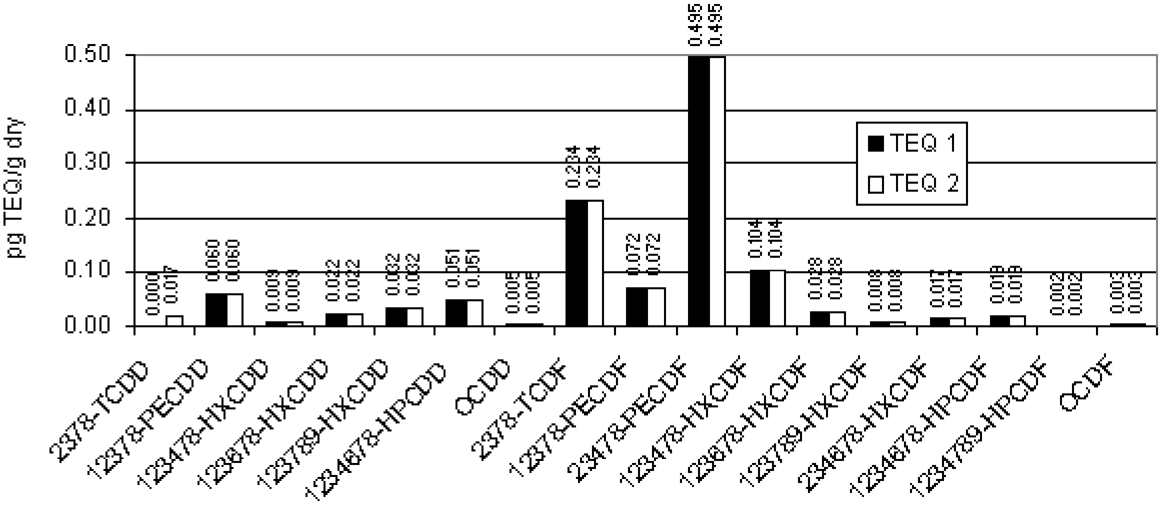


PCDD/PCDF Concentration Profiles for Site 9

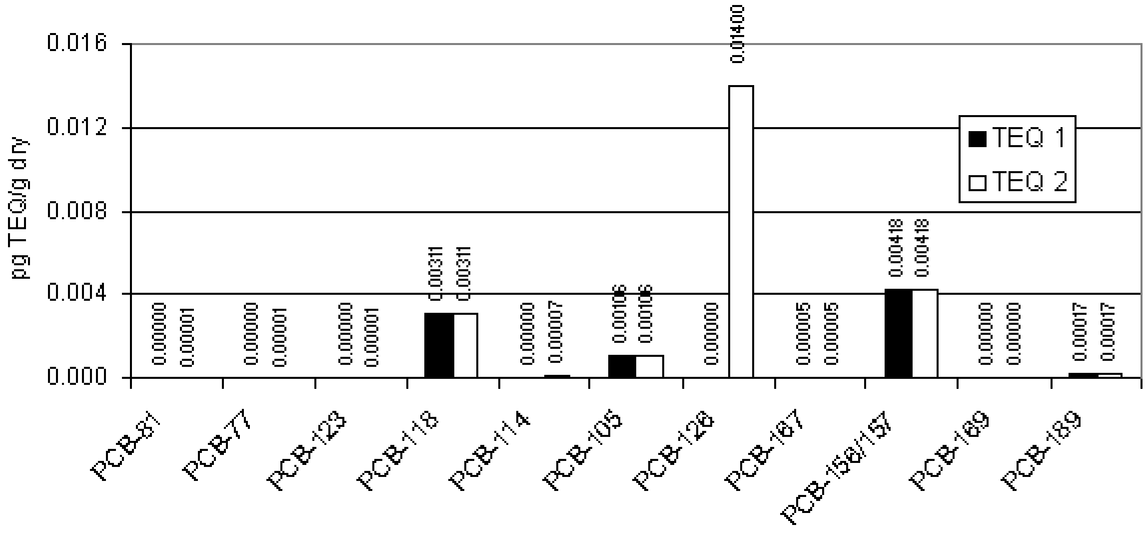


Dioxin-like PCB Concentration Profiles for Site 9

Site 9
Keystone State Park, OK

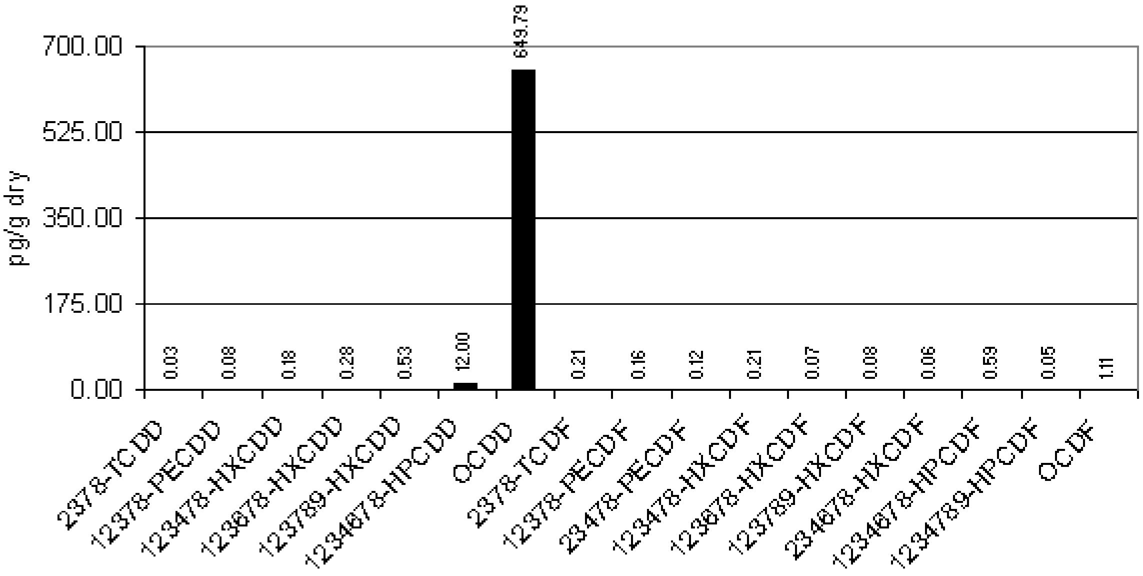


PCDD/PCDF TEQ Profiles for Site 9

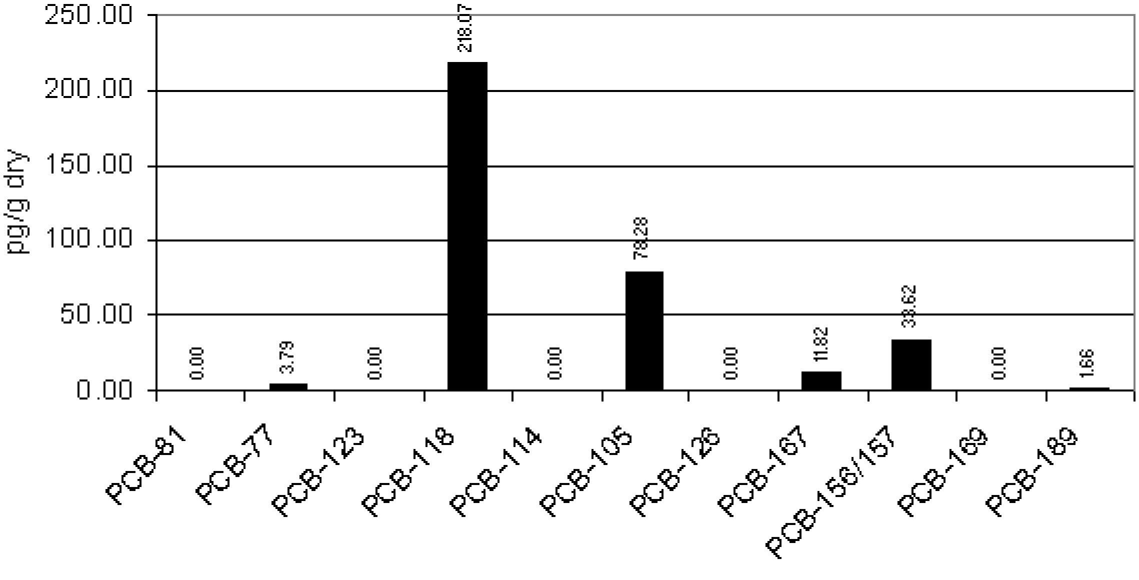


Dioxin-like PCB TEQ Profiles for Site 9

Site 10
Arkadelphia, AR

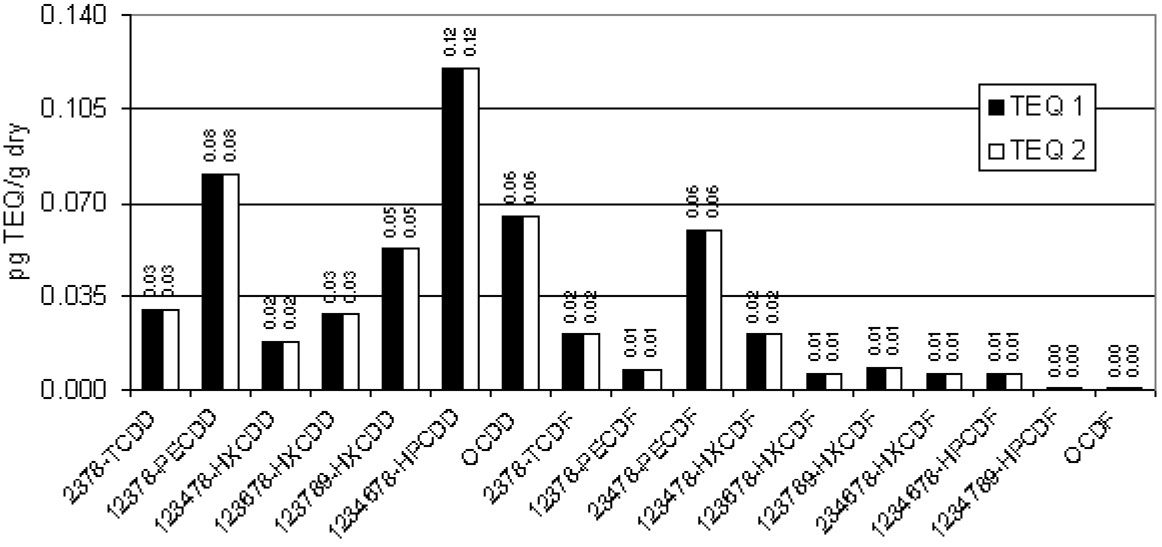


PCDD/PCDF Concentration Profiles for Site 10

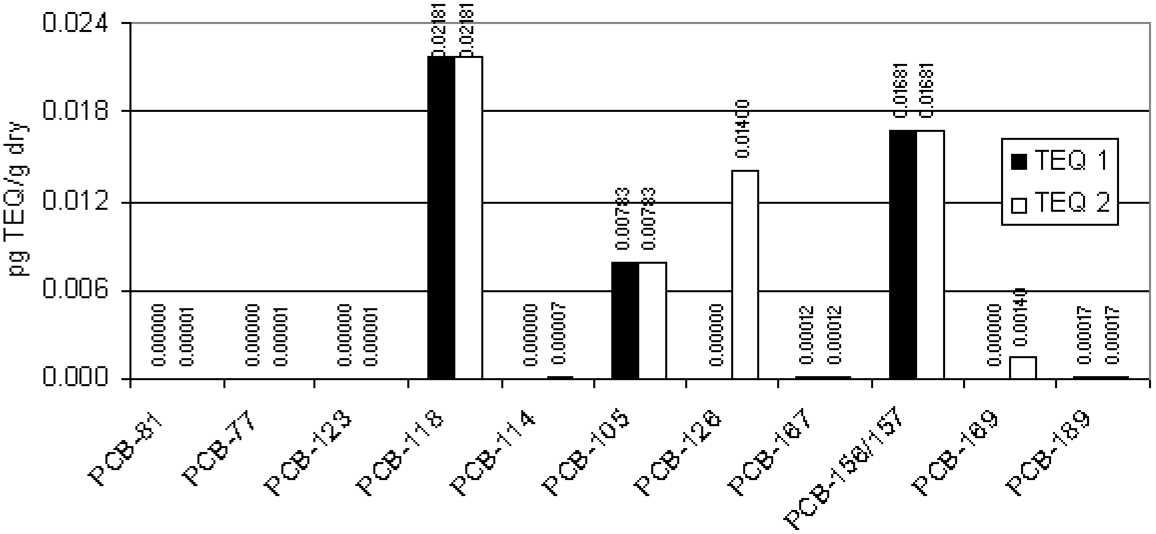


Dioxin-like PCB Concentration Profiles for Site 10

Site 10
Arkadelphia, AR

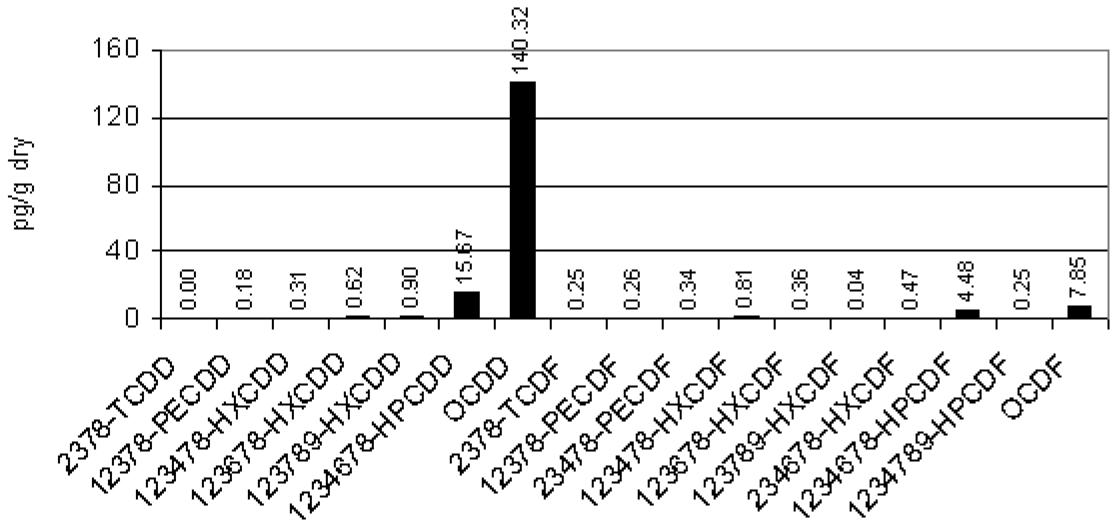


PCDD/PCDF TEQ Profiles for Site 10

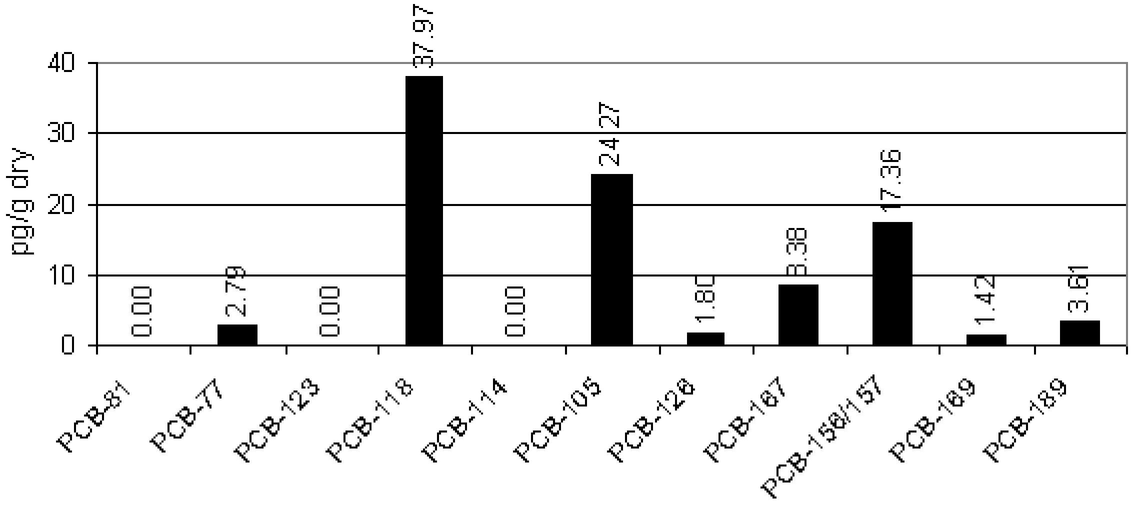


Dioxin-like PCB TEQ Profiles for Site 10

Site 11
Bennington, VT

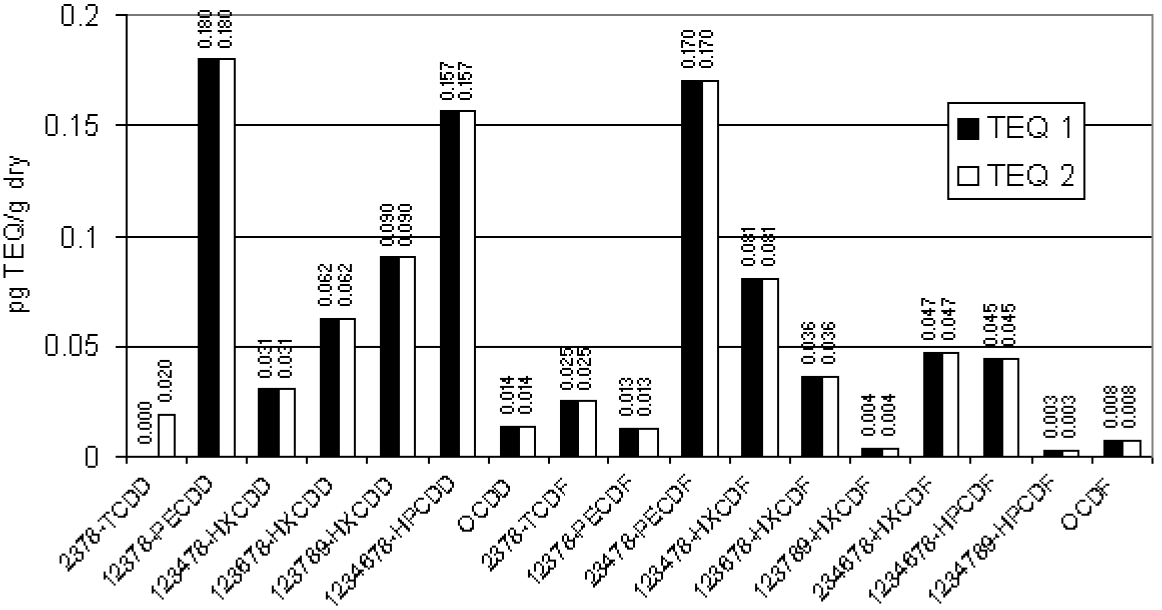


PCDD/PCDF Concentration Profiles for Site 11

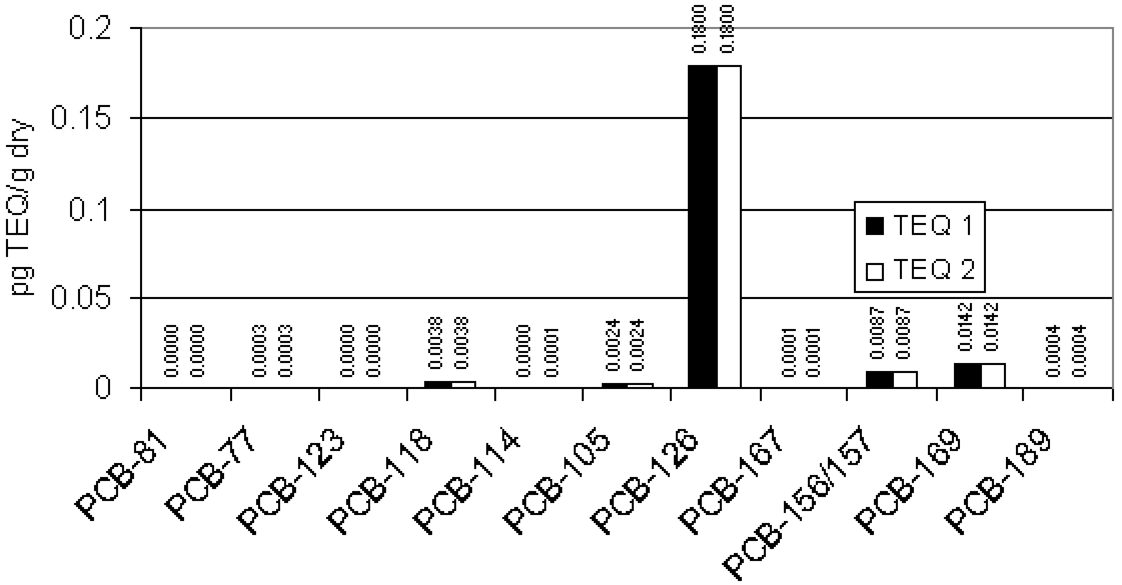


Dioxin-like PCB Concentration Profiles for Site 11

Site 11
Bennington, VT

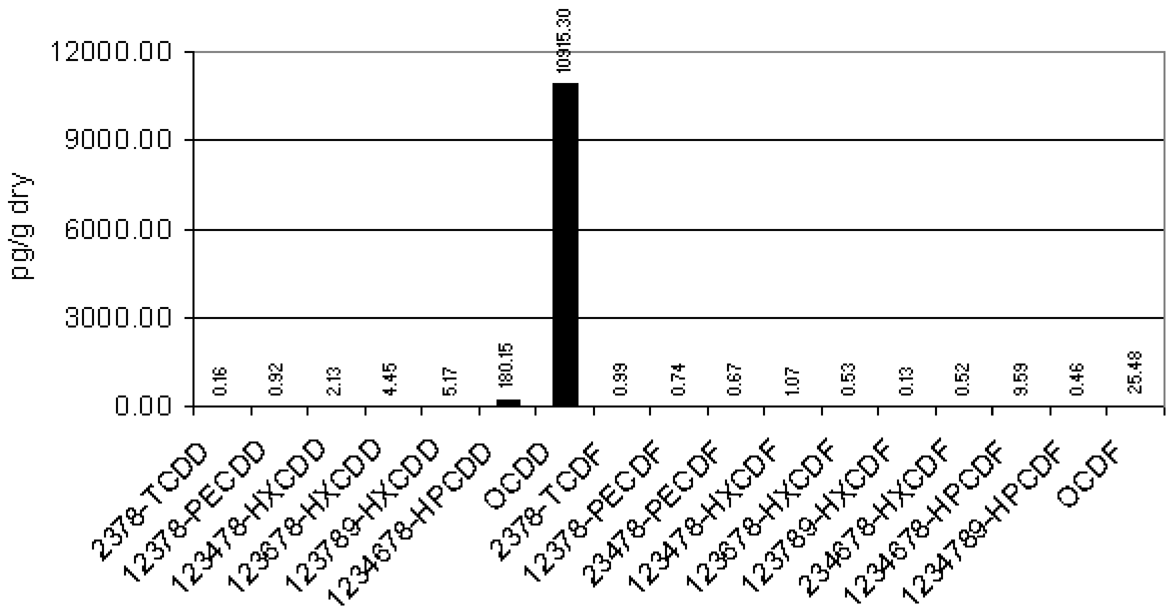


PCDD/PCDF TEQ Profiles for Site 11

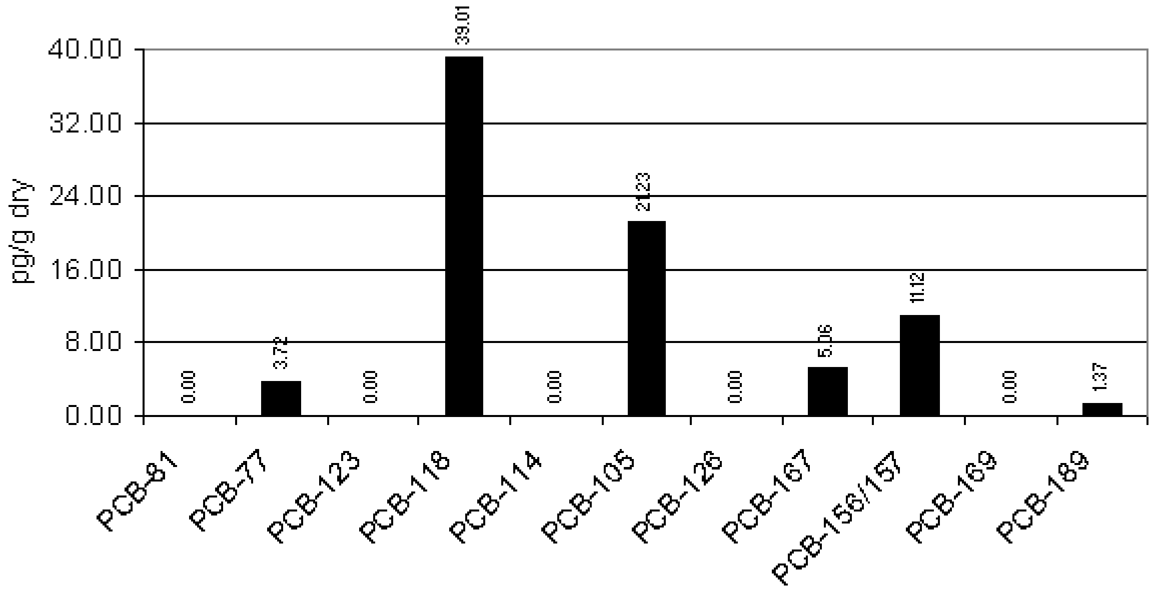


Dioxin-like PCB TEQ Profiles for Site 11

Site 12
Jasper, NY

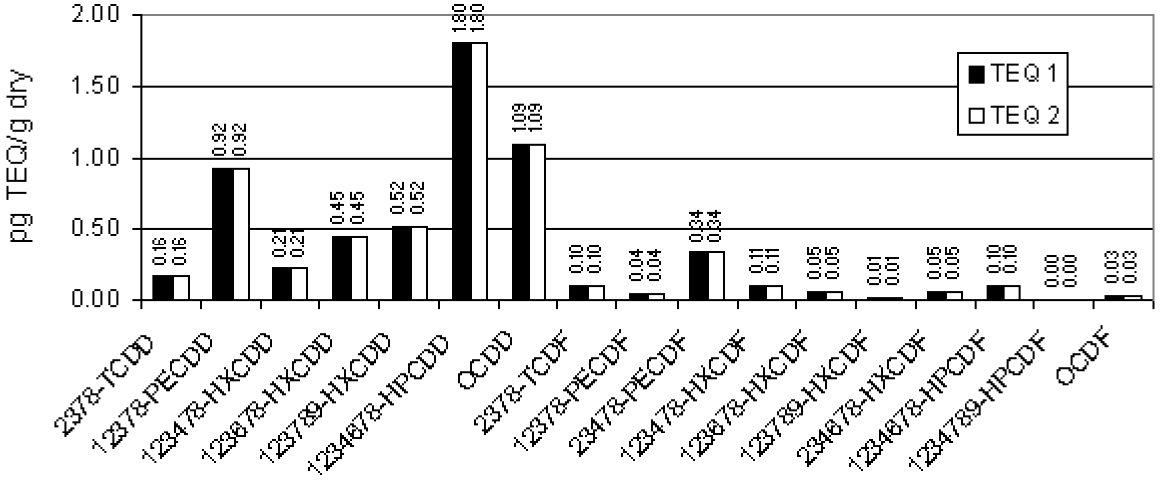


PCDD/PCDF Concentration Profiles for Site 12

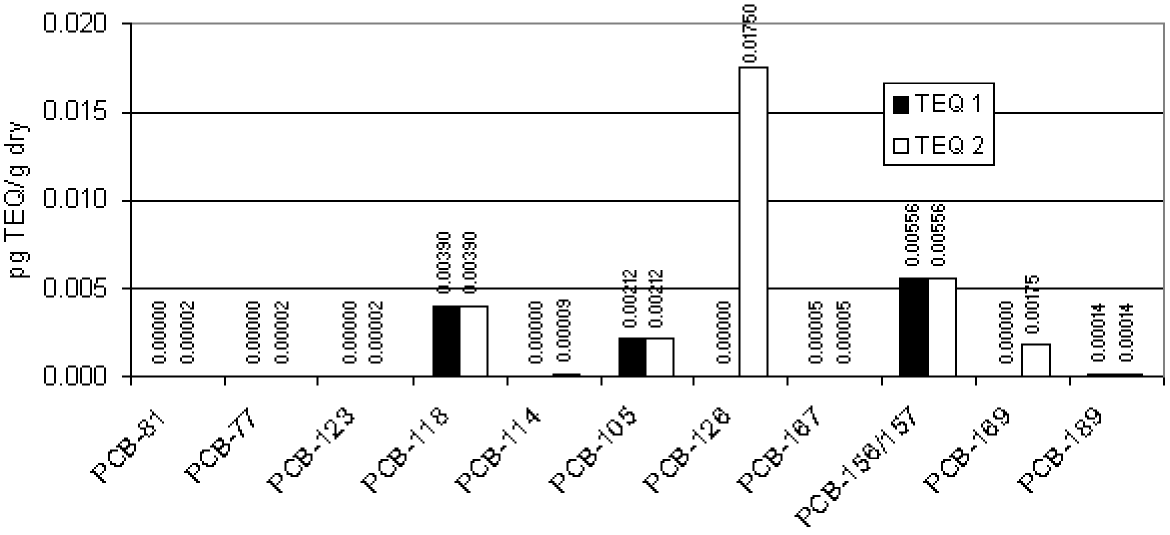


Dioxin-like PCB Concentration Profiles for Site 12

Site 12
Jasper, NY

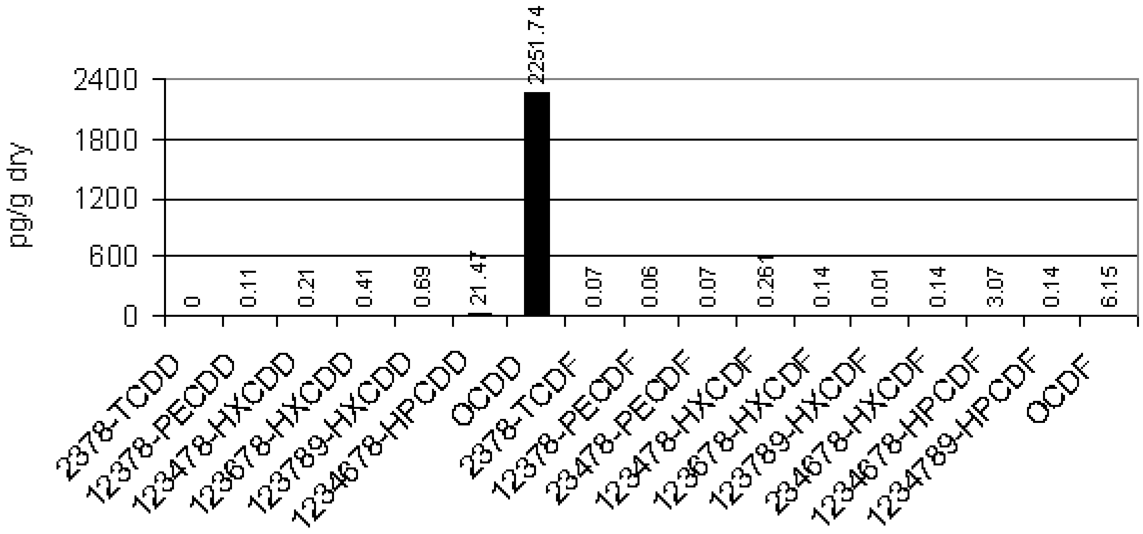


PCDD/PCDF TEQ Profiles for Site 12

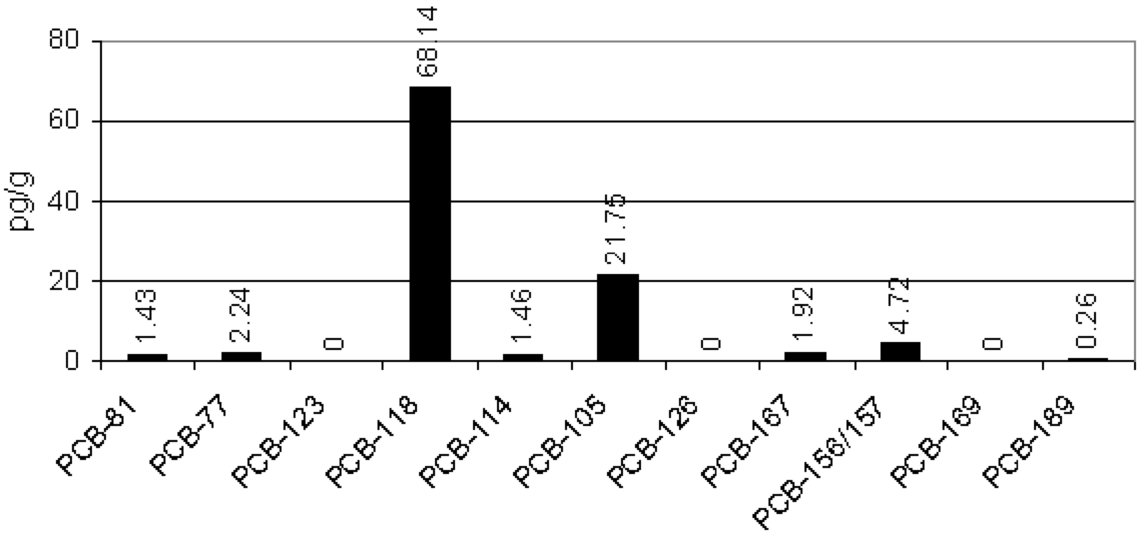


Dioxin-like PCB TEQ Profiles for Site 12

Site 14
Caldwell, OH

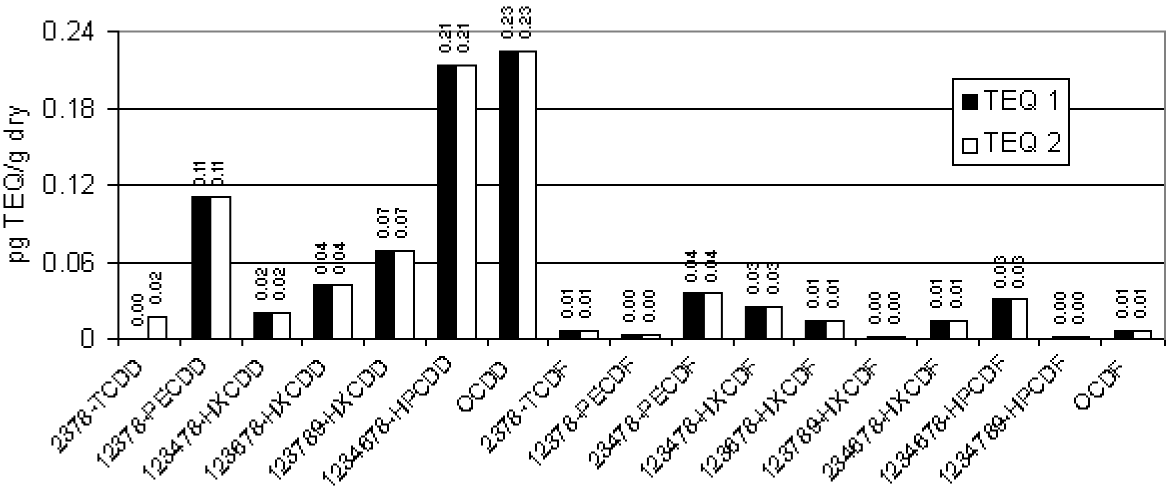


PCDD/PCDF Concentration Profiles for Site 14

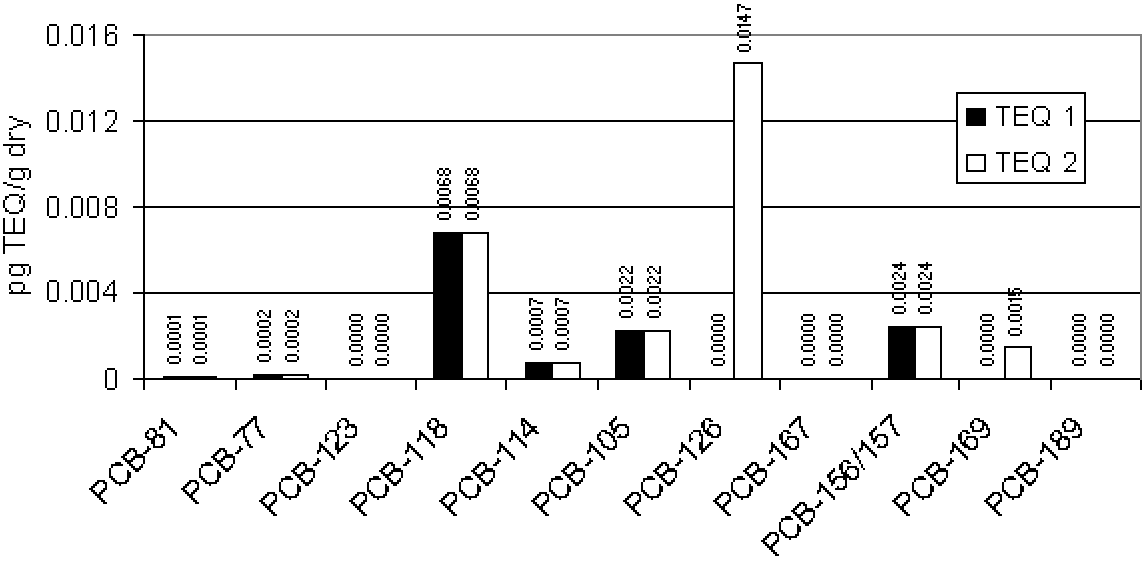


Dioxin-like PCB Concentration Profiles for Site 14

Site 14
Caldwell, OH

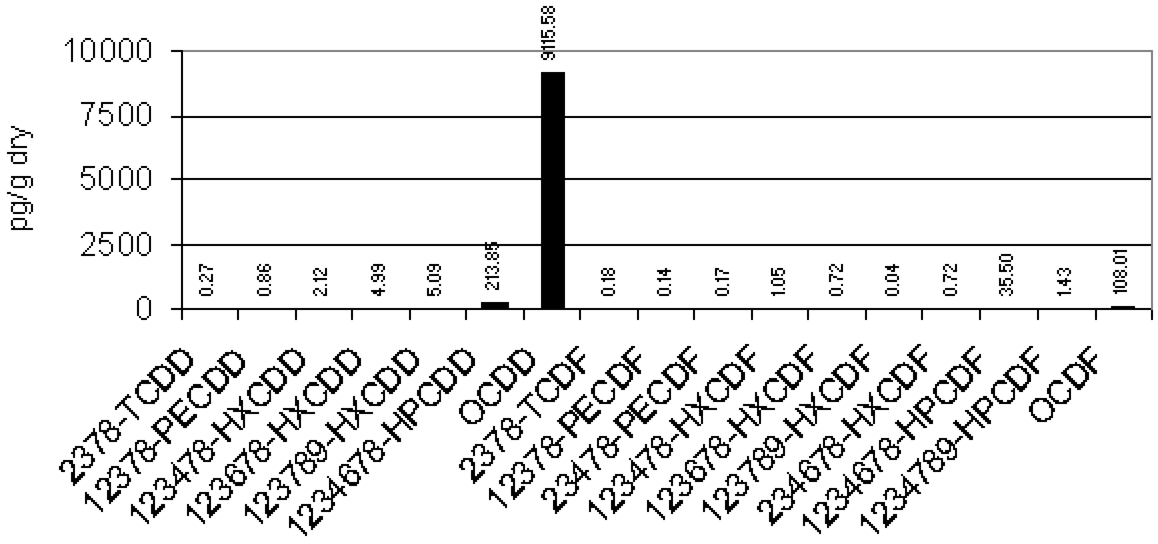


PCDD/PCDF TEQ Profiles for Site 14

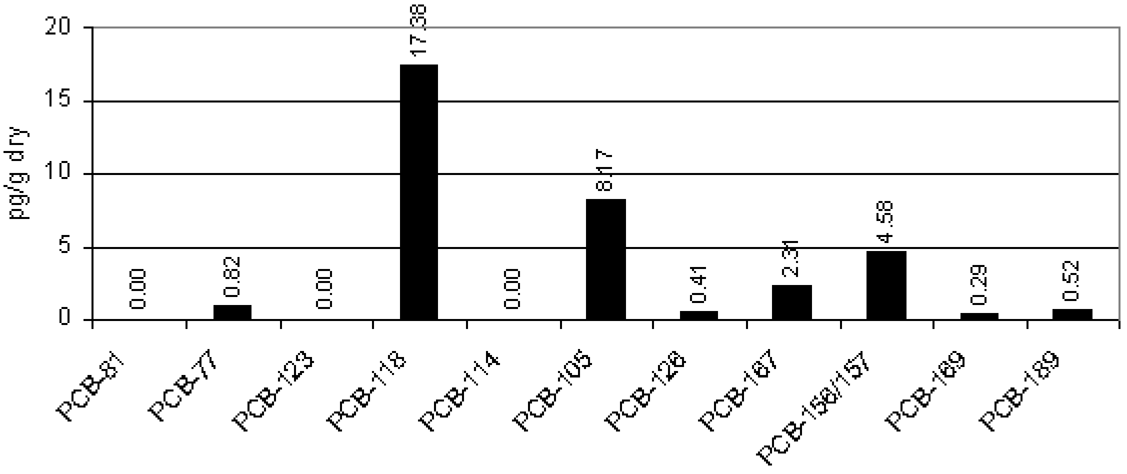


Dioxin-like PCB TEQ Profiles for Site 14

Site 16
Dixon Springs, IL

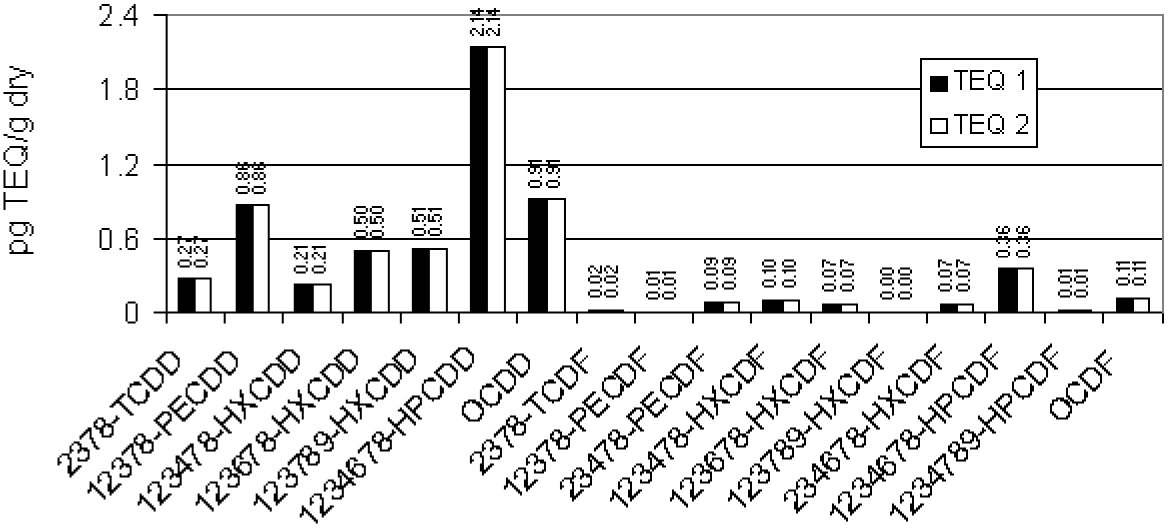


PCDD/PCDF Concentration Profiles for Site 16

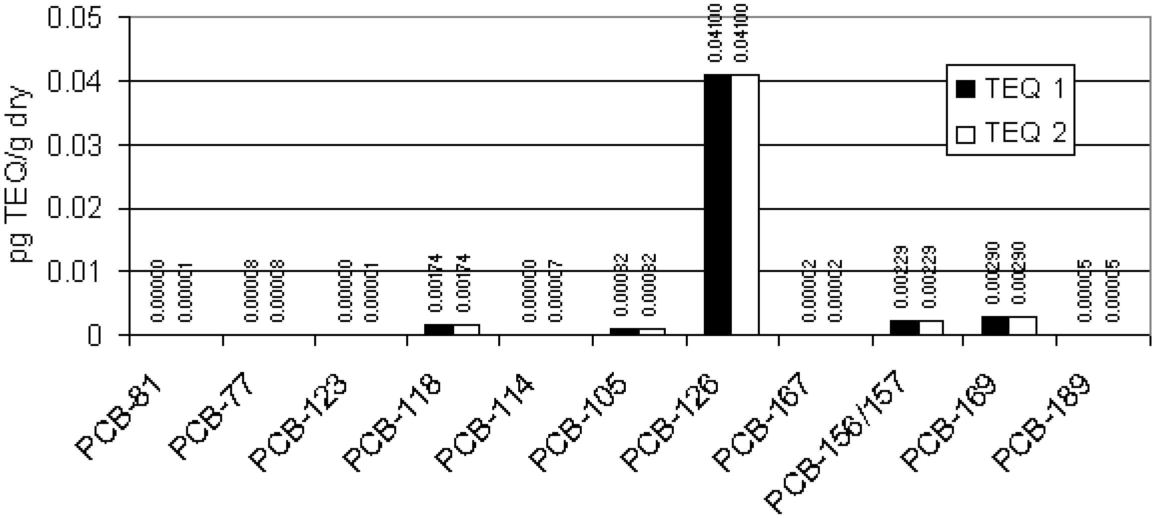


Dioxin-like PCB Concentration Profiles for Site 16

Site 16
Dixon Springs, IL

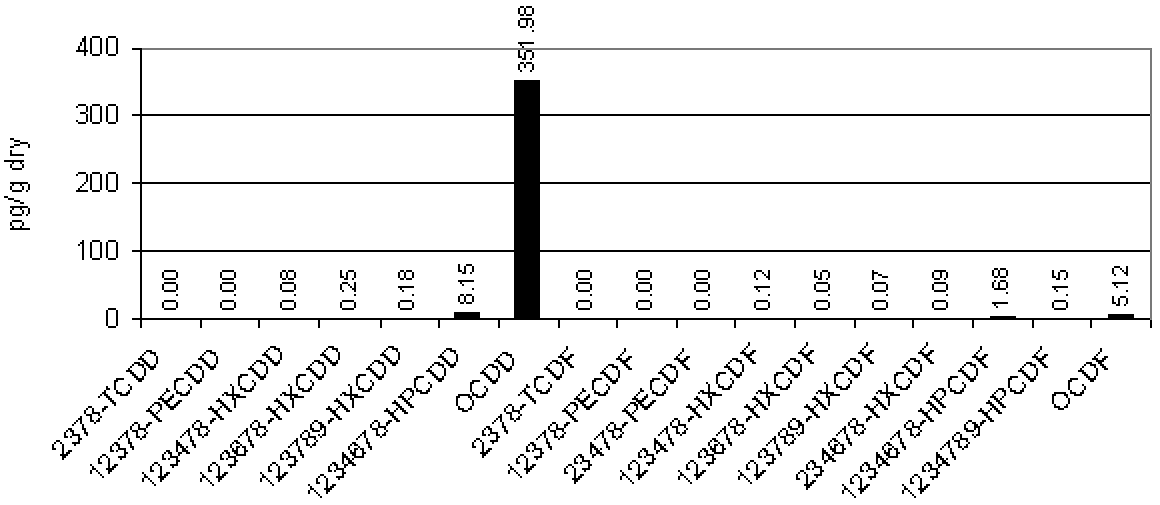


PCDD/PCDF TEQ Profiles for Site 16

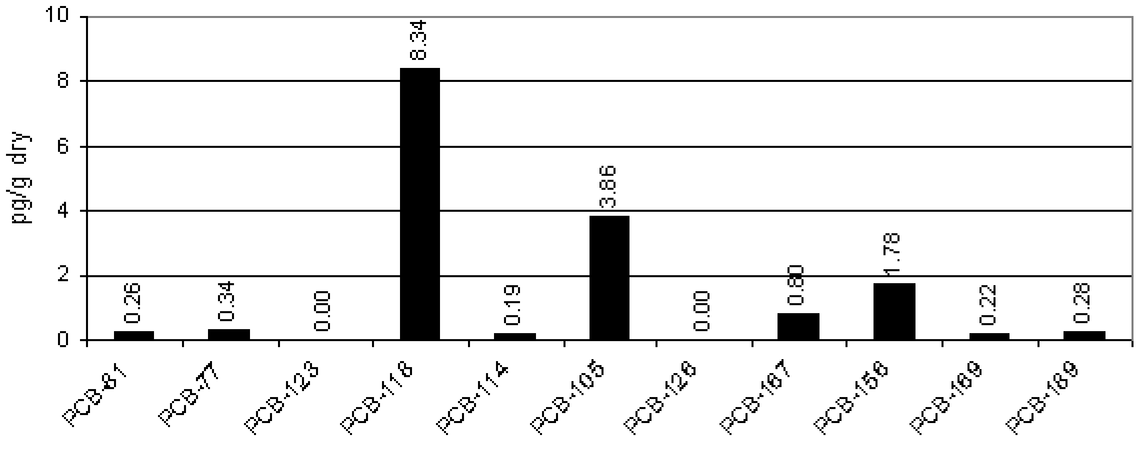


Dioxin-like PCB TEQ Profiles for Site 16

Site 17
Quincy, FL

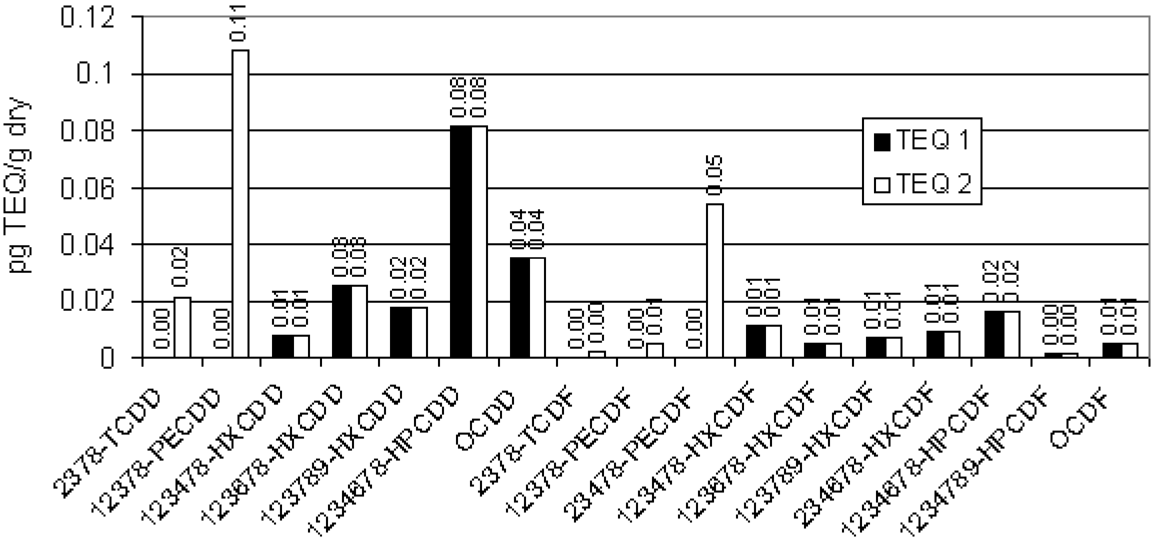


PCDD/PCDF Concentration Profiles for Site 17

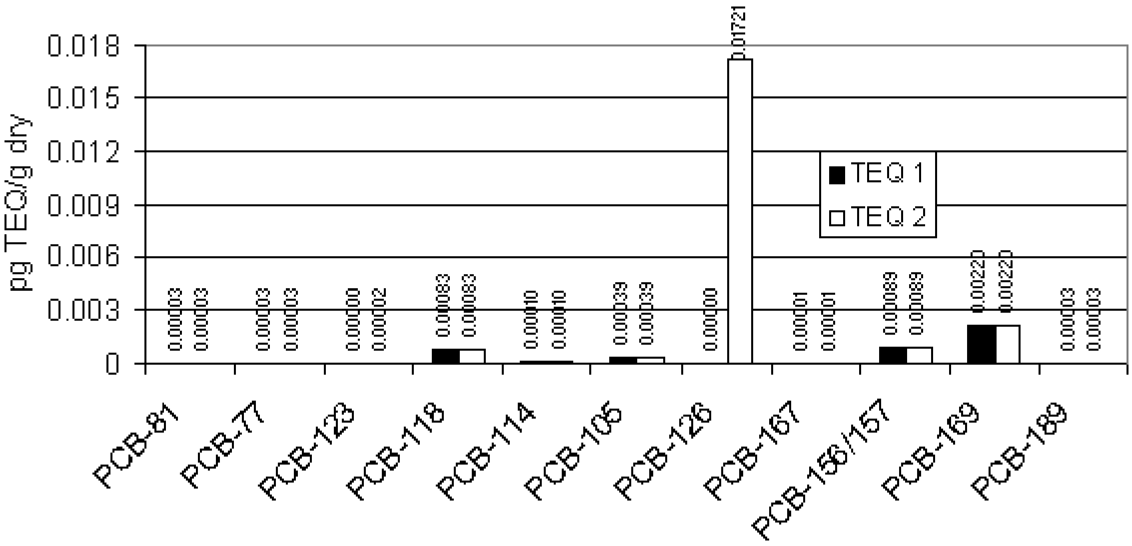


Dioxin-like PCB Concentration Profiles for Site 17

Site 17
Quincy, FL

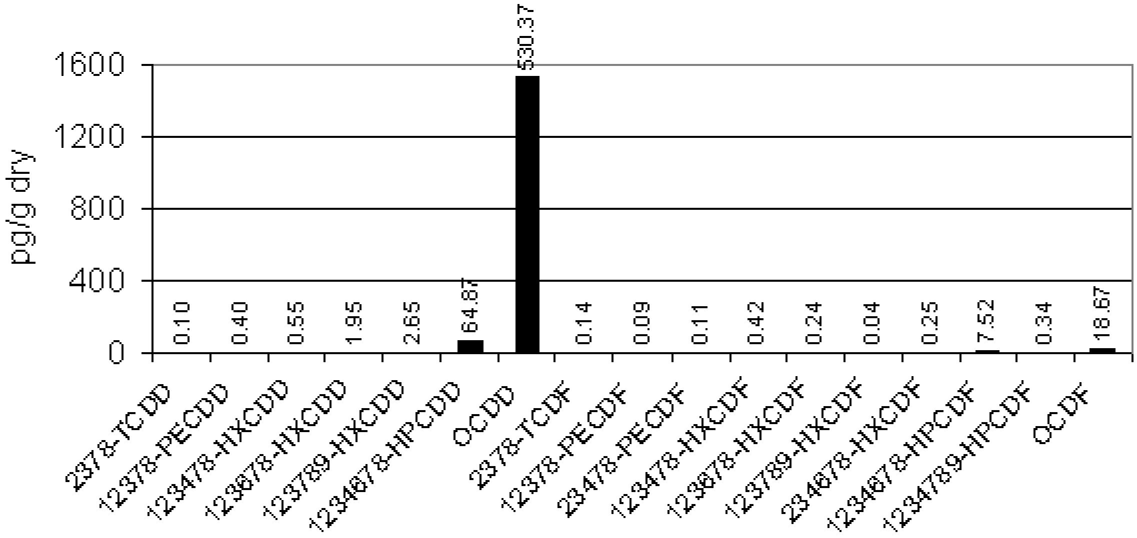


PCDD/PCDF TEQ Profiles for Site 17

