BASELINE CHEMICAL CHARACTERIZATION OF SAGINAW BAY WATERSHED SEDIMENTS

A Report to the Office of the Great Lakes
Michigan Department of Environmental Quality

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Introduction

General: Persistent bioaccumulative compounds have historically been released from industrial entities in the Saginaw Bay watershed. Dioxins and furans, polychlorinated and polybrominated biphenyl compounds (PCBs and PBBs), pesticides, herbicides, and other persistent bioaccumulative compounds have been identified as significant pollutants in the Saginaw River and the Saginaw Bay (U.S.EPA, 1995).

In September of 2000, the Waste Management Division of the Michigan Department of Environmental Quality (MDEQ) was awarded a grant of $88,775.00 from the Michigan Great Lakes Protection Fund to conduct a study entitled: ìBaseline Chemical Characterization of Saginaw Bay Watershed Sediments (Baseline Study).î The Baseline Study area is shown below as Figure 1. A copy of the approved grant proposal and budget is attached as Appendix 1 of this Report.

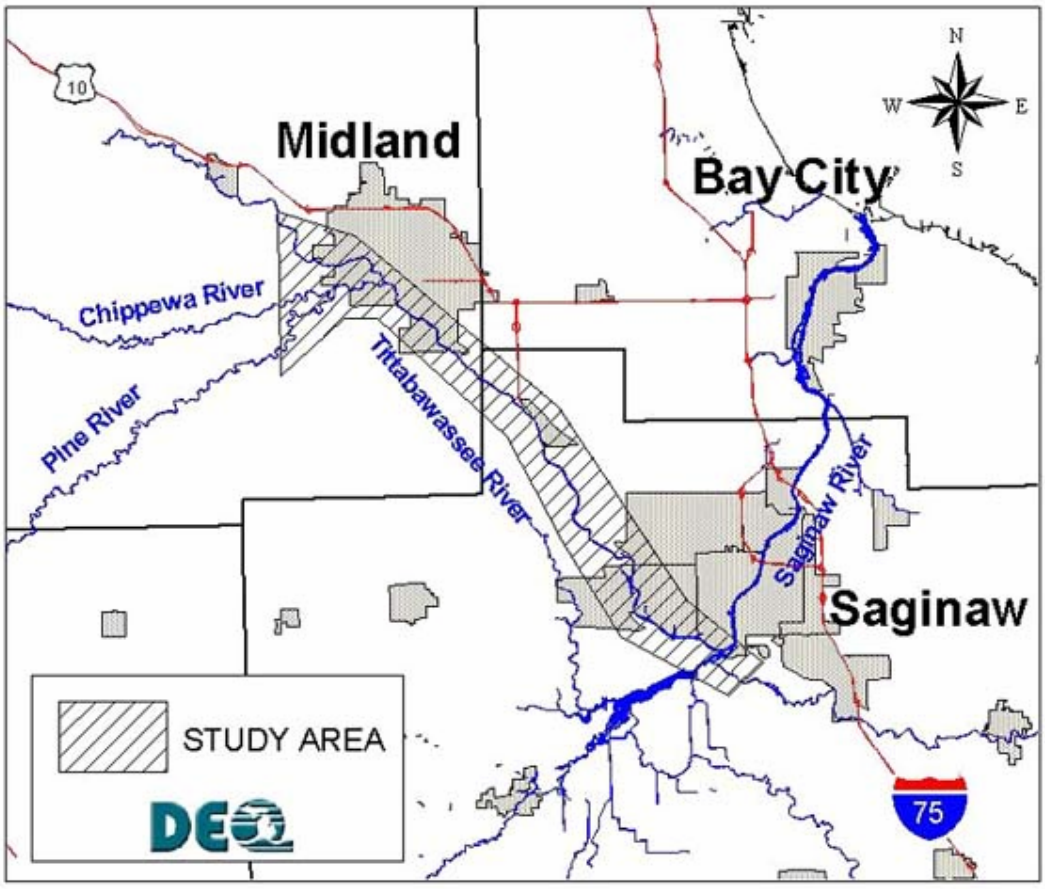

Figure 1

The focus of the Baseline Study is to characterize the sediments in the identified study area beginning upstream of Midland Michigan on the Pine, Chippewa, and Tittabawassee Rivers, and continuing downstream on the Tittabawassee to its confluence with the Saginaw River. The data and sampling locations from this study establish a year 2001 baseline level of contamination of watershed sediments that provides a benchmark against which future improvements in sediment and water quality can be measured. The analytical data from this project is also intended to serve as a screening level evaluation of sediment quality in the Tittabawassee River; to determine if contaminants are present at levels of environmental concern; and to determine if Tittabawassee River sediments are a potential source of ongoing releases to Lake Huron. Finally, analytical results from the study can be used to form the basis for a request for corrective action, if necessary, from regulated facilities within the watershed.

The Baseline Study was developed and conducted in a phased manner that consisted of a review of existing literature and data; the development of a sampling and analysis plan; the collection of samples; the analysis of the data; and the preparation of this Report. The Michigan State University Aquatic Toxicology Laboratory (MSU ATL), under the direction of Dr. John Giesy, assisted in the completion of this project by conducting a detailed literature review, assisting with the development of the study sampling and analysis plan (SAP), and conducting the dioxin and furan related analyses on the samples.

Literature and Data Review

The MSU-ATL conducted a literature search to identify appropriate literature to support the Baseline Study. This included all general literature on polychlorinated diaromatic hydrocarbons and specifically all of the literature, either in the open or gray literature and reports on the Saginaw River drainage system. The literature is compiled into an electronic data base (Reference ManagerÆ). A hard copy of the literature search is attached as Appendix 2 of this study.