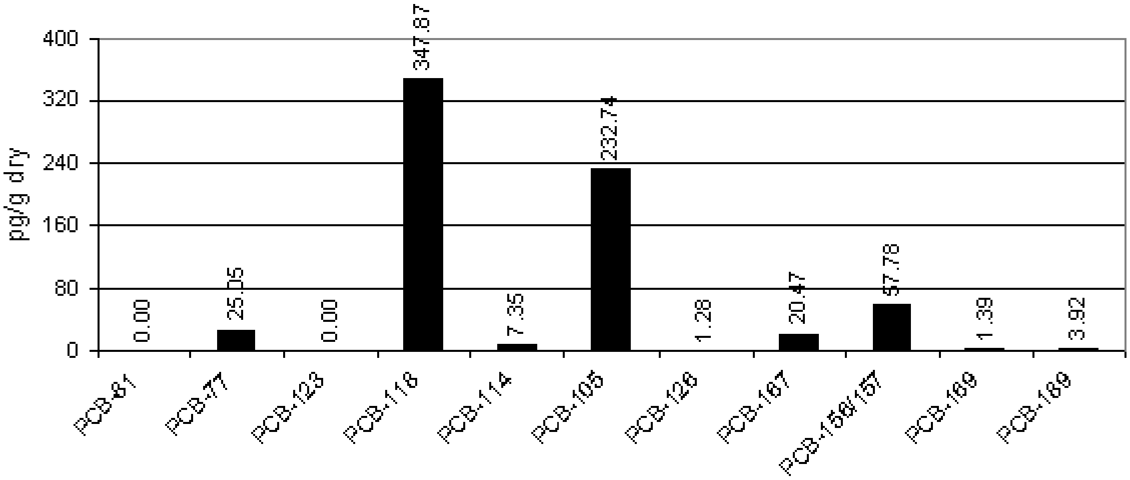


Dioxin-like PCB TEQ Profiles for Site 17

Site 18
Bay St. Louis, MS

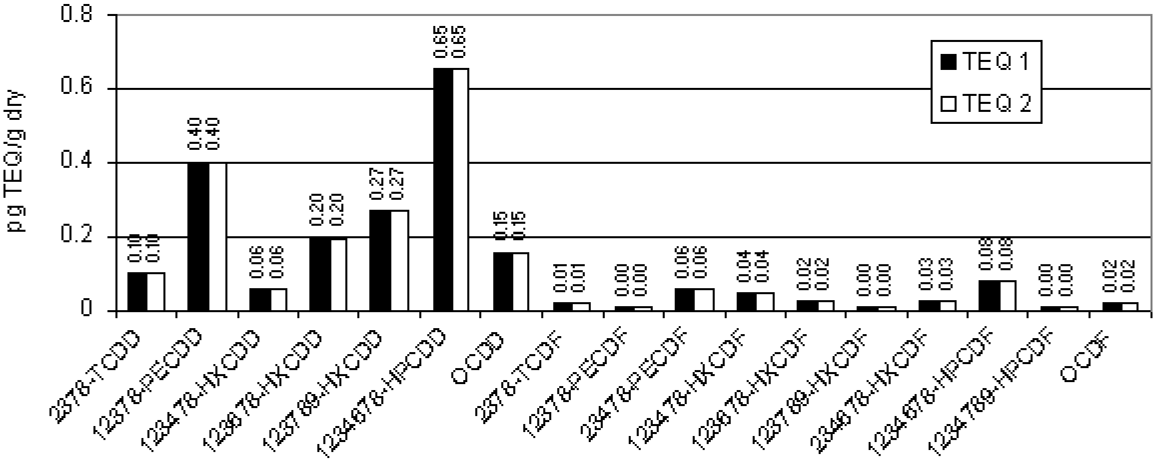


PCDD/PCDF Concentration Profiles for Site 18

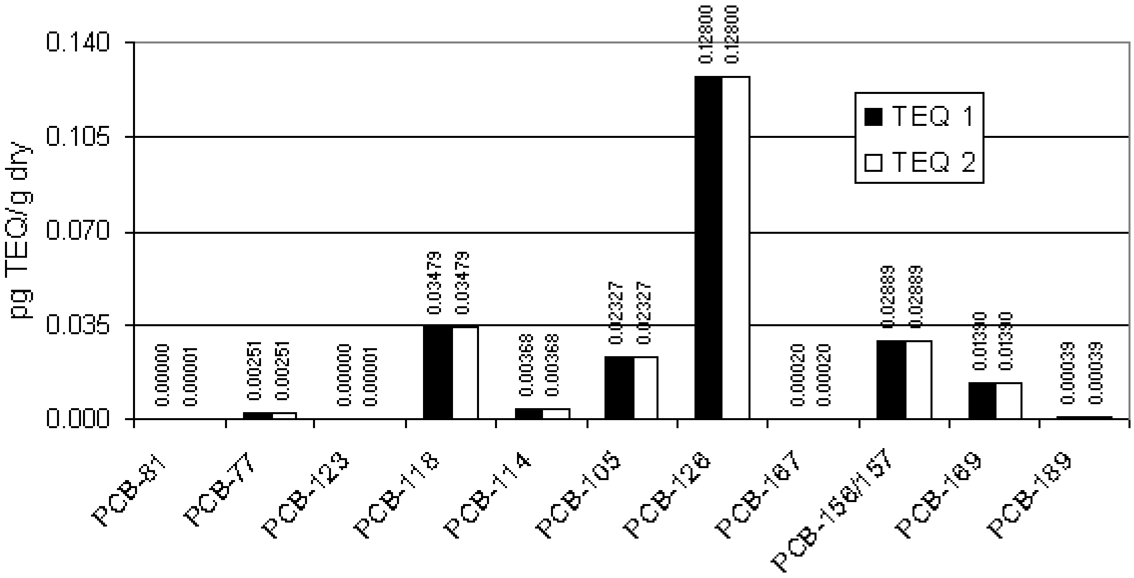


Dioxin-like PCB Concentration Profiles for Site 18

Site 18
Bay St. Louis, MS

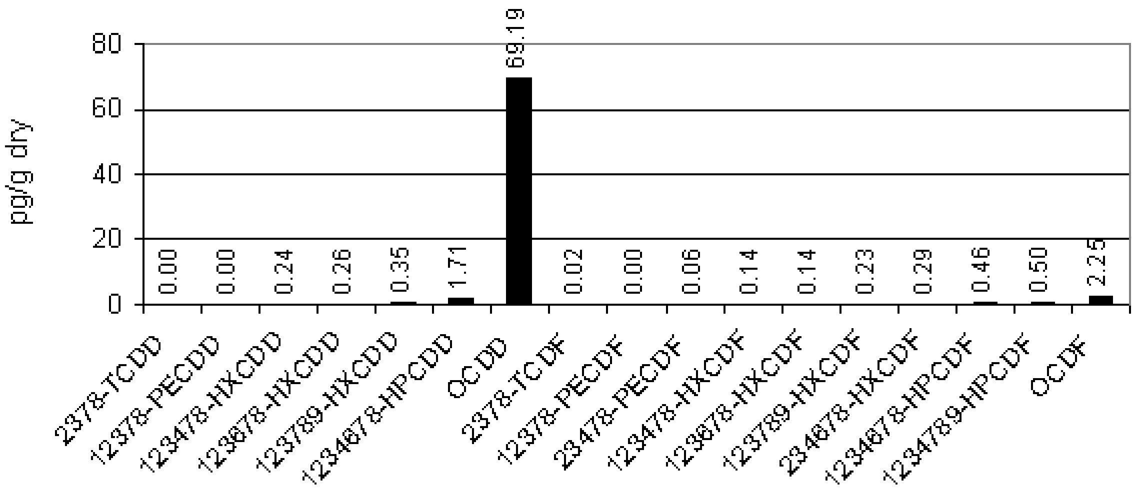


PCDD/PCDF TEQ Profiles for Site 18

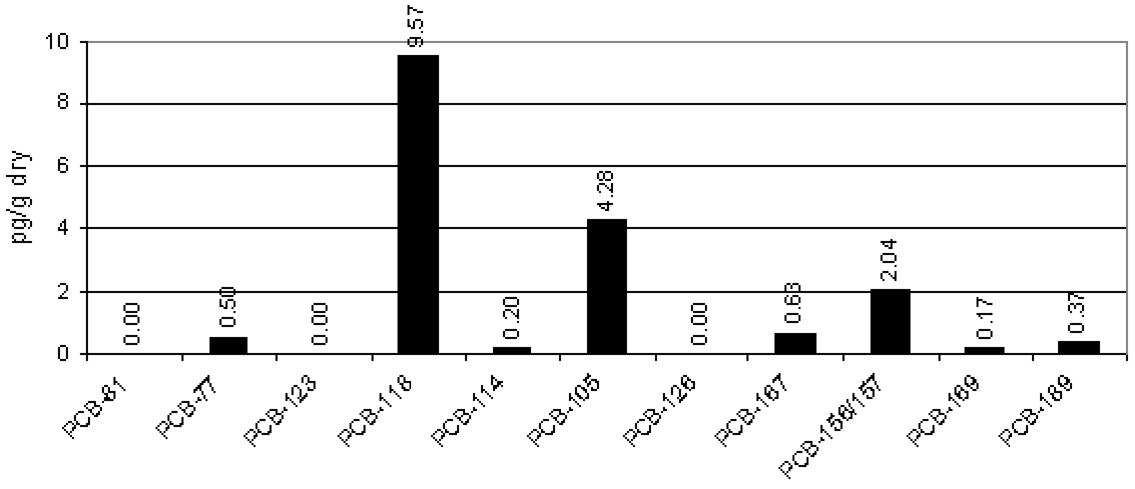


Dioxin-like PCB TEQ Profiles for Site 18

Site 19
Padre Island, TX

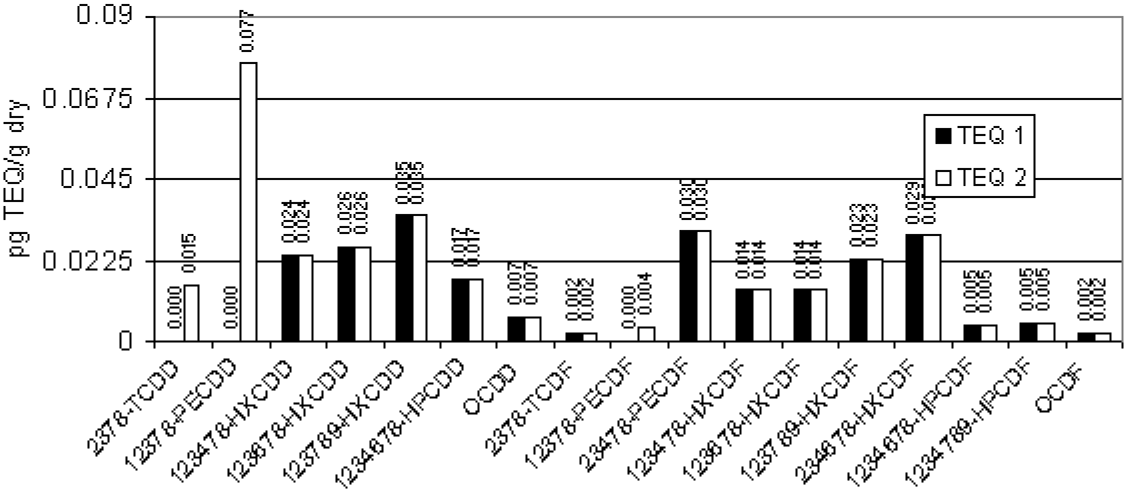


PCDD/PCDF Concentration Profiles for Site 19

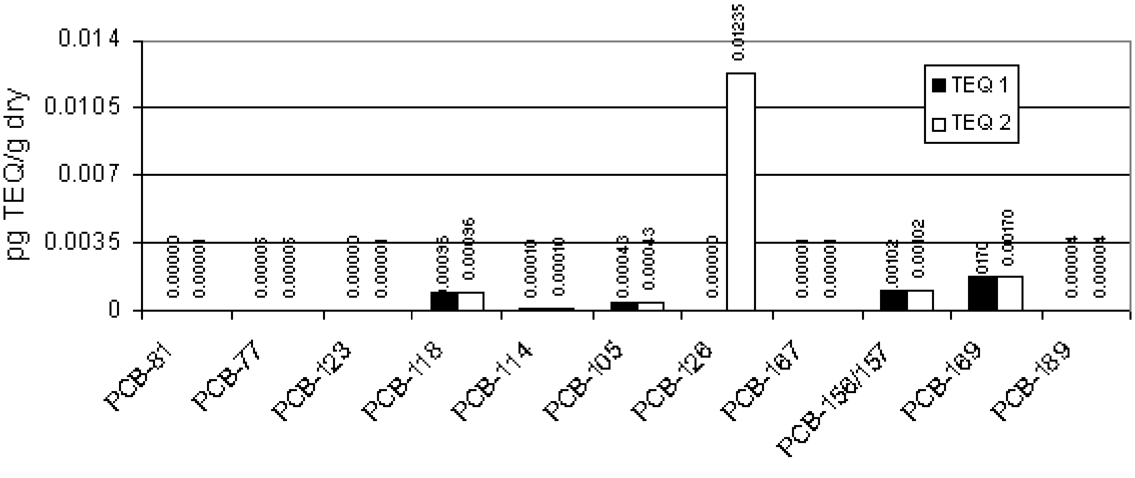


Dioxin-like PCB Concentration Profiles for Site 19

Site 19
Padre Island, TX

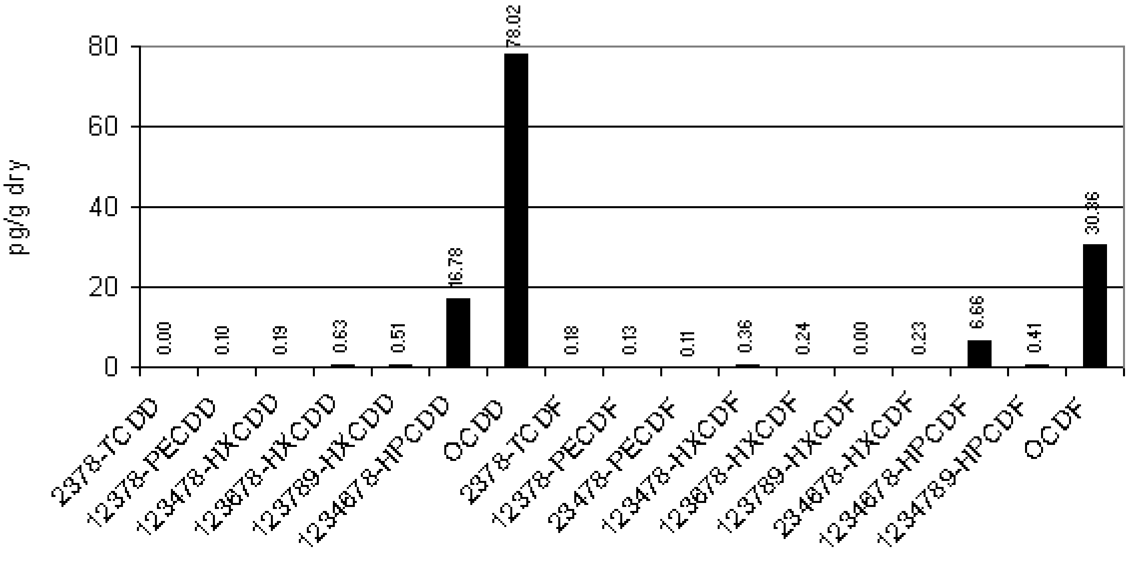


PCDD/PCDF TEQ Profiles for Site 19

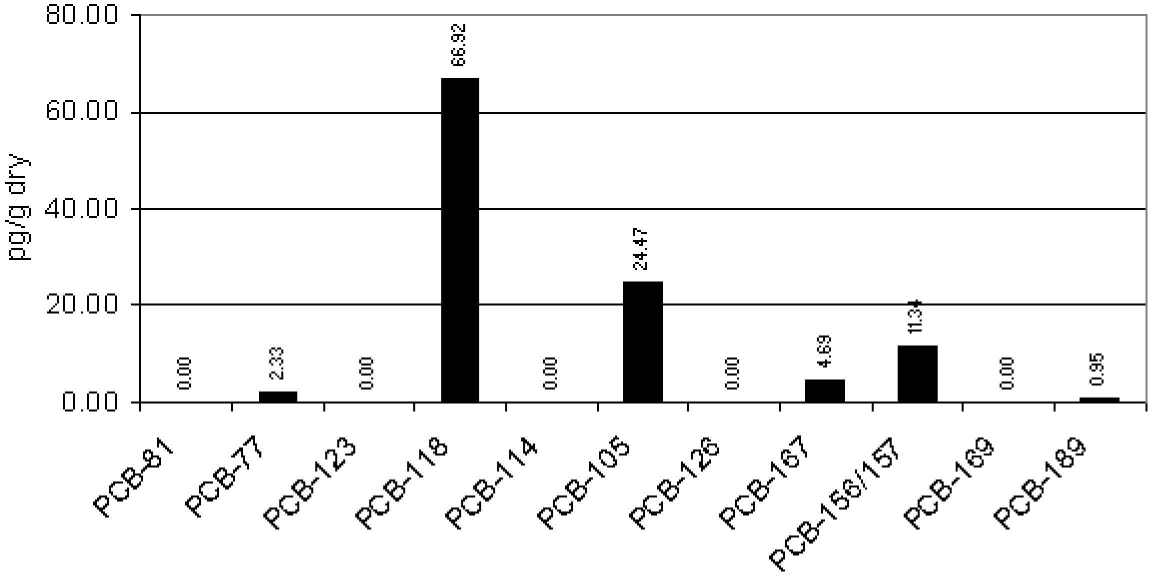


Dioxin-like PCB TEQ Profiles for Site 19

Site 20
Fond du Lac, MN

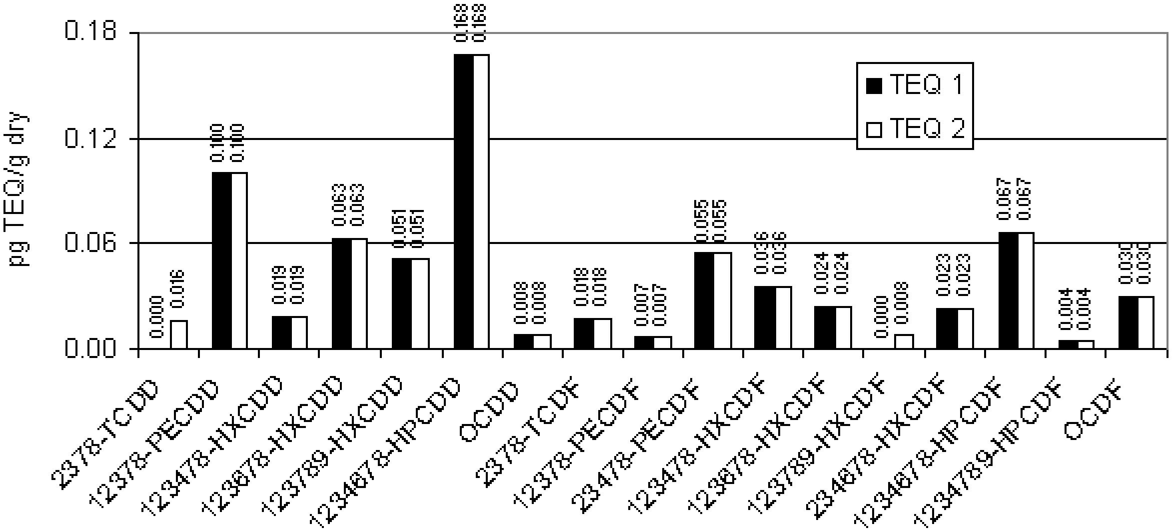


PCDD/PCDF Concentration Profiles for Site 20

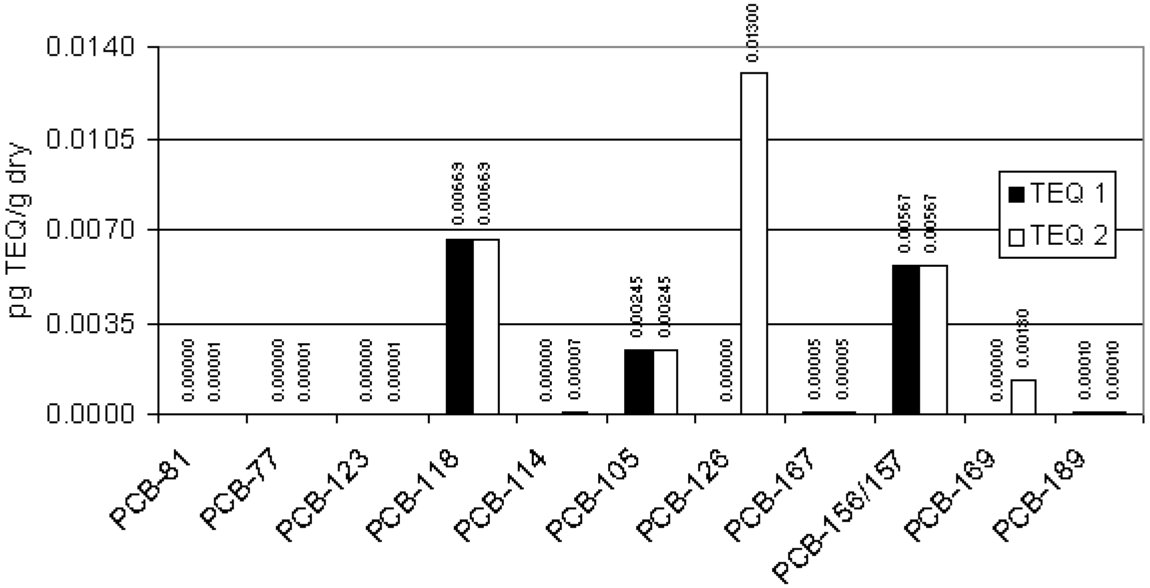


Dioxin-like PCB Concentration Profiles for Site 20

Site 20
Fond du Lac, MN

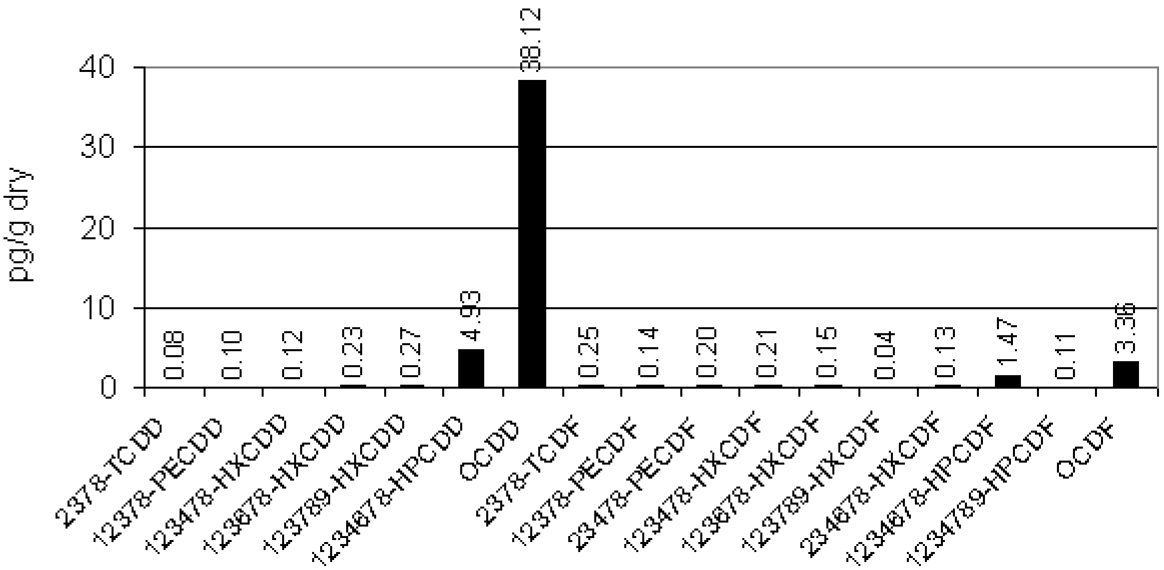


PCDD/PCDF TEQ Profiles for Site 20

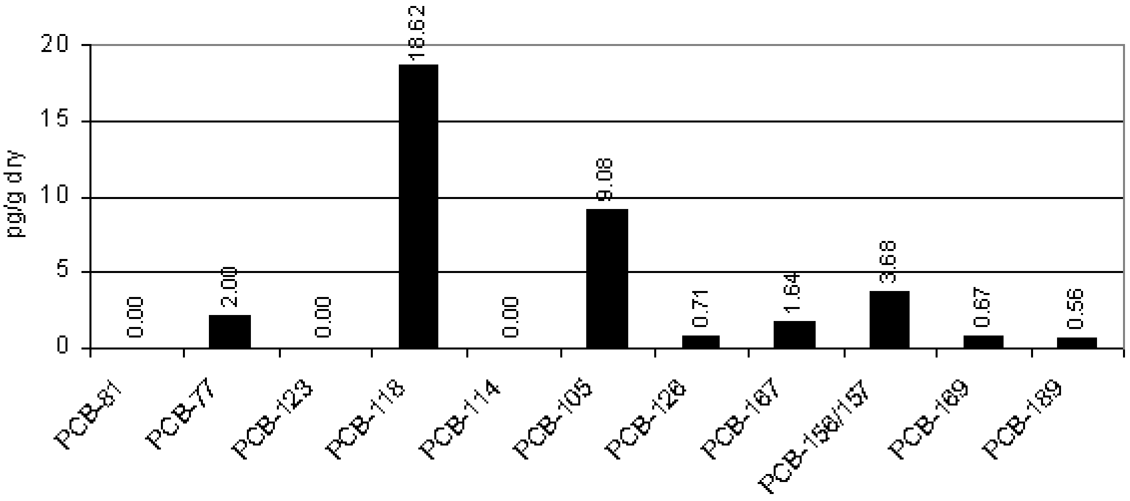


Dioxin-like PCB TEQ Profiles for Site 20

Site 21
North Platte, NE

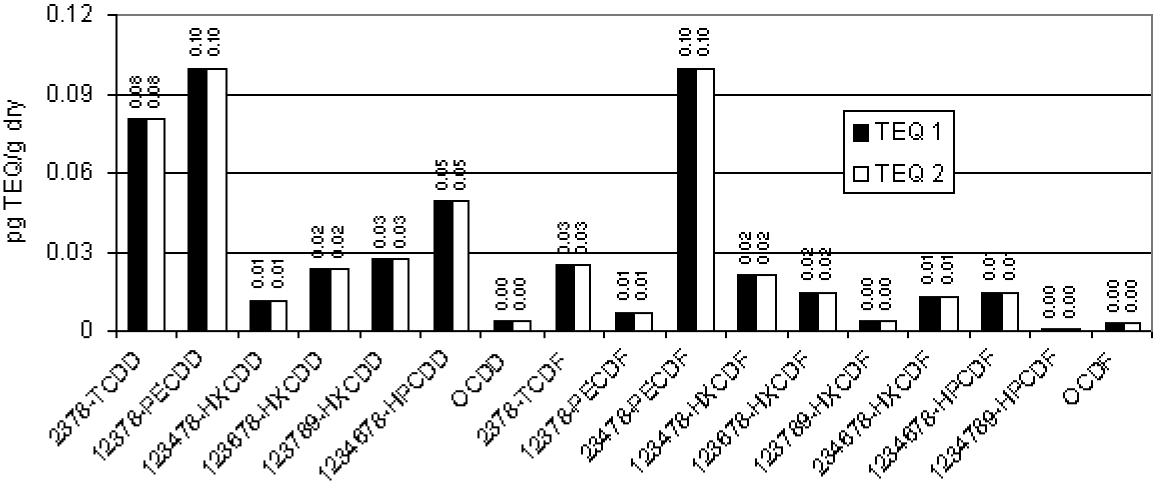


PCDD/PCDF Concentration Profiles for Site 21

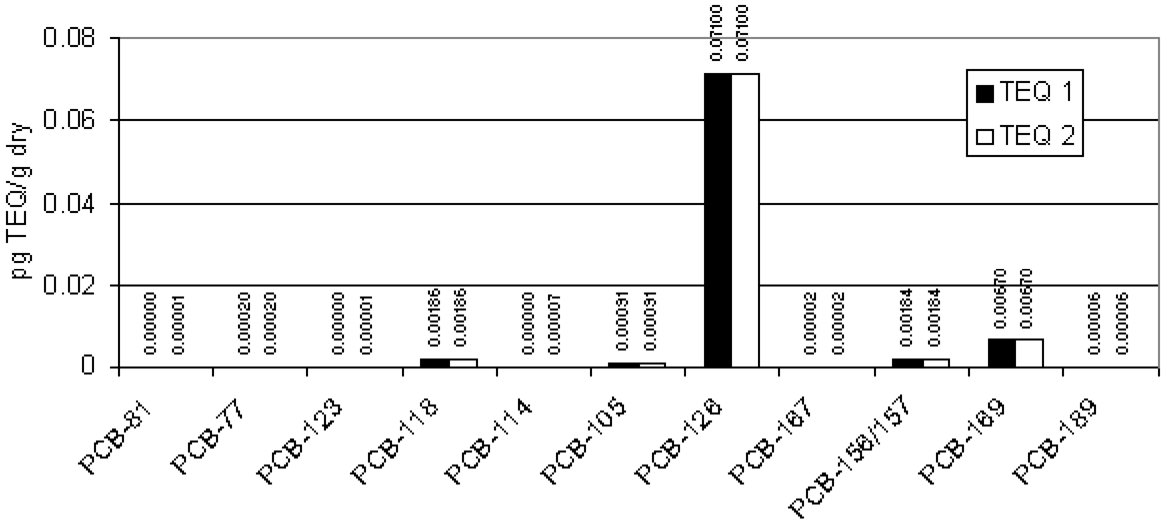


Dioxin-like PCB Concentration Profiles for Site 21

Site 21
North Platte, NE

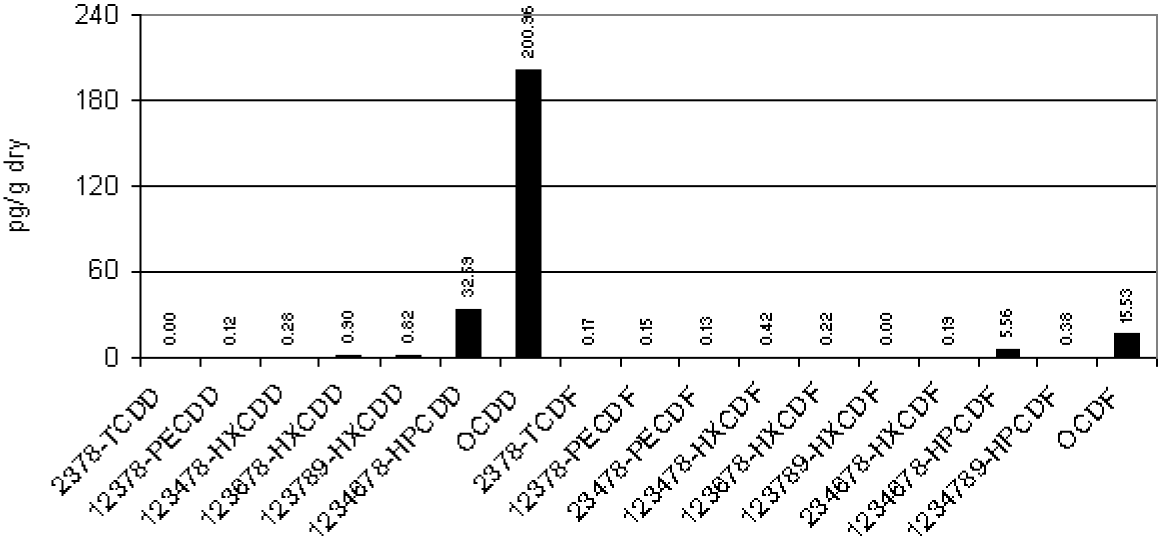


PCDD/PCDF TEQ Profiles for Site 21

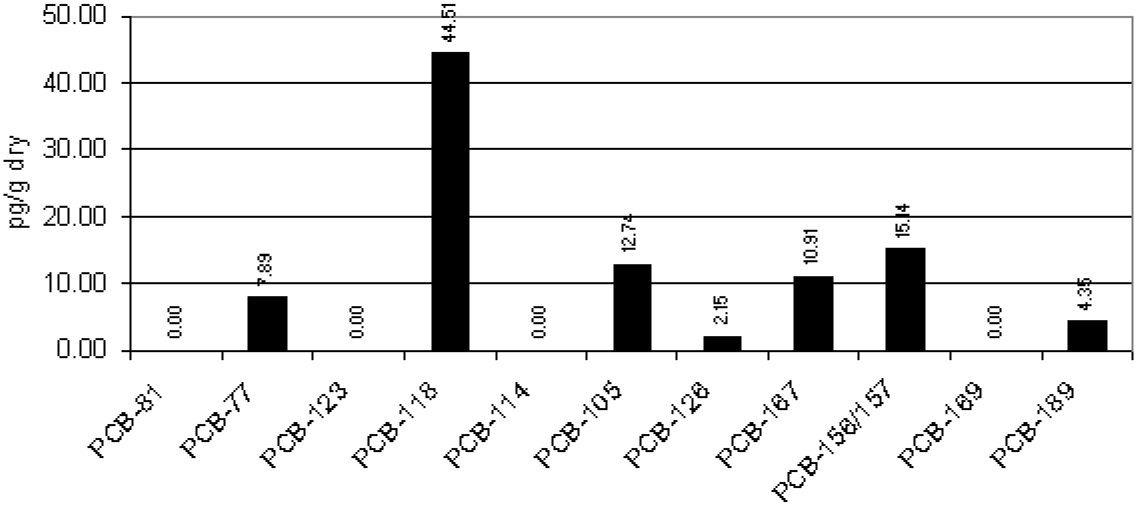


Dioxin-like PCB TEQ Profiles for Site 21

Site 22
Goodwell, OK

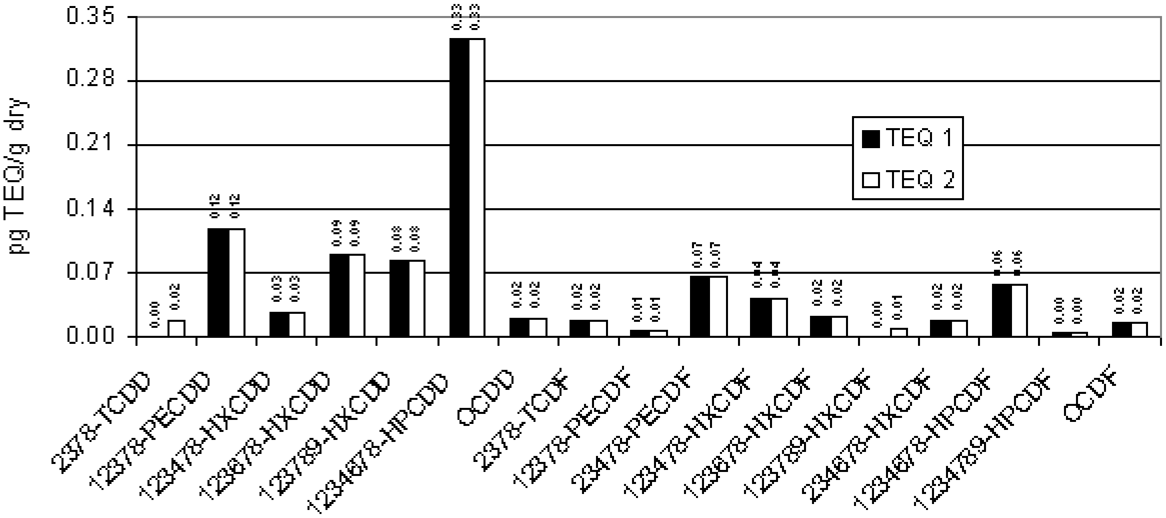


PCDD/PCDF Concentration Profiles for Site 22

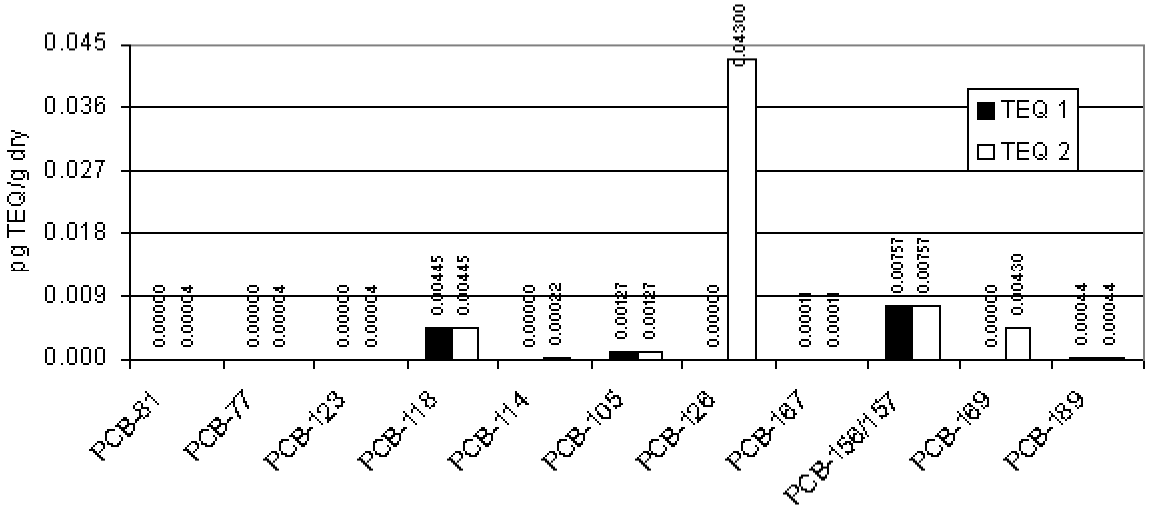


Dioxin-like PCB Concentration Profiles for Site 22

Site 22
Goodwell, OK

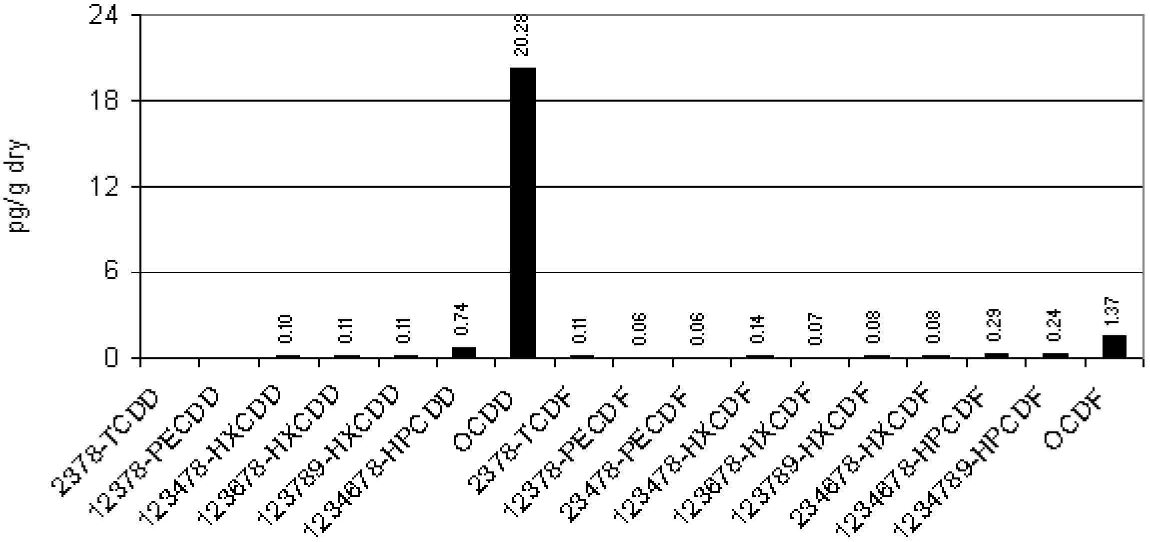


PCDD/PCDF TEQ Profiles for Site 22

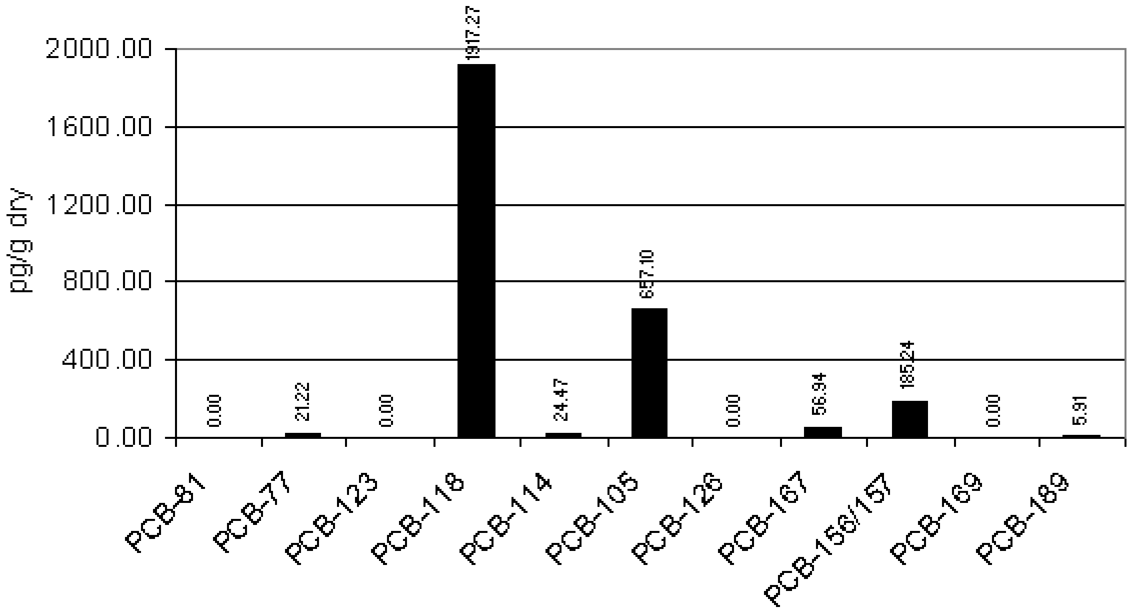


Dioxin-like PCB TEQ Profiles for Site 22

Site 23
Big Bend, TX

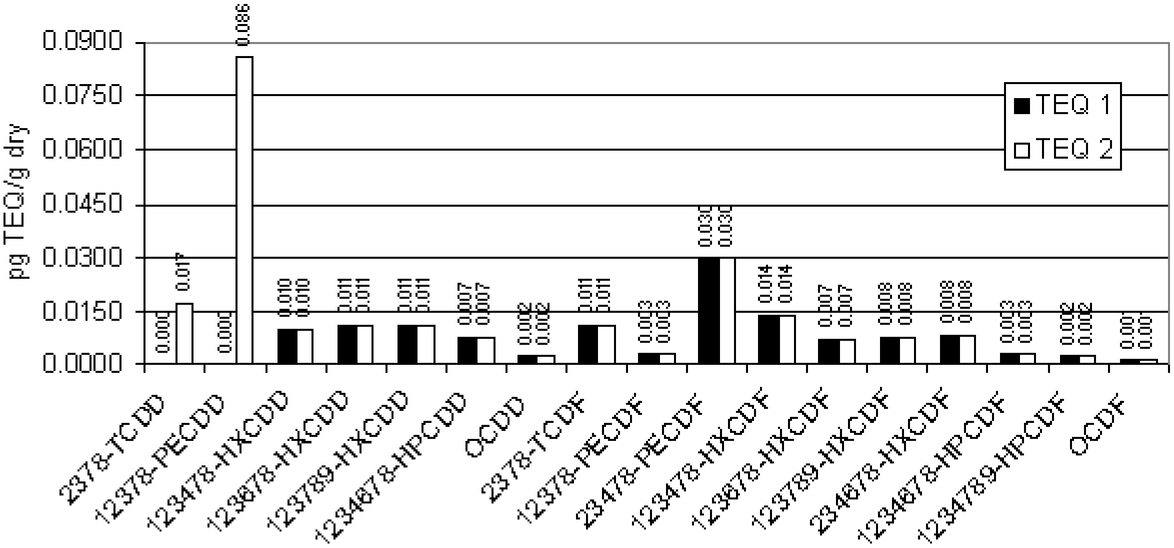


PCDD/PCDF Concentration Profiles for Site 23

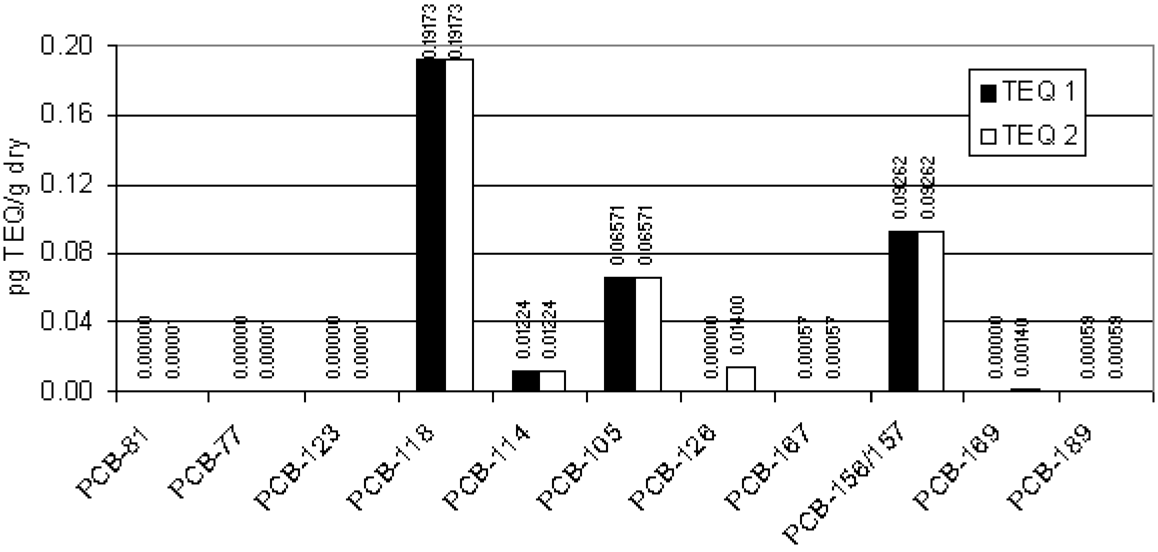


Dioxin-like PCB Concentration Profiles for Site 23

Site 23
Big Bend, TX

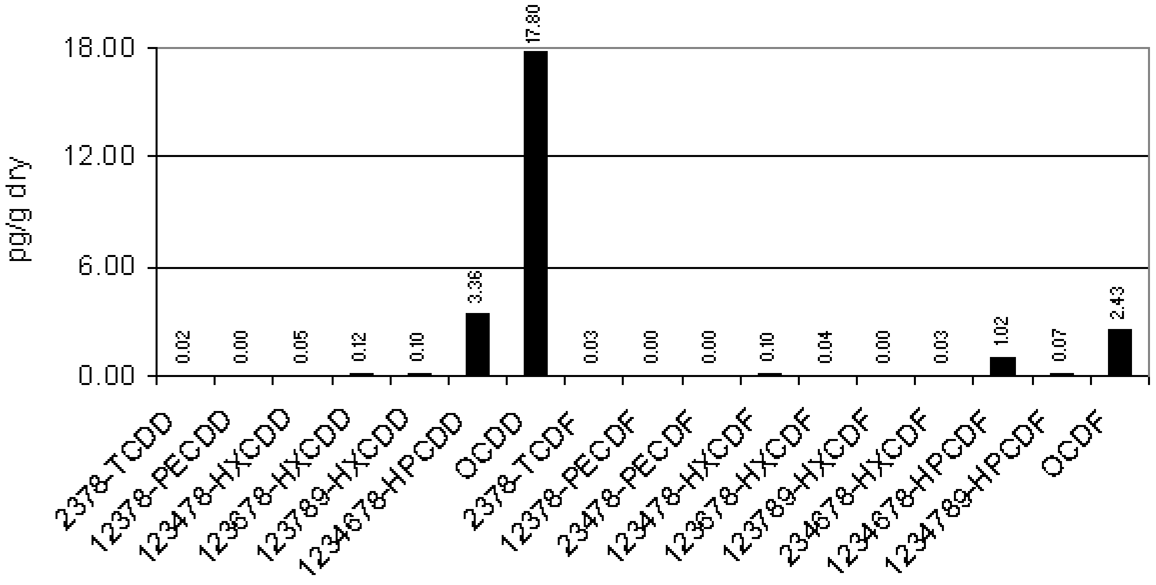


PCDD/PCDF TEQ Profiles for Site 23

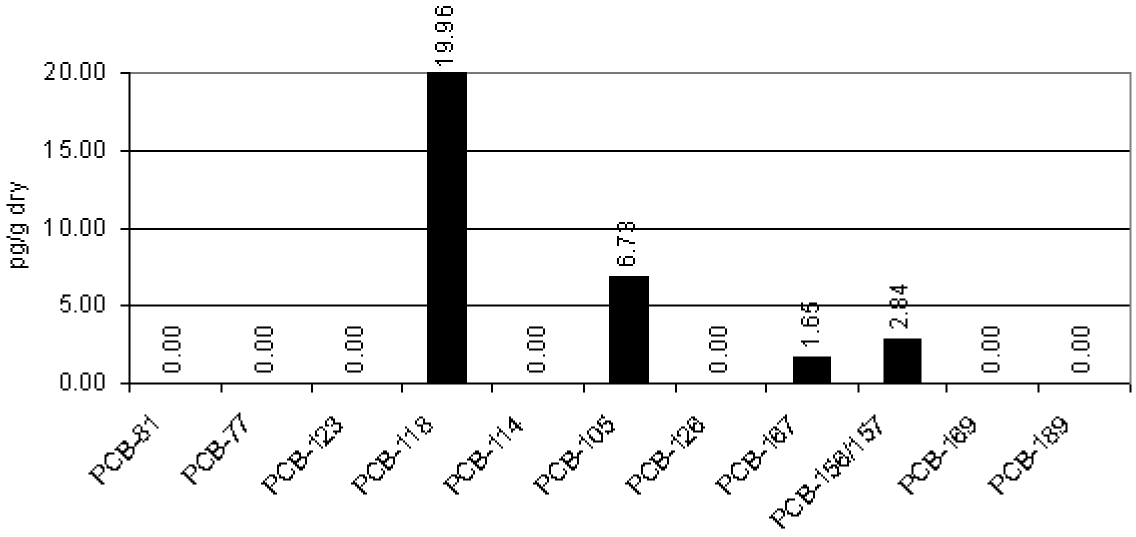


Dioxin-like PCB TEQ Profiles for Site 23

Site 24
Grand Canyon, AZ

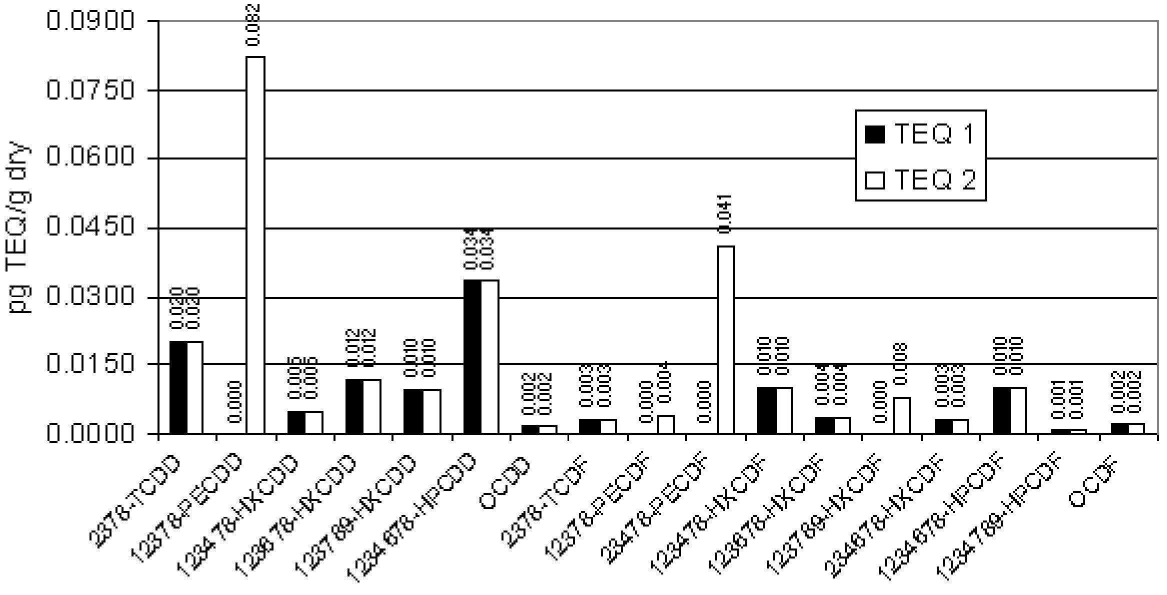


PCDD/PCDF Concentration Profiles for Site 24

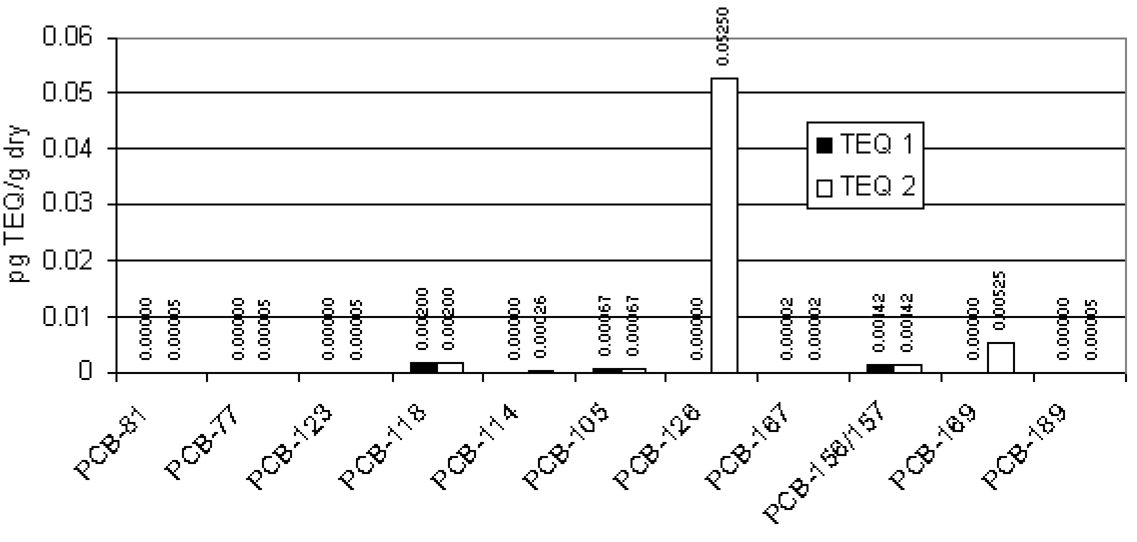


Dioxin-like PCB Concentration Profiles for Site 24

Site 24
Grand Canyon, AZ

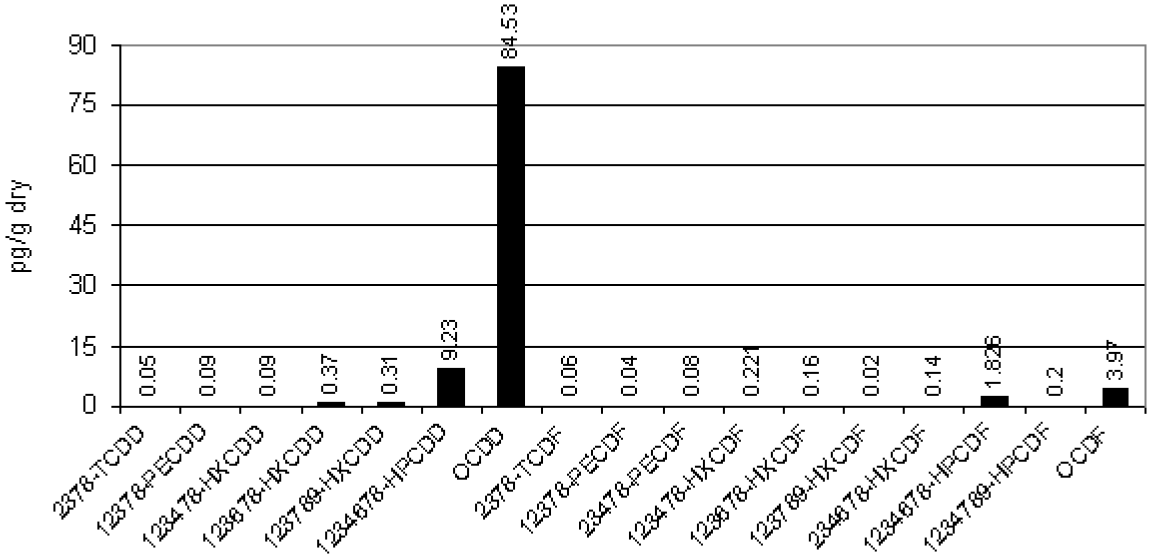


PCDD/PCDF TEQ Profiles for Site 24

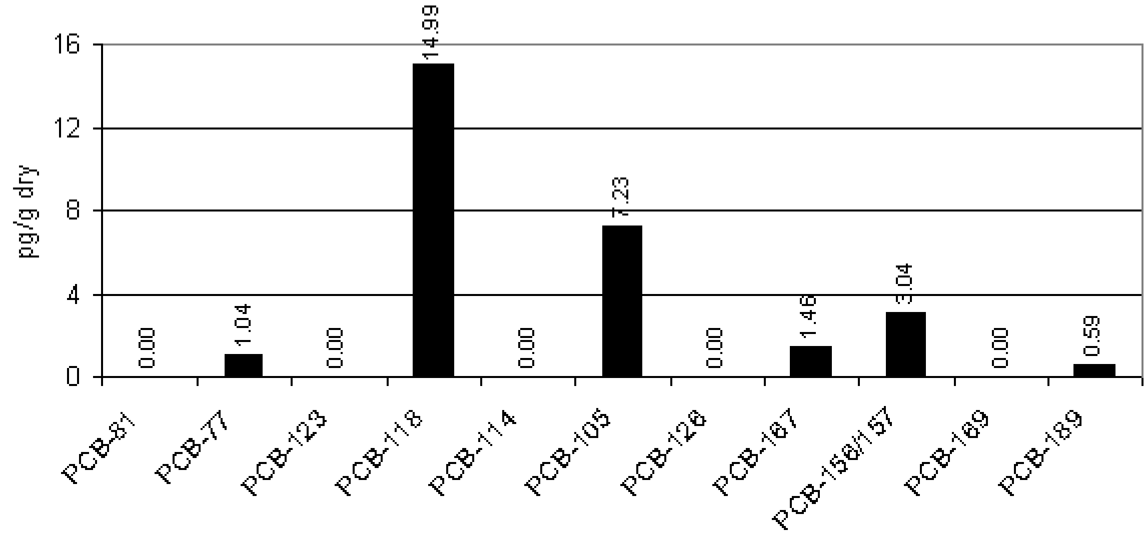


Dioxin-like PCB TEQ Profiles for Site 24

Site 25
Theodore Roosevelt, ND

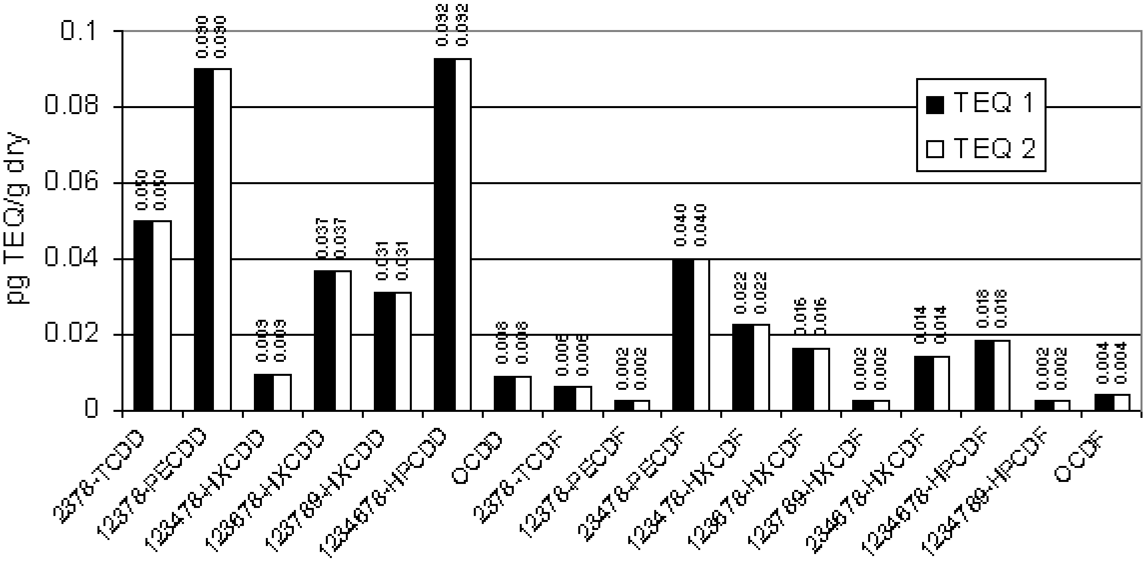


PCDD/PCDF Concentration Profiles for Site 25

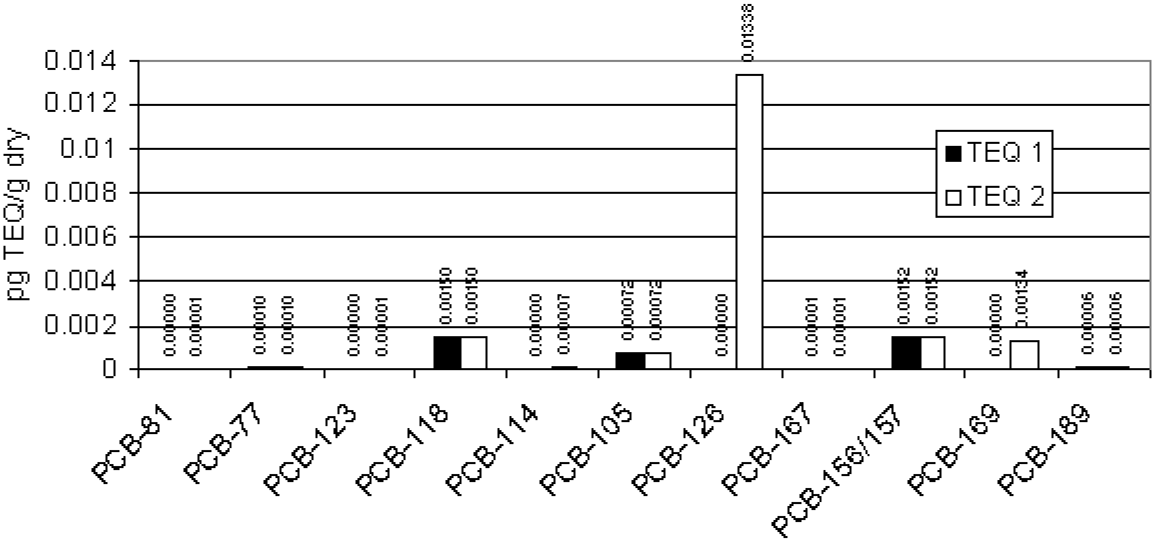


Dioxin-like PCB Concentration Profiles for Site 25

Site 25
Theodore Roosevelt, ND

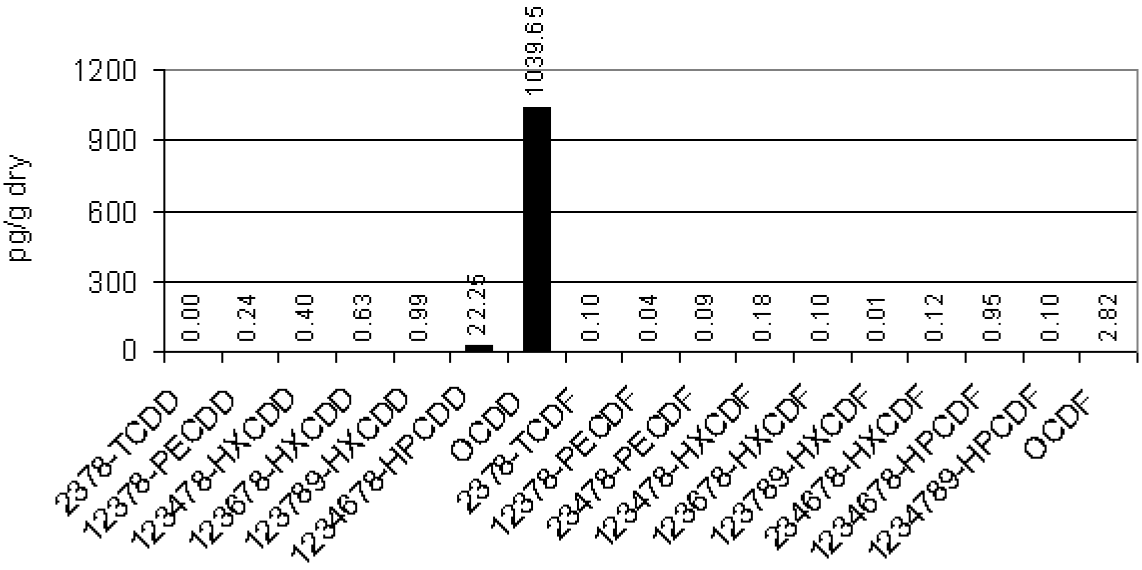


PCDD/PCDF TEQ Profiles for Site 25

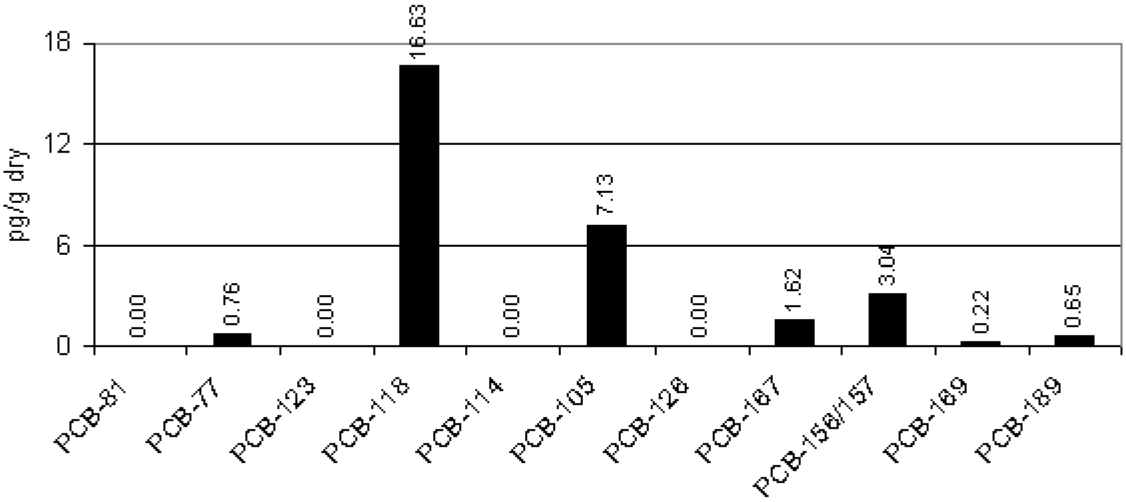


Dioxin-like PCB TEQ Profiles for Site 25

Site 27
Chiricahua, AZ

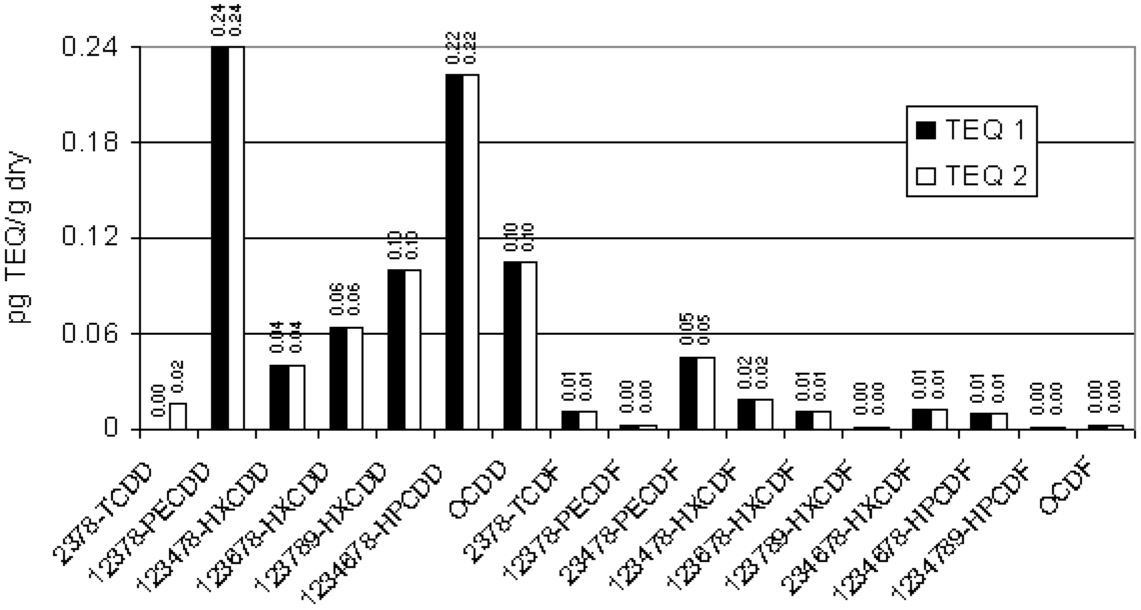


PCDD/PCDF Concentration Profiles for Site 27

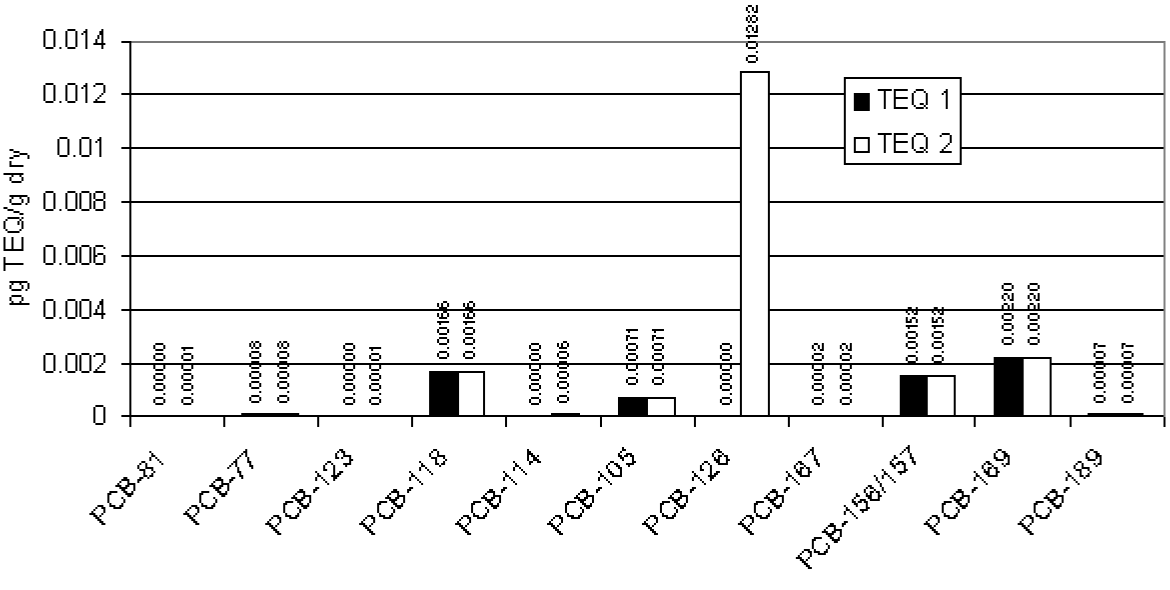


Dioxin-like PCB Concentration Profiles for Site 27

Site 27
Chiricahua, AZ

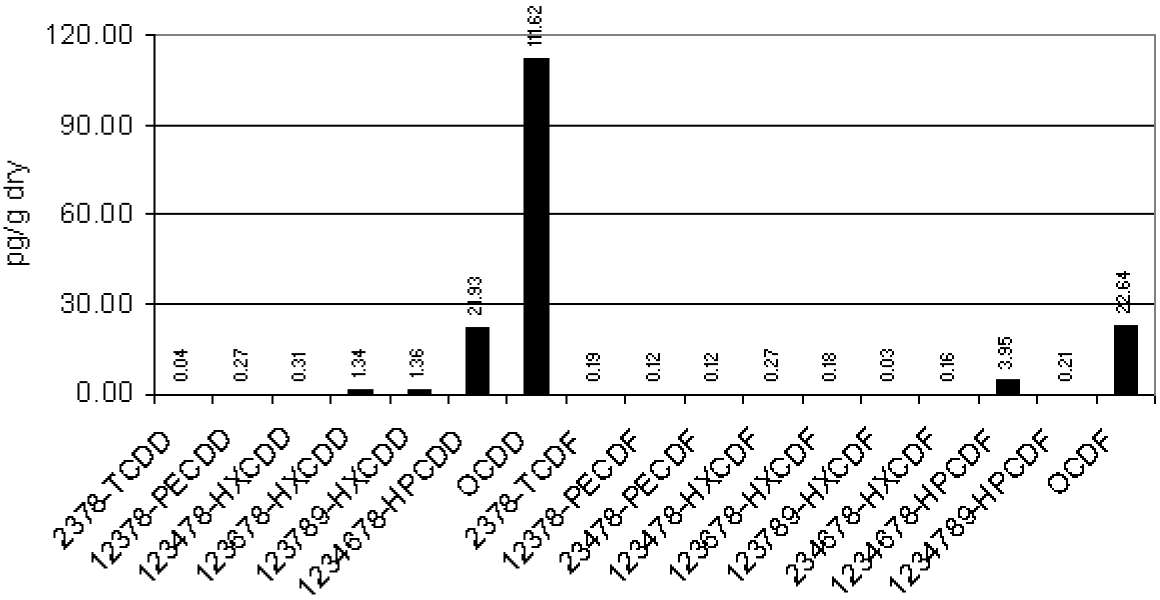


PCDD/PCDF TEQ Profiles for Site 27

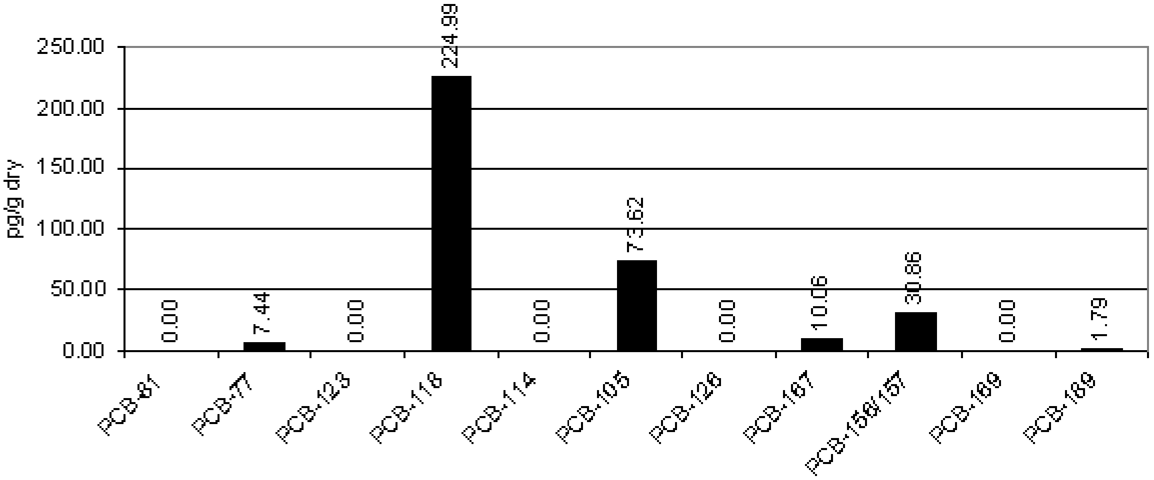


Dioxin-like PCB TEQ Profiles for Site 27

Site 28
Rancho Seco, CA

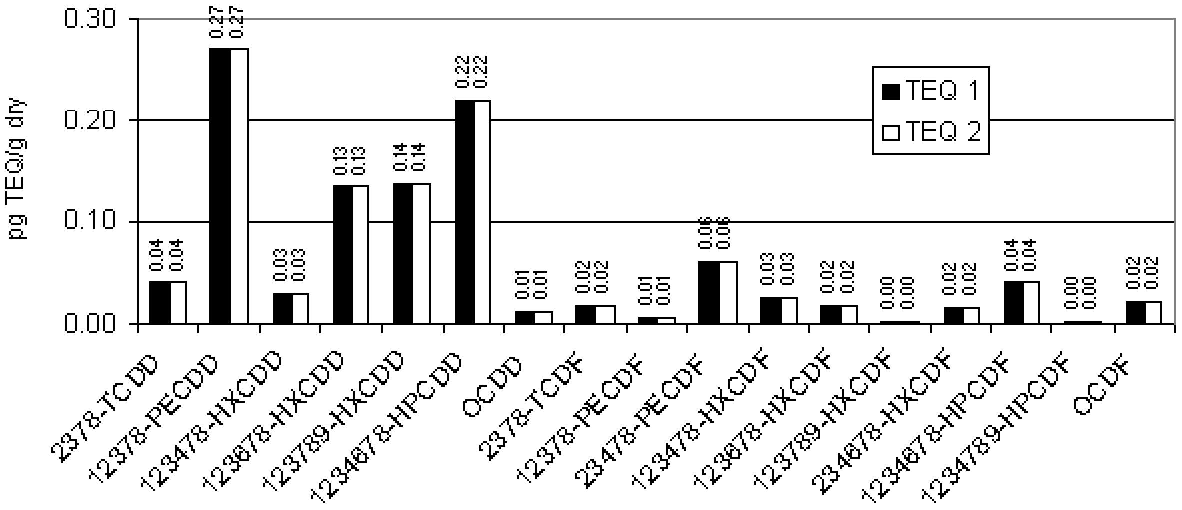


PCDD/PCDF Concentration Profiles for Site 28

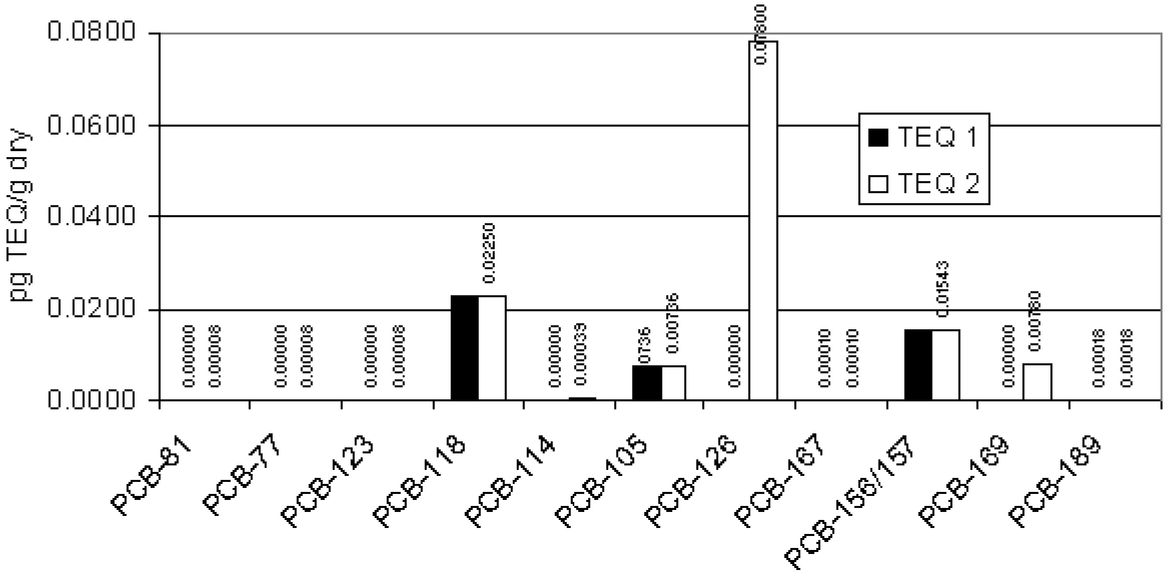


Dioxin-like PCB Concentration Profiles for Site 28

Site 28
Rancho Seco, CA

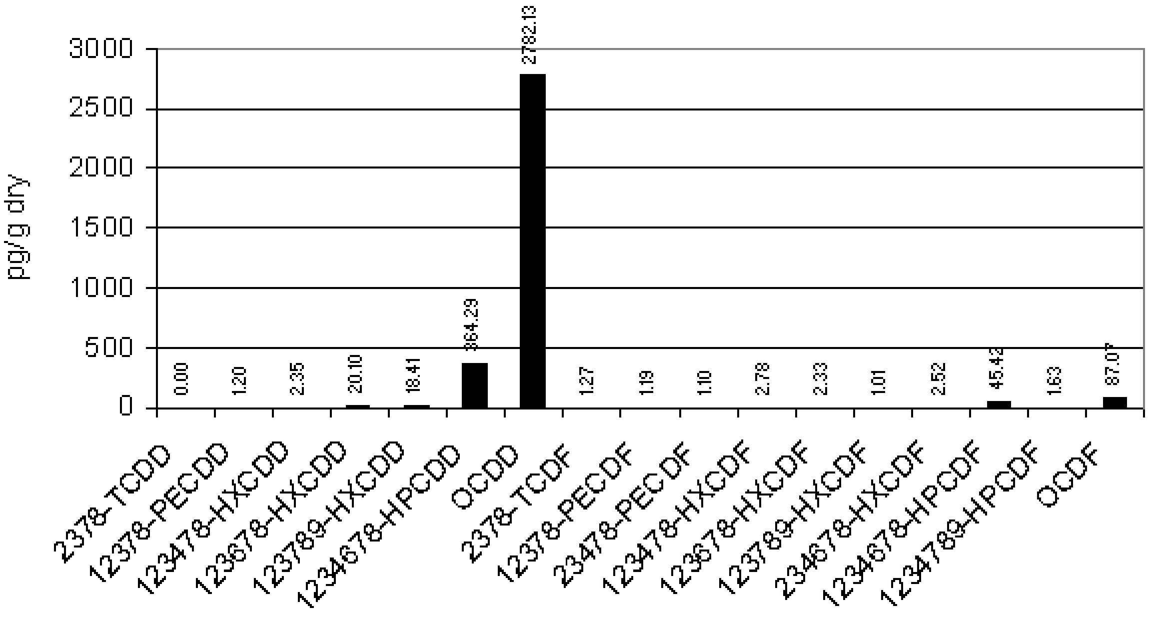


PCDD/PCDF TEQ Profiles for Site 28

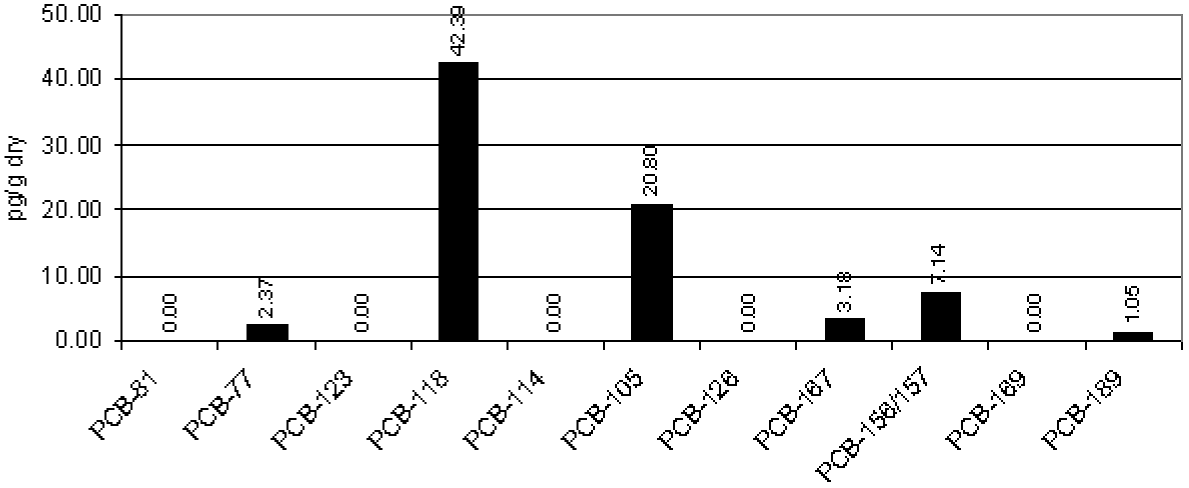


Dioxin-like PCB TEQ Profiles for Site 28

Site 29
Marvel Ranch, OR

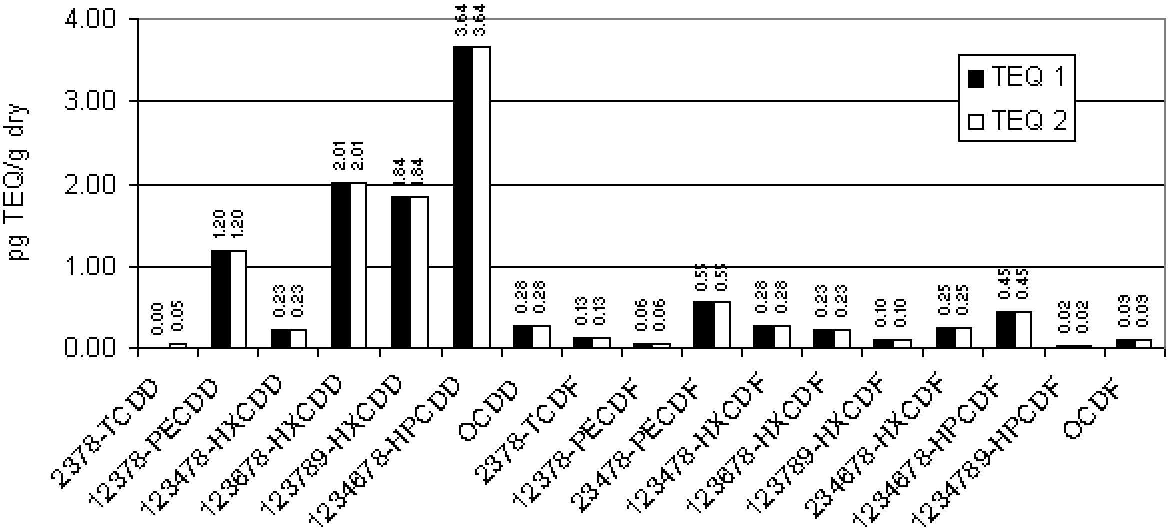


PCDD/PCDF Concentration Profiles for Site 29

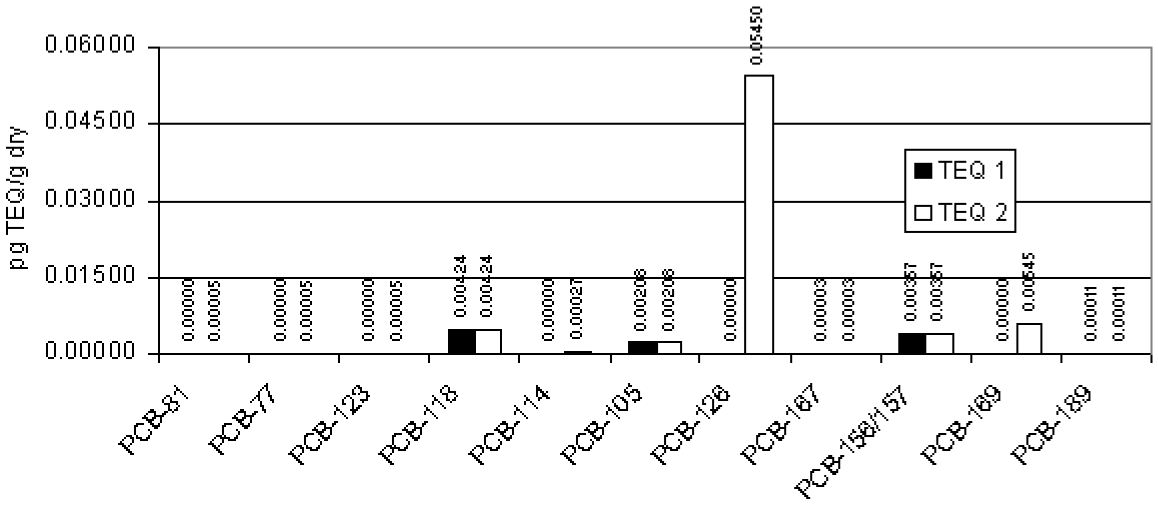


Dioxin-like PCB Concentration Profiles for Site 29

Site 29
Marvel Ranch, OR

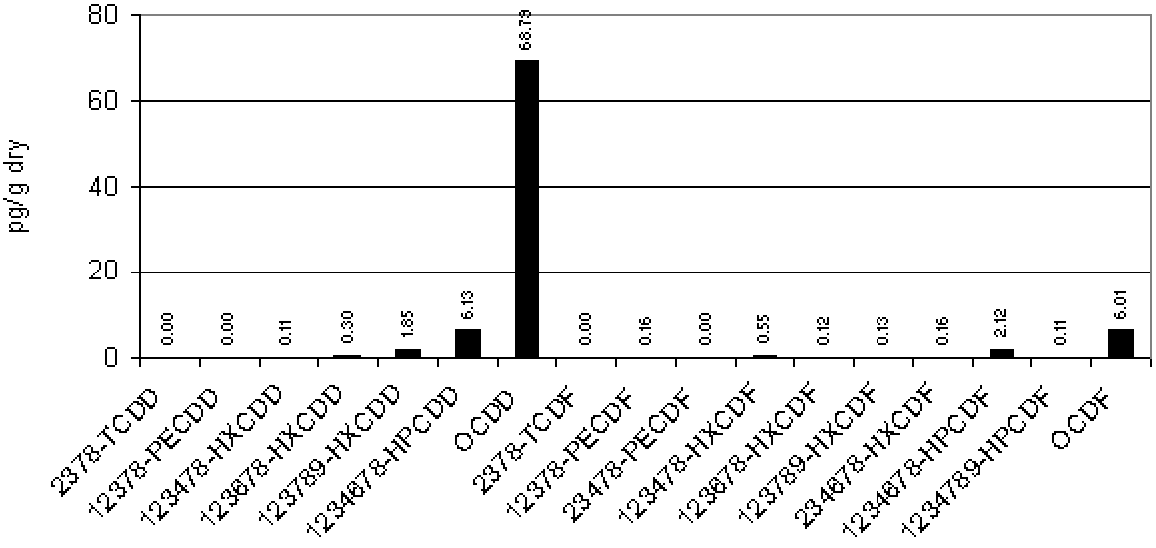


PCDD/PCDF TEQ Profiles for Site 29

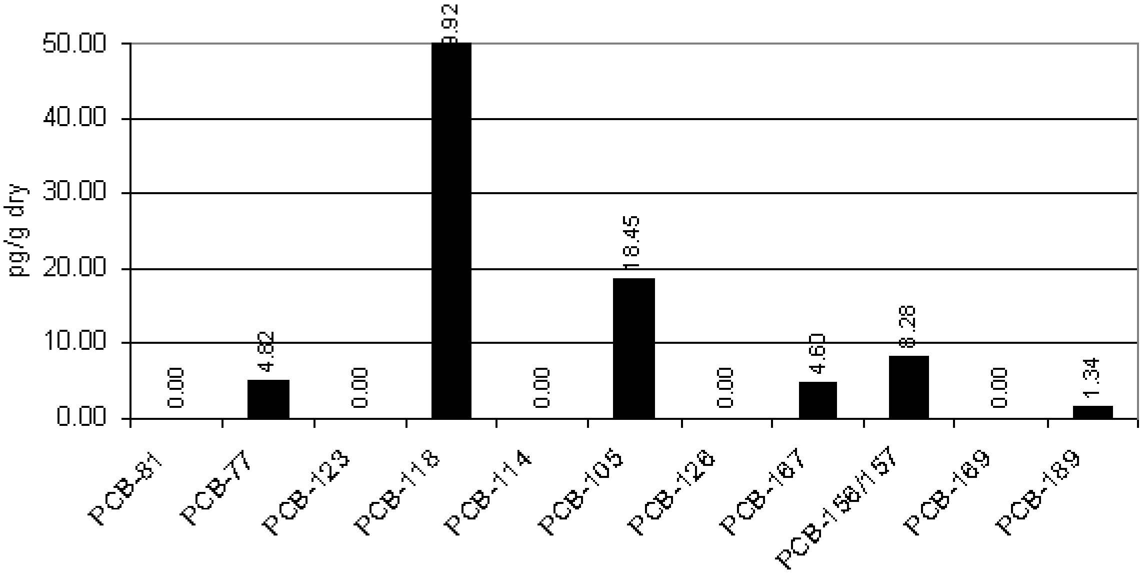


Dioxin-like PCB TEQ Profiles for Site 29

Site 30
Ozette Lake, WA

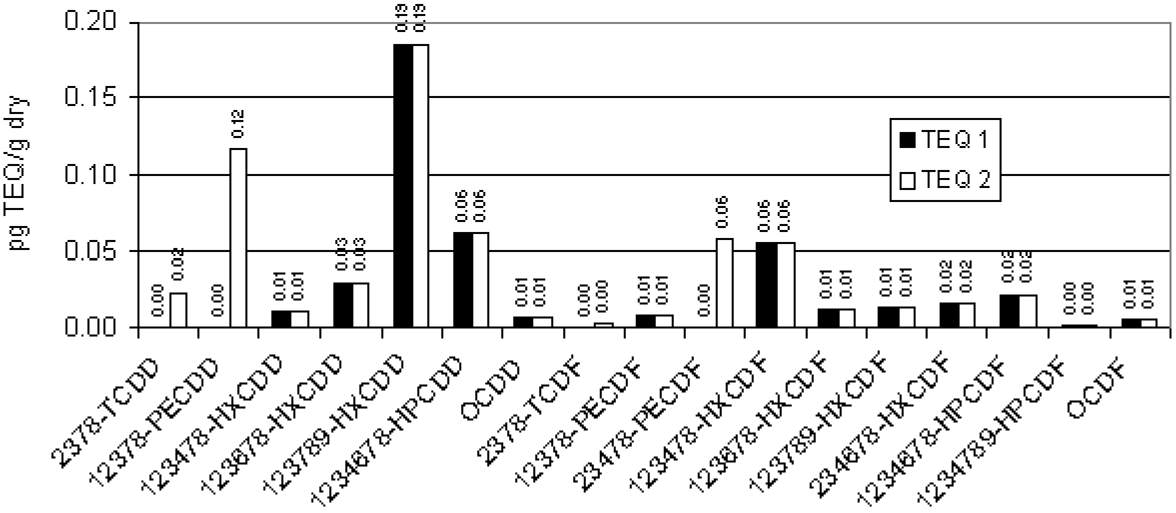


PCDD/PCDF Concentration Profiles for Site 30

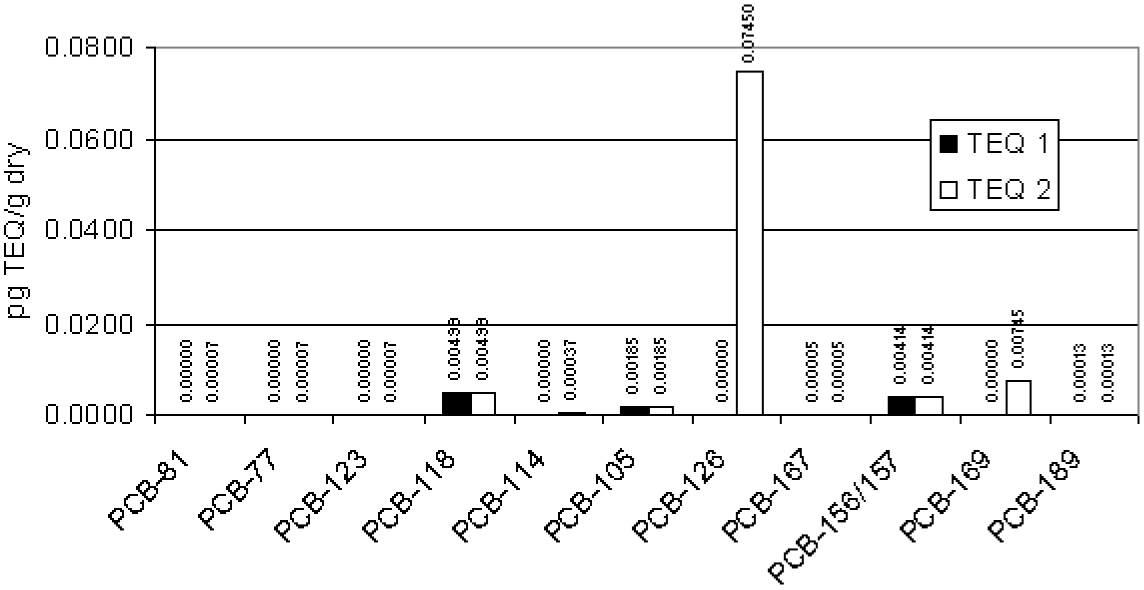


Dioxin-like PCB Concentration Profiles for Site 30

Site 30
Ozette Lake, WA

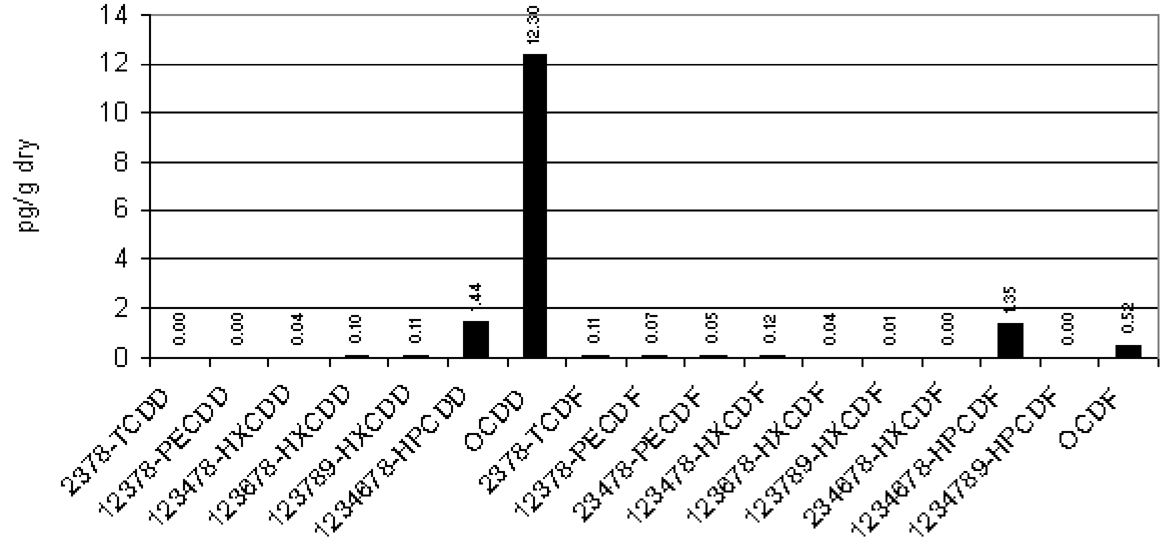


PCDD/PCDF TEQ Profiles for Site 30

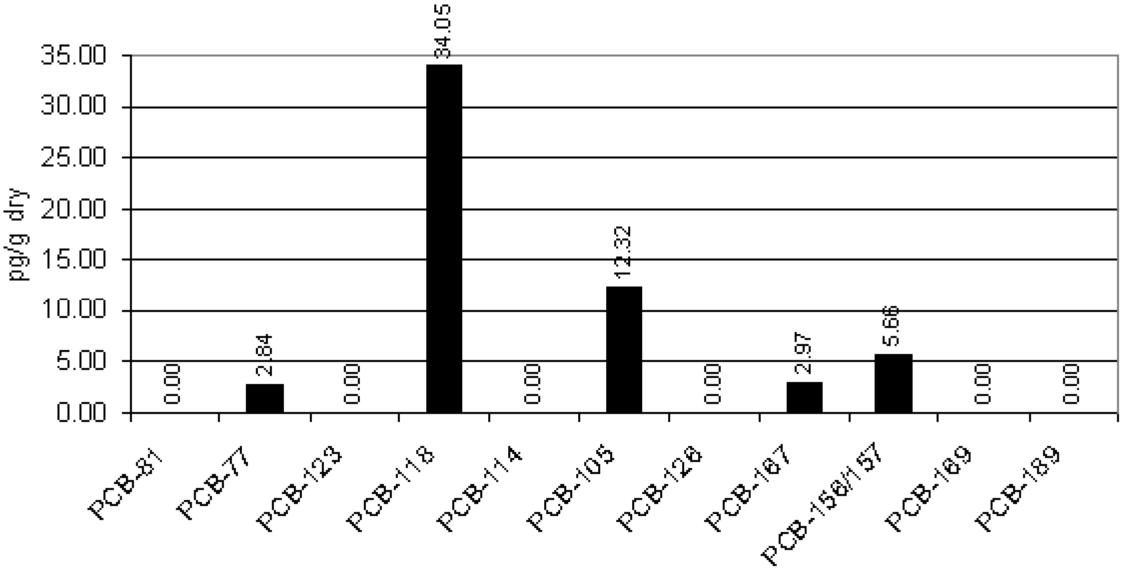


Dioxin-like PCB TEQ Profiles for Site 30

Site 34
Trapper Creek, AK

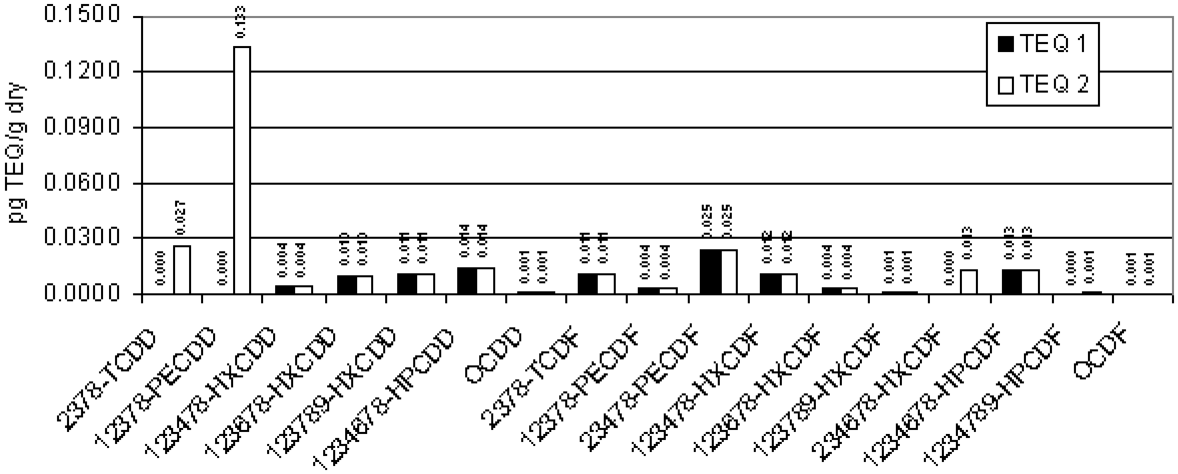


PCDD/PCDF Concentration Profiles for Site 34

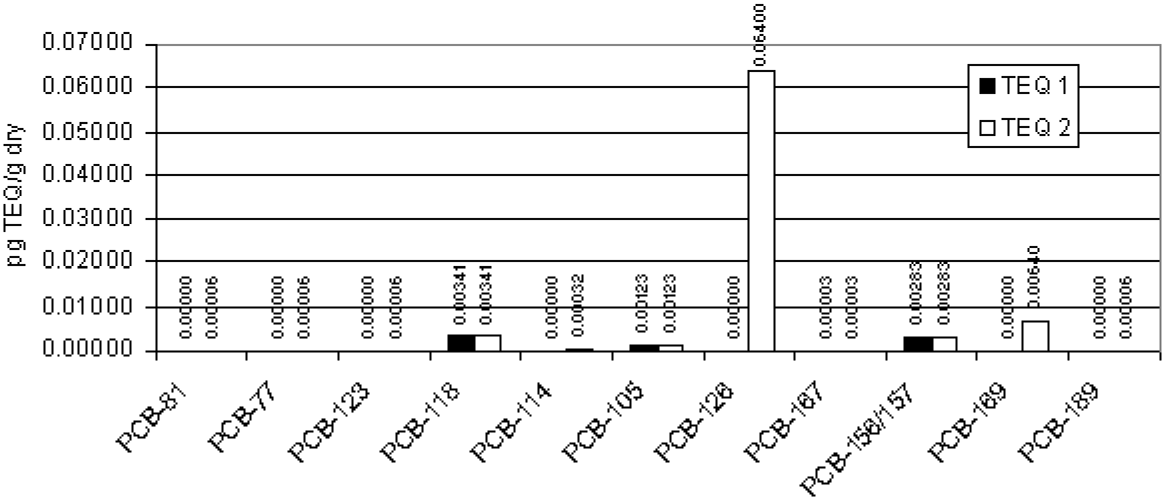


Dioxin-like PCB Concentration Profiles for Site 34

Site 34
Trapper Creek, AK



PCDD/PCDF TEQ Profiles for Site 34

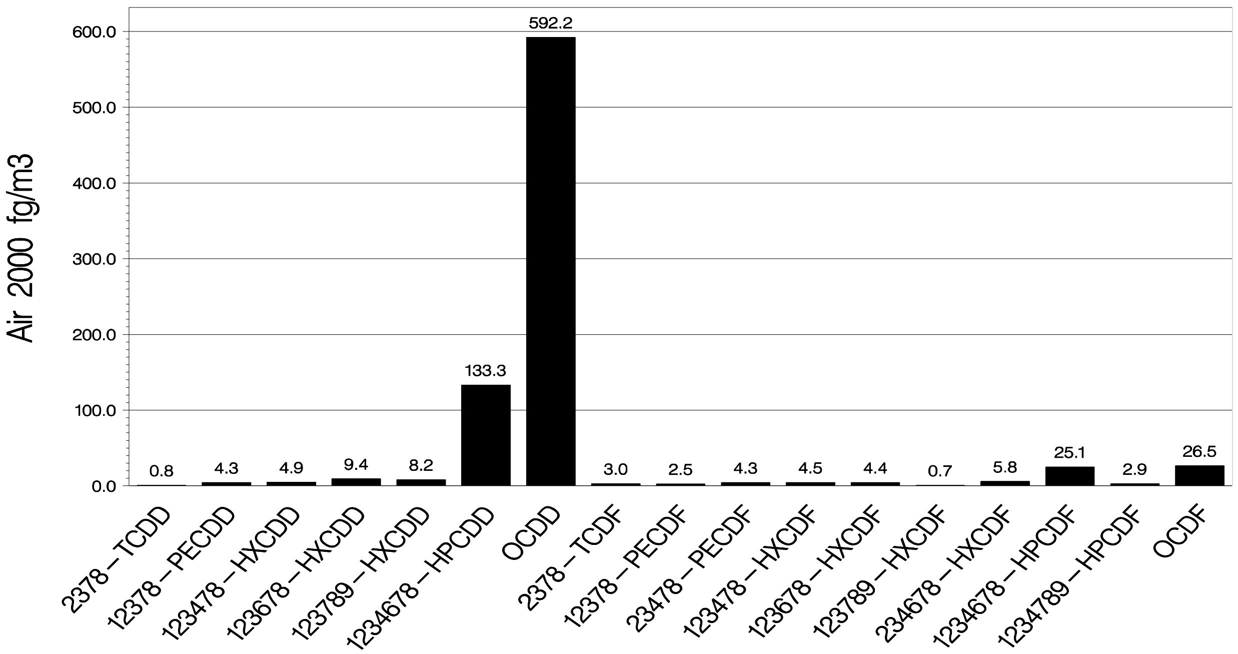


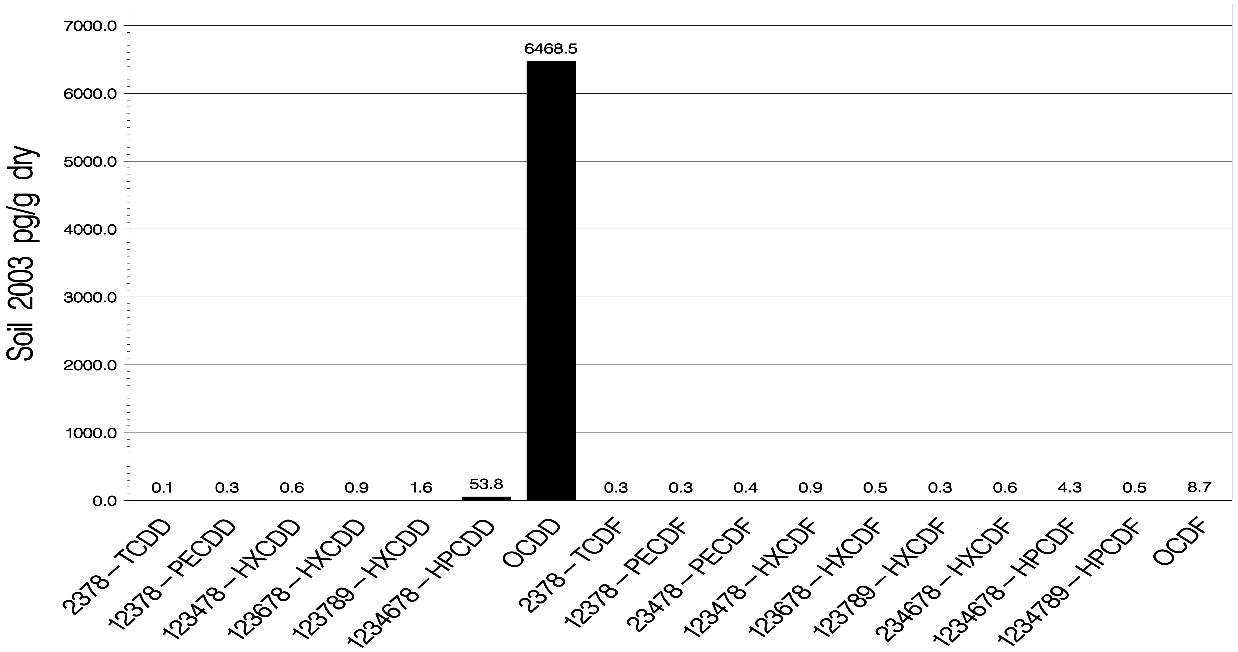
Dioxin-like PCB TEQ Profiles for Site 34