The literature review indicates that significant sediment investigation work has been downstream of the study area in the Saginaw River and in the Saginaw Bay. In particular, the United States Environmental Protection Agency (U.S. EPA) has identified the Saginaw River and Saginaw Bay as an Area of Concern (U.S. EPA, 1995) as part of the Assessment and Remediation of Contaminated Sediments Program (ARCS Program). The ARCS Program resulted in the development of detailed sediment quality information on the lower Saginaw River and Saginaw Bay. Grab and core sediment samples from the ARCS study were analyzed for polyaromatic hydrocarbons, PCBs, chlorinated pesticides, polychlorinated dibenzo-p-

dioxins, polychlorinated dibenzofurans, and metals. General conclusions regarding the presence and risk of PCBs and certain metals in the sediments of the Area of Concern are presented in this study. In addition, to the ARCS Program, the Army Corps of Engineers (1999) has also conducted fairly extensive sampling and analysis of sediments in the navigable channel of the Saginaw associated with dredging projects.

Much less data is available on sediment quality in the Tittabawassee, Pine, and Chippewa Rivers upstream of the confluence of the Tittabawassee and the Saginaw Rivers. The Michigan Department of Community Health has issued fish advisories based on elevated levels of dioxins and furans and PCBs found in fish tissues in the Tittabawassee and Saginaw Rivers downstream of Midland. Fish advisories for PBBs and DDT have been issued for all species of fish on the Pine River downstream of the St. Louis impoundment.

Amendola and Barna (1986) reported of dioxin concentrations at up to 16 parts per billion (OCDD) in Tittabawassee River sediments in 1984. Dioxins were not detected in sediments upstream of The Dow Chemical Company facility in Midland, Michigan. Two studies have analyzed Tittabawassee River sediments for PCBs (MDNR, 1971, 1988). The Michigan Department of Natural Resources summaries of this work indicate that the sediment data from these studies is relatively sparse.

The MDEQ Surface Water Quality Division conducted an extensive review of existing information on the Tittabawassee River in 1993 during the evaluation of a potential Natural Resources Damage Assessment claim against The Dow Chemical Company (RCG/Hagler, Bailly Inc., 1993). That review identified data gaps in water quality and sediment quality with respect to dioxins and furans and PCBs.

The MDEQ conducted limited sediment sampling of the Tittabawassee and Chippewa Rivers in 1996 for a broad range of organic compounds, including dioxins and furans, and metals (MDEQ, 1996). This sampling event was concentrated adjacent to The Dow Chemical Company facility and immediately upstream of the facility.

Based on a review of existing data, it was clear that no comprehensive sediment characterization program has been conducted on the Tittabawassee River and that there is no program which routinely analyzes sediments to track changes in environmental quality of this portion of the Saginaw Bay watershed. It was also apparent that dioxins and furans were a significant issue in this watershed.

Methodology

Sampling and Analysis Plan: A SAP was developed for the collection and analysis of the samples. A copy of the SAP is attached as Appendix 3 to this Report. Unless otherwise stated, analyses were conducted in accordance with "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication SW-846, Third Edition, November 1986, and its updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), and IIIA (April 1998) (SW-846)

Study Design: The goals of this project were to (1) obtain a baseline chemical characterization of Tittabawassee River sediments and floodplain soils; and (2) to choose and document sampling locations so that the study might be replicated at a future date. These goals, in concert with financial and logistical realities, resulted in the sampling and analysis approach utilized in this project.

The parameters of interest in this study consisted of heavy metals, volatile and semivolatile organic compounds, pesticides, PCBs, dioxins, and furans. These parameters were chosen by the principal investigators as being the best indicators of the overall environmental health of the river system. A complete list of individual parameters is included in Table 1. In addition, analyses of physical parameters [Total Organic Carbon (TOC) and Total Organic Matter] were performed on all samples.

In addition to conventional dioxin and furan instrumental analysis (SW-846 Method 8290), each of the transect, composite, floodplain soils, and selected individual reach samples described below were analyzed using H4IIE-luc bioassay to determine relative dioxin-like activity. A subset of these samples were further analyzed in order to perform mass balance calculations to determine if other compounds are contributing significant dioxin like activity to Tittabawassee River sediments and soils.

The persistent bioaccumulative compounds of concern in this study are most frequently associated with fine particulate matter in the environment. Due to this association, an attempt was made to preferentially sample areas of fine particulate deposition. Preferred sampling locations included zones immediately downstream of large snags, bridge pilings, or other man-made structures, the inside bend of river meanders and other areas where fine particulates were observed to accumulate. Field personnel frequently tested possible sampling locations by poling with a boat oar to determine if there was suitable sediment accumulation. Figure 2 illustrates typical river sampling locations.

The location of each sample collected during this study was memorialized using hand-held Global Positioning System technology. Location coordinates in decimal degrees latitude and longitude for each sampling location in this study are identified in Table 2.

Figure 2. Typical River Sample Locations

Transect Sampling Locations: Sampling locations were chosen to provide geographic coverage of the study area and to preferentially sample depositional areas where the parameters of concern were most likely to be found. Nine locations (hereinafter referred to as transects) were chosen in order to provide coverage along the approximately 22 miles of river comprising the study area. Transect locations were tentatively identified by reviewing maps of the study area and locations finalized by the identification of depositional zones in the field. Transect locations are shown in Figure 3.

Two types of sampling were typically performed at each transect location. A sediment core sample was obtained by driving a four-inch diameter acetate or polyvinyl chloride (PVC) tube into the river bottom to a depth of one and a half to two feet (0.45 to 0.6 meter) at one discrete location per transect. Stones larger than º inch and leaves and twigs were removed from the sample before compositing in a stainless steel bowl. Aliquots of the composite sample were taken for analysis for each of the study parameters. Figure 4 illustrates a typical core sample.

Figure 4. Typical Core sample

A composite sample of the surficial sediments was obtained by using a petite Ponar dredge to collect a sample of the upper one to two inches of sediment at three to five locations per transect along a line between each bank of the river. The number of locations per transect was a field decision based on stream width, particle size of the sediment, and practical considerations in obtaining a sample. The individual dredge samples were composited by mixing with a stainless steel scoop in a

stainless steel bowl. Stones larger than º inch and leaves and twigs were removed from the sample before compositing in a stainless steel bowl. Figure 5 illustrates a typical upper composite sample. Aliquots of the composite sample were taken for analysis for each of the study parameters.