APPENDIX L

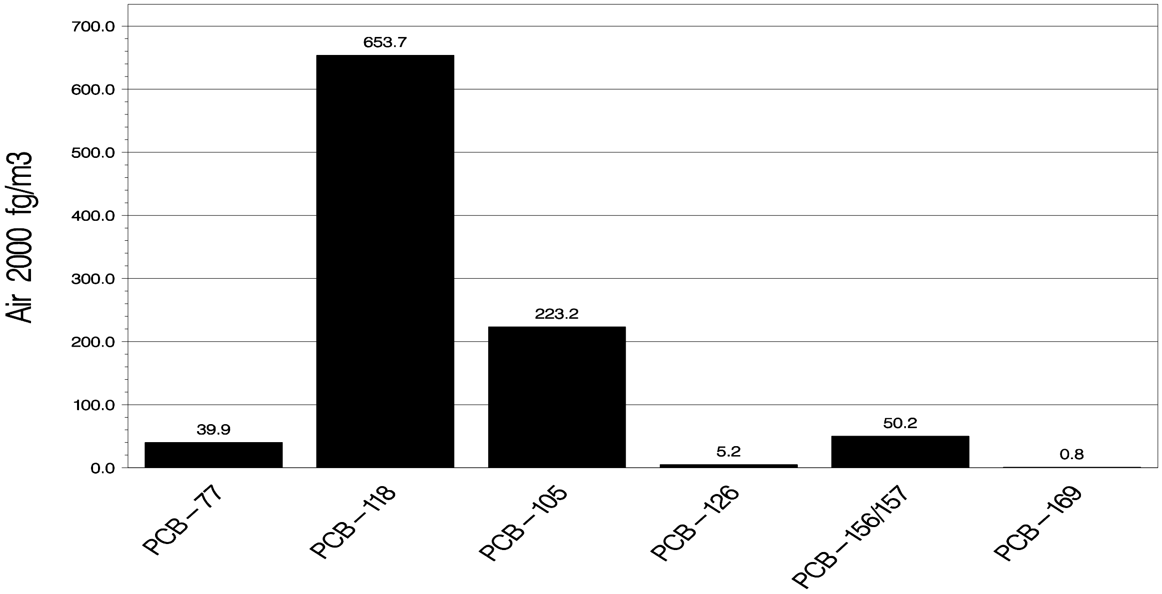
PAIRED AIR/SOIL CONGENER PROFILES

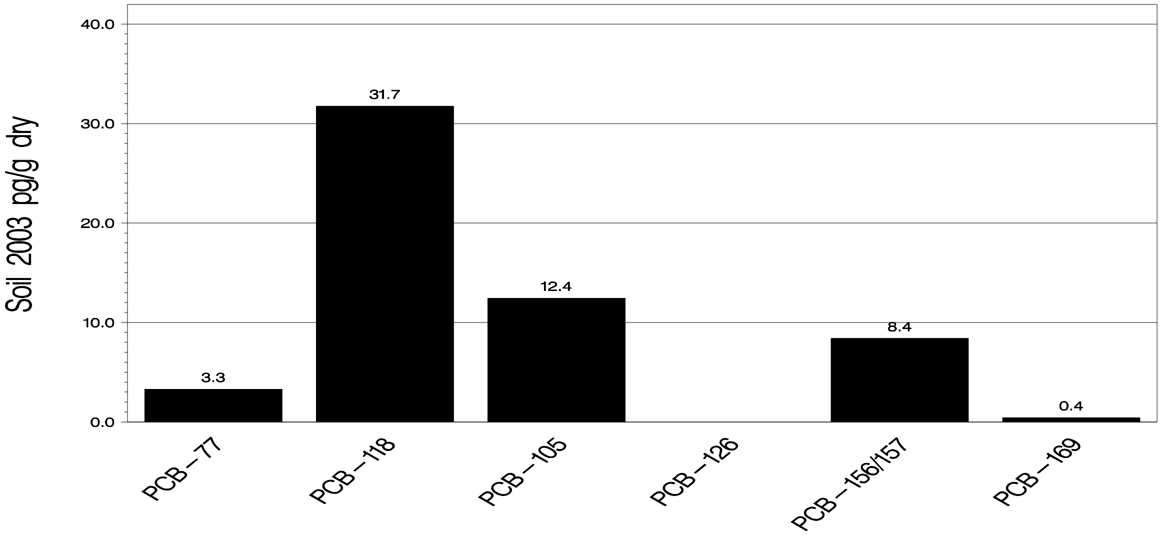
Site 1
Penn Nursery, PA



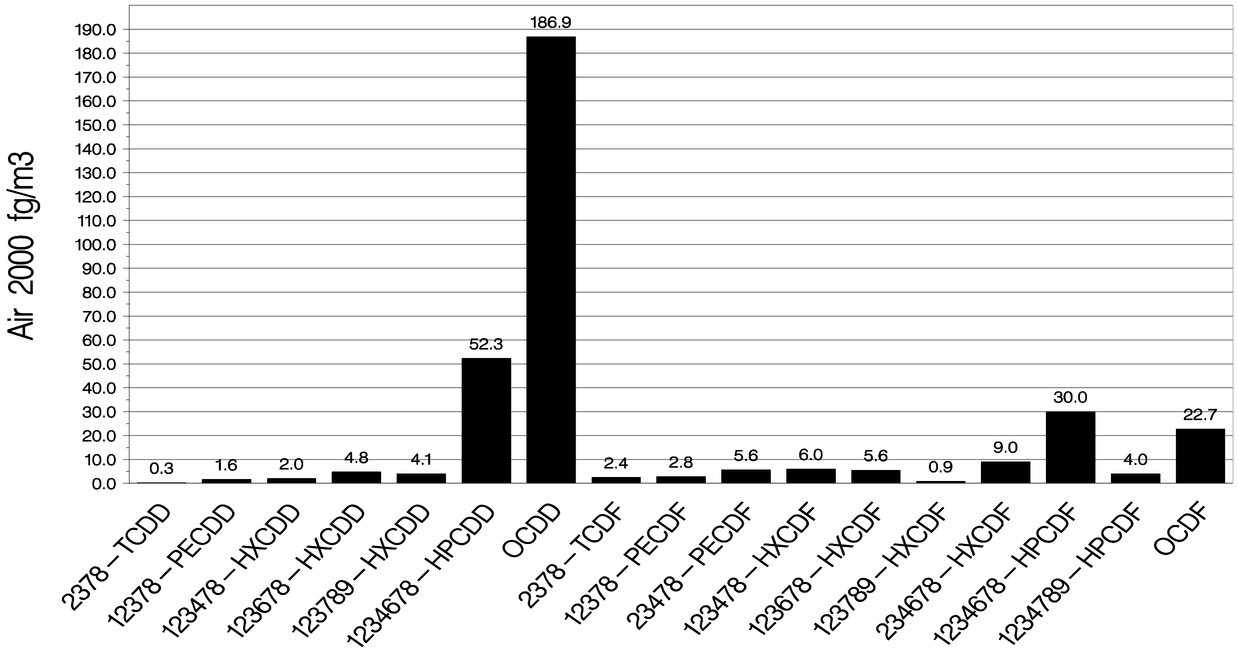


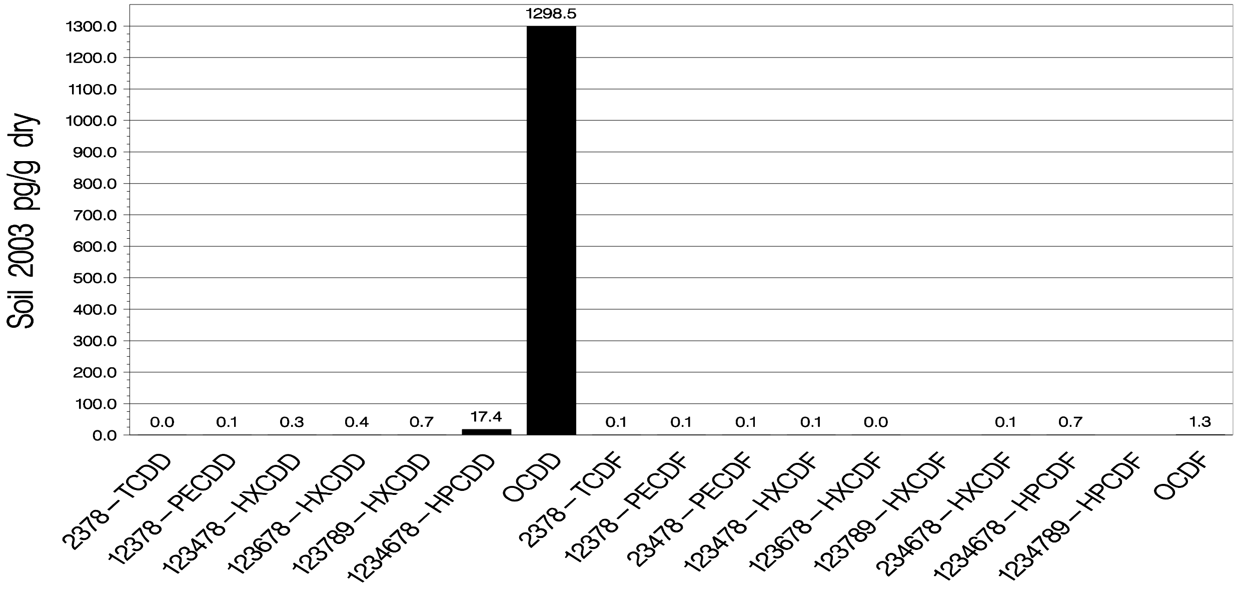
Site 1
Penn Nursery, PA



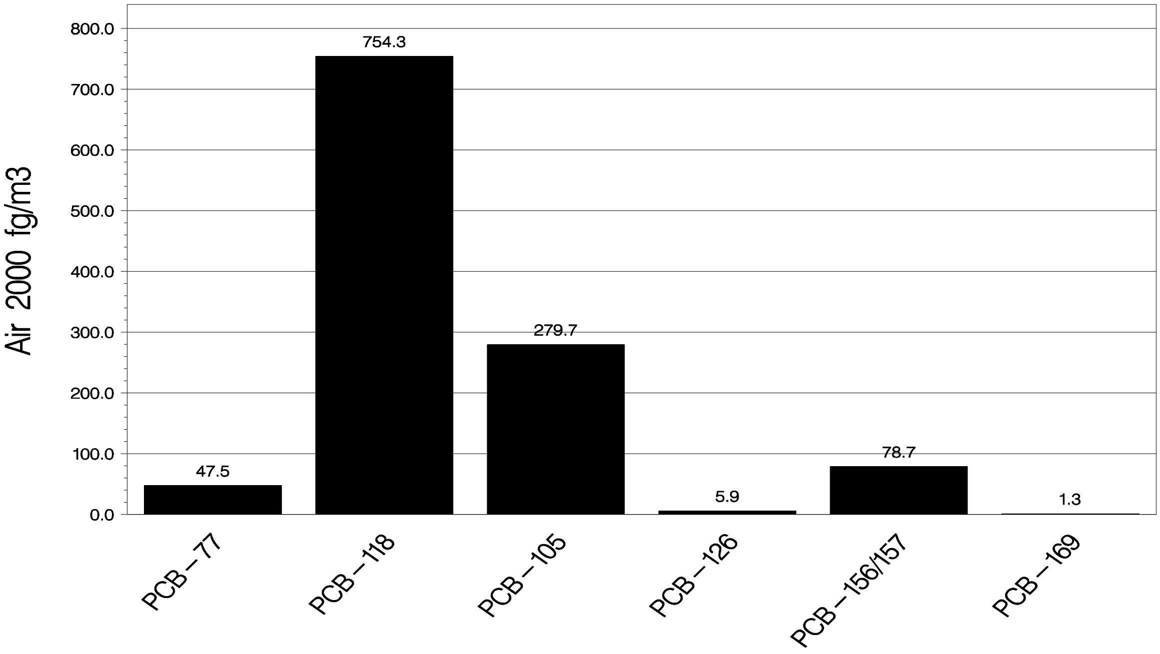


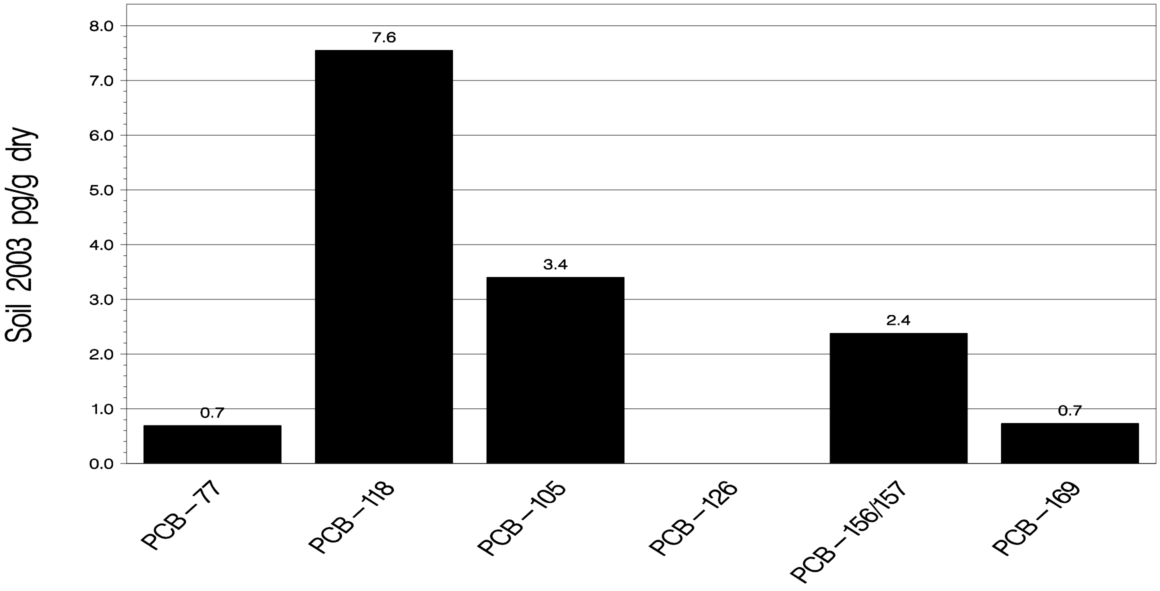
Site 2
Clinton Crops, NC



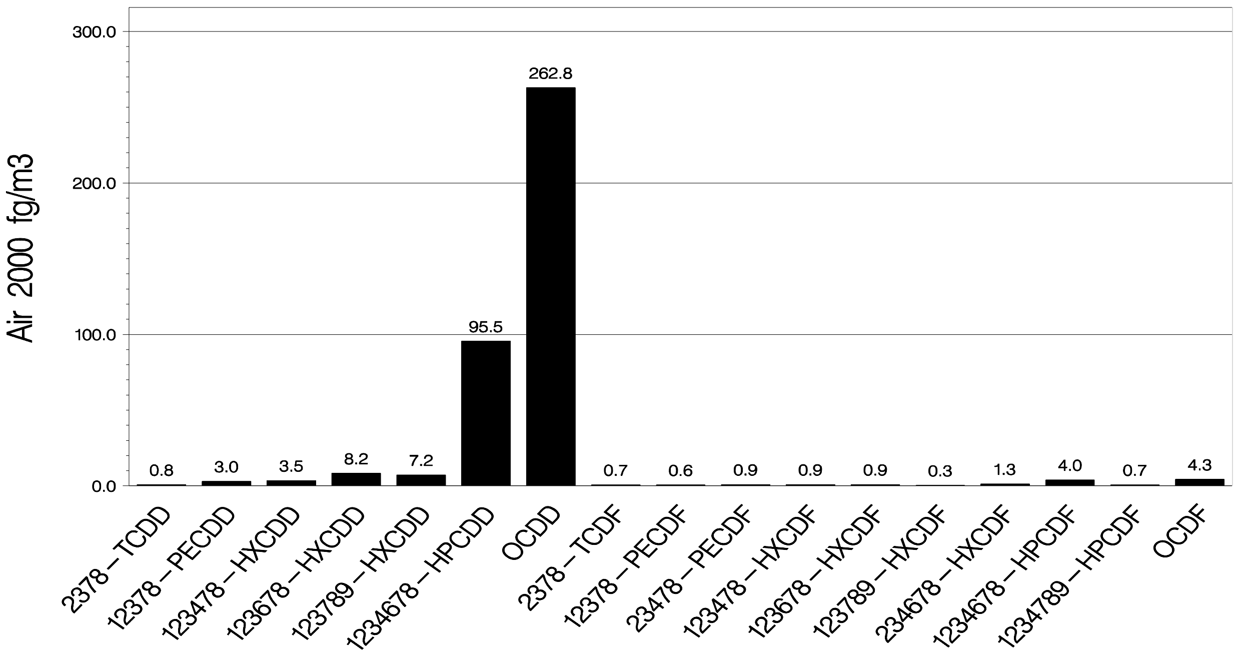


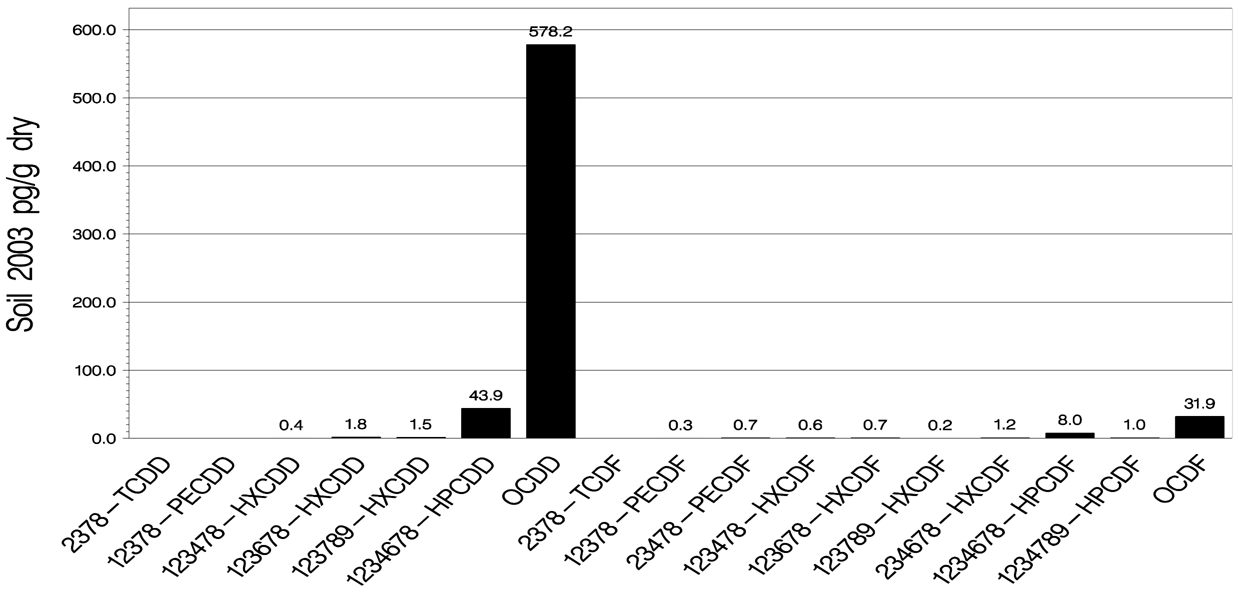
Site 2
Clinton Crops, NC



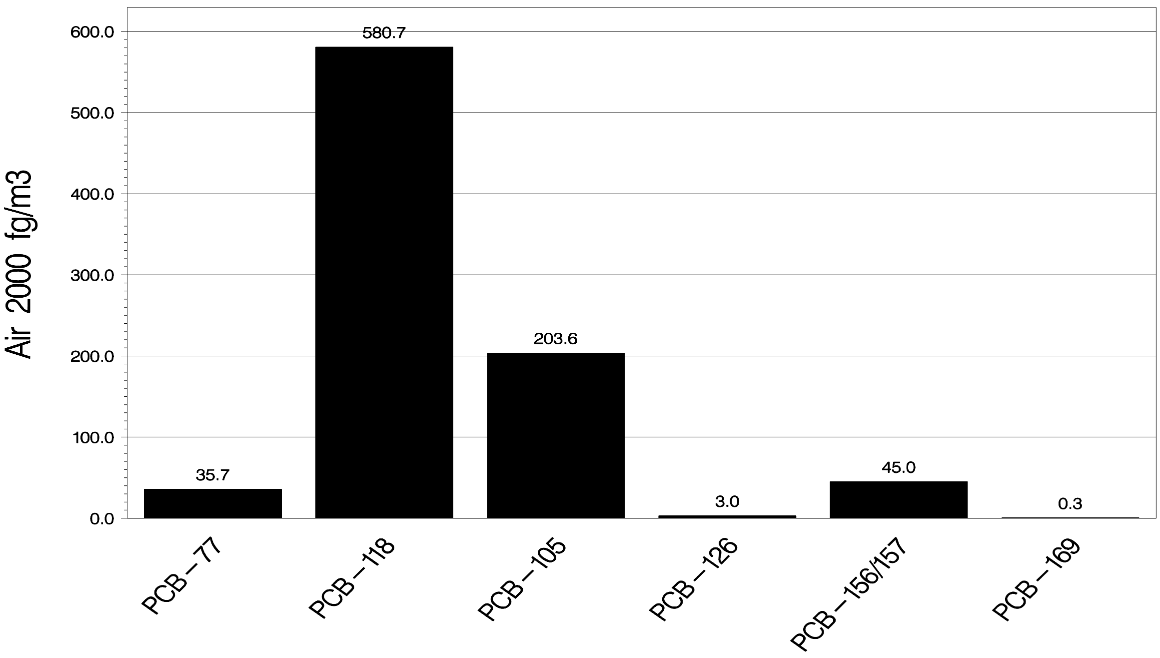


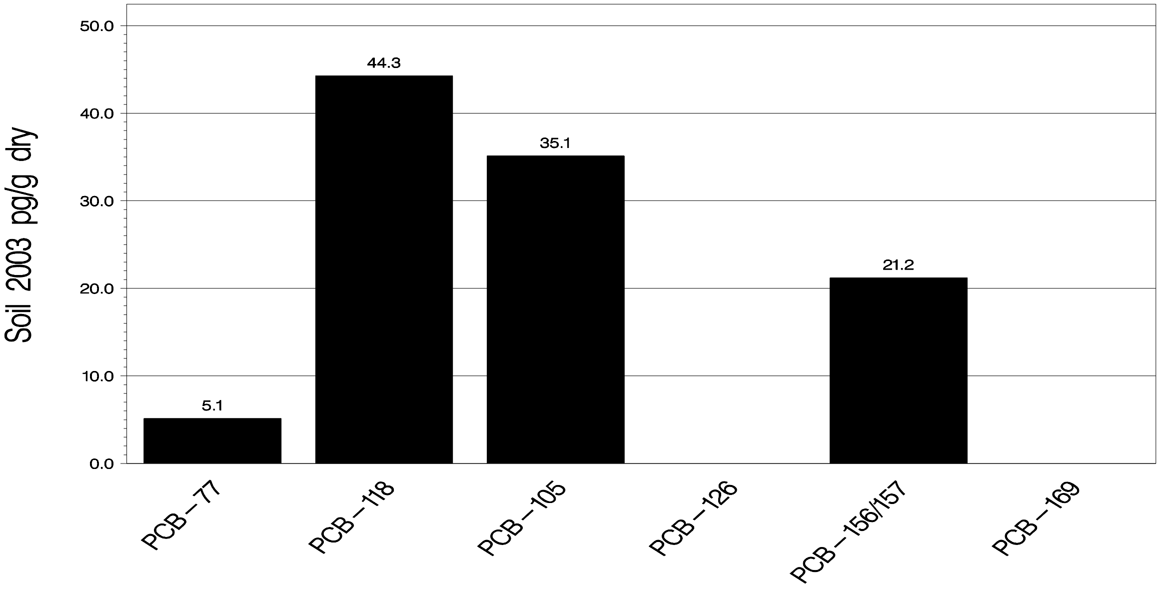
Site 4
Everglades, FL



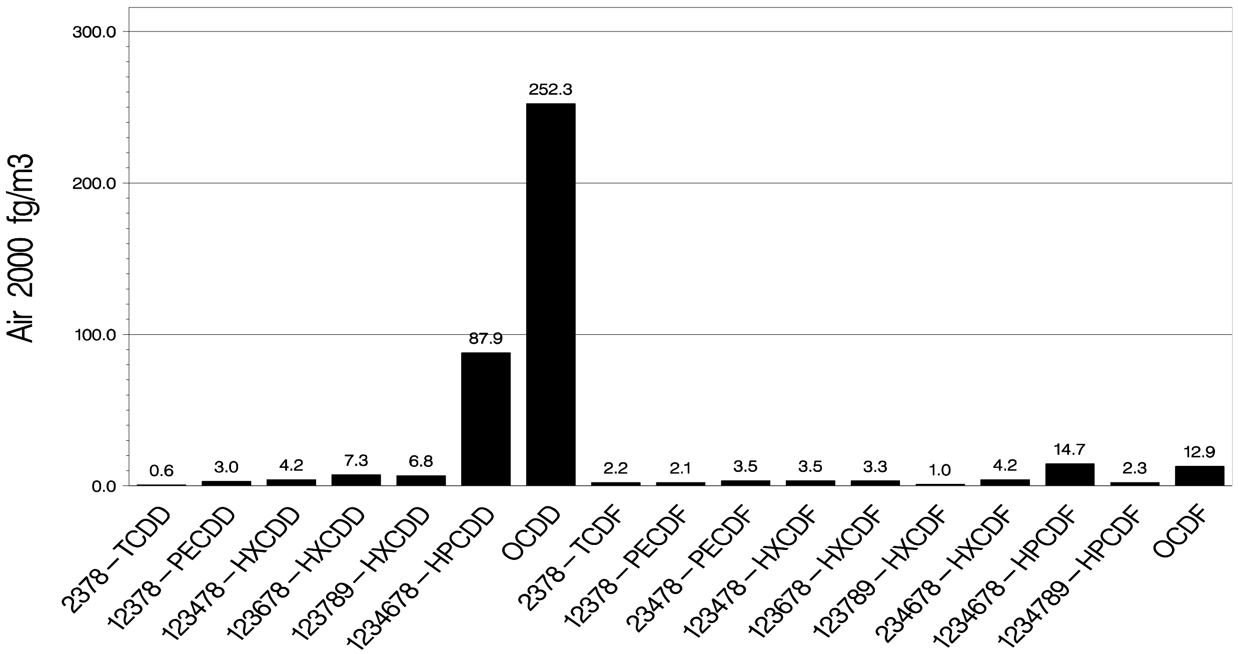


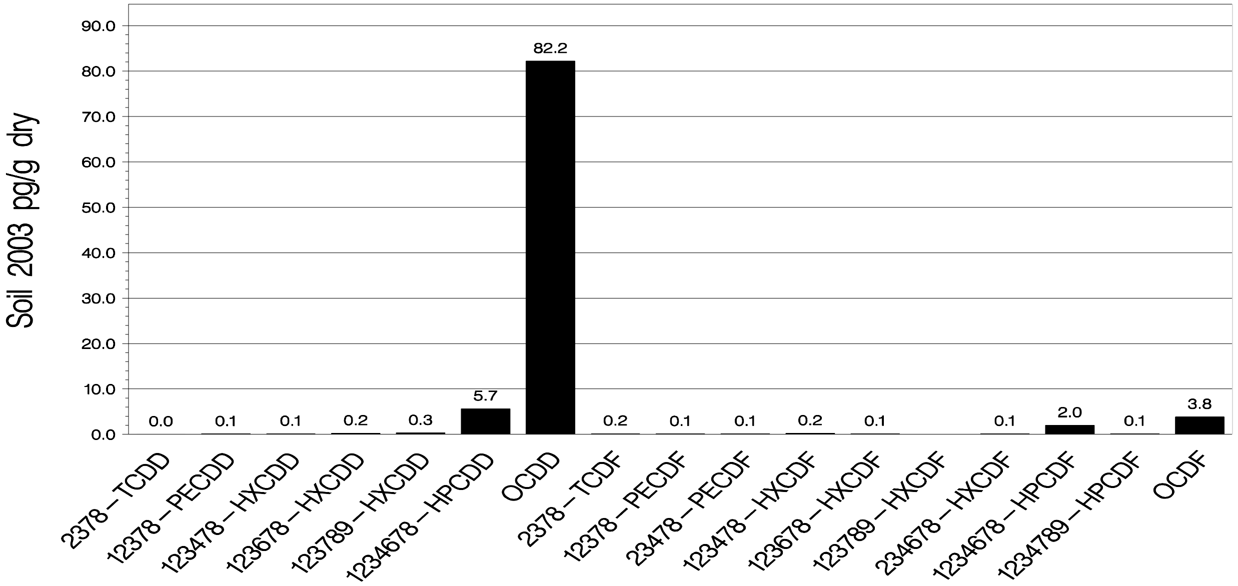
Site 4
Everglades, FL



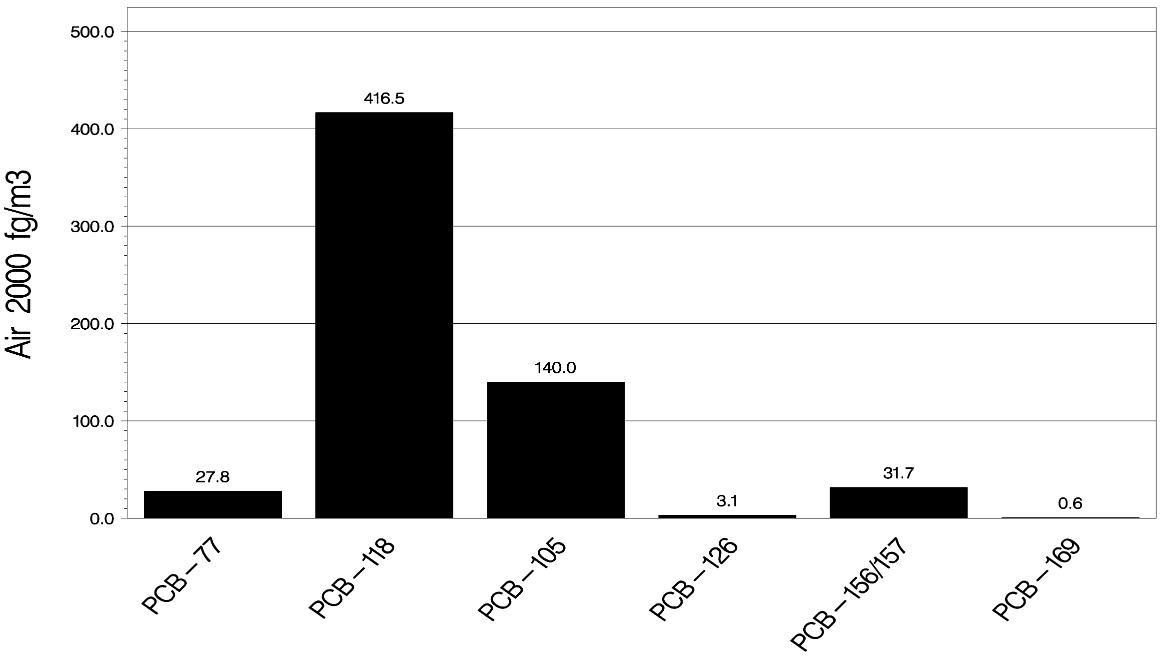


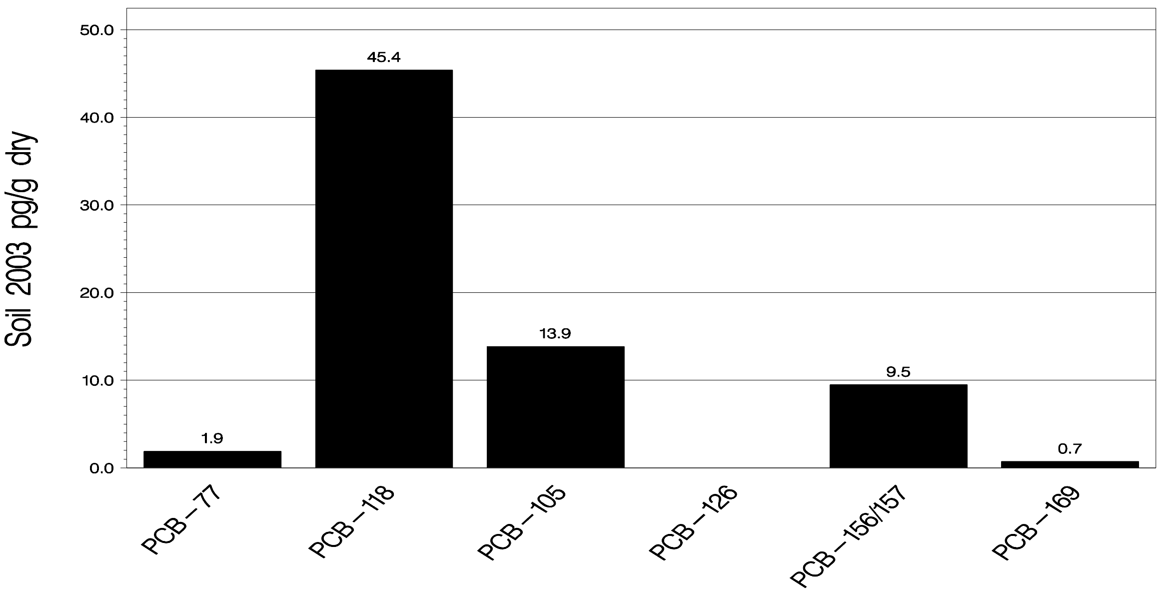
Site 5
Lake Dubay, WI



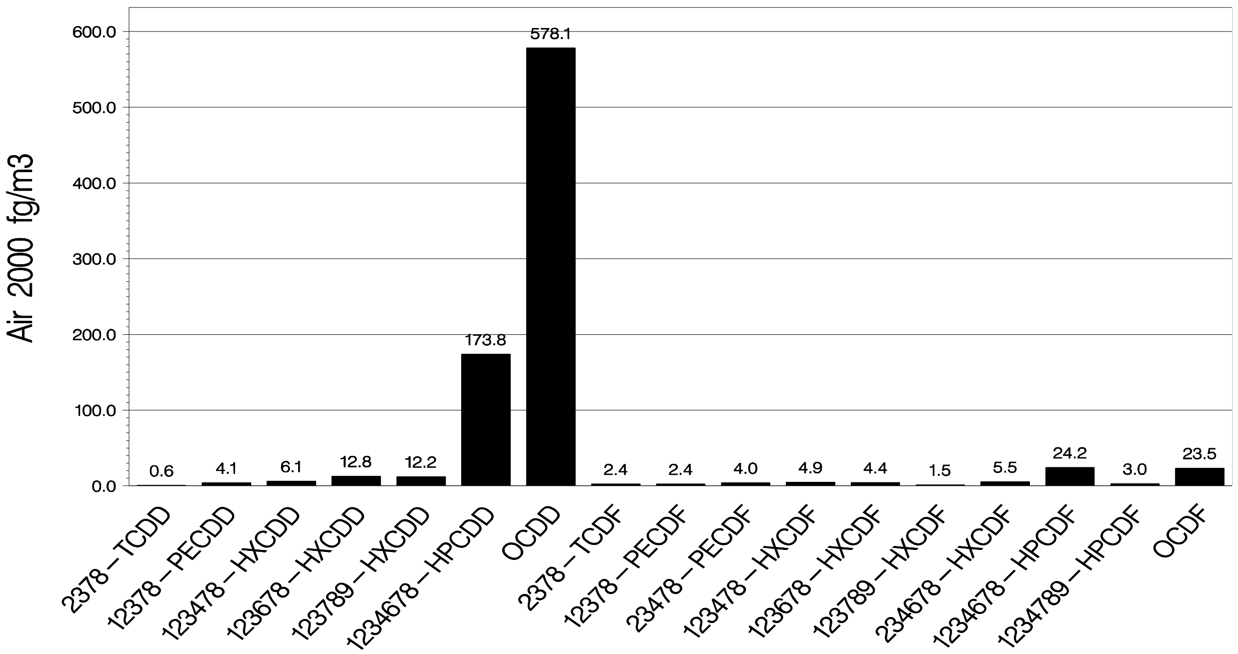


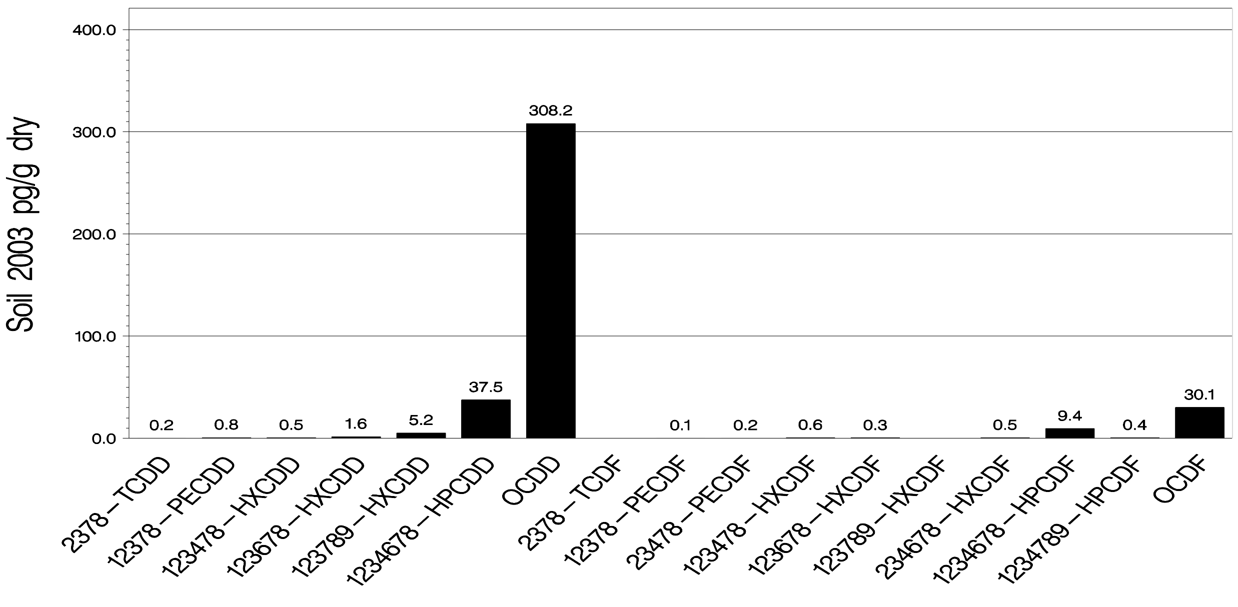
Site 5
Lake Dubay, WI