Figure 5. Typical Composite Sample

Discrete Sediment Samples: A series of discrete sediment samples were collected at locations between transects to characterize individual reaches of the river and obtain good geographic coverage of the study area. Between three and 20 reach samples were taken between each transect. Reach sample locations were identified in the field and were preferentially located in depositional zones. Reach samples consisted of the upper one to two inches of sediment collected with a petite Ponar dredge. Stones larger than º inch and leaves and twigs were removed from the sample before compositing in a stainless steel bowl. Reach sample locations are identified in Figure 3. Because of resource limitations and holding time limitations, reach samples were analyzed for dioxins, furans and physical parameters only.

Adjacent individual reach samples were carefully split and mixed in the lab to form a composite sample prior to analysis. A total of 19 reach ìcompositeî samples were analyzed for dioxins and furans. The individual reach samples and the reach composites are shown on Figure 3 and listed in Table 3.

In addition to the composites, individual analyses were completed on each of the reach samples that make up Composites #4, #11, #15, and #17. These composites were selected for additional analysis based on the results of the H4IIE-luc bioassay as described in further detail below. An individual analysis was also performed on reach sample #23 which was distinguished from the other samples by a much higher TOC content.

Floodplain Soil Samples: Ten floodplain soil samples were also collected as part of the study. Low lying areas immediately adjacent to the river were selected, based on field judgment, as likely sites of significant fine particle deposition. The locations of the floodplain soil samples are identified in Figure 3. Latitudes and longitudes of floodplain soil sample locations are included in Table 2. Floodplain soil samples were collected from a one square foot location. The top one to two inches of soil was collected using a stainless steel scoop and thoroughly mixed in a stainless steel bowl. Large organic matter such as leaves and twigs were removed. Aliquots were taken for analysis of each of the study parameters listed in Table 1. A typical floodplain soil sampling location is shown in Figure 6.

Figure 6. Typical Floodplain Soil Sampling Location

Field Quality Control: As a quality control measure, duplicate samples were taken at river sediment location TF-C and floodplain soil location SS-7. These duplicate samples were analyzed for all study parameters.

Reference Locations: As the study area was the Tittabawassee River between the confluence of the Pine and Chippewa Rivers in the northwest to the Shiawassee River in the southeast, a transect core, composite, and

several discrete (reach) sediment and floodplain soil samples were taken above the confluence of the Tittabawassee and the Pine and the Tittabawassee and the Chippewa to serve as upstream controls. These upstream reference locations are listed in Table 4.

Additional Samples

Due to a broken chain-of-custody, dioxin and furan samples had to be recollected at transect sampling locations TC-C and TC-UC, and discrete reach sampling locations 1, 2, 3, and 11. Additional aliquots for the other study parameters were taken at the transect locations and serve as quality control replicates. The second set of samples collected from these locations is designated by an asterisk (1*, 2*, 3*, 11*, TC-C* and TC-UC*).

Analytical Methods

Analyses of sediments and flood plain soils at the selected locations were conducted using generally accepted sediment sampling and analytical techniques as identified below. Table 1 summarizes the analyses conducted on each sample and lists individual analytical parameters.

Volatile Organics: River sediment and floodplain soil samples for analysis of volatile organic compounds (VOCs) were methanol preserved in the field. Extraction was according to SW-846 Method 5035. Extracts were analyzed according to SW-846 Method 8260B. The list of analytes and detection limits is provided with the results in Table 5. Due to the lack of detections, no samples for VOCs were collected upstream of Transect F.

Semivolatile Organics: Semivolatile Organics (base neutral and acid extractable compounds and polynuclear aromatic hydrocarbons) were extracted using SW-846 Method 3510 and analyzed according to SW-846 Method 8270. The list of analytes and detection limits is provided with the results in Table 6.

Pesticides and PCBs: Pesticides and PCBs were extracted using SW-846 Method 3510 and analyzed according to SW-846 Method 8070. The list of analytes and detection limits is provided with the results in Table 7.

Metals: Samples were analyzed for metals using SW-846 6000 and 7000 series methods. The list of analytes and detection limits is provided with the results in Table 8.

Dioxins and Furans Chemical Analysis: Concentrations of seventeen 2,3,7,8-substituted polychlorinated dibenzo-p-dioxins (PCDDs) and

dibenzofurans (PCDFs) were analyzed by modifications of previously described methods (Yamashita et al., 2000; Kannan et al., 2001; Im et al., 2002). A copy of MSU-ATLís ìStandard Operating Procedure for the Extraction and Analysis of 2,3,7,8-substituted PCDDs and PCDFs in Sediments using High Resolution Gas Chromatography ñ High Resolution Mass Spectrometryî is attached as Appendix 4 of this Report.

In Vitro Bioassay Analysis: H4IIE-luc bioassay was used to determine total dioxin-like activity (TCDD-EQs) in sediments. The bioassay procedures are described in detail in Appendix 5 of this Report, and in Villeneuve et al., 2000 and Hilscherova et al. 2001. Samples were tested as raw extracts and as acid-treated extracts using the *in vitro* H4IIE-luc recombinant cells for dioxin-like activity. A mass balance analysis of dioxin-like activity derived from instrumental and bioassay analyses was used to test for the presence of other dioxin-like compounds that can bind to the aromatic hydrocarbon receptor (AhR). Mass balance analysis (or potency balance analysis) was also used to examine whether or not the known composition of a sample (identified by instrumental analysis) can account for the magnitude or potency of biological response observed.

Total Organic Matter: Sediment and floodplain soil samples were analyzed for Total Organic Matter by the Michigan State University Soil Analysis Laboratory using their ìLoss by Ignitionî protocol.