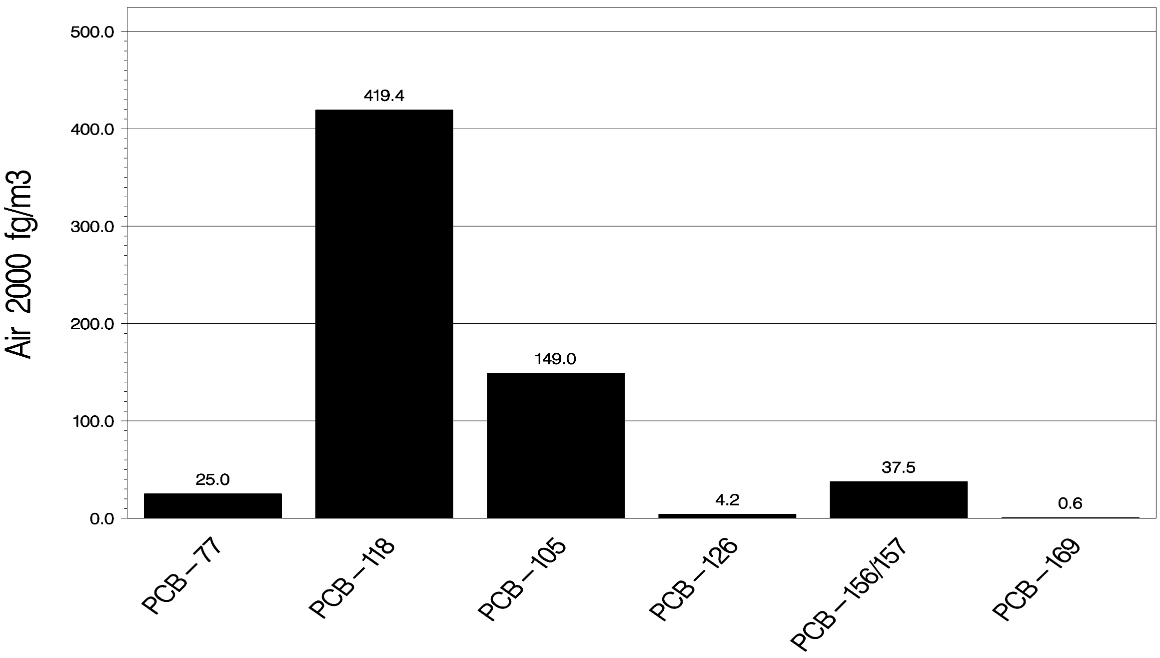


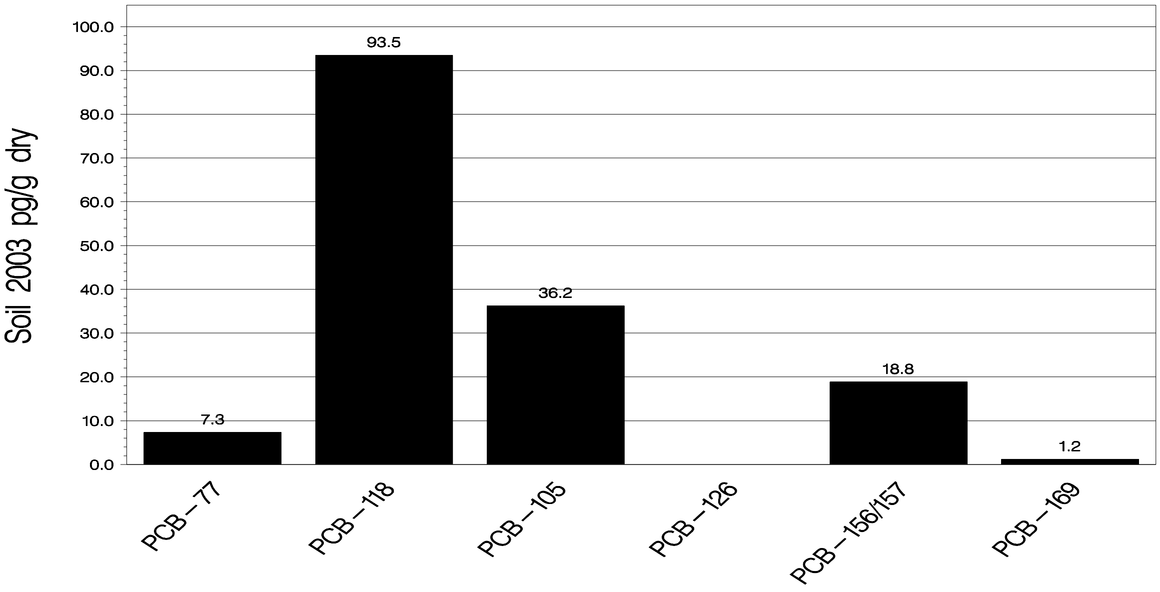
Site 6
Monmouth, IL



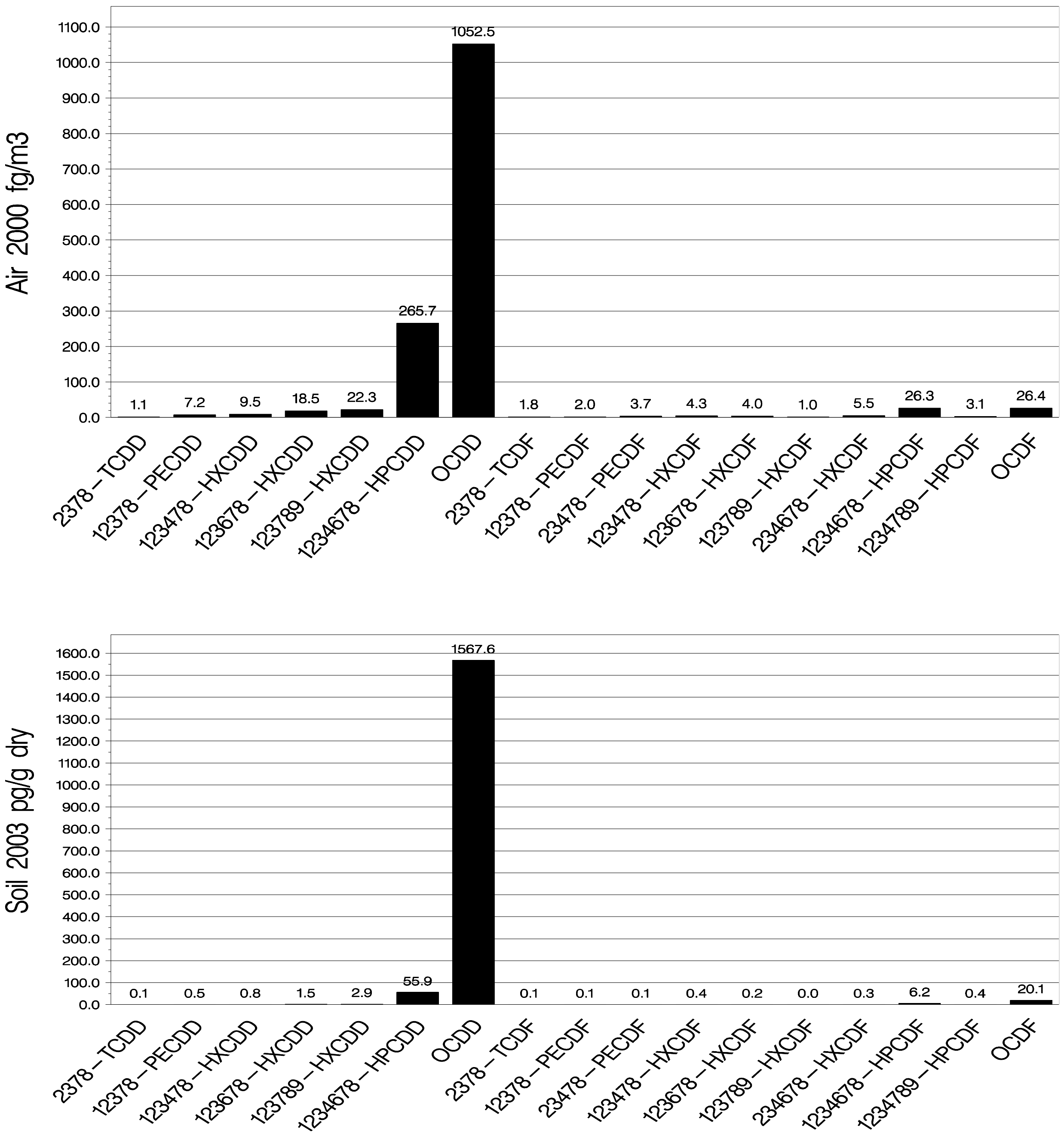


Site 6
Monmouth, IL

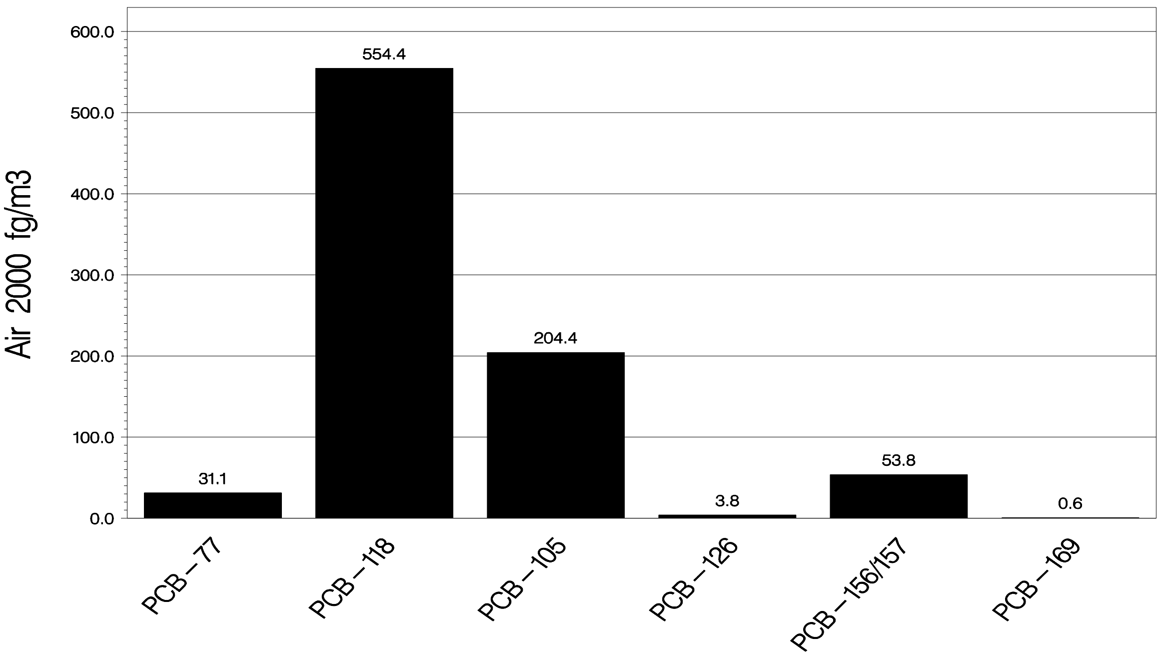


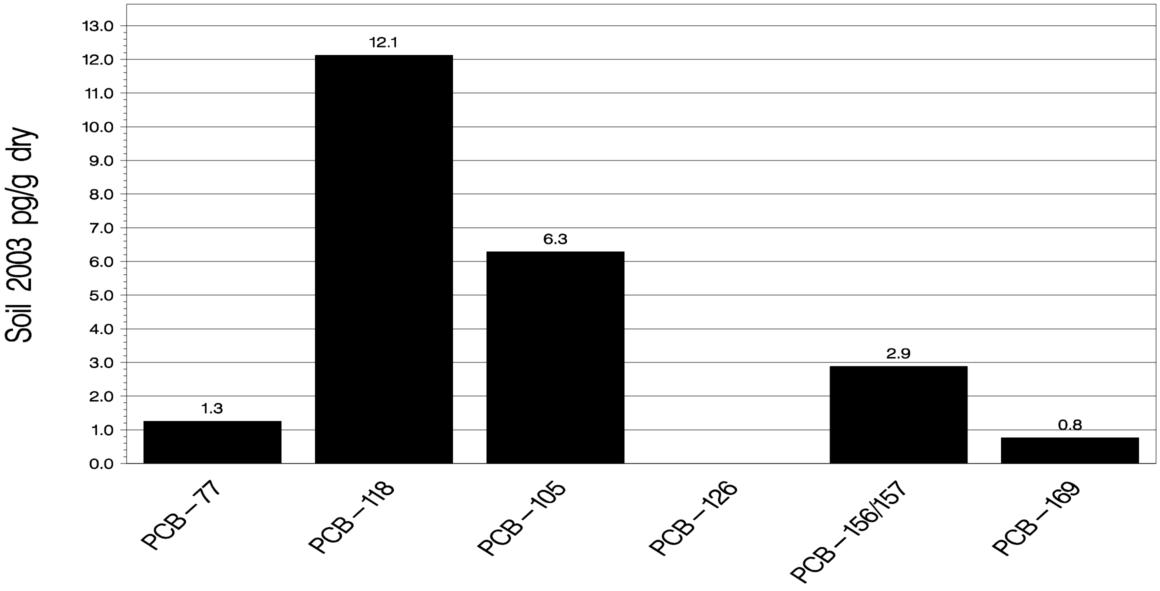


Site 7
McNay Farms, IA

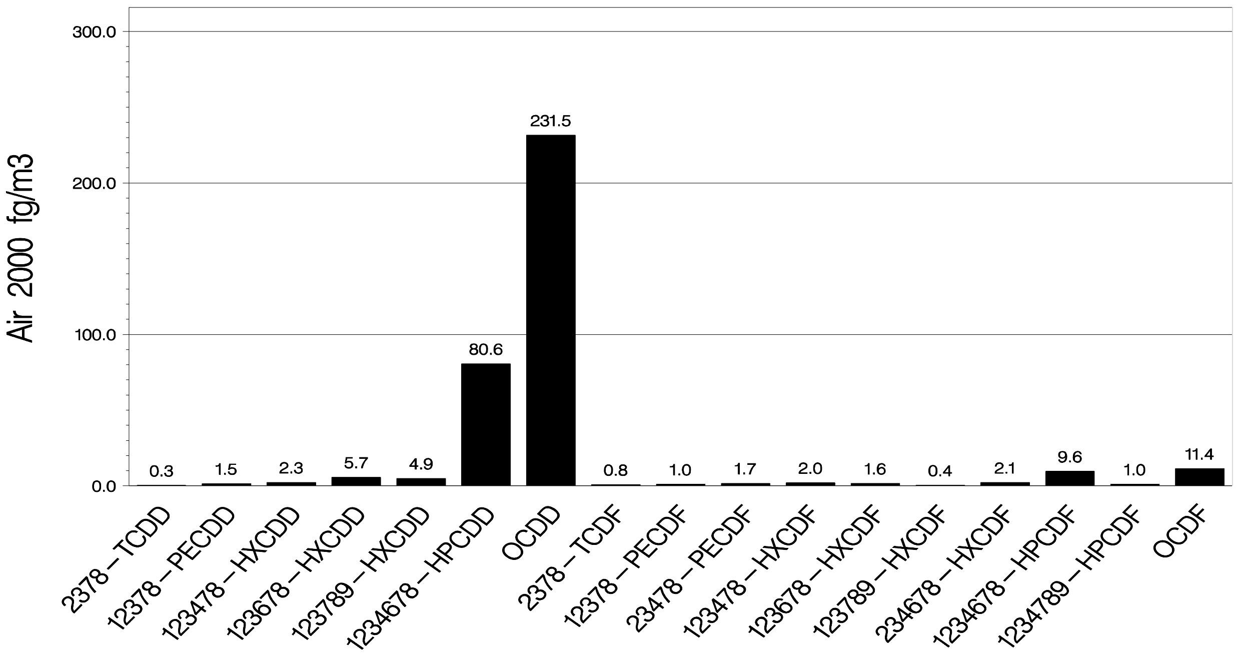


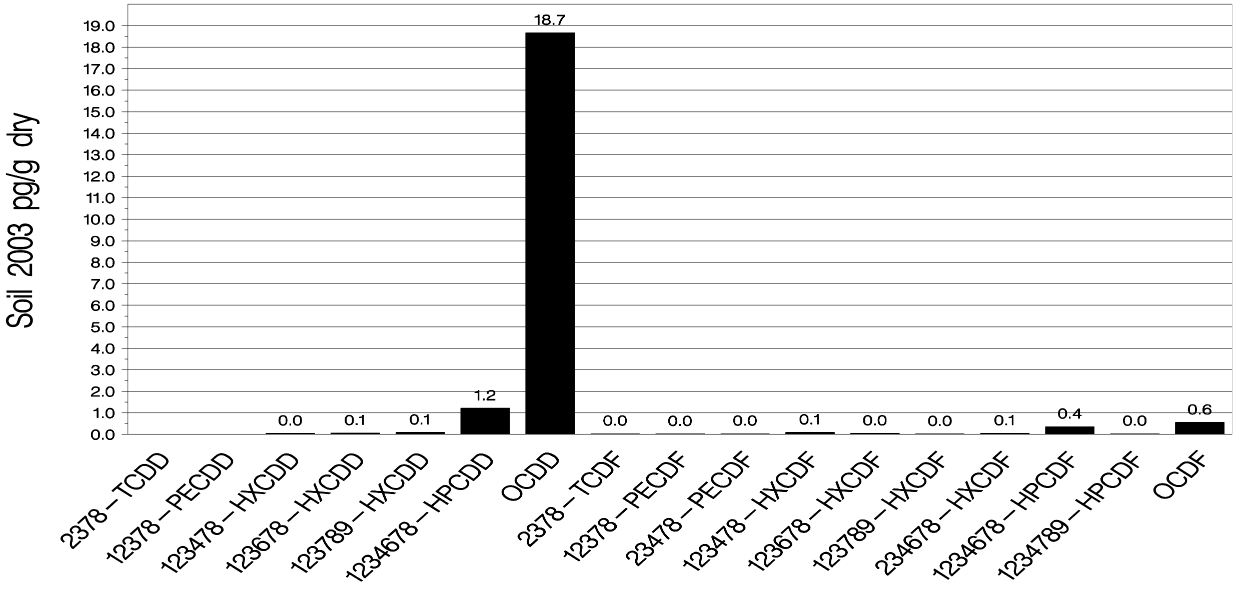
Site 7
McNay Farms, IA



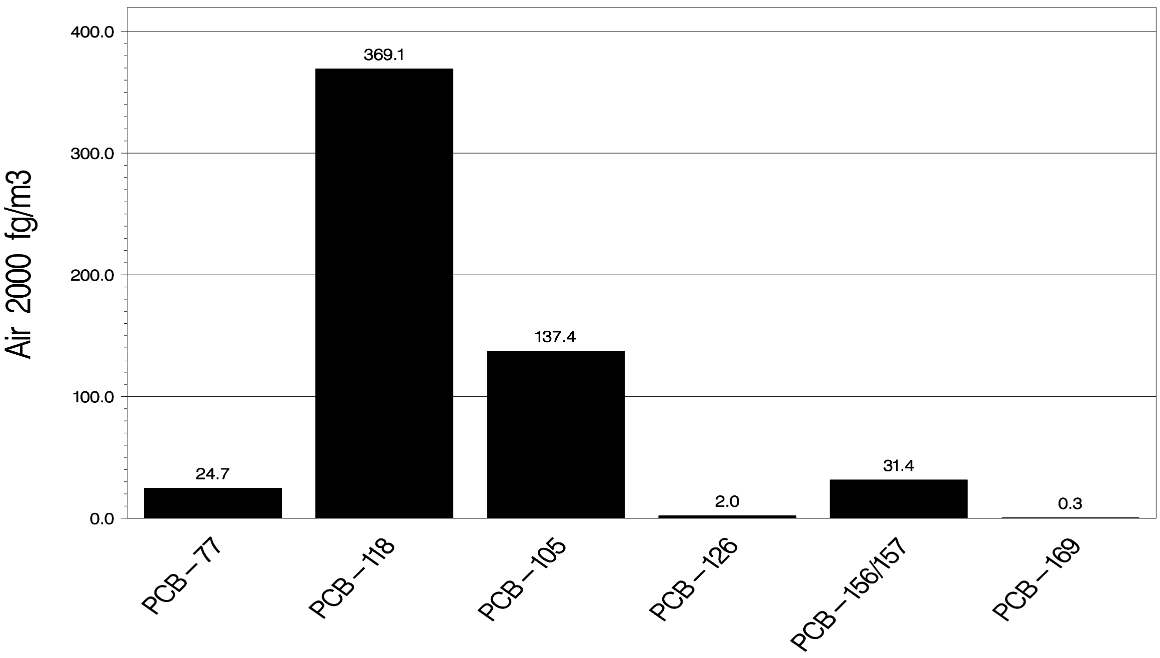


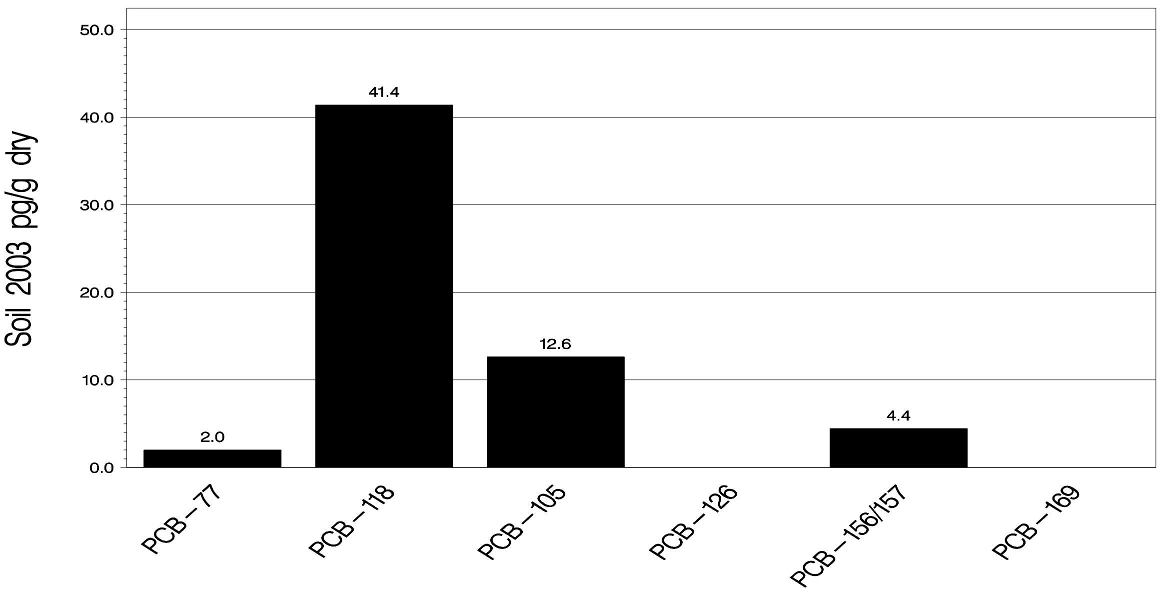
Site 8
Lake Scott, KS



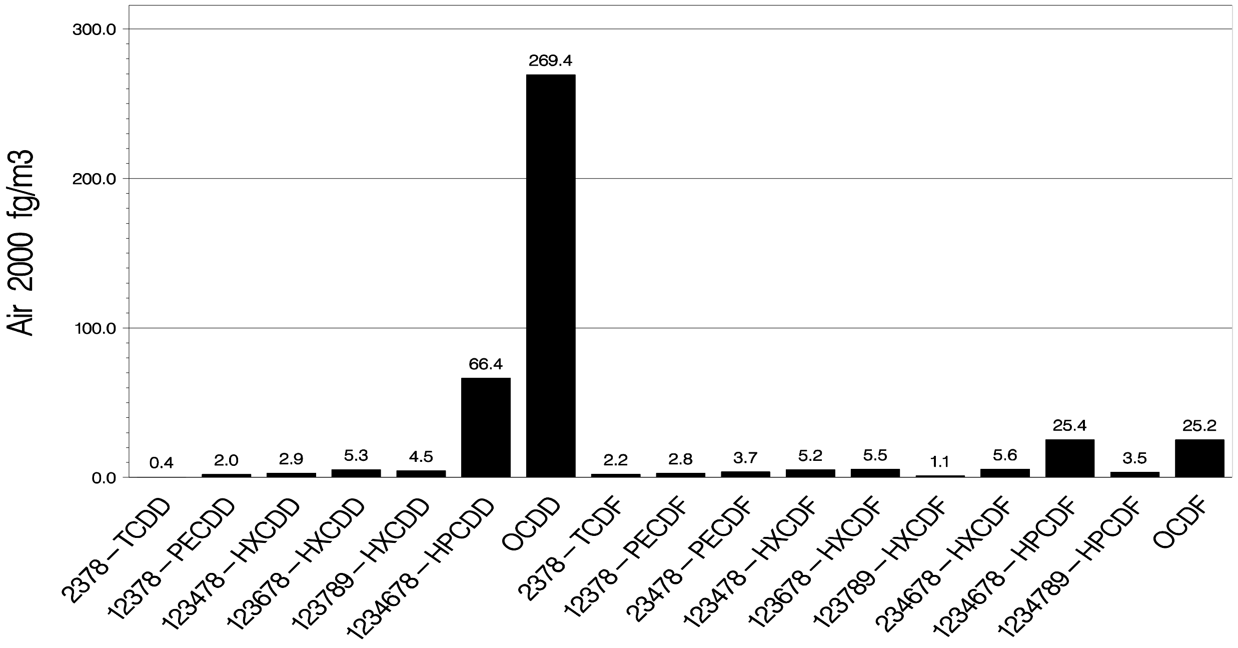


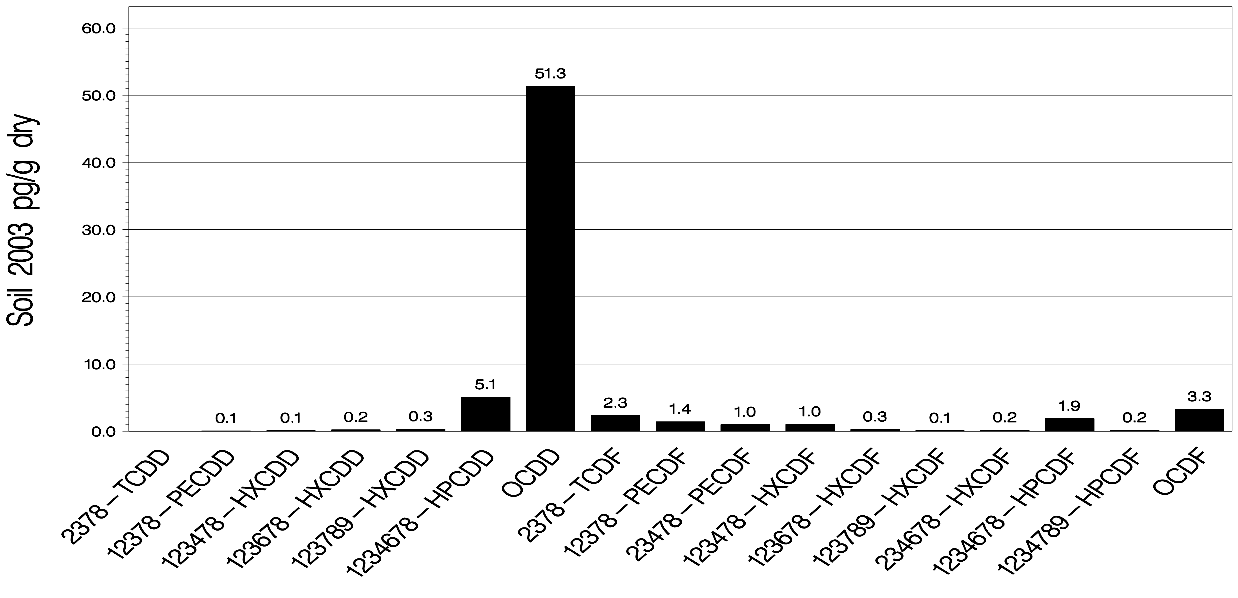
Site 8
Lake Scott, KS



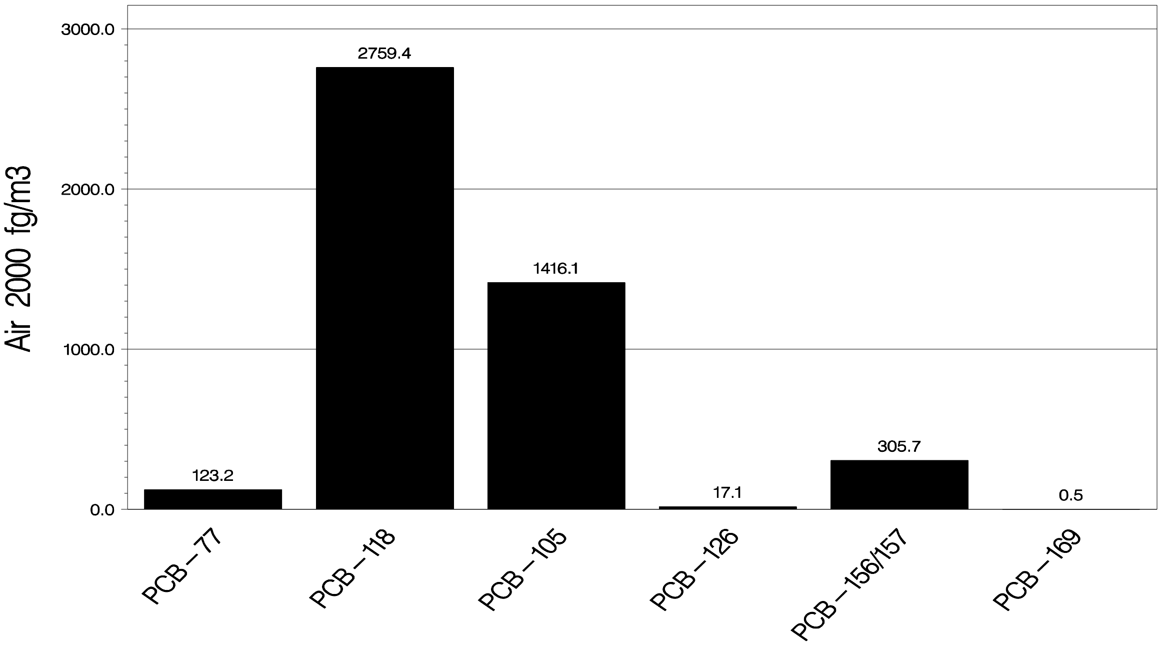


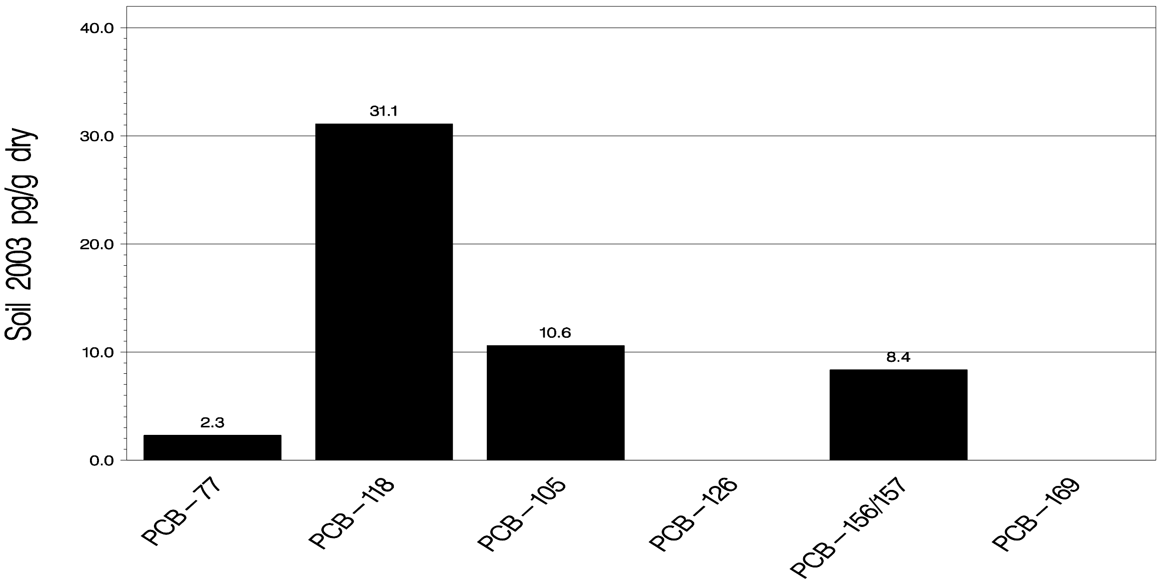
Site 9
Bixby, OK - 2000 Air
Keystone State Park, OK - 2003 Soil



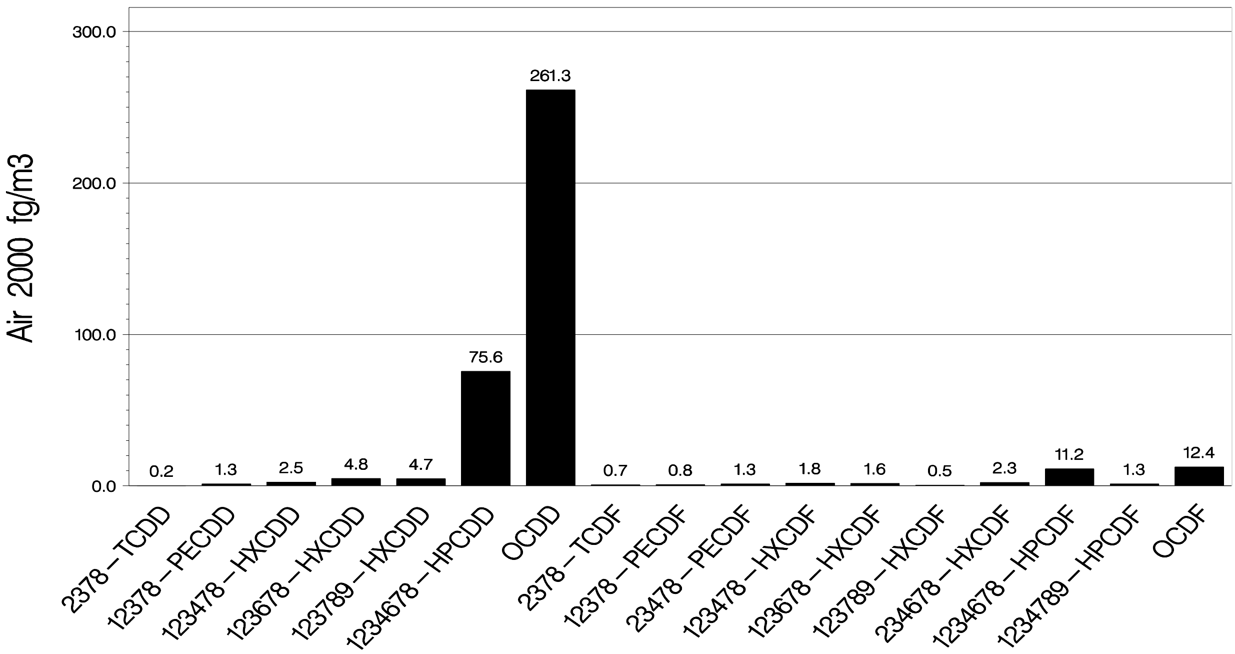


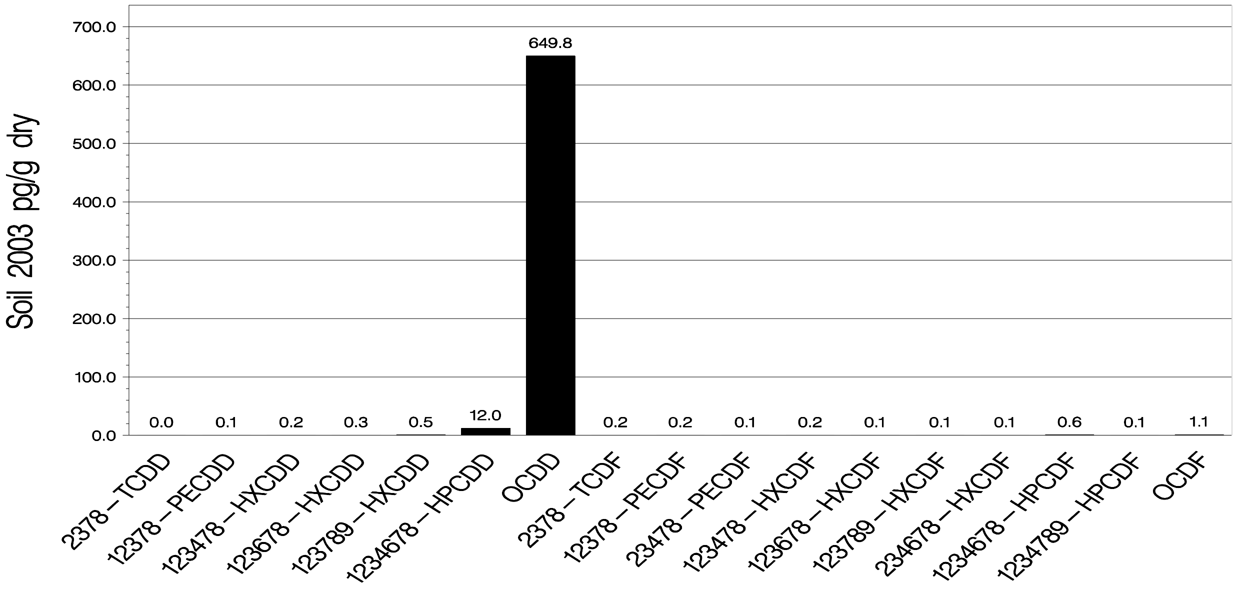
Site 9
Bixby, OK - 2000 Air
Keystone State Park, OK - 2003 Soil



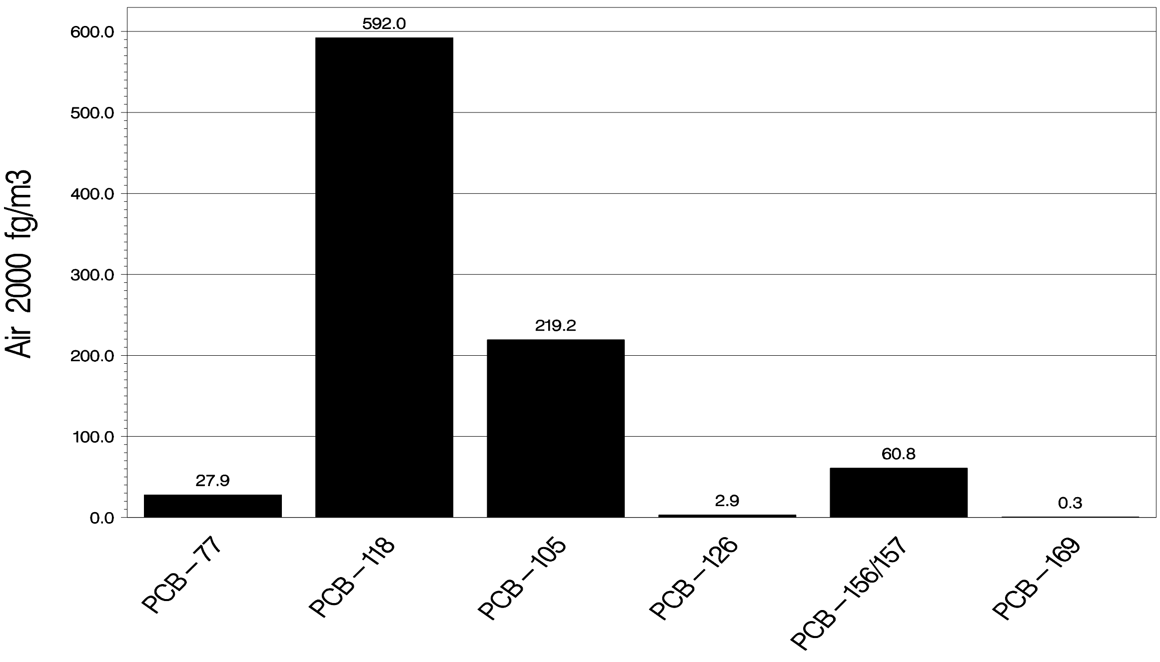


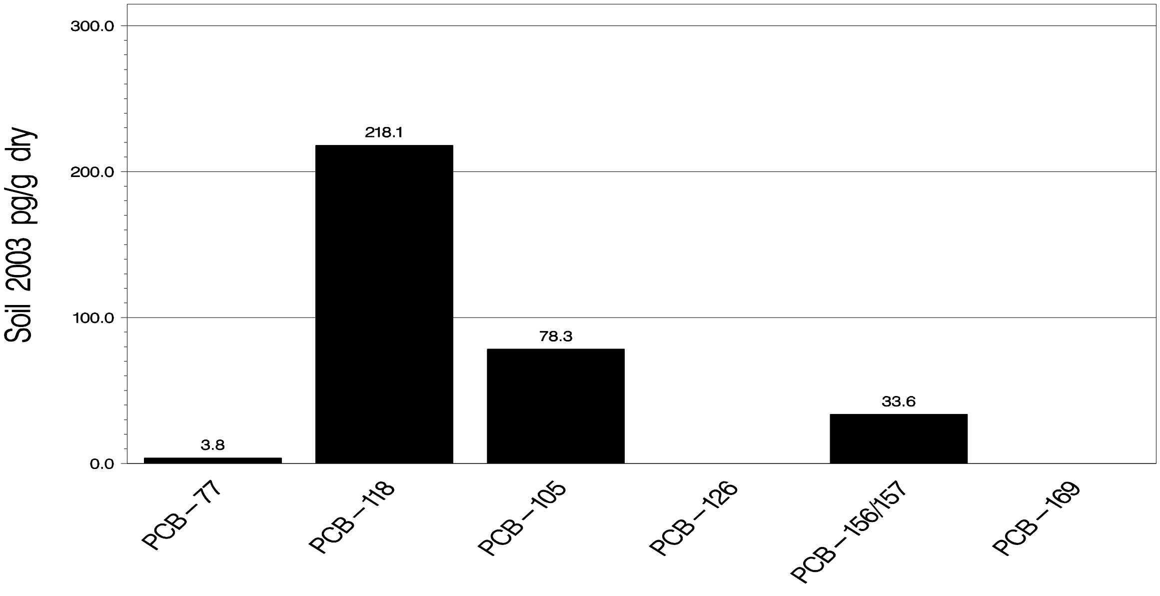
Site 10
Arkadelphia, AR



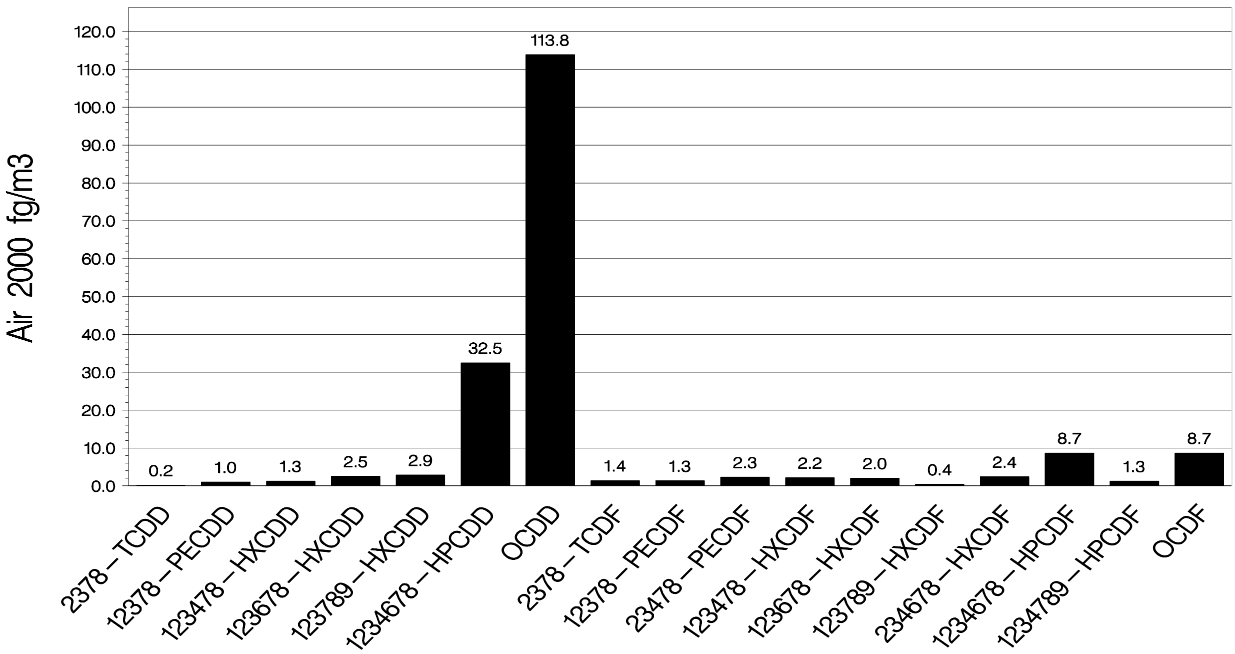


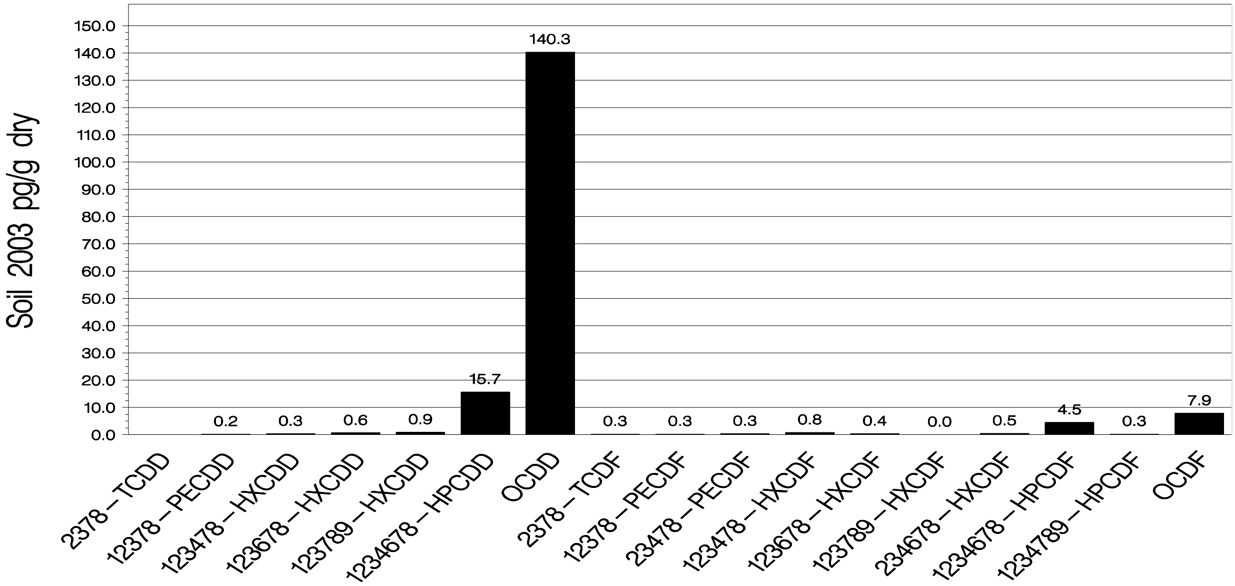
Site 10
Arkadelphia, AR



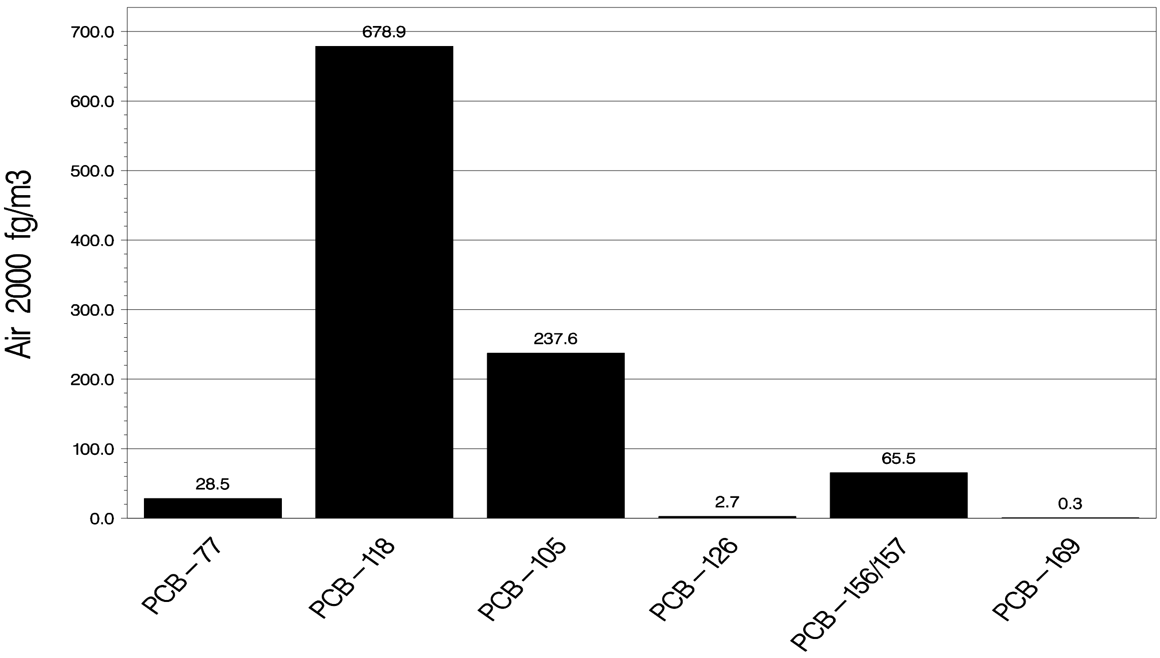


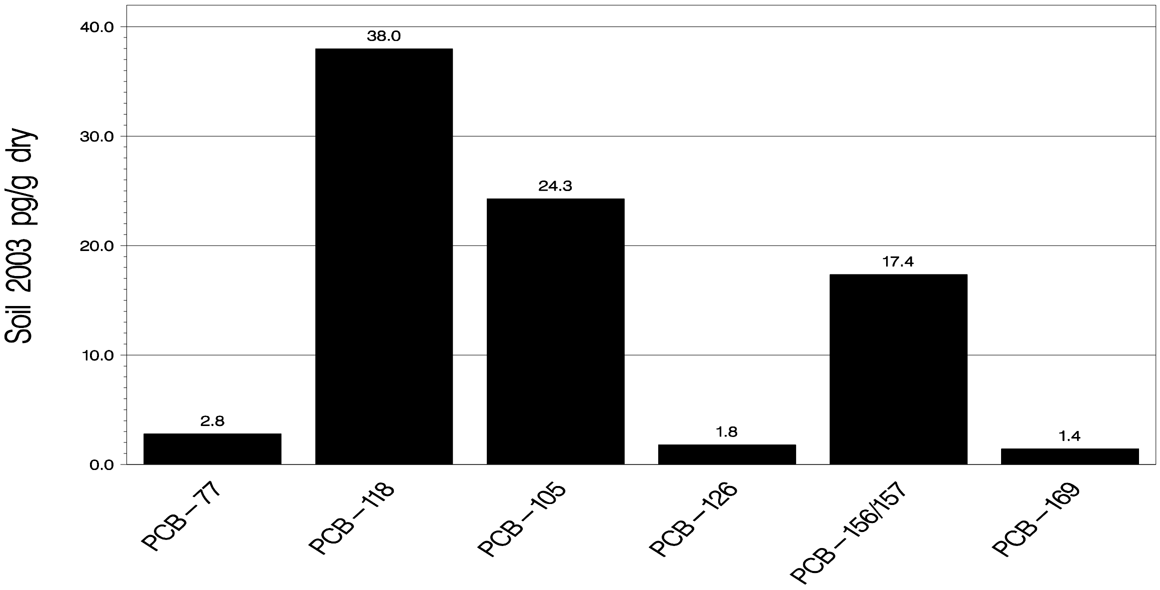
Site 11
Bennington, VT



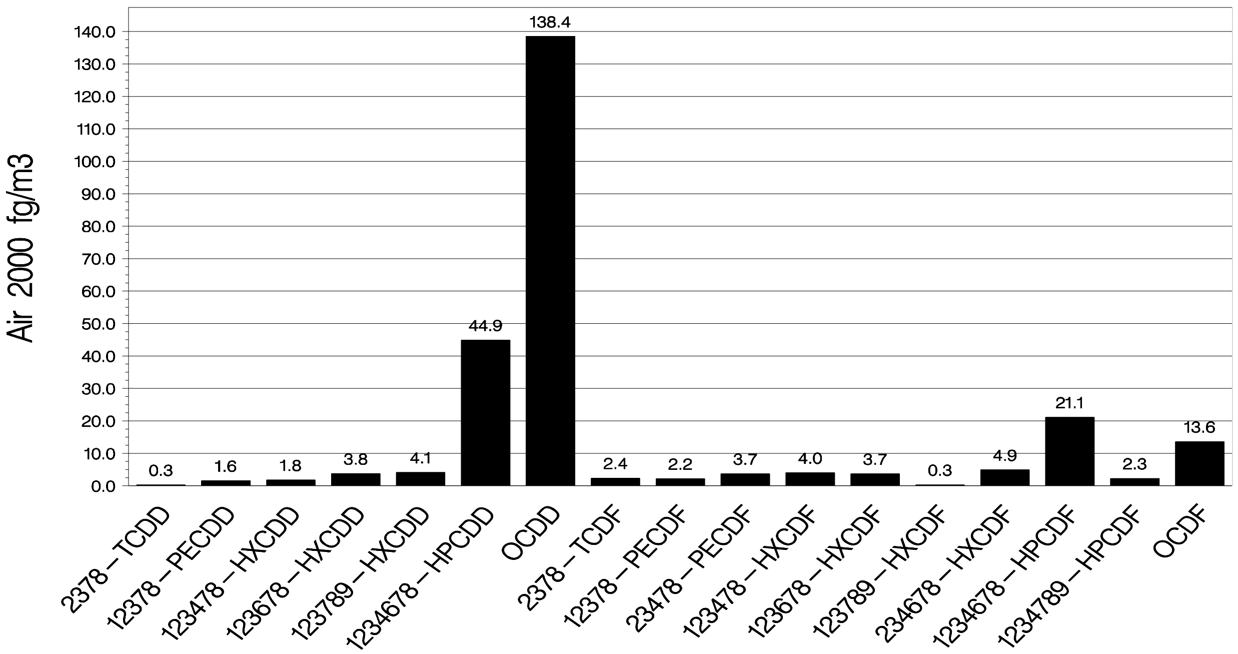


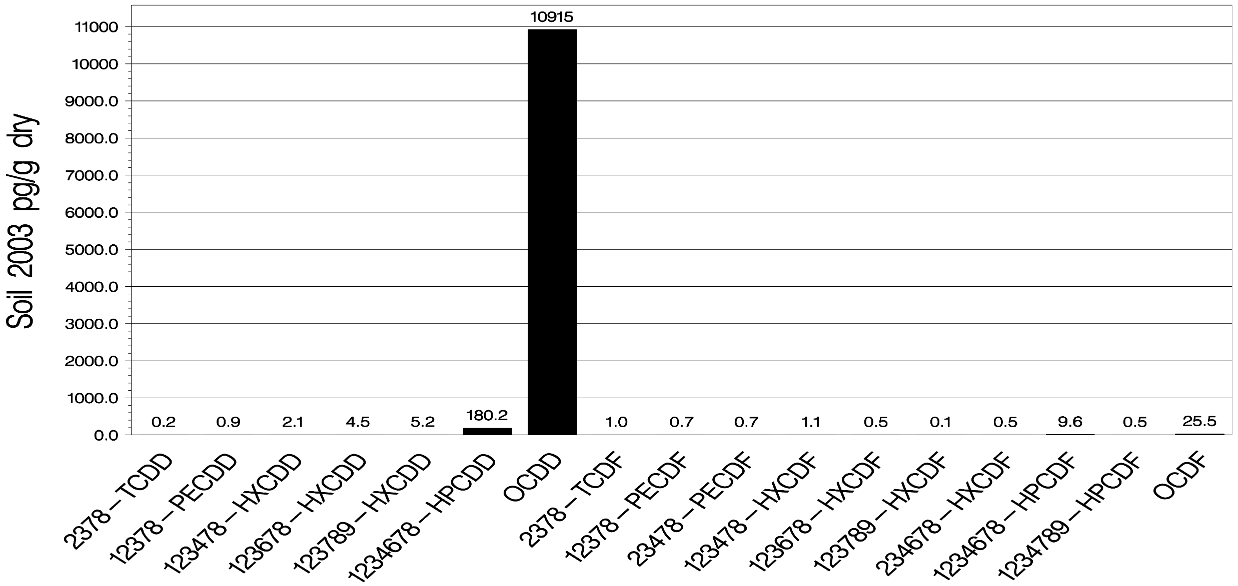
Site 11
Bennington, VT



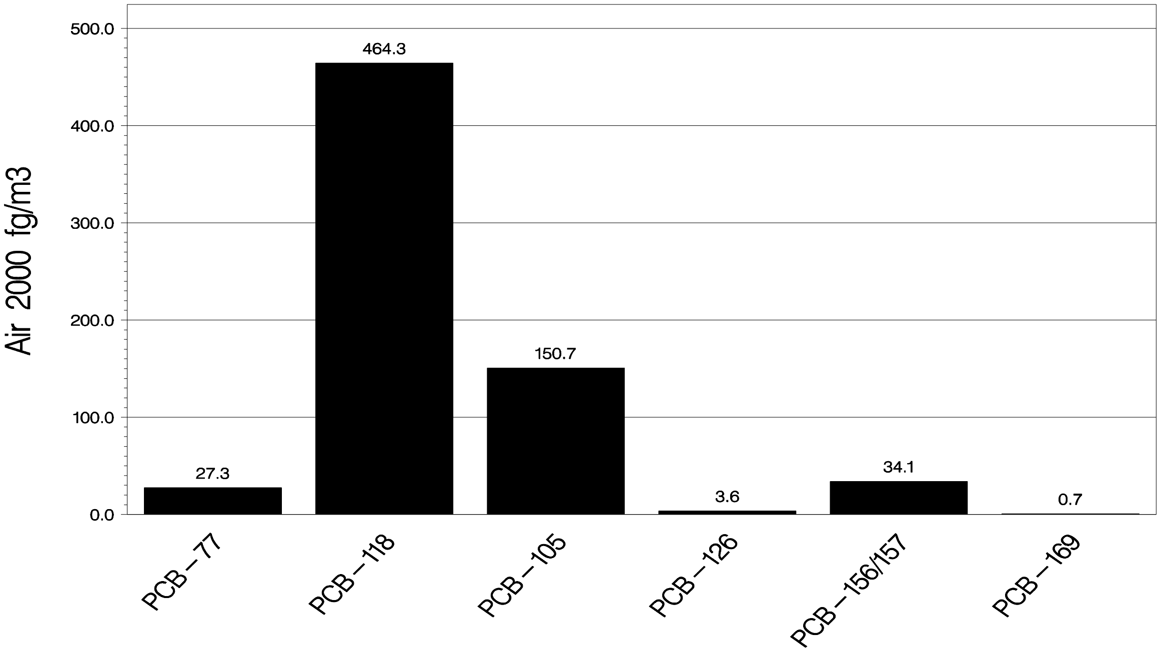


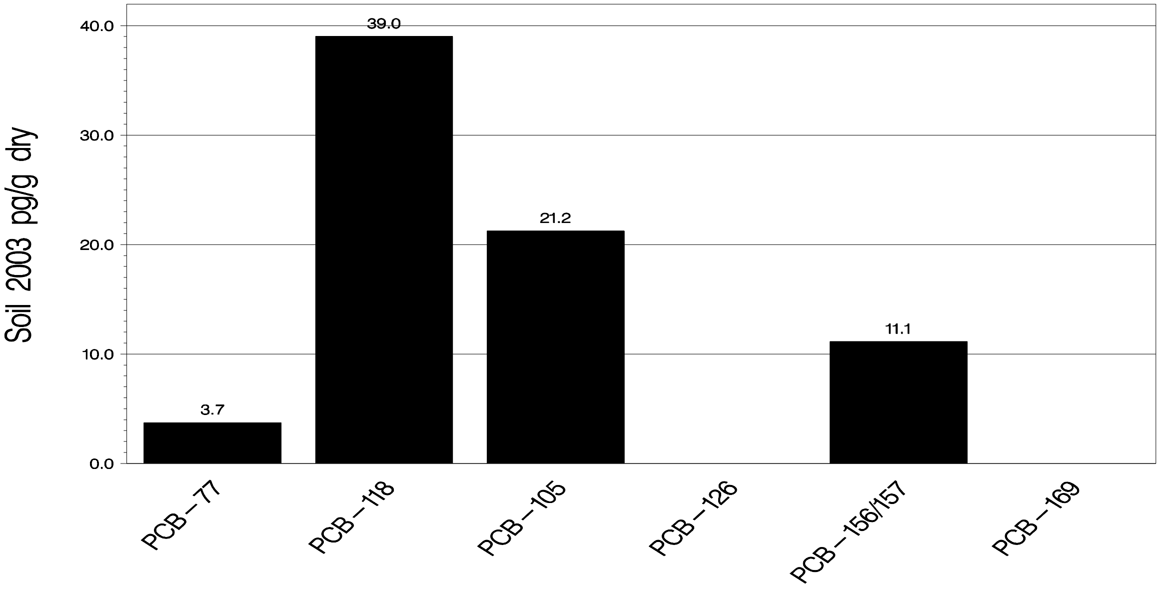
Site 12
Jasper, NY



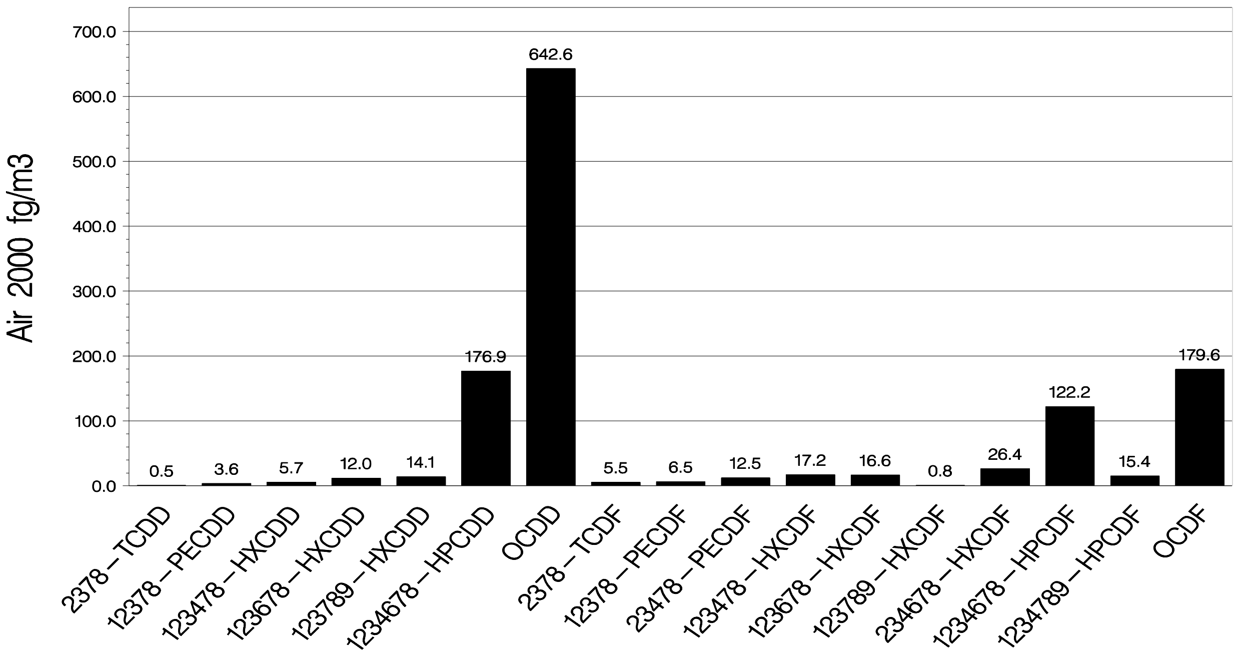


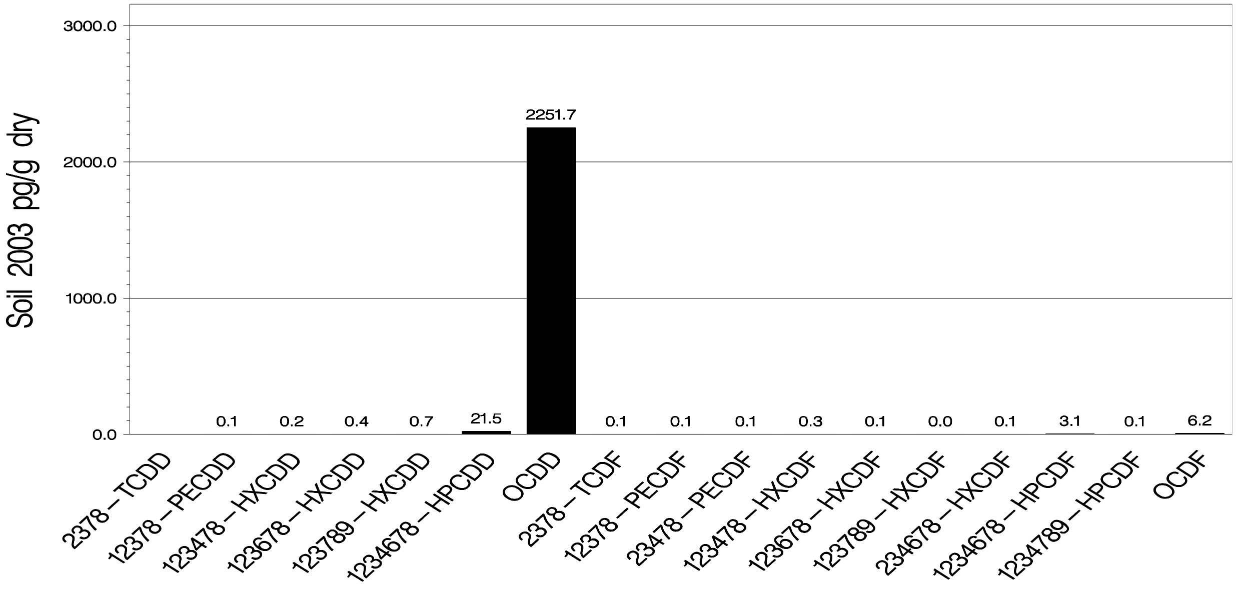
Site 12
Jasper, NY



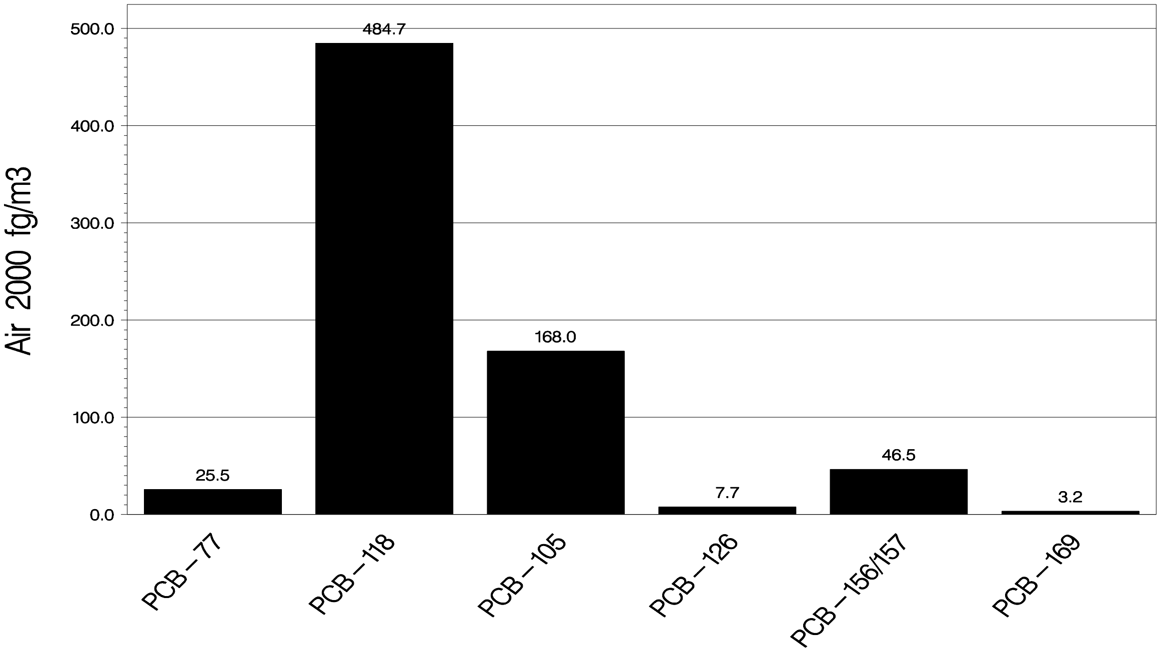


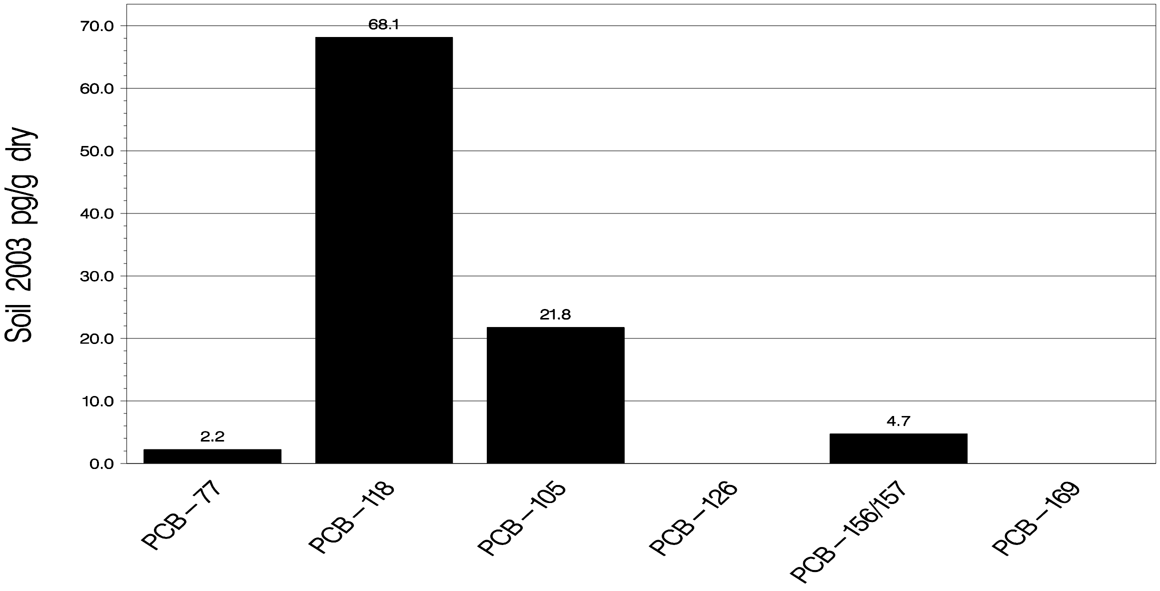
Site 14
Caldwell, OH



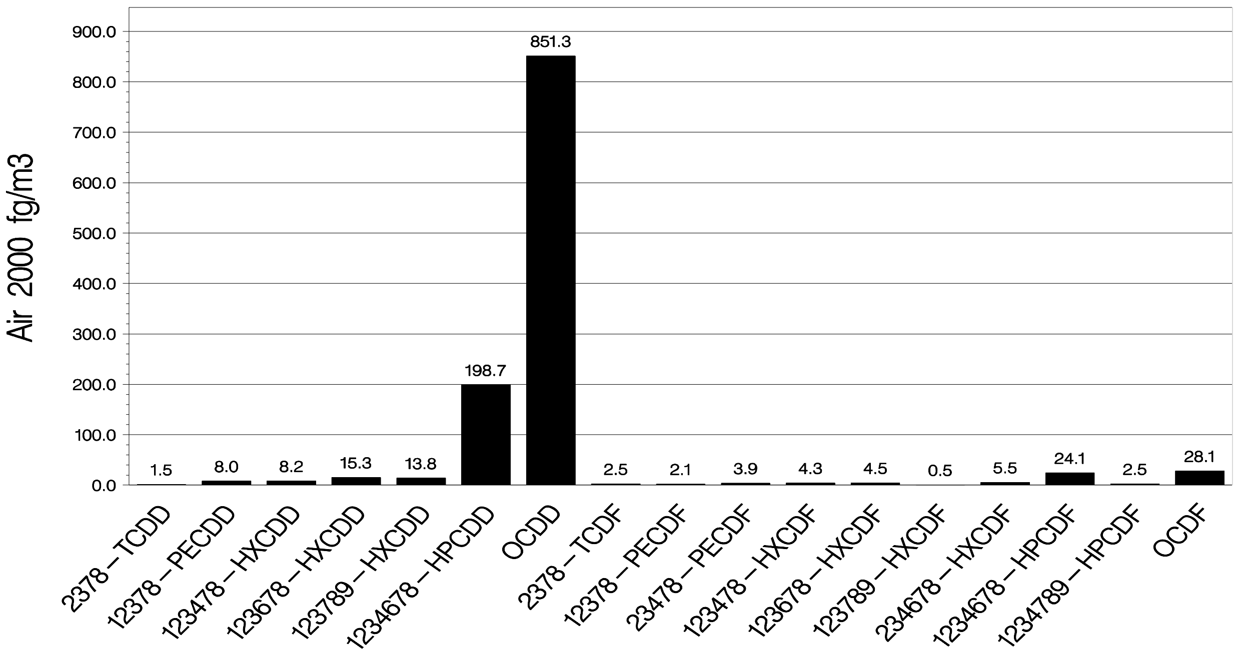


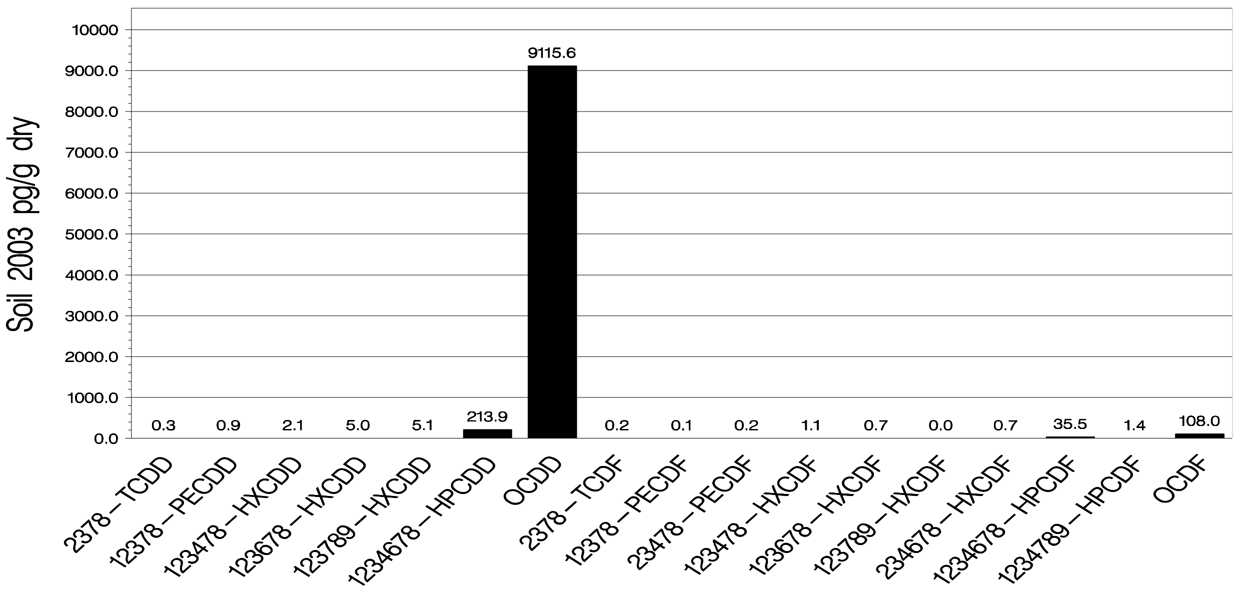
Site 14
Caldwell, OH



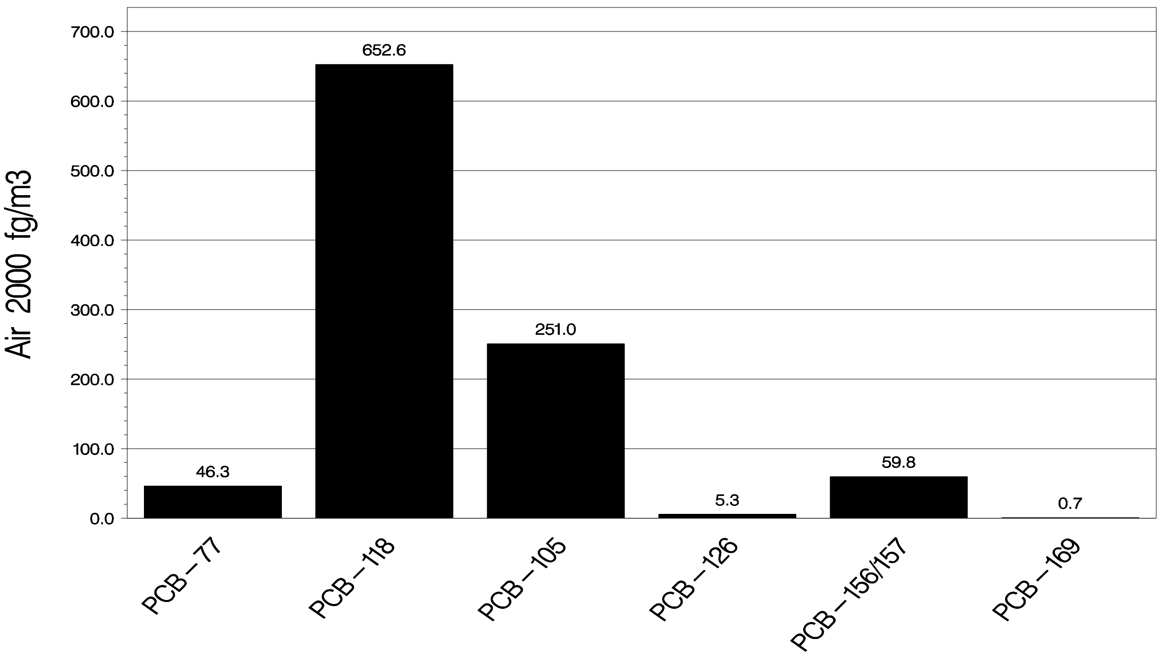


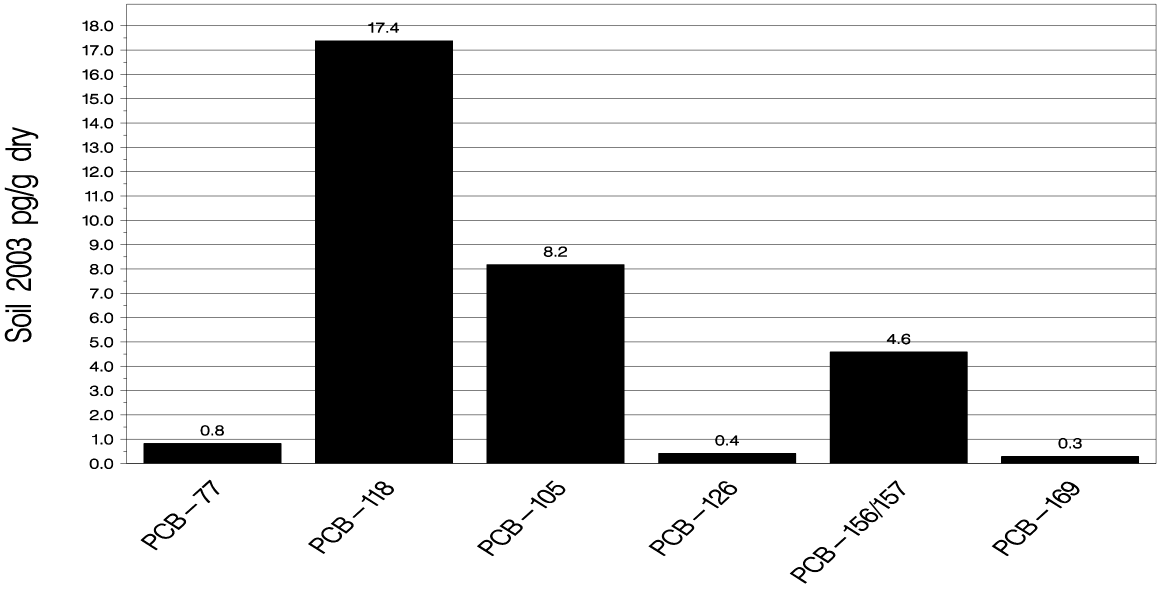
Site 16
Dixon Springs, IL



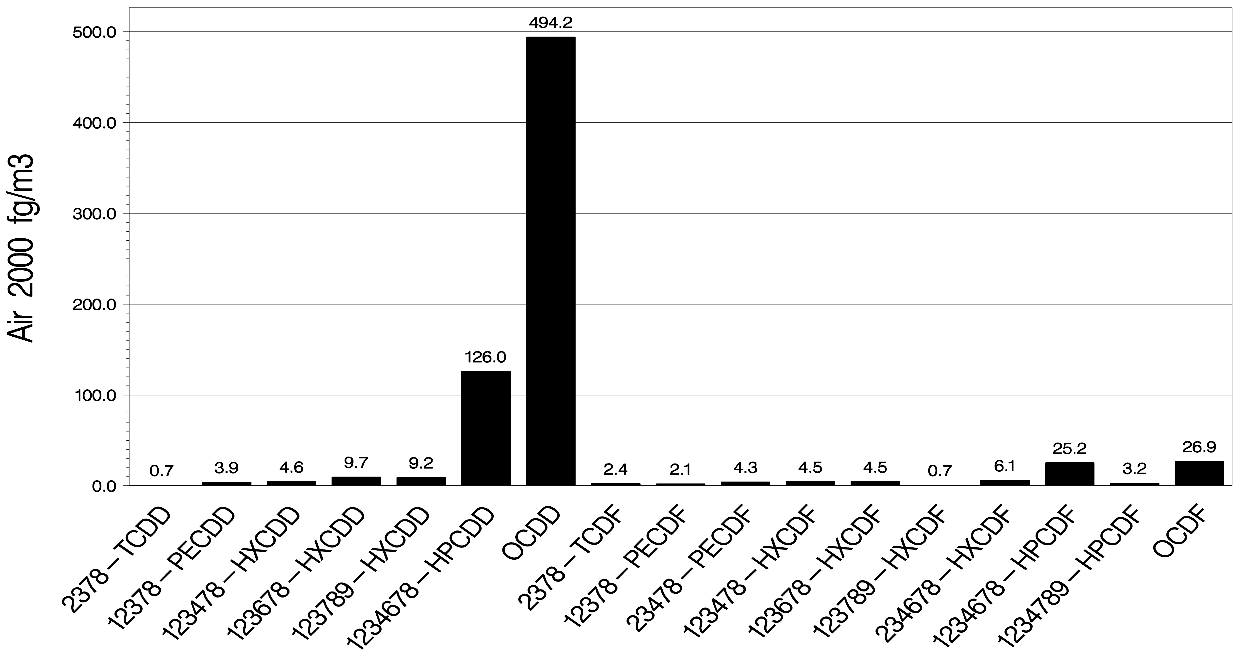


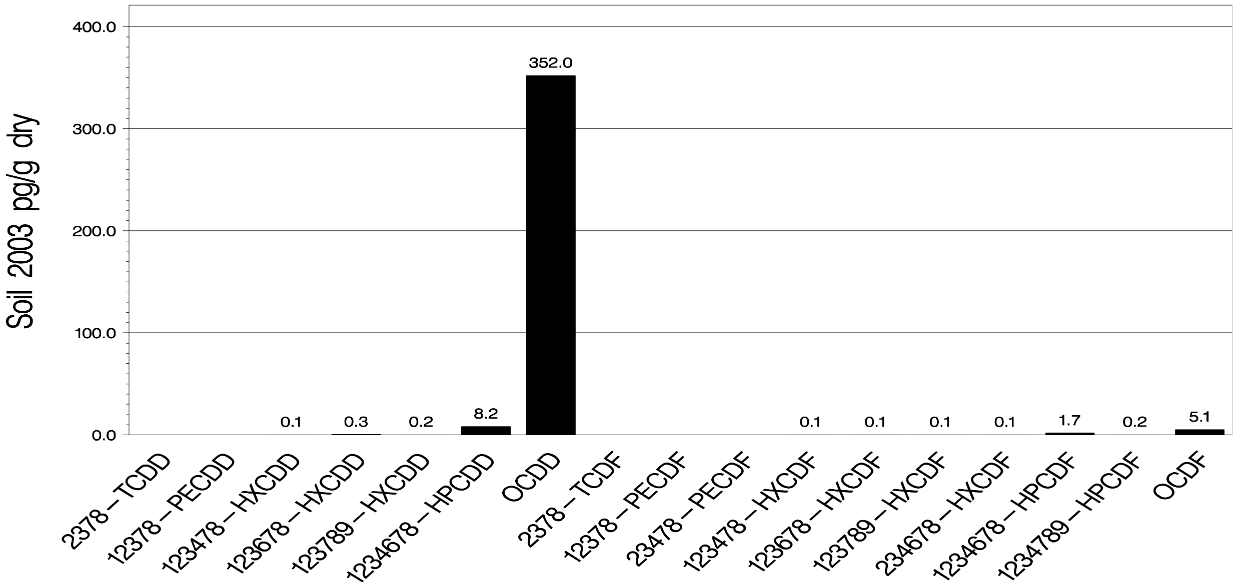
Site 16
Dixon Springs, IL



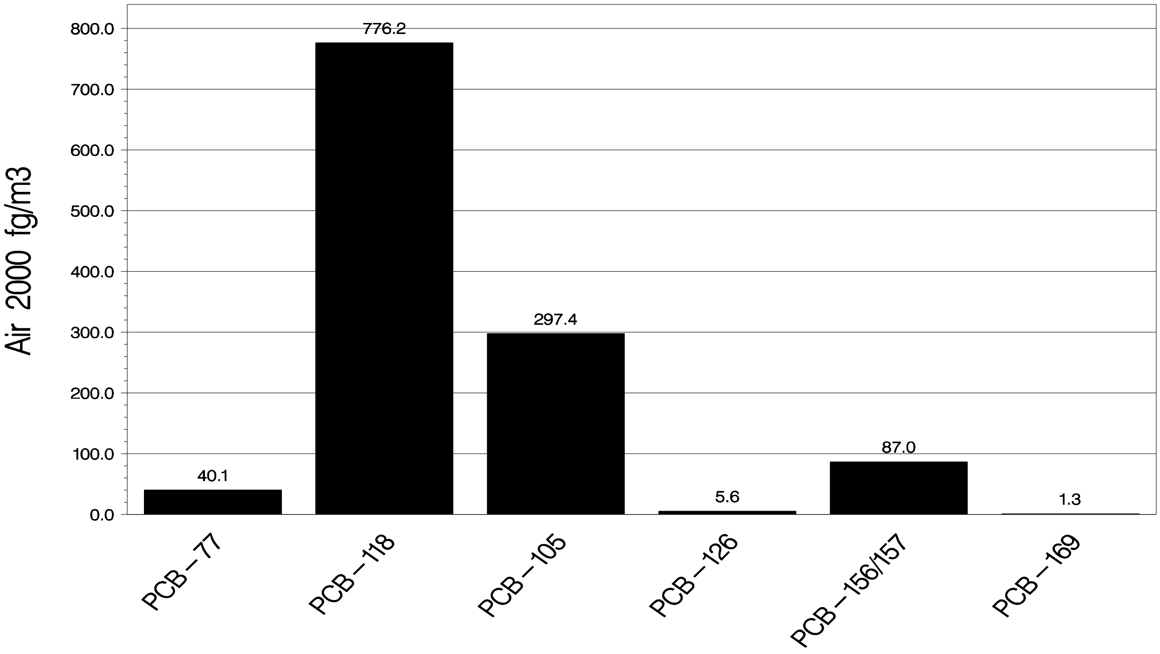


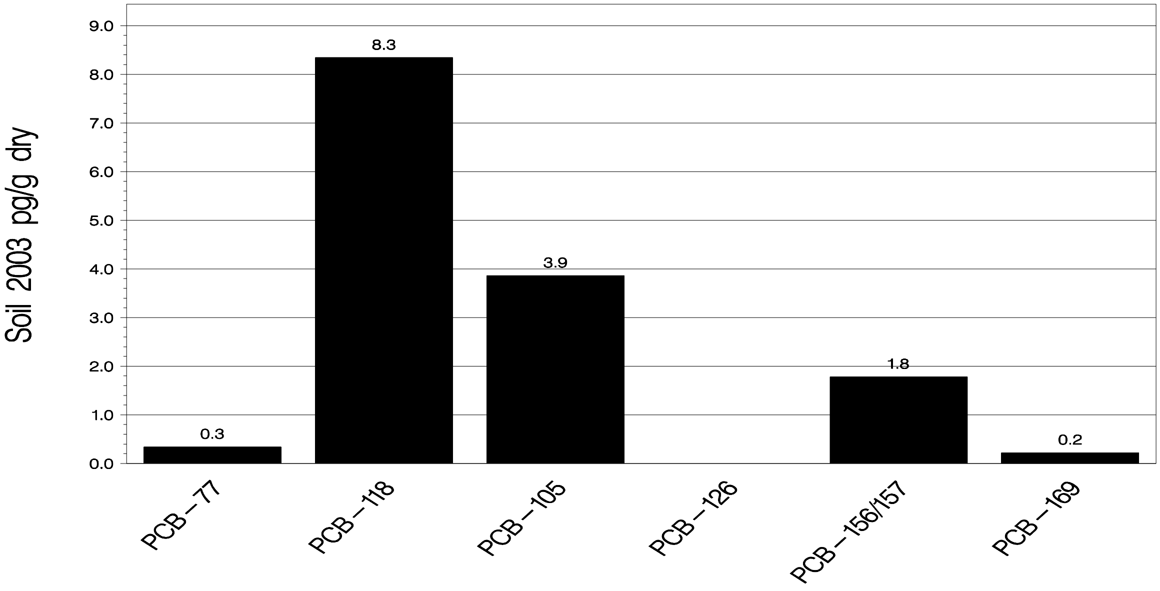
Site 17
Quincy, FL



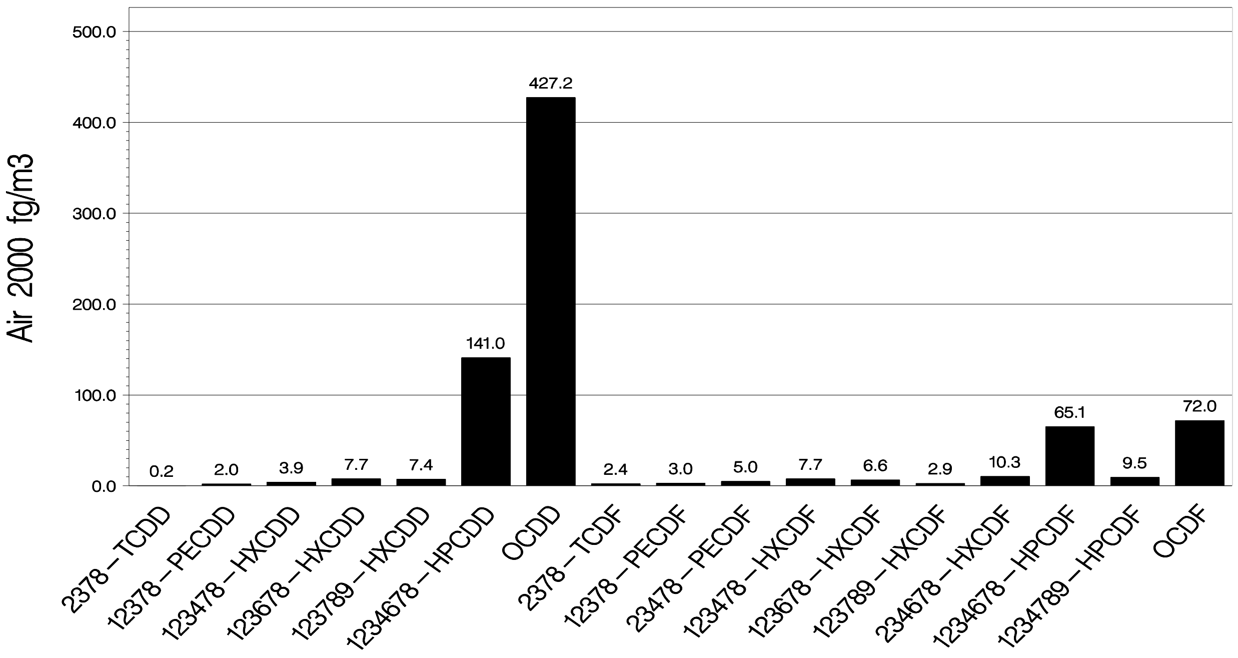