Total Organic Carbon: Sediment and floodplain soil samples were analyzed for TOC by the Michigan State University Soil Analysis Laboratory using their ìDry Combustion using Microcarbon Analyzerî protocol.

Results

Results for VOCs, semivolatile organic compounds, pesticides, PCBs, and metals are presented in Tables 5, 6, 7 and 8, respectively. Geographic distribution of semivolatiles, PCBs, and pesticides are presented in Figure 7.

Dioxin and furan results are presented in Table 9. A full copy of the dioxin and furan analytical and bioassay results and quality assurance data is being retained by the MDEQ Waste Management Division. Table 9 also includes the results of the TOC and total organic matter analyses of these samples. The geographic distribution of dioxins and furans is presented in Figure 8.

Discussion

Volatiles:
Only one VOC was detected during this study. Tetrachloroethene was detected at 95 ppb at sample location TC-UC. Due to the flow regime of the Tittabawassee River, the lack of detectable volatile organic compounds is not surprising. The Tittabawassee is a shallow, fast moving river with considerable turbulence and scour during storm events. Volatile compounds, should they be released to the river, are not likely to persist in sediments that are frequently agitated. Given the lack of detects, the decision was made to cease sampling for VOCs above Transect F. None of the floodplain soil samples showed detectable levels of VOCs.

Semivolatiles:
Semivolatile organic compounds, when found, were typically present at very low concentrations. Semivolatile organic compounds, including phenanthrene, fluoranthene, pyrene, benzo(a)anthracene, and chrysene, were detected at sample locations TC-C, TF-CR, TE-C, TF-C, TF-UC, and TG-UC at levels below 400 ug/kg. Sample location TF-CR, in addition to having phenanthrene, fluoranthene, pyrene, and chrysene at very low levels, displayed hexachlorobenzene at 1300 ug/kg, a level that might indicate potential adverse impact to benthic organisms. Floodplain soil results were similar in displaying low levels of the same six semivolatile compounds at low levels (all below 510 ug/kg).

Pesticides and PCBs:
No PCBs were detected in sediment or floodplain soil samples. 4,4-DDT at 1,100 ug/kg and 4,4 DDD at 83 ug/kg in sediment sample TI-UC were the only target pesticides detected in sediment samples. Low levels of DDT and its breakdown products were detected at floodplain soil sampling locations SS-4 (Pine River), SS-5, and SS-7. There is a historical source of DDT on the Pine River that may have contributed to these detections. Hexabromobenzene was also detected at low levels at floodplain soil sampling locations SS-2, SS-4, and SS-7R. Detected levels of pesticides are not expected to have any significant impact on human health or the environment.

Metals:
Levels of metals in sediments are generally consistent with background levels. Of the floodplain soil samples, only SS-1 and SS-2 had concentrations that indicated the potential for minor aquatic life impacts (arsenic, chromium, copper, lead, mercury, nickel, and zinc). It is notable that SS-2 is immediately adjacent to and SS-1 is downstream of a former plate glass manufacturing facility which is known to have released metals to the river.

Dioxins and Furans:
Dioxins and furans were detected at each of the transect, reach, and individual sediment sampling locations analyzed during this study. Dioxins and furans were also present in each of the flood plain soil samples analyzed during this study. The concentrations of each of the 17 2,3,7,8-substituted PCDD and PCDF congeners, the calculated total toxic equivalent concentration (TEQ) relative to 2,3,7,8-TCDD, and the concentrations of the total tetra, penta, hexa, and hepta isomer groups are presented in Table 9 for each dioxin and furan sampling location.

The geographic distribution of the TEQ results is presented on Figure 8.

In general, the geographic distribution of dioxins and furans in the study area points to a source in the Midland area. The concentrations of dioxin in river sediments were less than 5 parts per trillion (ppt) TEQ in the seven reference samples that were taken above the junction of the Chippewa and Tittabawassee Rivers. Below the junction of the Chippewa and Tittabawassee Rivers, TEQ concentrations ranged from 5 to 2,000 ppt.

The concentrations of dioxins and furans were not significantly correlated with the TOC content of the samples. Correlation with the grain size of the samples with dioxin concentration was not completed in this study because the high levels of dioxins and furans in the samples resulted in an unanticipated health and safety concern during grain size analysis which involves drying and shaking of the samples. It is recommended that this evaluation be completed under the appropriate laboratory conditions.

Transect Results:
These samples are identified on Figure 8 by green boxes. The core composite samples are identified with a ìCî suffix (e.g. TA-C). The upper composite (dredge composite) samples are identified with a ìUCî suffix (e.g., TA-UC). The core sample results represent a mix of the surficial sediments and deeper sediments. The upper composite results represent only the top several inches of sediment.

In general, the concentrations of dioxins and furans were much higher in transect samples collected below the junction of the Tittabawassee and Chippewa Rivers than in the upstream reference samples. The transect reference samples were each less than 5 ppt total TEQ. Samples collected below the junction of the Tittabawassee River ranged in TEQ concentration from 5.1 ppt to 2000 ppt. It should be noted that the 5.1 ppt TEQ transect sample was collected at location TG-UC. No core sample could be collected from this location because of the coarse grained nature of the sediments. It is possible that the coarse grained nature of the sediments resulted in a relatively low concentration at this location.

The TEQ of the upper composite samples was similar to the core concentrations in most of the samples, with the exception of transects TC and TD where the concentration of dioxin and furans in the deeper cores was significantly higher than the upper composite samples. This suggests that in some parts of the study area, a reservoir of dioxin and furans may be present in deeper sediments. Further detailed coring work should be completed to determine if higher levels of dioxins and furans are present at lower depths in the river sediments.

Composite Results:
These samples show the distribution of dioxin and furans in the upper several inches of sediment in sediment accumulation areas in the study area. Due to resource limitations, adjacent sediment samples were carefully split and mixed in the lab to form a composite sample prior to analysis. The results of these analyses are shown on the maps as ìComposite #1 through Composite #19.î

These results show that the composite concentrations are significantly elevated and quite variable below the junction of the Tittabawassee and Chippewa Rivers. This variability is probably reflective of the flow and depositional characteristics of the different segments of the river system.