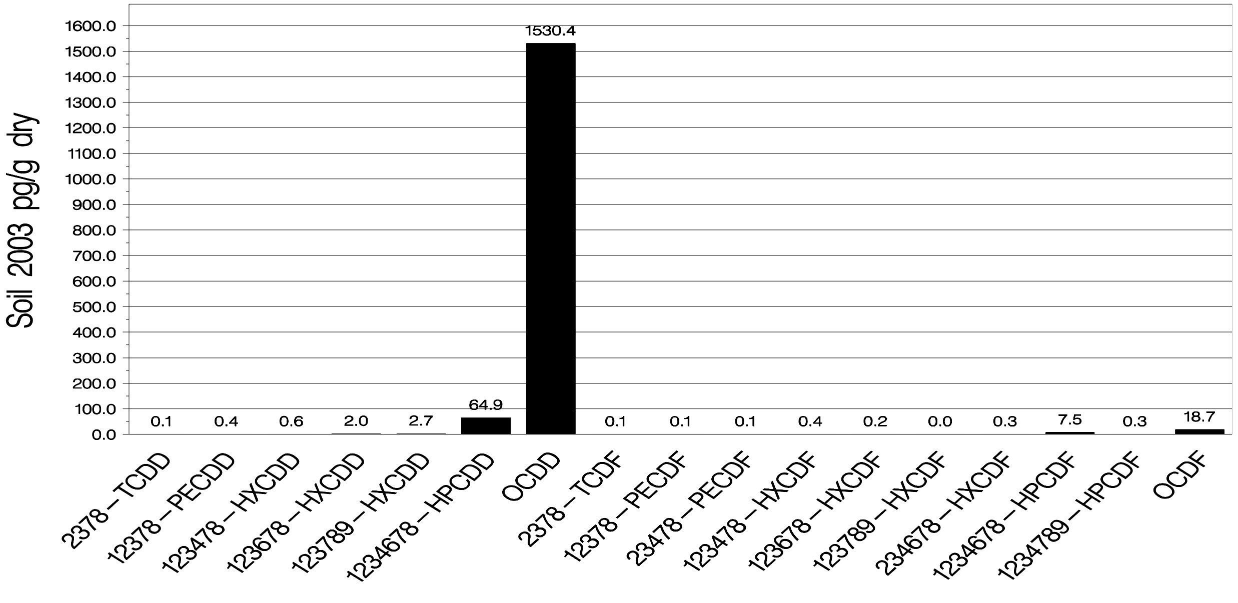
Site 17
Quincy, FL



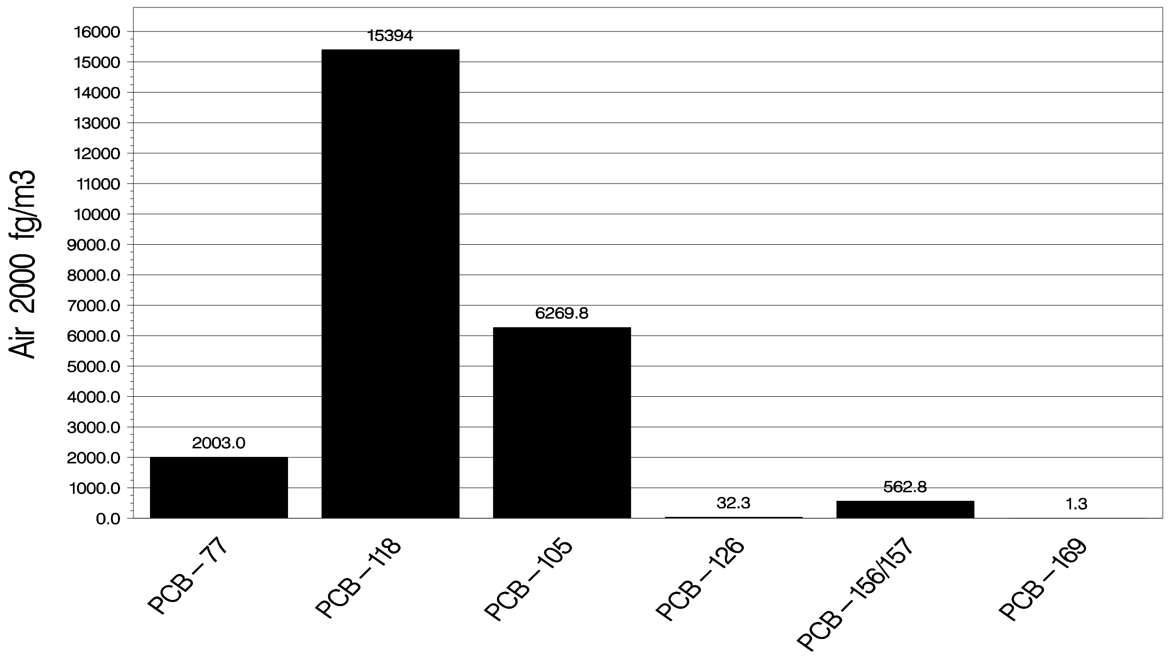


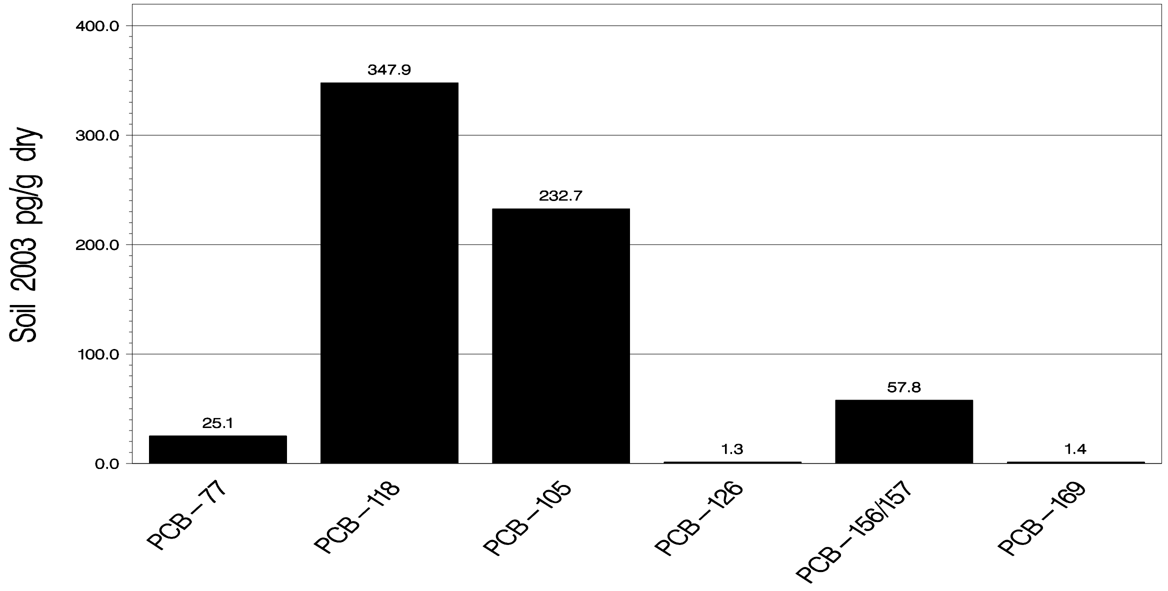
Site 18
Bay St. Louis, MS



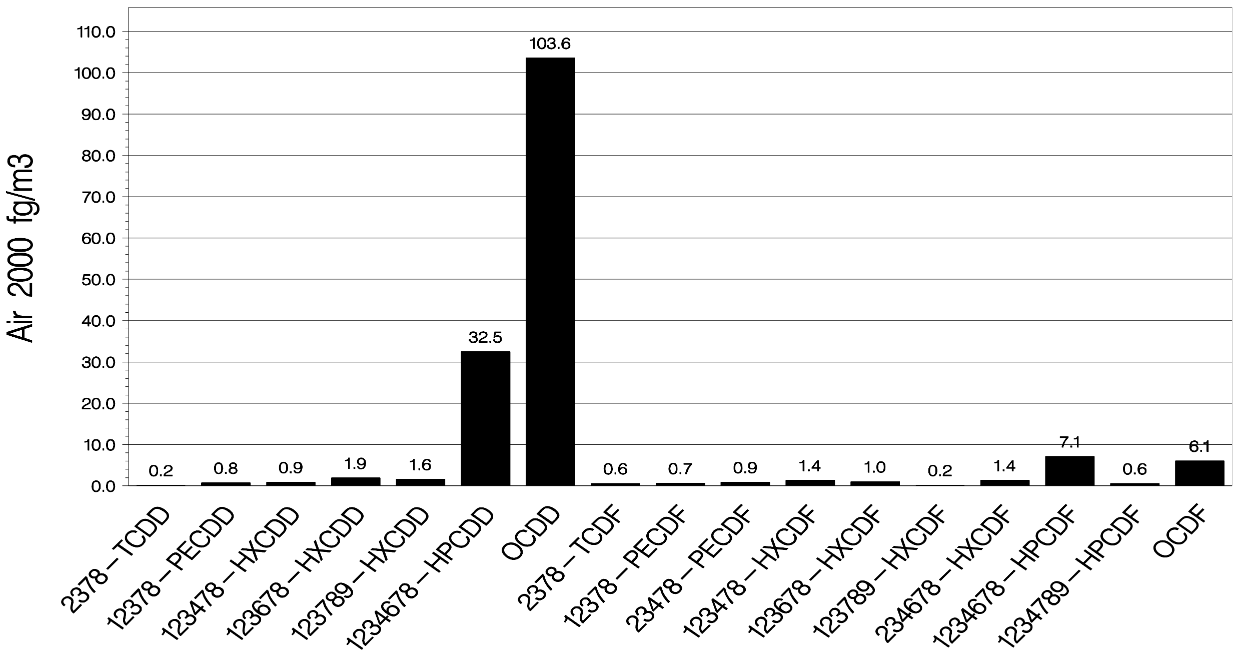


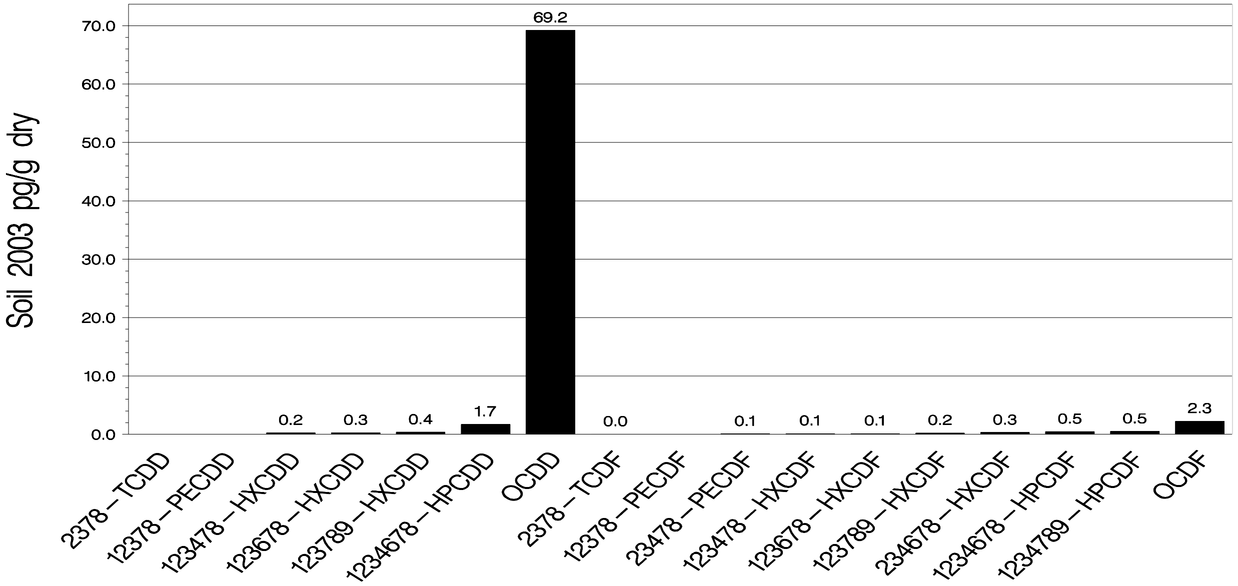
Site 18
Bay St. Louis, MS



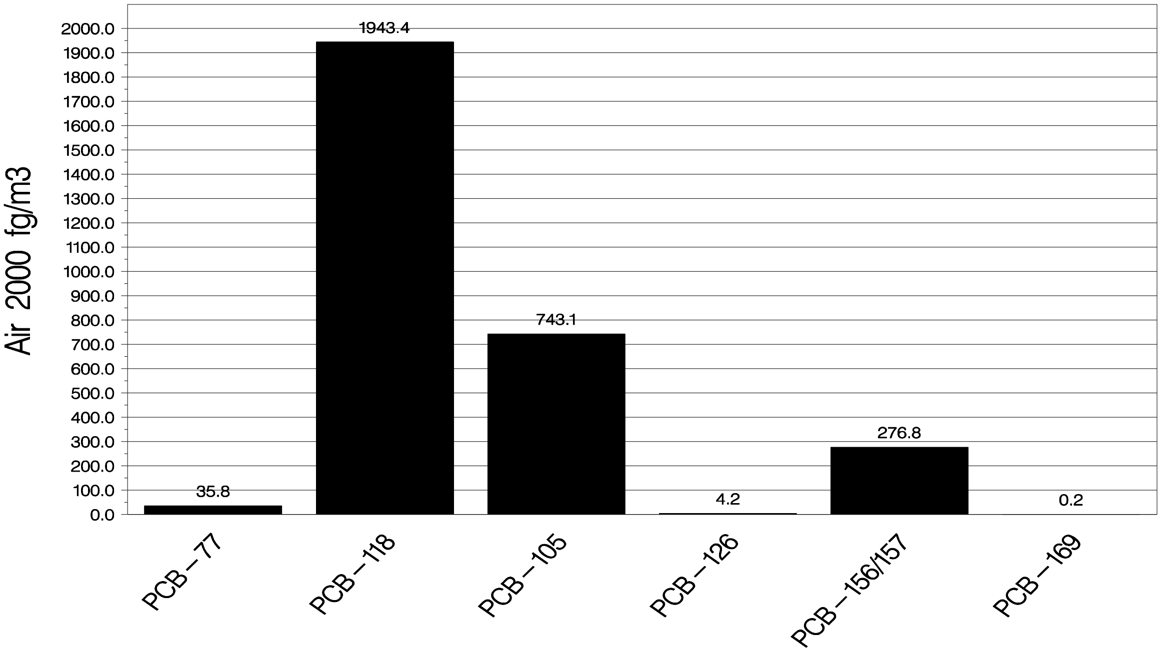


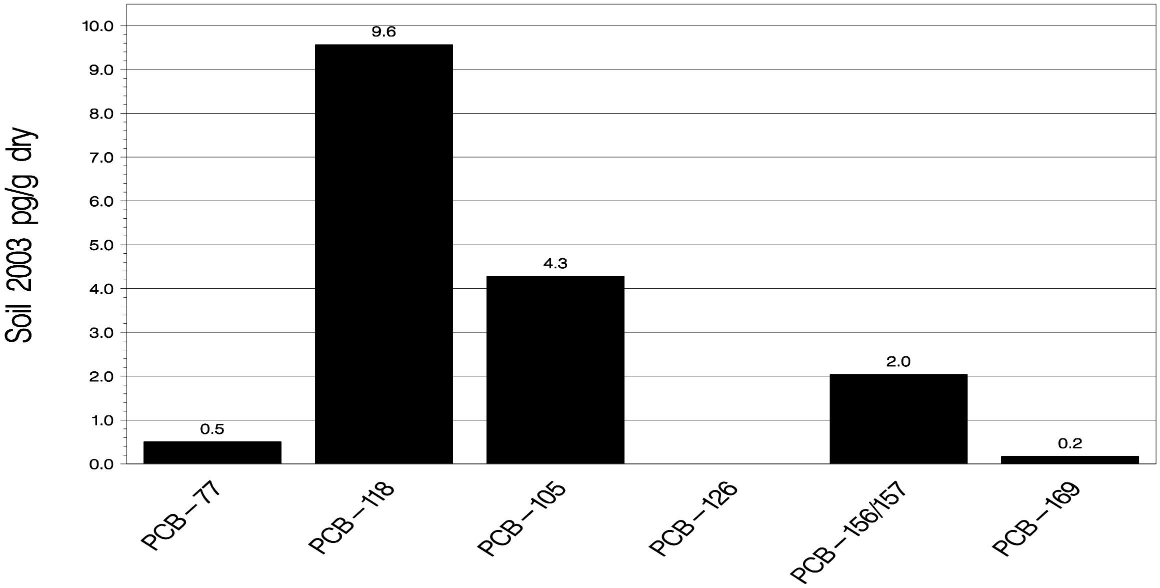
Site 19
Padre Island, TX



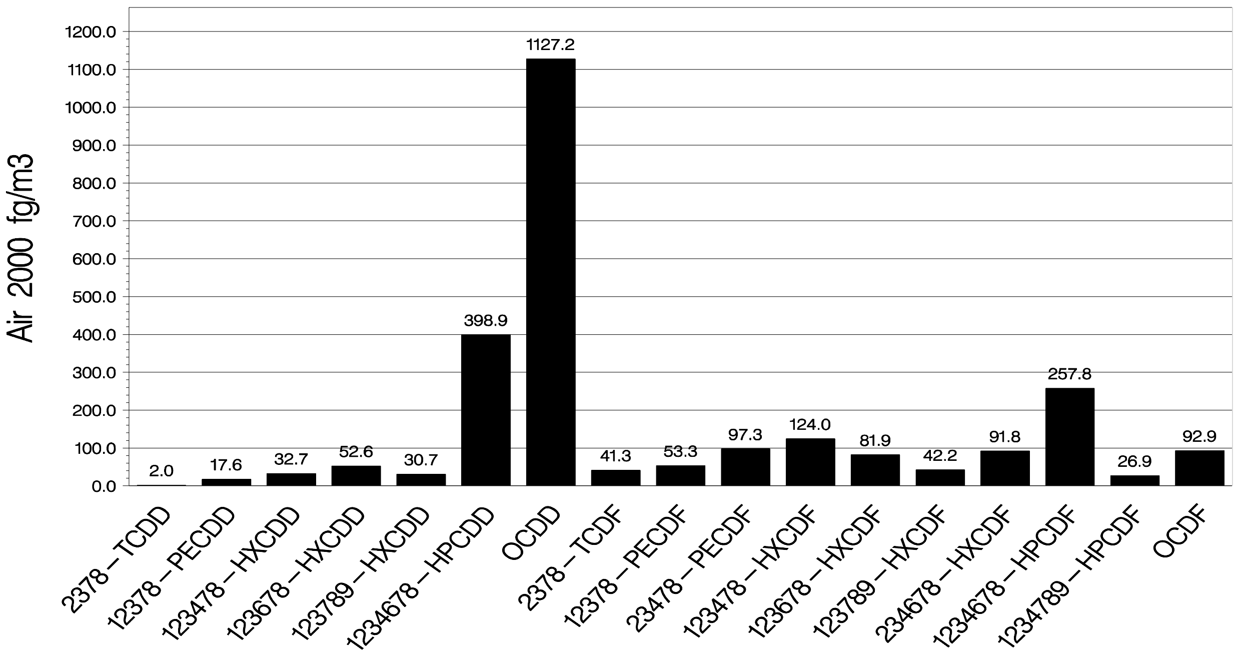


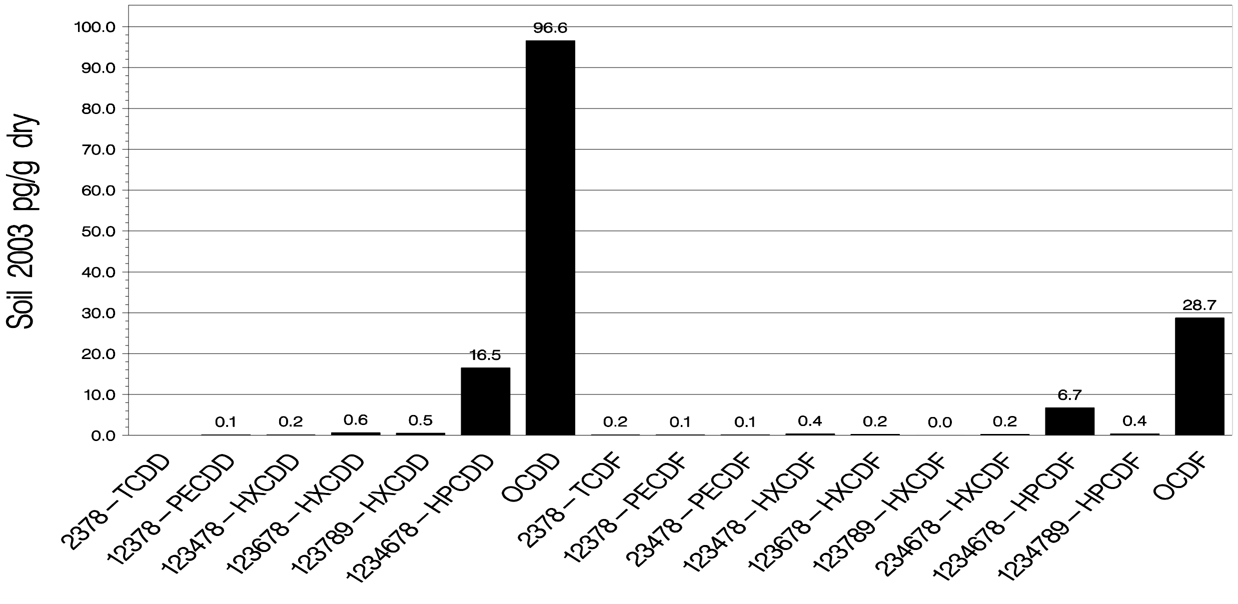
Site 19
Padre Island, TX



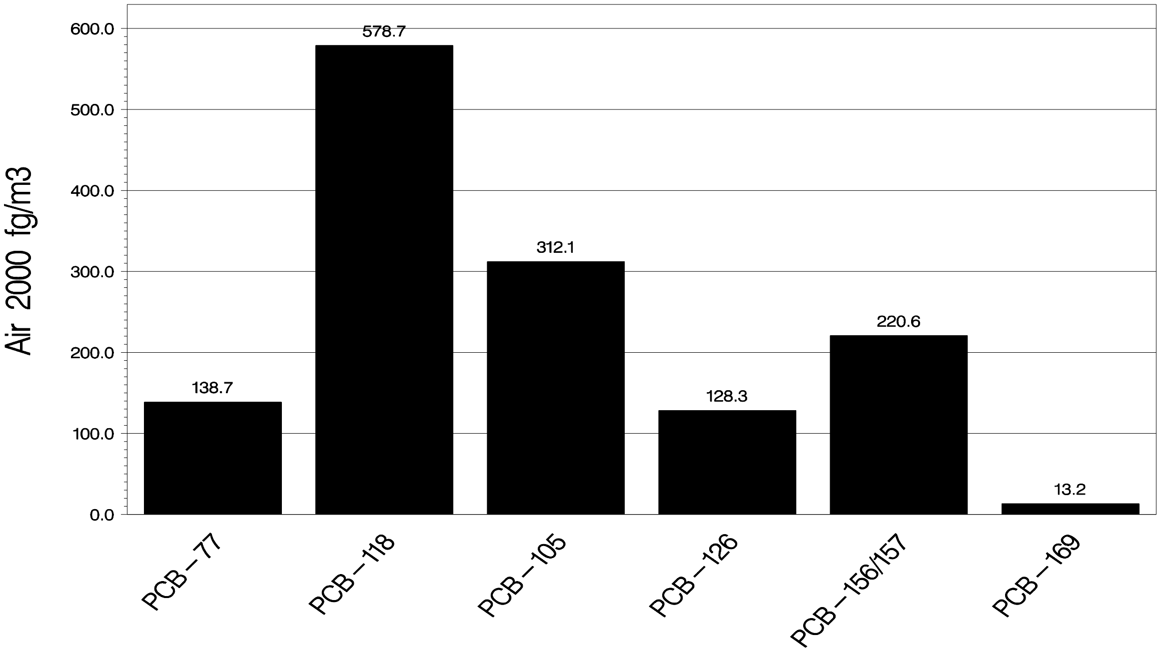


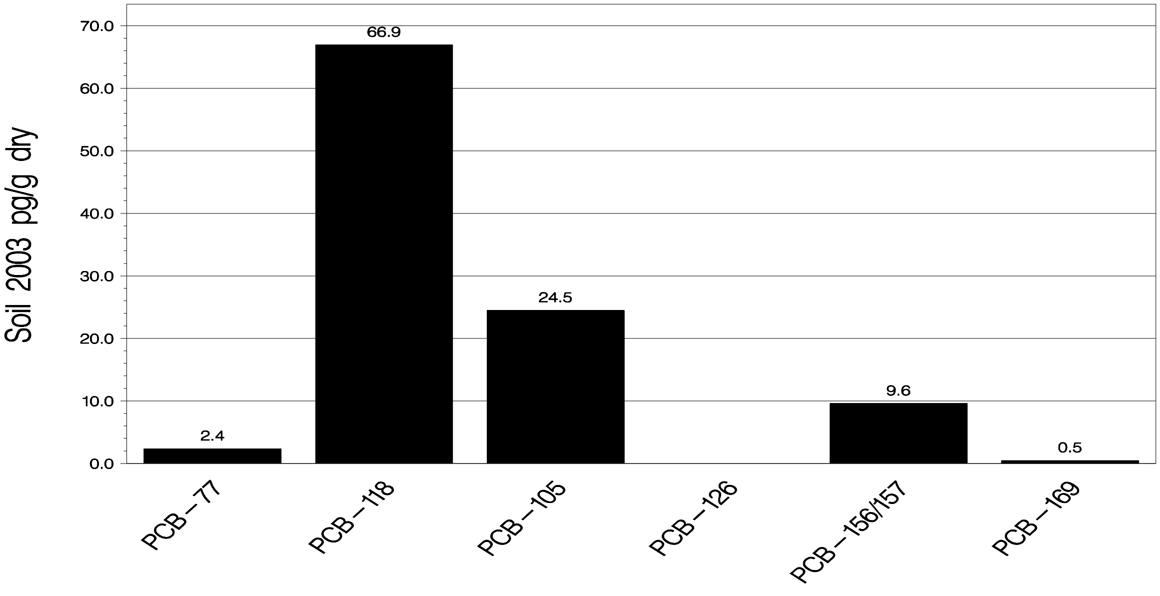
Site 20
Fond Du Lac, MN



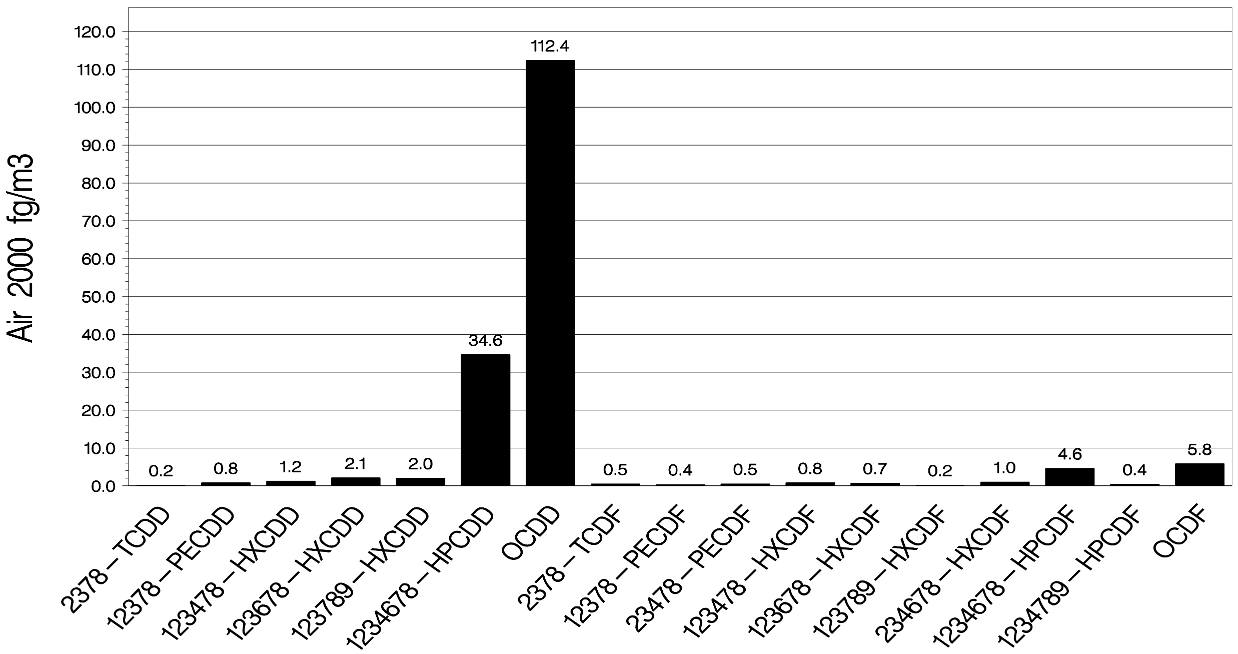


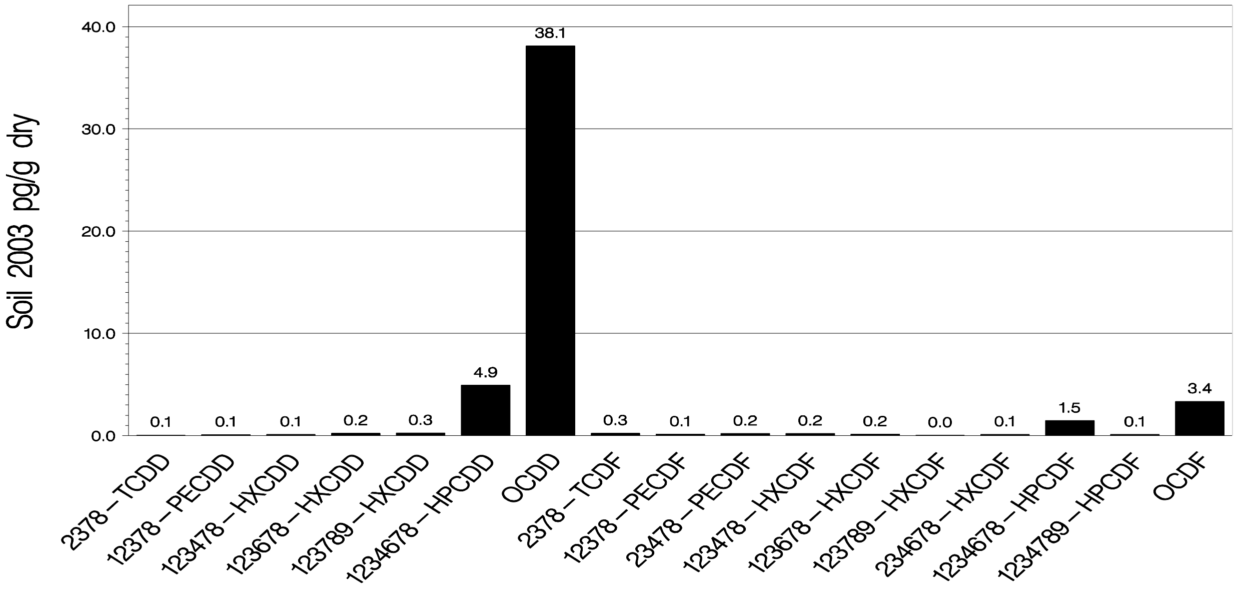
Site 20
Fond Du Lac, MN



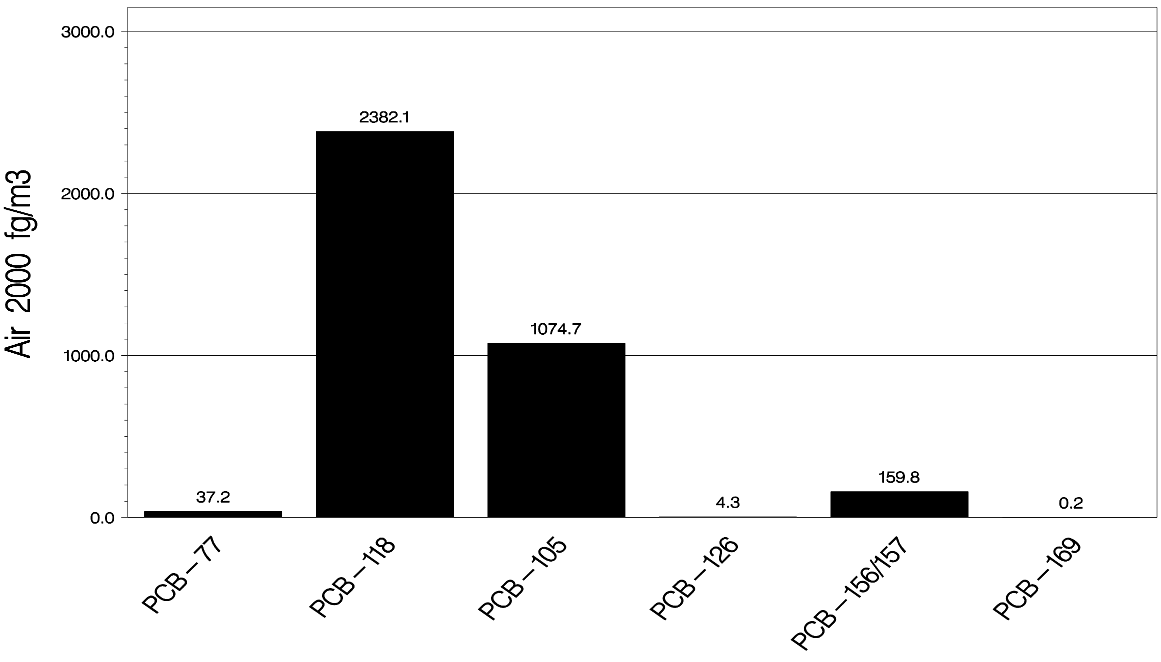


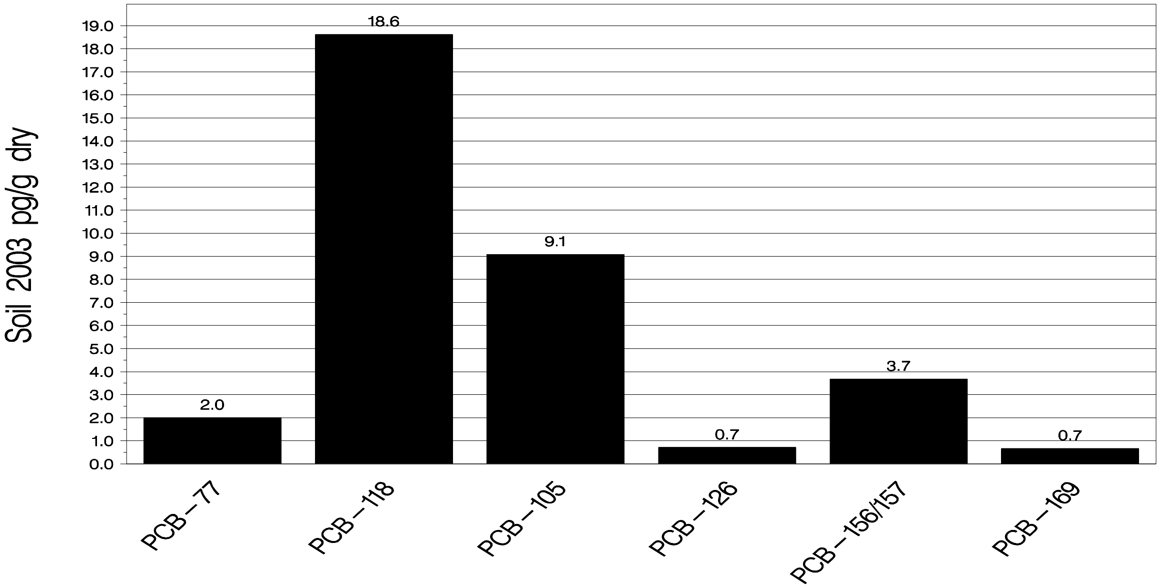
Site 21
North Platte, NE



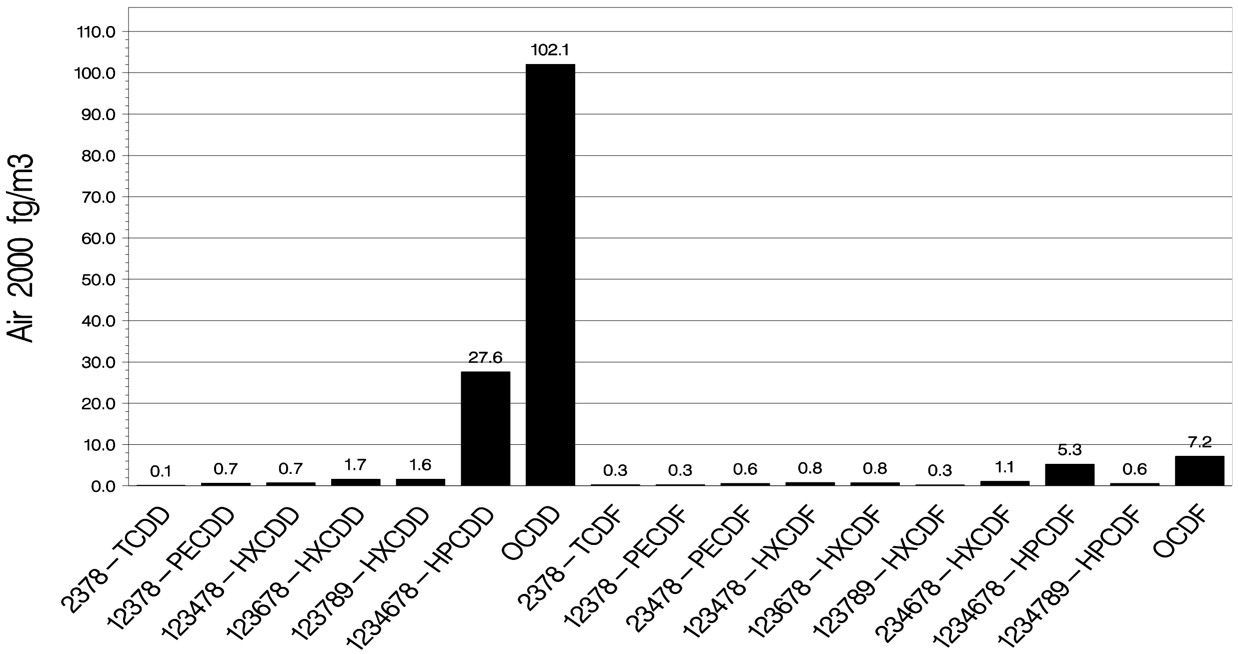


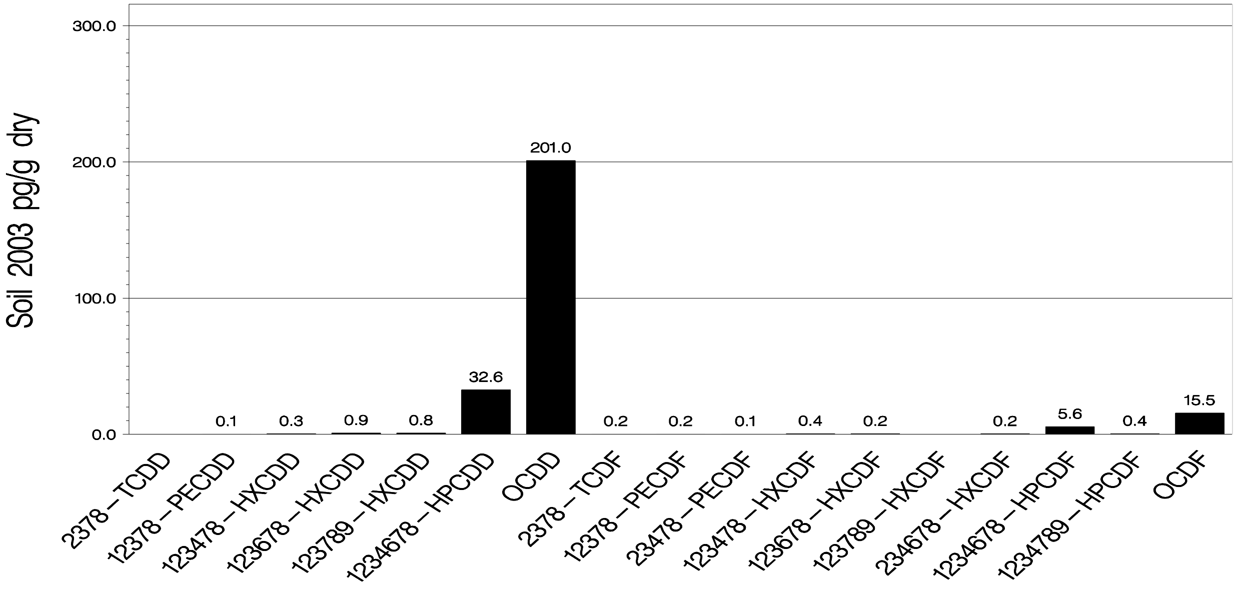
Site 21
North Platte, NE



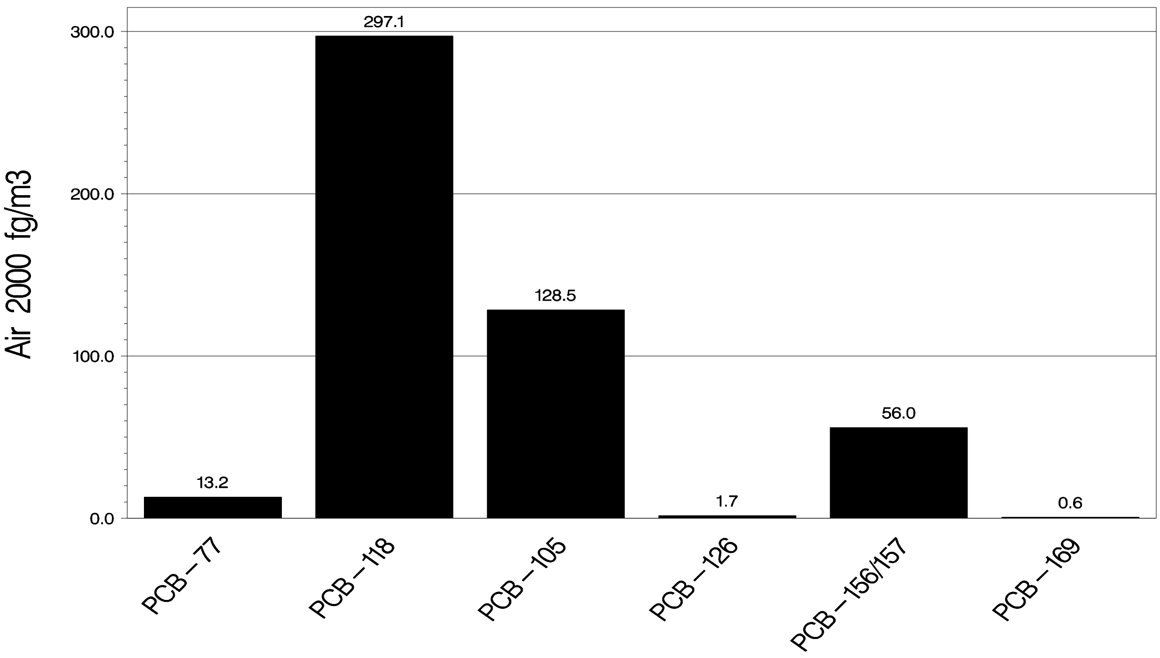


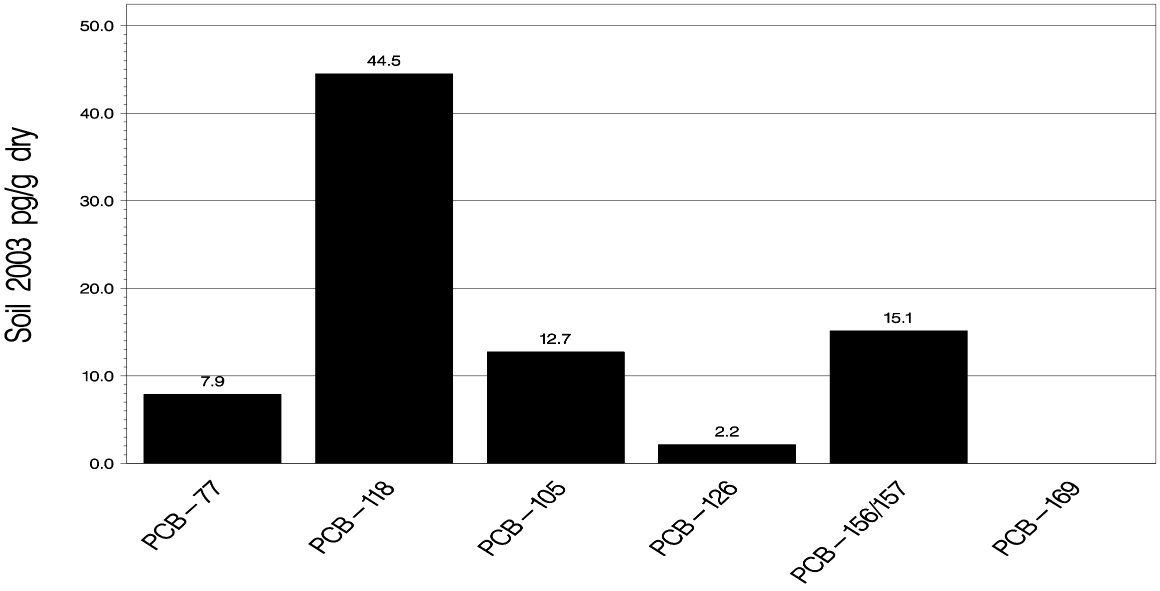
Site 22
Goodwell, OK



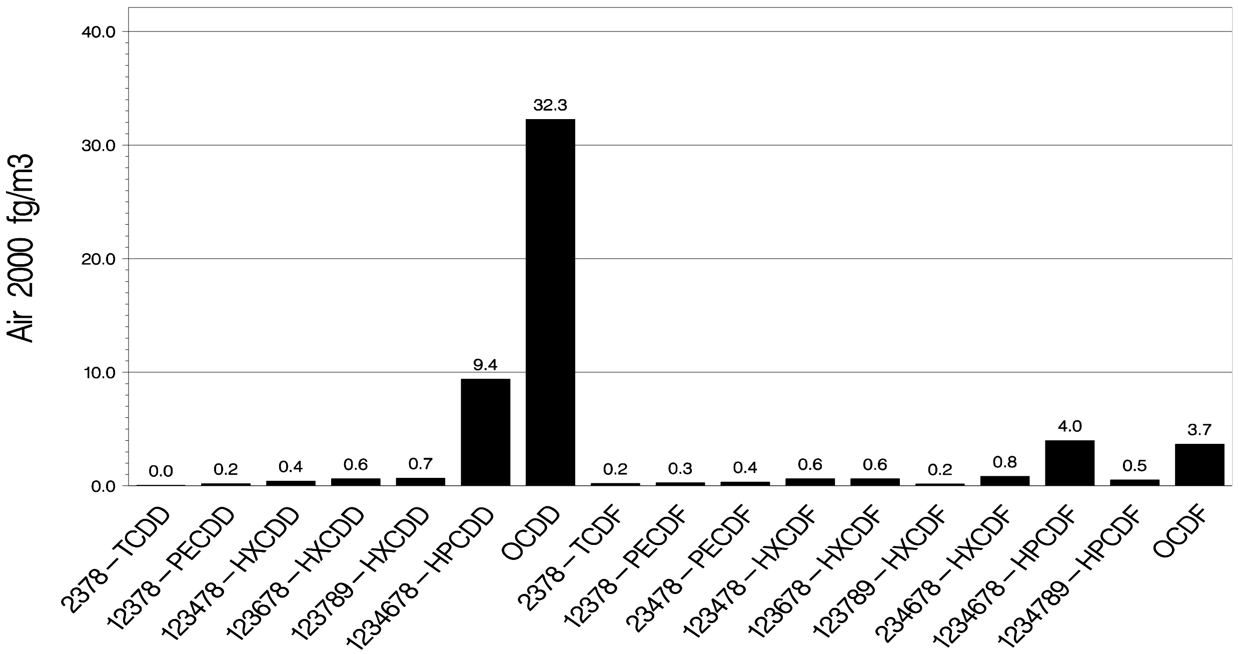


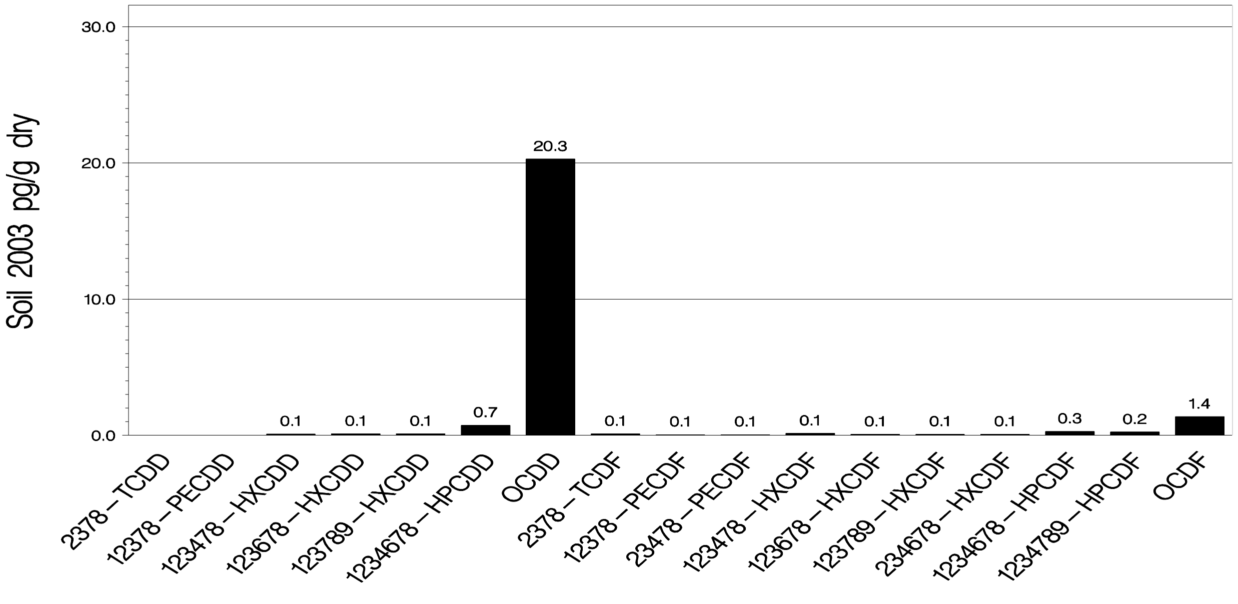
Site 22
Goodwell, OK



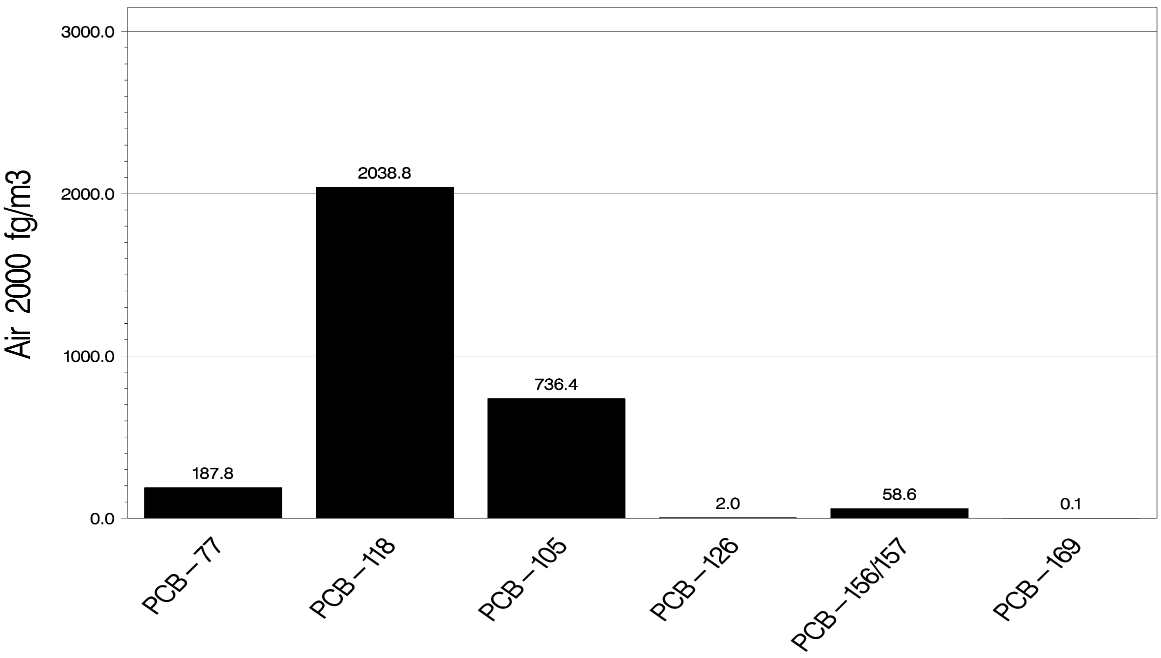