In order to evaluate the variability within the composite locations, individual reach samples were analyzed at four locations in addition to the composite analyses. This analysis revealed that there can be considerable variability in the TEQ concentrations of the individual reach samples that were used to form a composite sample. In the case of Composite #15, the concentrations varied from 22 to 2000 ppt TEQ with a composite average analysis of 960 ppt. Composite #11 varied from 130 to 1000 ppt TEQ with a composite average analysis of 480 ppt TEQ. Based on the recognized variability, any future studies that are conducted to more fully understand of the distribution of dioxin contamination should avoid compositing samples.

Flood Plain Soil Samples:
Ten flood plain soil samples were taken as a part of this study. These samples are identified on Figure 8 by red hexagons. These samples were collected by scraping the upper one inch of soil from a one square foot area in selected low lying flood plain areas adjacent to the Tittabawassee River. Sample locations were also based on property access agreements.

Dioxin results in floodplain soils ranged from 2 to 11 ppt TEQ in control samples above the junction of the Chippewa and Tittabawassee Rivers. Below the junction, TEQ concentrations ranged from 300 to1500 ppt. With the exception of SS#6, all of the samples were taken in the flood plain

within 100 feet of the edge of the river. SS#6 was taken from a recently tilled farm field several hundred yards away from the edge of the river.

The upstream reference sample results are consistent with Michiganís soil background dioxin levels. The soil concentrations below the confluence of the Tittabawassee and Chippewa are significantly elevated over Michiganís background soil concentration and exceed Michiganís Part 201, Environmental Remediation, of the Natural Resources and Environmental Protection Act, 1994 PA 451, as amended, generic residential soil protection criterion of 90 ppt TEQ.

The downstream soil concentrations also exceed the Agency for Toxic Substances and Disease Registry (ATSDR) interim policy guidelines for PCDDs/PCDFs in residential soils near or on hazardous waste sites (De Rosa et al., 1997). When concentrations of TEQs exceed 50 pg /g TEQ dry weight, the ATSDR recommends evaluation of site-specific factors such as pathway analysis and soil cover. When soil concentrations exceed 1000 pg/g TEQ dry weight, the ATSDR recommends health surveillance and exposure investigations.

Based on the results of this investigation and follow up work conducted by the MDEQ in 2002, additional work is needed to determine if the level of exposure in the flood plain is resulting in health and/or ecological impacts.

Bioassay and Mass Balance Results:
H4IIE-luc bioassay was used to determine total dioxin-like activity (TCDD-EQs) in sediments and to direct the instrumental analyses. A mass balance analysis of dioxin-like activity derived from instrumental and bioassay analyses was used to test for the presence of other dioxin-like compounds that can bind to the aromatic hydrocarbon receptor (AhR). Earlier studies have demonstrated that the H4IIE-luc bioassay coupled with instrumental analysis is useful in the integrated assessment of dioxin-like activity in sediment (Khim et al., 1999; Hilscherova et al., 2000; Khim et al., 2001).

The instrumental and bioanalytical approaches provide different and complementary information. While instrumental analysis is a useful tool to identify the compounds of interest and to evaluate the concentrations of environmental contaminants, it provides little information regarding the integrated biological relevance of a complex mixture of compounds associated with environmental samples such as sediment. Where appropriate, bioassay-directed fractionation and mass balance analysis is a powerful tool to characterize the causative agents responsible for bioassay responses observed. Recent studies have indicated that organic extracts of sediments elicit both dioxin-like responses significantly *in vitro,* although the chemical concentrations often did not explain the bioassay

activities observed (Khim et al., 1999; Kannan et al., 2000; Hilscherova et al., 2001). Empirical bioassay results and mass balance analyses can suggest the magnitude of contribution of target organic compounds to total dioxin-like activity of sediment extracts. Thus, the use of bioassay-based toxicity identification and evaluation (TIE) and mass balance analysis are important approach to assess sediment contamination since the sediment extracts may contain many potentially AhR-active compounds, which were not analyzed by instrumental methods.

The results of this study showed that the results of the bioassay analyses matched very well with the results of the instrumental analysis. Therefore, the bioassay technique proved valuable in determining which samples showed significant dioxin-like activity. The bioassay technique could be used to direct further investigation in the watershed by screening out low activity samples and identifying areas of high dioxin like activity that would warrant follow up chemical confirmation.

Appendix 5 contains the results of the bioassay analyses and the mass balance calculations. The results suggest that PCDDs/PCDFs, are the major sources of dioxin-like activity in sediments/soils from the Tittabawassee River and that little, if any, activity was due to other compounds, such as PCBs, that exhibit dioxin-like activity. This was further supported by the lack of detectable concentrations of PCBs in soils and sediments.

Sources of Dioxins and Furans in the Tittabawassee River Watershed:

Figures 9, 10, 11, and 12 are congener profiles of the dioxin and furan samples collected during this study. The TEQ concentration of each sample has been normalized to 100 percent (%) and the normalized toxic equivalent concentration of each congener is plotted on the resulting bar chart. Congeners that were not detected at a specific sampling location were assigned a value of zero. The resulting bar chart is a ìfingerprintî of the 17 dioxins and furans that exhibit dioxin-like toxicity. The upstream reference samples are plotted at the top of each of the graphs and are marked with an asterisk.

As can be seen by review of the graphs, all of the samples taken from below the confluence of the Chippewa and Tittabawassee Rivers are fairly similar to each other and markedly different from the upstream reference samples. The chemical fingerprint of the sediment samples is similar to the chemical fingerprint of the floodplain soil samples, indicating that the dioxins and furans in these media are likely from the same source(s).