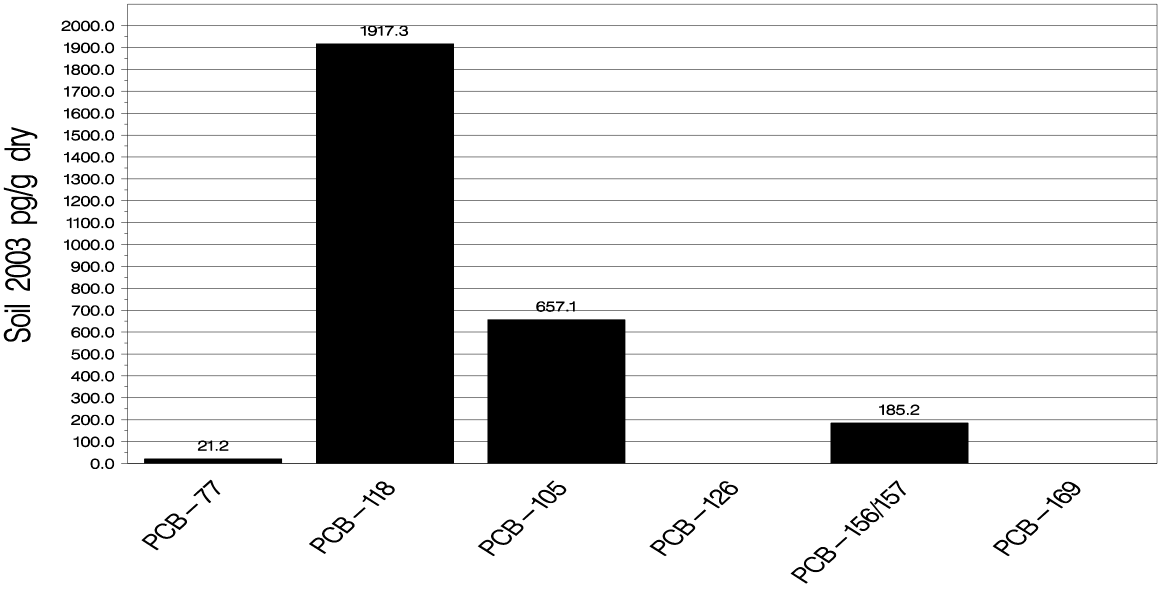
Site 23
Big Bend, TX



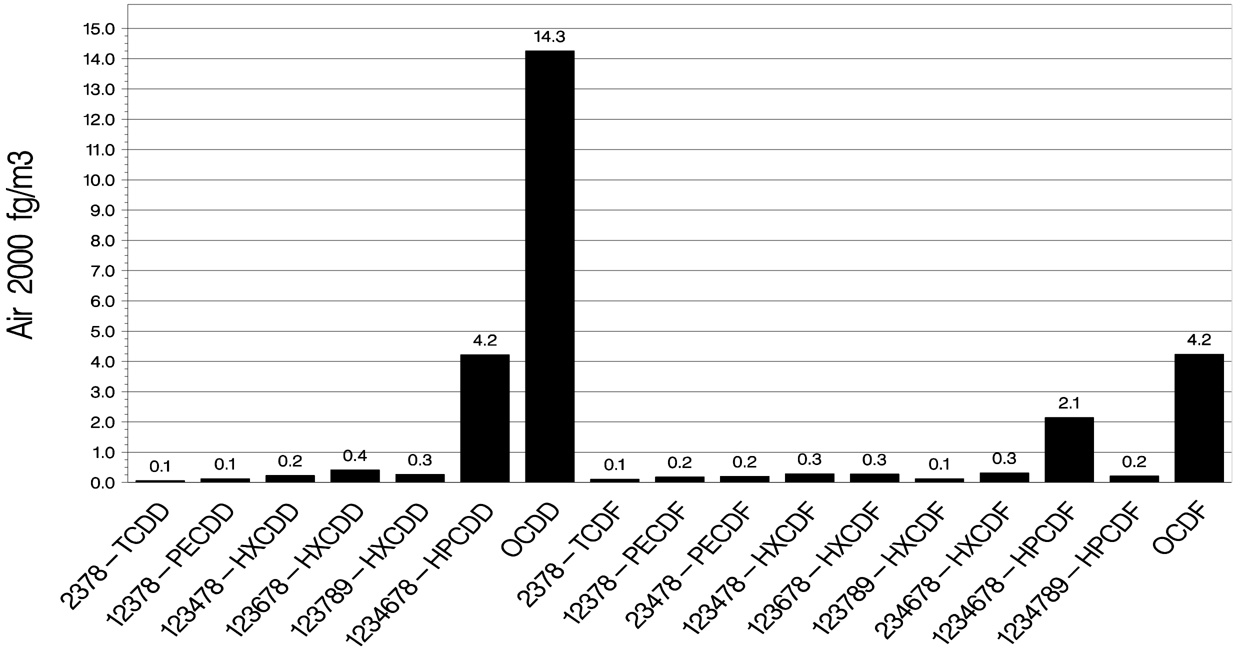


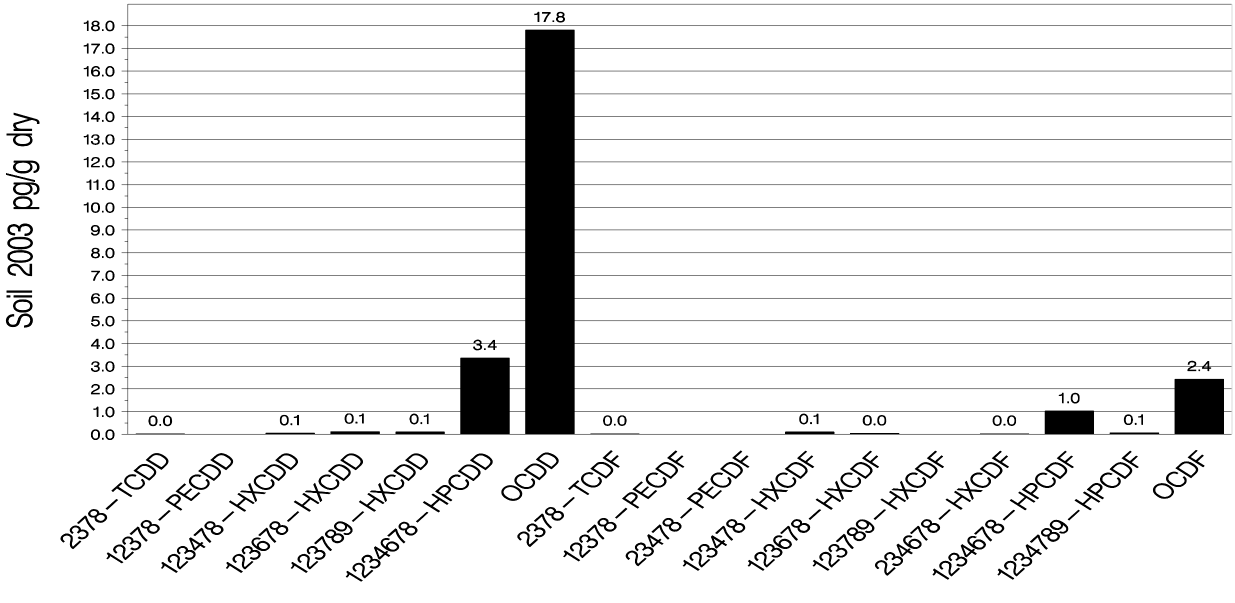
Site 23
Big Bend, TX



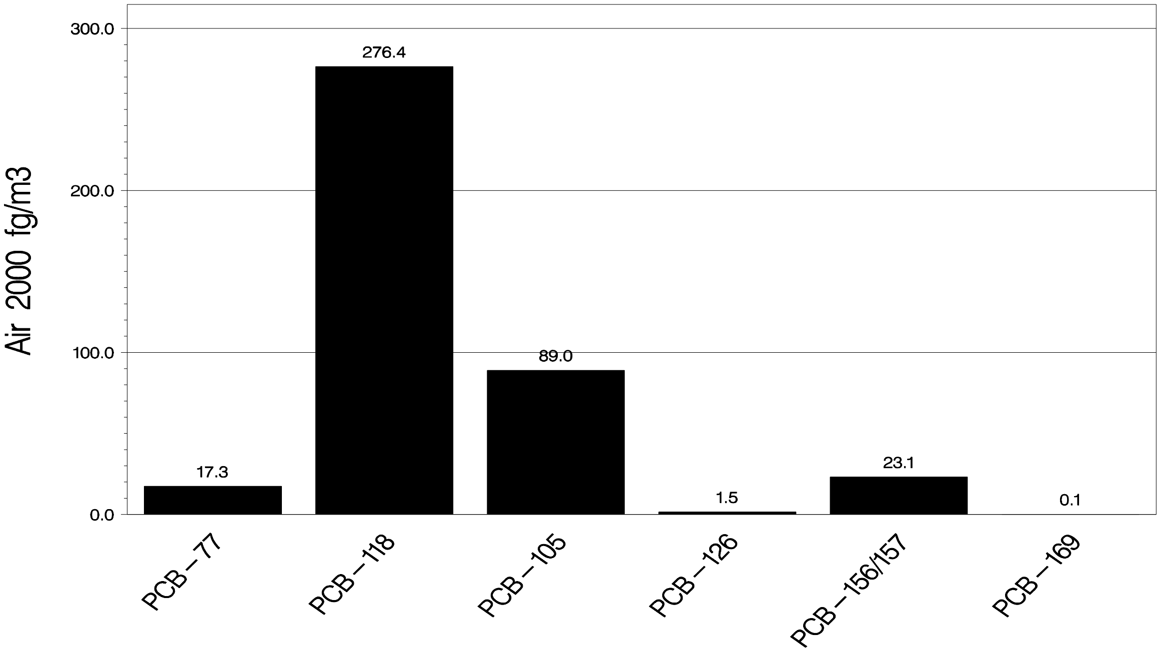


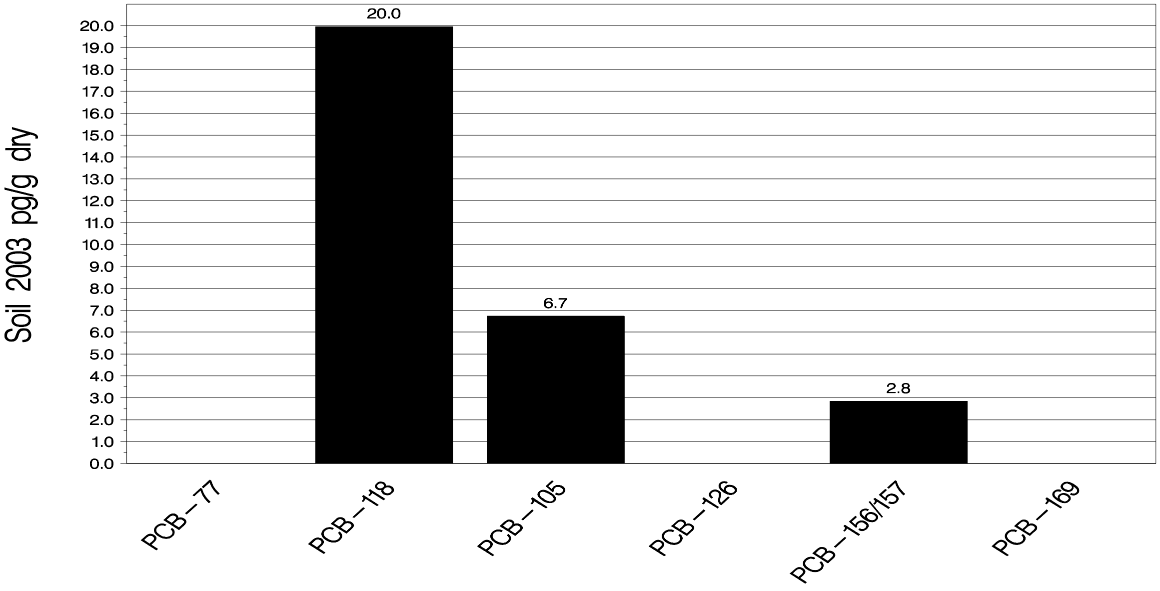
Site 24
Grand Canyon, AZ



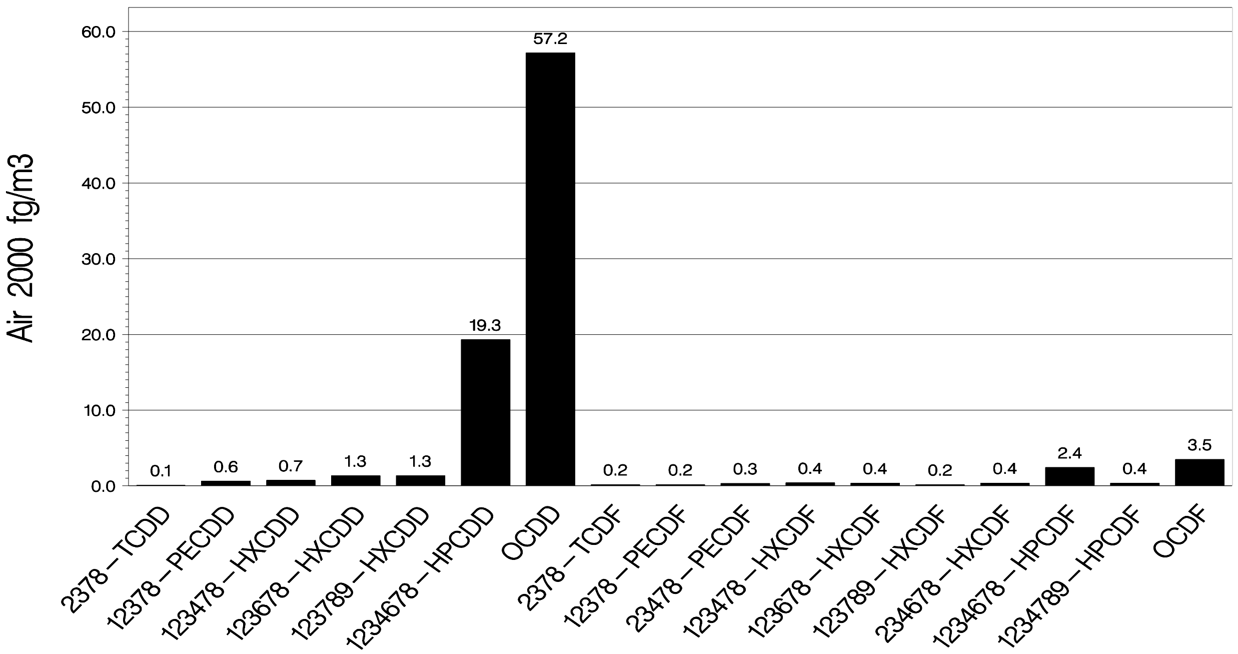


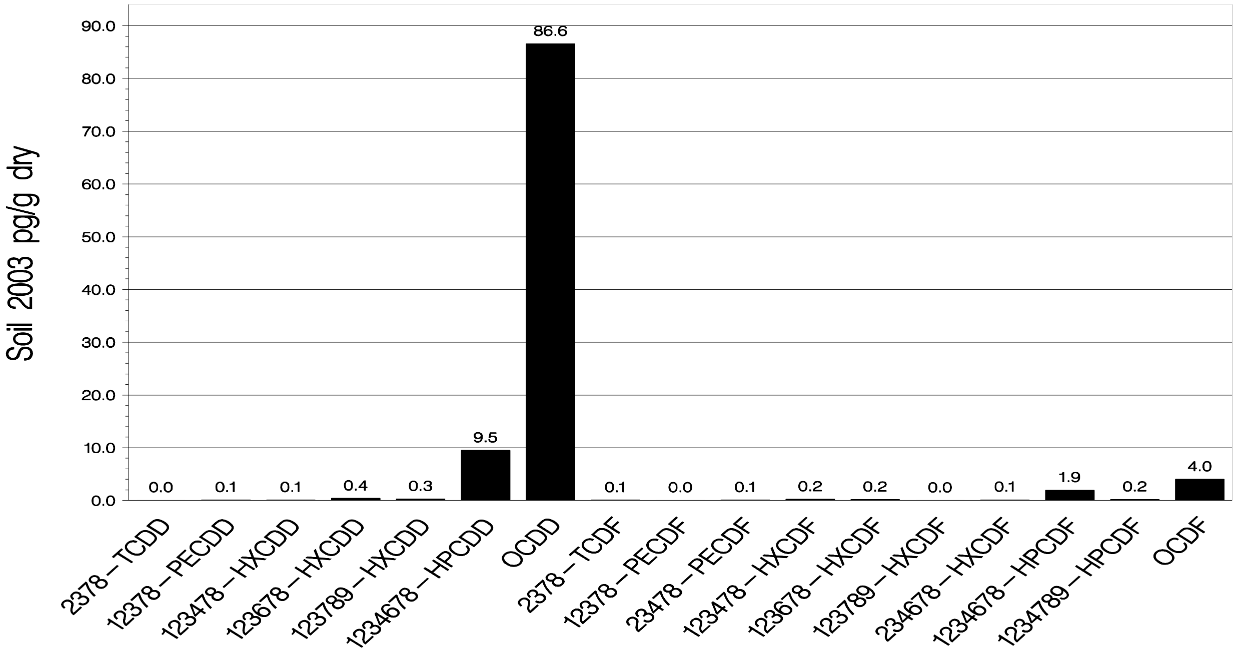
Site 24
Grand Canyon, AZ



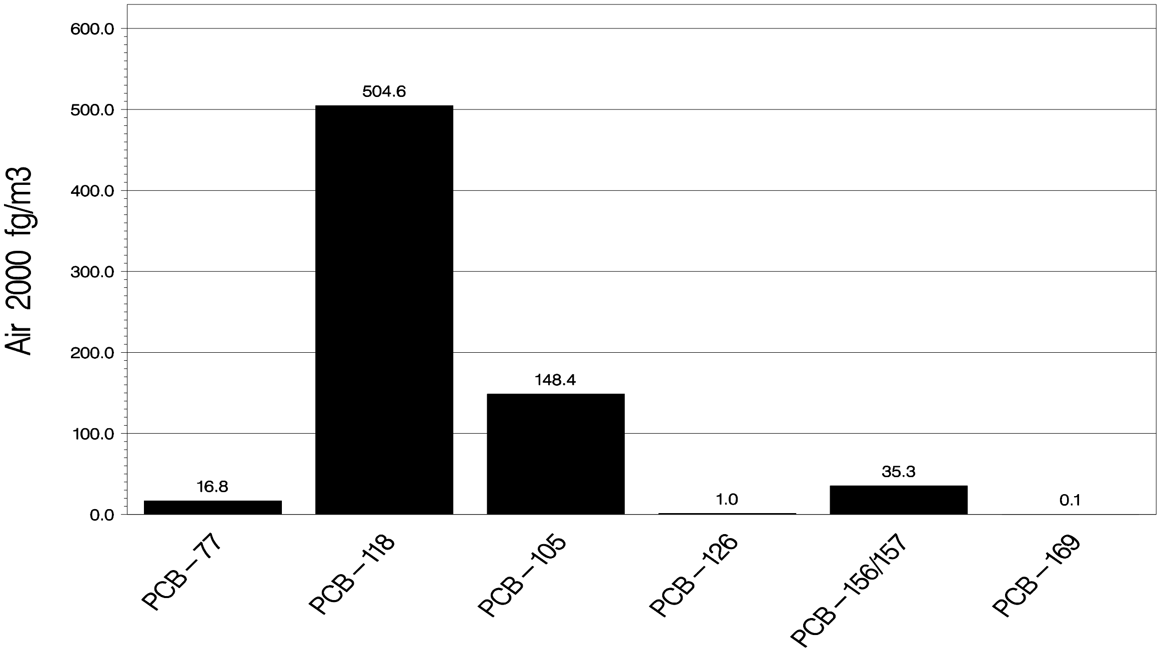


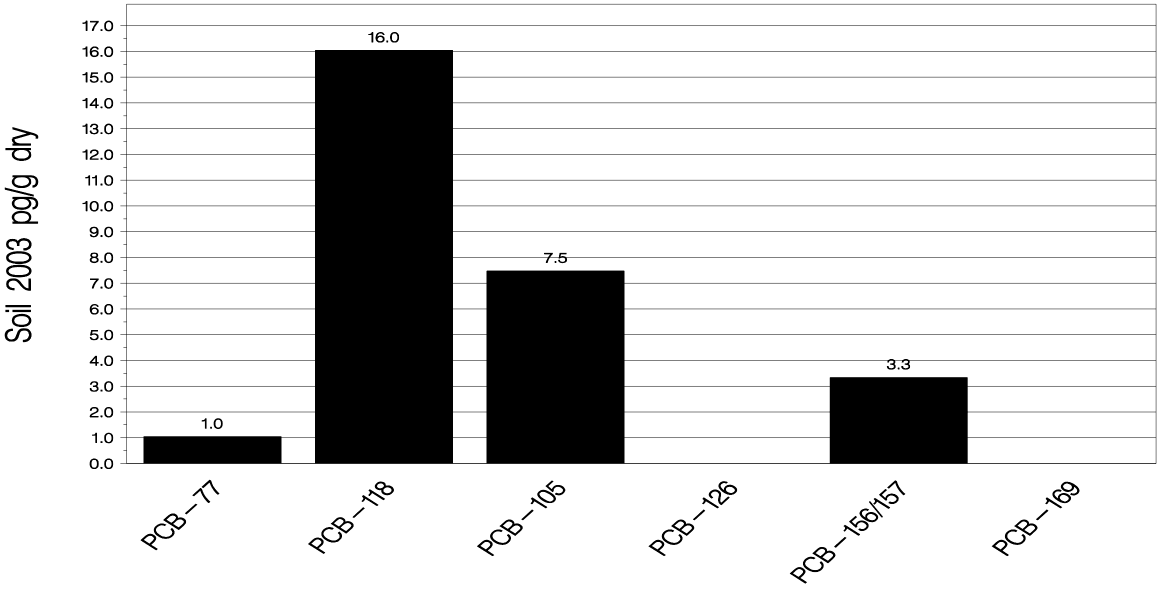
Site 25
Theodore Roosevelt, ND



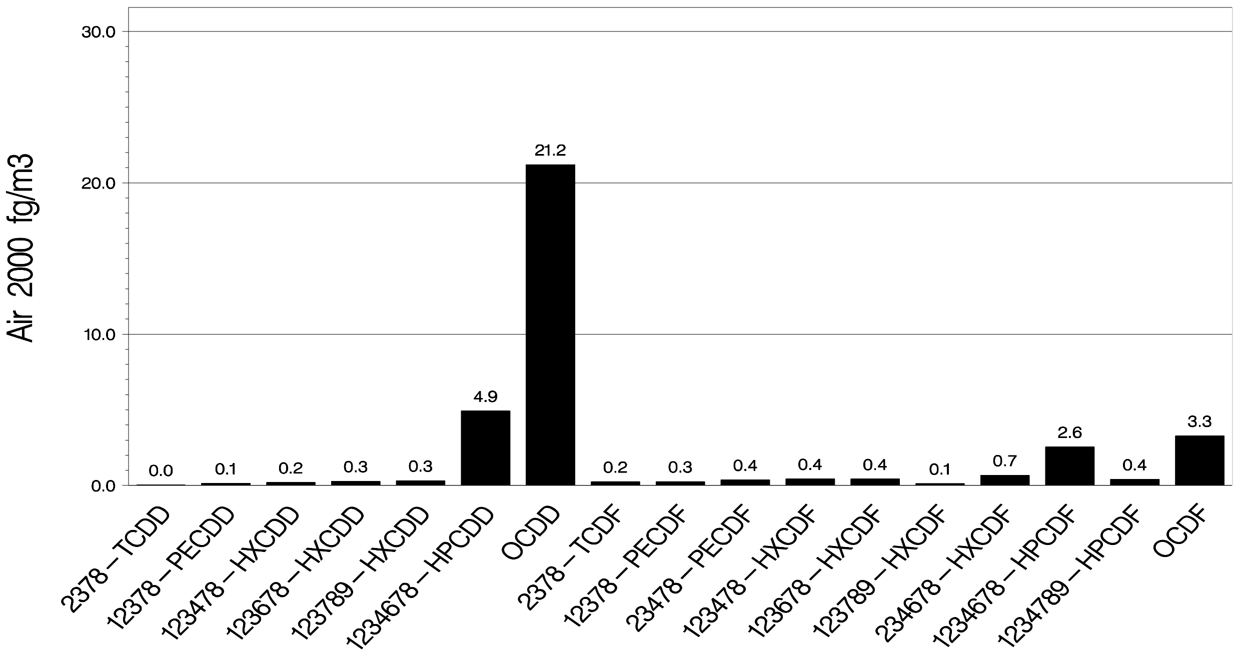


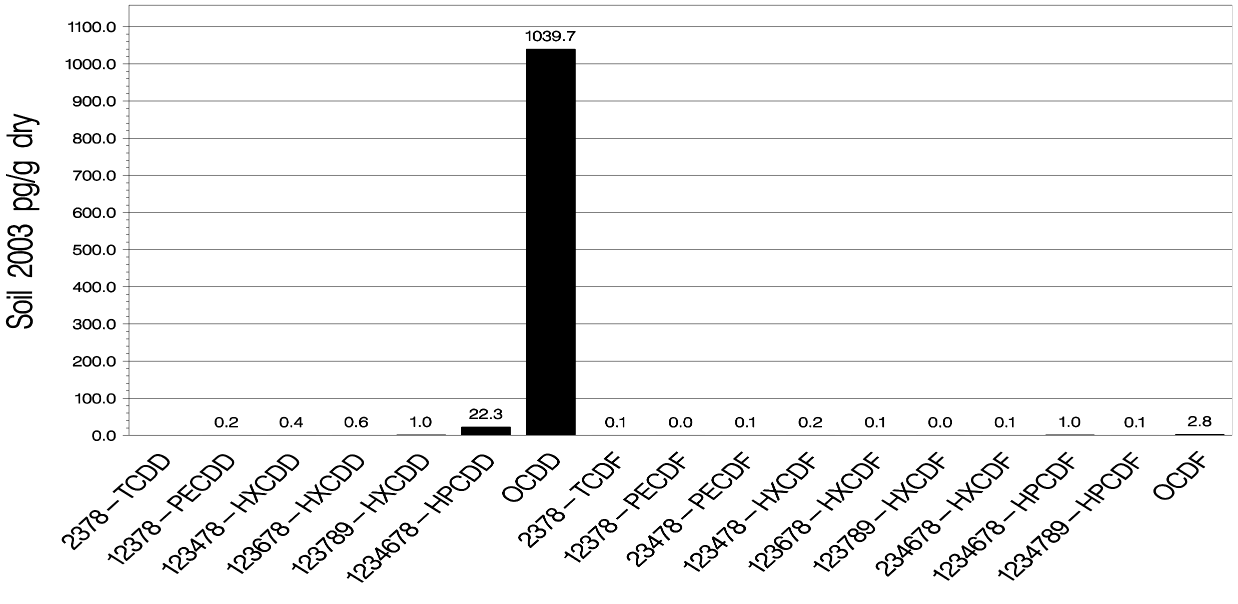
Site 25
Theodore Roosevelt, ND



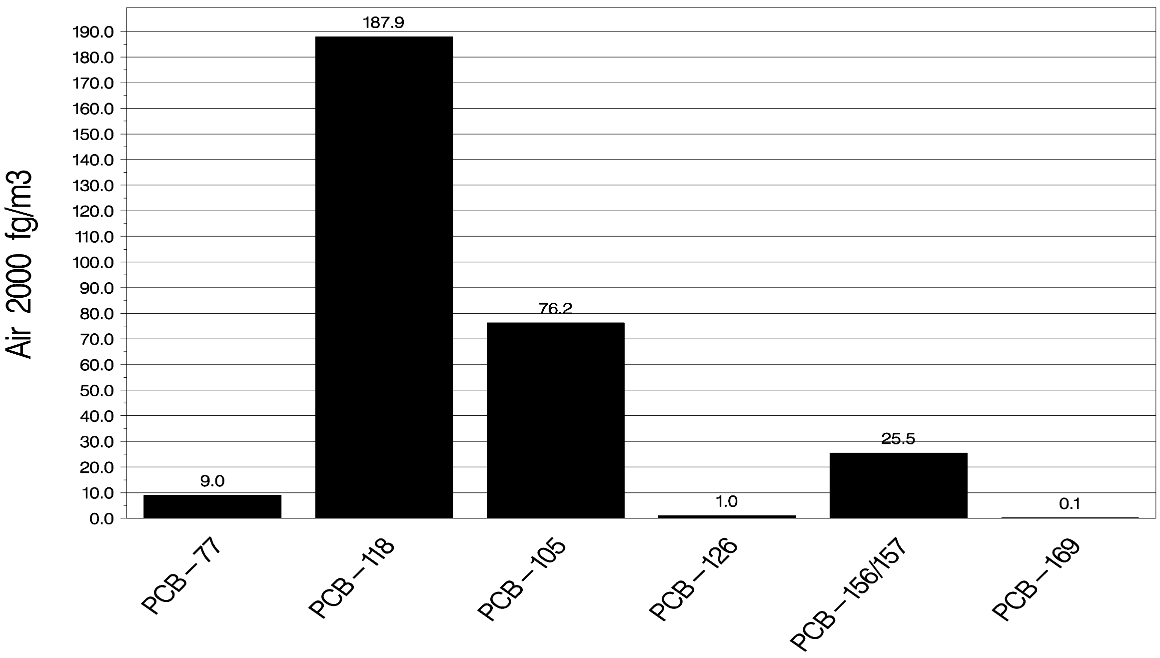


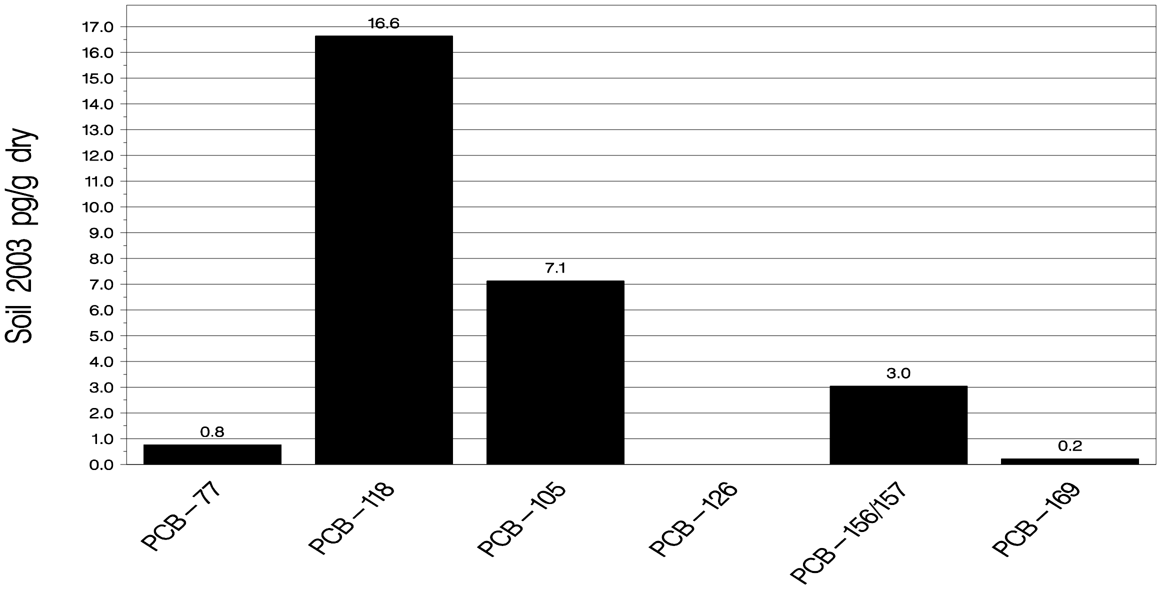
Site 27
Chiricahua, AZ



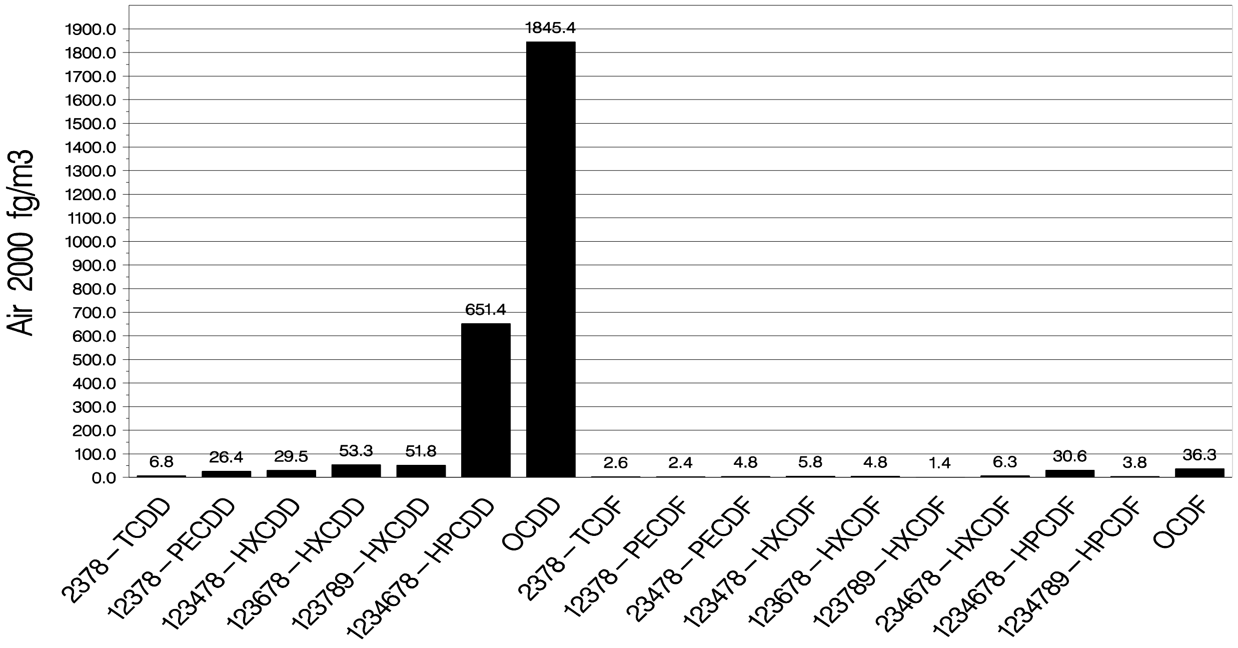


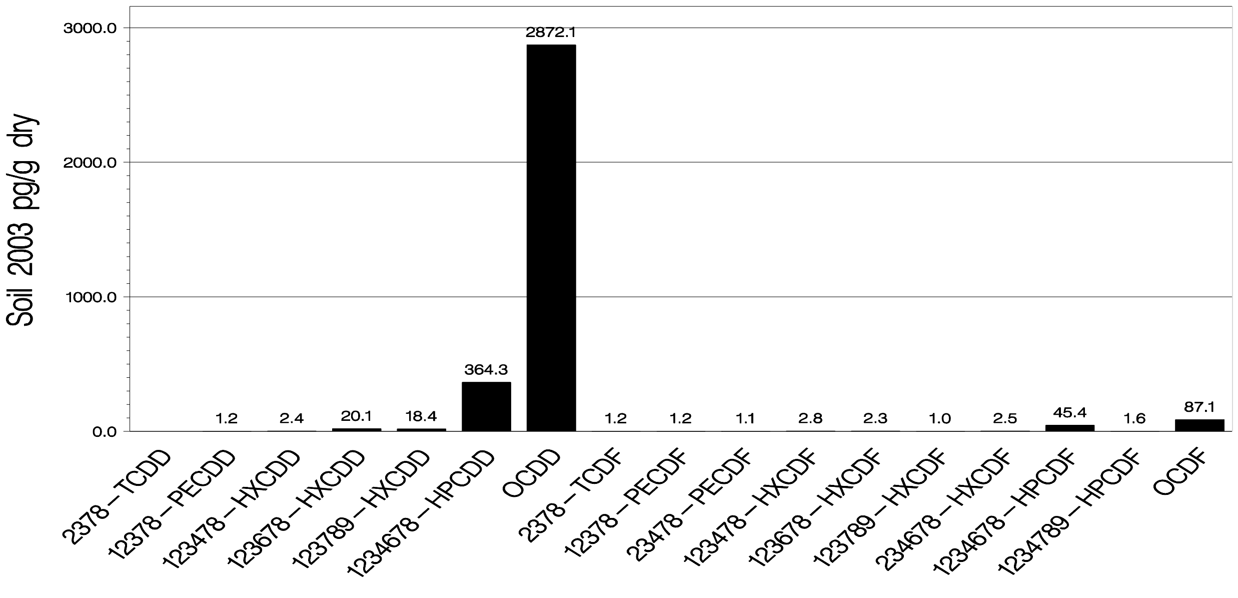
Site 27
Chiricahua, AZ



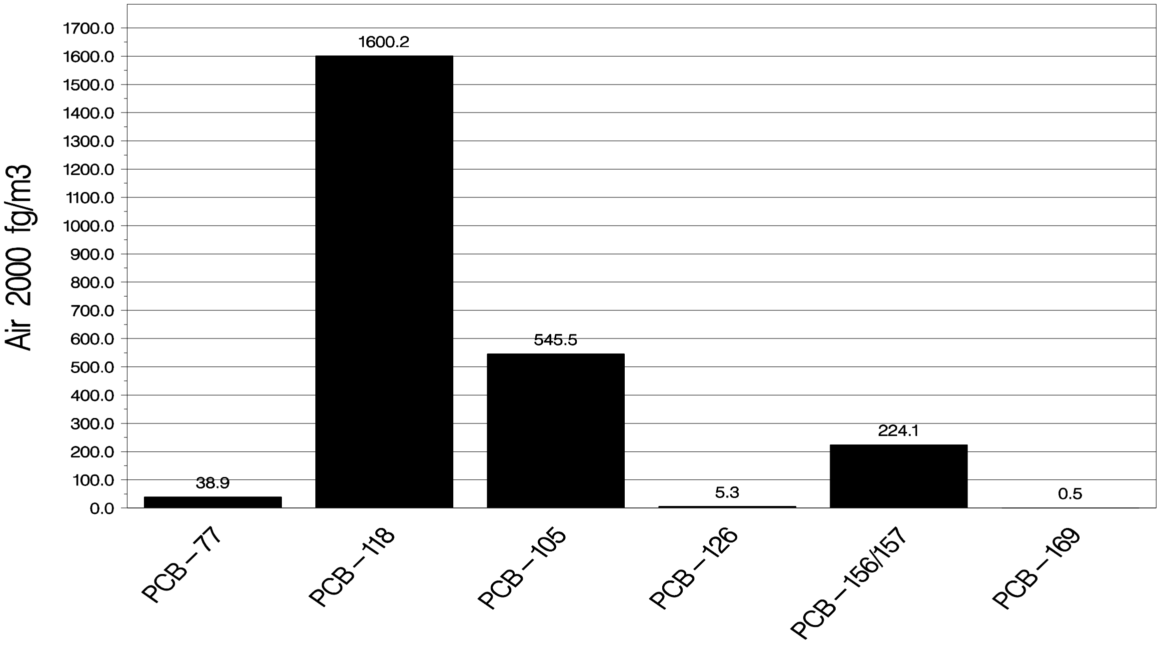


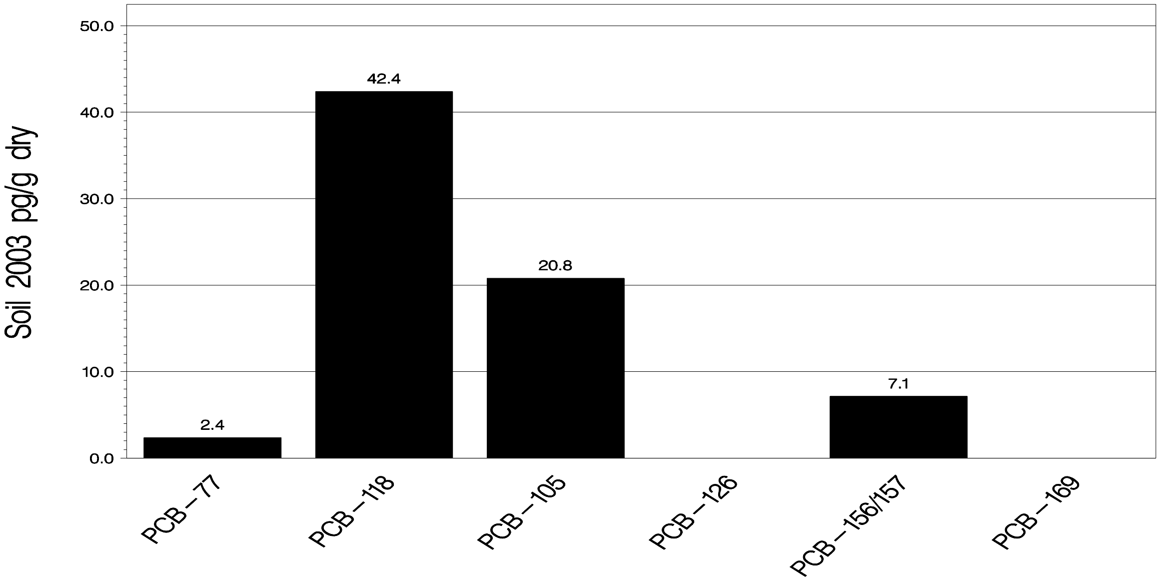
Site 29
Hyslop Farm, OR - 2000 Air
Marvel Ranch, OR - 2003 Soil



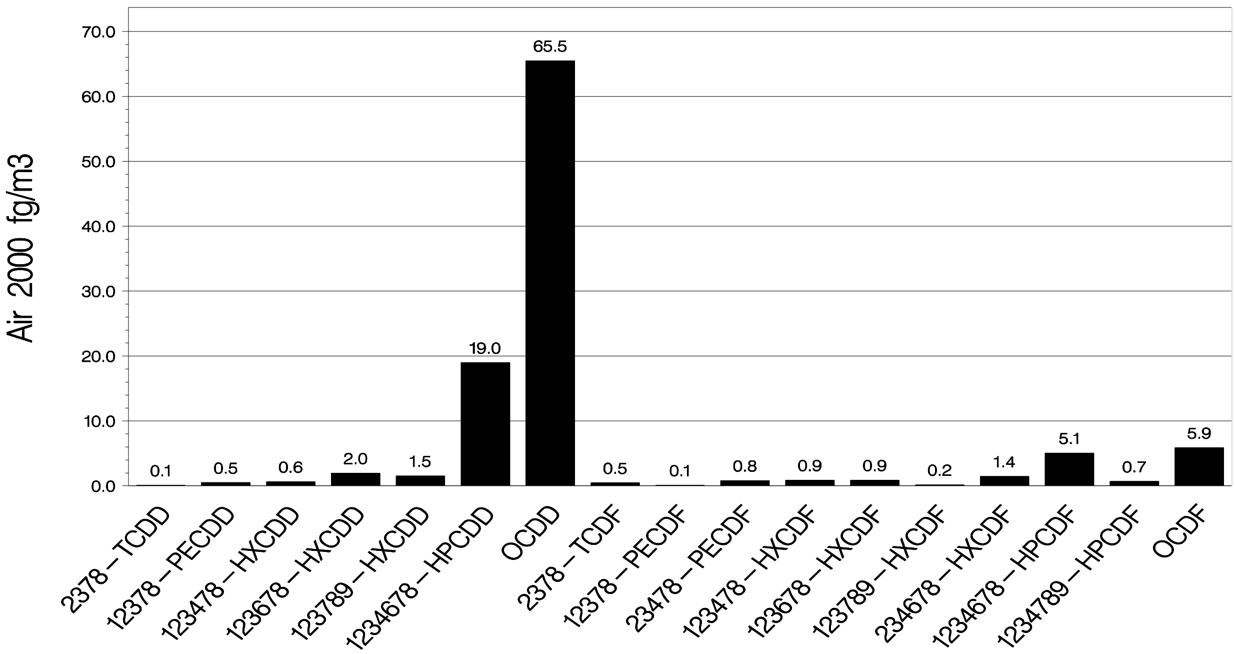


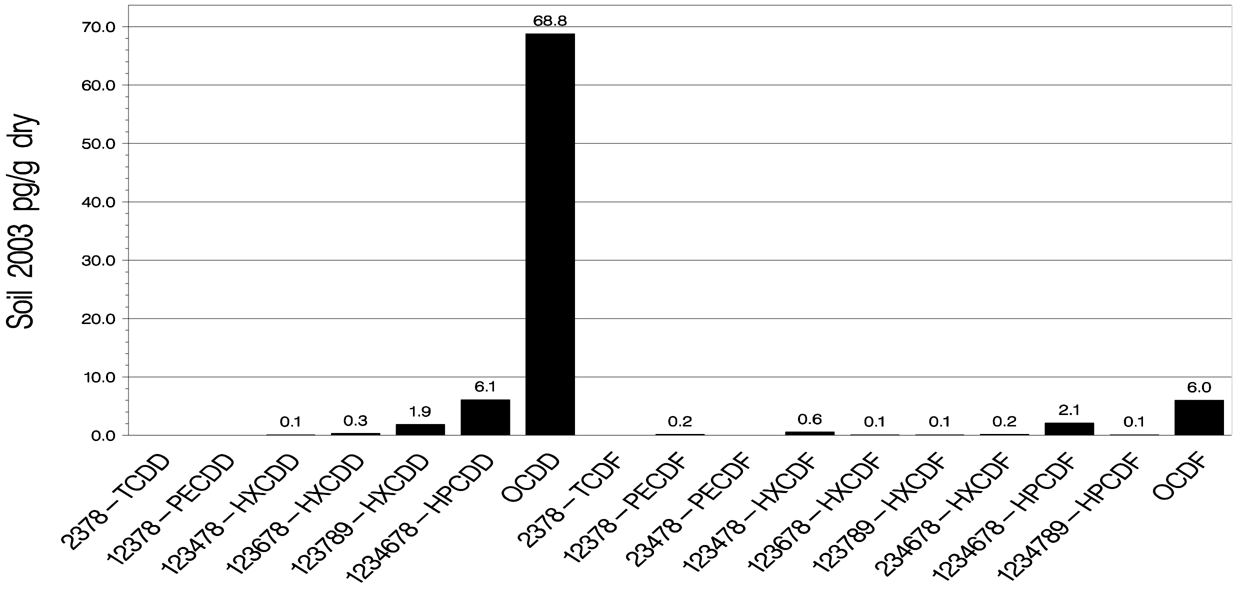
Site 29
Hyslop Farm, OR - 2000 Air
Marvel Ranch, OR - 2003 Soil





Site 30
Ozette Lake, WA





Site 30
Ozette Lake, WA