The bulk of the dioxin-like toxicity in the downstream samples is contributed by furan congeners. 2,3,7,8-TCDF and 2,3,4,7,8-PeCDF

contribute the bulk of the dioxin-like toxicity in the study area downstream of the confluence of the Tittabawassee and Chippewa Rivers. 1,2,3,7,8 ñ PeCDF and 1,2,3,4,7,8-HxCDF also make significant contributions to the dioxin-like toxicity of downstream sediment and soil samples. 2,3,7,8-TCDD, the most potent dioxin-like compound, typically contributes less than five percent of the TEQ in the downstream samples.

Different sources of PCDDs/PCDFs are characterized by different congener and homologue patterns (Kannan et al., 1998). Furthermore, differences in the physicochemical (mobility, solubility, etc.) and biological (biodegradation, bioaccumulation, etc.) properties may alter the congener profiles.

The fingerprints of PCDD and PCDF congeners in sediments collected from the Tittabawassee River downstream of Midland are all similar, suggesting the presence of a single major source. As noted above, the pattern of relative concentrations of PCDD/PCDF congeners was also different in soils and sediment collected downstream of Midland than in those collected upstream of the reference locations. A large proportion of OCDD and HpCDD has been suggested to be due to the sources originating from chlorophenol-related sources (Masunaga et al., 2001). Greater proportions of TCDFs suggest sources originating from PCB mixtures, chlorobenzenes, chlor-alkali processes, and incineration of PCBs and polyvinyl chloride (Wakimoto et al., 1988; Masunaga et al., 2001; Swami et al., 1992; Kannan et al., 1998). Total concentrations of PCBs in sediments from the Tittabawassee River were less than 150 nanograms/gram. This suggests that PCBs are not the source of the PCDD/PCDFs, but rather other sources such as chlorophenol and chlorobenzene production, incineration, or chlor-alkali processes are the sources of the PCDFs found in Tittabawassee River sediments collected below Midland.

The Dow Chemical Company (Dow) has a long history as a major manufacturer of chlorobenzenes, chlorophenols, and chlor-alkali products in Midland, Michigan. Additionally, Dow has conducted chemical waste incineration for many years. The geographic distribution of the contaminants combined with the dioxin and furan congener profile information strongly suggests that Dowís Midland facility is the most likely source of the elevated levels of dioxins and furans in the Tittabawassee River.

Conclusions and Recommendations

Summary: The data reviewed indicates that the potential for impacts to aquatic life to occur from the sediment chemicals analyzed, other than dioxins and furans, is minimal.

With regard to dioxins and furans, the following conclusions can be drawn:

1. The concentrations of dioxins and furans in sediments and soils represent a potential environmental and human health issue in the Tittabawassee River watershed that requires further study.

2. In the study area, the bulk of the dioxin-like toxicity in sediments and soils is contributed by the polychlorinated dibenzo-p-dioxins and furans. In particular, 2,3,7,8-TCDF and 2,3,4,7,8-PeCDF contribute the bulk of the dioxin-like toxicity to the samples collected from below the confluence of the Tittabawassee and Chippewa Rivers. Little dioxin-like toxicity appears to be contributed by other compounds that exhibit dioxin-like activity, such as PCBs.

3. The downstream extent of dioxin and furan contamination in sediments and floodplain soils has not been completely defined.

4. Deeper sediment cores are needed to determine the vertical extent of sediment contamination in the Tittabawassee and downstream in the Saginaw River.

5. The distribution of dioxins and furans in the study area sediments appears heterogeneous. This is most likely related to the flow regime of the Tittabawassee River. Any follow up sampling that is conducted should not involve compositing sediment samples. The relationship of grain size to dioxin and furan concentrations in the study area needs to be investigated.

6. The H4IIE-luc bioassay technique can be successfully used as a tool to direct further investigation of dioxin and furan contamination in the study area.

7. The most probable historic source of dioxins and furans in the Tittabawassee River watershed, based on the geographic distribution of the contaminants and the chemical profiles of the dioxin and furan congeners, is located in Midland, Michigan.

Recommendations:
Based on these conclusions, additional sampling is recommended to more completely define the horizontal and vertical distribution of dioxins and furans in the Tittabawassee River and Saginaw River watersheds. Periodic resampling should be conducted to determine if concentrations

are changing over time. The existing information should be used along with other available information to conduct an assessment of the risk to human health and the environment from the elevated concentrations of dioxins and furans present in the study area.

TABLES

TABLE 1

ANALYTICAL OVERVIEW

Sample Type	Analyses												
	Volatile Organics		Semi volatile organics		Pesticides & PCB's		Metals		Dioxins and Furans (instrumental analyses)		TOC and TOM		Dioxins and Furans (bioassay)
Transect Upper Composite	X*		X		X		X		X		X		X
Transect Core	X*		X		X		X		X		X		X
Floodplain Soils			X		X		X		X		X		X
Reach Composites									X		X		X
Discrete Reach Samples									Selected		Selected		X

Transect Upper Composite Samples = TA-UC, TB-UC, TC-US, TD-US, TE-US, TF-US, TG-UC, TH-US, TI-UC
(TG-UC, TH-UC AND TI-UC WERE NOT ANALYZED FOR VOLATILE ORGANIC COMPOUNDS)

Transect Core Samples = TA-C, TB-C, TC-C, TD-C, TE-C, TF-C, TF-CR, TH-C, TI-C
(*TH-C AND TI-C WERE NOT ANALYZED FOR VOLATILE ORGANIC COMPOUNDS).

Floodplain Soil Samples = SS-1, SS-2,SS-3, SS-4, SS-5, SS-6, SS-7, SS-8, SS-9, SS-10

Reach Composites = Composite #1, Composite #2, Composite #3, Composite # 4, Composite # 5, Composite #6, Composite #7, Composite #8, Composite #9, Composite #10, Composite #11, Composite #12, Composite #13, Composite #14, Composite #15, Composite #16, Composite #17, Composite #18

Discrete Reach Samples (selected) = 8, 13, 14, 15, 16, 23, 37, 38, 39, 52, 53, 54, 55, 59, 60,

REPLACE THIS PAGES WITH :

TABLE 1- ANALYTICAL OVERVIEW

TABLE 1

VOLATILE ORGANICS

Method 624/8260

1,1,1,2-Tetrachloroethane
1,1,1-Trichloroethane
1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethylene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloroethylene (cis)
1,2-Dichloroethylene (trans)
1,2-Dichloropropane
1,3,5-Trimethylbenzene(Mesitylene)
1,3-Dichlorobenzene
1,3-Dichloropropene (cis)
1,3-Dichloropropene (trans)
1,4-Dichloro-2-butene(trans)
1,4-Dichlorobenzene
2-Butanone (MEK)
2-Hexanone
2-Methylnaphthalene
Acetone (2-Propanone)
4-Methyl-2-Pentanone (MIBK)
Acrylonitrile
Benzene
Bromobenzene
Bromochloromethane
Bromodichloromethane
Bromoform
Bromomethane
Carbon disulfide
Carbon tetrachloride
Chlorobenzene
Chloroethane
Chloroform
Chloromethane
Dibromochloromethane
Dibromomethane
Dichlorodifluoromethane
Diethyl ether
Ethylbenzene
Hexachloroethane
Isopropylbenzene
m&p-Xylene
Methyl Iodide (Iodomethane)
Methyl Tertiary Butyl Ether (MTBE)
Methylene chloride
Naphthalene
n-Butylbenzene
n-Propylbenzene
o-Xylene
p-Isopropyl Toluene (p-Cymene)
sec-Butylbenzene
Styrene
tert- Butylbenzene
Tetrachloroethylene
Tetrahydrofuran
Toluene
Trichloroethylene
Trichlorofluoromethane
Vinyl chloride

TABLE 1

(continued)

BTEX/MTBE VOLATILES

Method 624/8260

Benzene
Toluene
Ethylbenzene
m & p-Xylene
0 -Xylene
Methyl Tertiary Butyl Ether

PESTICIDES

Method 608/8081

Aldrin
a-BHC
b-BHC
d-BHC
g-BHC (lindane)
BP-6 (PPB)
a-Chlordane
g-Chlordane
4,4'-DDD
4,4'-DDE
4,4'-DDT
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan Sulfate
Endrin
Endrin Aldehyde
Endrin Ketone
Heptachlor
Heptachlor epoxide
Hexabromobenzene
Methoxychlor
Mirex
Toxaphene

PCB'S

Methods 608/8082

PCB-1016
PCB-1221
PCB-1232
PCB-1242
PCB-1248
PCB-1254
PCB-1260
PCB-1262
PCB-1268

TABLE 1

(continued)

SEMIVOLATILE ORGANICS

BASE/NEUTRAL/ACIDS

Method 625/8270

1,2,4-Trichlorobenzene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
2,4,5-Trichlorophenol
2,4,6-Trichlorophenol
2,4-Dichlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
2,4-Dinitrotoluene
2,6-Dinitrotoluene
2-Chloronaphthalene
2-Chlorophenol
2-Methyl-4,6-dinitrophenol
2-Methylnaphthalene
2-Methylphenol
2-Nitroaniline
2-Nitrophenol
3/4-Methylphenol
3-Nitroaniline
4-Bromophenyl phenylether
4-Chloro-3-methylphenol
4-Chlorophenyl phenylether
4-Nitroaniline
4-Nitrophenol
Acenaphthene
Acenaphthylene
Anthracene
Azobenzene
Benz(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(g,h,i)perylene
Benzo(k)fluoranthene
Bis(2-chloroethoxy)methane
Bis(2-chloroethyl)ether
Bis(2-chloroisopropyl)ether
Bis(2-ethylhexyl)phthalate
Butyl benzyl phthalate
Carbazole
Chrysene
Dibenz(a,h)anthracene
Dibenzofuran
Diethyl phthalate
Dimethyl phthalate
Di-n-butyl phthalate
Di-n-octyl phthalate
Fluoranthene
Fluorene
Hexachlorobenzene
Hexachlorobutadiene
Hexachlorocyclopentadiene
Hexachloroethane
Indeno(1,2,3-cd)pyrene
Isophorone
Naphthalene
Nitrobenzene
N-Nitrosodimethylamine
N-Nitrosodi-n-propylamine
N-Nitrosodiphenylamine
Pentachlorophenol
Phenanthrene
Phenol
Pyrene

TABLE 1

(continued)

METALS

Aluminum
Antimony
Arsenic
Barium
Beryllium
Boron
Cadmium
Calcium
Chromium
Chromium VI
Cobalt
Copper
Iron
Lead
Lithium
Magnesium
Manganese
Mercury
Molybdenum
Nickel
Potasium
Selenium
Silver
Sodium
Strontium
Titanium
Thallium
Vanadium
Zinc

PCDD'S AND PCDF'S

PCDF	PCDD
2,3,7,8-TCDF	2,3,7,8-TCDD
1,2,3,7,8-PeCDF	1,2,3,7,8-PeCDD
2,3,4,7,8-PeCDF	1,2,3,4,7,8-HxCDD
1,2,3,4,7,8-HxCDF	1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	1,2,3,7,8,9-HxCDD
1,2,3,7,8,9-HxCDF	1,2,3,4,6,7,8-HpCDD
2,3,4,6,7,8-HxCDF	OCDD
1,2,3,4,6,7,8-HpCDF	
1,2,3,4,7,8,9-HpCDF	
OCDF	

TABLE 2

SAMPLE LOCATION COORDINATES

SAMPLE ID.	LATITUDE	LONGITUDE
TA-C	N 43∞23.045	W 083∞58.532
TA-UC	N 43∞23.045	W 083∞58.532
#1	N 43∞23.054	W 083∞58.464
#1*	N 43∞38.420	W 083∞97.410
#2	N 43∞23.063	W 083∞58.378
#2*	N 43∞38.563	W 083∞96.851
#3	N 43∞23.124	W 083∞58.299
#4	N 43∞23.198	W 083∞59.049
#5	N 43∞23.537	W 083∞59.650
#6 and #6R	N 43∞23.541	W 084∞00.006
#7	N 43∞23.591	W 084∞00.638
#8	N 43∞23.599	W 084∞00.887
#9	N 43∞23.625	W 084∞00.904
#10	N 43∞23.658	W 084∞01.169
#11	N 43∞23.755	W 084∞01.312
#11*	N 43∞39.597	W 084∞02.182
#12	N 43∞23.941	W 084∞01.528
TB-C	N 43∞24.150	W 084∞01.956
TB-UC	N 43∞24.150	W 084∞01.956
#13	N 43∞24.208	W 084∞02.011
#14	N 43∞24.274	W 084∞02.243
#15	N 43∞24.350	W 084∞02.502
#16	N 43∞24.496	W 084∞02.768
#17	N 43∞24.927	W 084∞02.701
#18	N 43∞24.985	W 084∞02.676
TC-C	N 43∞25.254	W 084∞02.608
TC-UC	N 43∞25.254	W 084∞02.608
TC-C*	N 43∞42.104	W 084∞02.608
TC-UC*	N 43∞42.104	W 084∞02.608
#19	N 43∞25.254	W 084∞02.847
#20	N 43∞25.638	W 084∞02.997
#21	N 43∞25.867	W 084∞02.916
#22	N 43∞26.023	W 084∞02.880
#23	N 43∞26.264	W 084∞02.839
#24	N 43∞26.510	W 084∞03.301
TD-C	N 43∞27.242	W 084∞04.993
TD-UC	N 43∞27.242	W 084∞04.993
#25	N 43∞27.585	W 084∞05.045
#26	N 43∞27.692	W 084∞05.534
#27	N 43∞27.816	W 084∞05.613
TE-C	N 43∞28.094	W 084∞05.798
TE-UC	N 43∞28.094	W 084∞05.798
#28	N 43∞28.337	W 084∞05.601
#29	N 43∞28.510	W 084∞05.473
#30	N 43∞29.482	W 084∞06.062
#31	N 43∞29.621	W 084∞06.193
#32	N 43∞29.748	W 084∞06.257
#33	N 43∞30.092	W 084∞07.006

TABLE 2

Sample Location Coordinates (continued)

SAMPLE ID.	LATITUDE	LONGITUDE
#34	N 43∞30.268	W 084∞07.119
#35	N 43∞30.376	W 084∞07.115
#36	N 43∞30.562	W 084∞07.157
#37	N 43∞30.820	W 084∞07.262
#38	N 43∞51.729	W 084∞12.333
#39	N 43∞52.239	W 084∞12.484
#40	N 43∞53.216	W 084∞13.525
#41	N 43∞53.399	W 084∞13.718
#42	N 43∞53.508	W 084∞13.789
#43	N 43∞53.692	W 084∞14.203
#44	N 43∞54.172	W 084∞14.674
#45	N 43∞54.424	W 084∞15.513
TF-C	N 43∞54.785	W 084∞16.014
TF-UC	N 43∞54.785	W 084∞16.014
#46	N 43∞54.452	W 084∞15.714
#47	N 43∞54.679	W 084∞15.943
#48	N 43∞55.556	W 084∞17.574
#49	N 43∞56.011	W 084∞18.271
#50	N 43∞56.540	W 084∞18.754
#51	N 43∞56.744	W 084∞19.049
#52	N 43∞56.887	W 084∞19.432
#53	N 43∞57.053	W 084∞19.659
#54	N 43∞57.201	W 084∞20.131
#55	N 43∞57.869	W 084∞20.549
TG-UC	N 43∞58.375	W 084∞20.799
#56	N 43∞58.490	W 084∞20.986
#57	N 43∞58.915	W 084∞22.420
#58	N 43∞59.637	W 084∞23.610
TH-C	N 43∞61.598	W 084∞25.137
TH-UC	N 43∞61.598	W 084∞25.137
#59	N 43∞61.423	W 084∞25.040
#60	N 43∞61.223	W 084∞25.002
#61	N 43∞61.044	W 084∞25.004
#62	N 43∞60.971	W 084∞25.305
TI-C	N 43∞61.208	W 084∞26.137
TI-UC	N 43∞61.208	W 084∞26.137
#63	N 43∞60.728	W 084∞27.545
#64	N 43∞61.000	W 084∞28.609
#65	N 43∞61.129	W 084∞24.659
#66	N 43∞60.741	W 084∞24.023
#67	N 43∞60.397	W 084∞23.976
SS #1	N 43∞38.387	W 083∞97.346
SS #2	N 43∞39.291	W 083∞99.866
SS #3	N 43∞60.379	W 084∞30.156
SS #4	N 43∞60.070	W 084∞29.683
SS #5	N 43∞57.124	W 084∞19.846
SS #6	N 43∞56.531	W 084∞18.429
SS #7	N 43∞45.321	W 084∞08.112
SS #8	N 43∞40.158	W 084∞02.975
SS #9	N 43∞39.243	W 084∞00.623
SS #10	N 43∞63.392	W 084∞32.204

TABLE 3

REACH SAMPLE COMPOSITES

COMPOSITE	COMPOSITED SAMPLES
C1	1*,2*,3,4
C2	5,6,7
C3	10,11*,12
C4	13,14,15,16
C5	17,18,19,20
C6	21,22,24
C7	25,26,27
C8	28,29
C9	30,31,32
C10	33,34,35,36
C11	37,38,39
C12	40,41,42,43
C13	44,45,46,47
C14	48,49,50,51
C15	52,53,54,55
C16	56,57,58
C17	59,60
C18	61,62,63,64
C19	65,66,67

TABLE 4

UPSTREAM REFERENCE SAMPLES

SAMPLE ID.	SAMPLE TYPE
TH-UC	Upper Sediment Composite
TH-C	Sediment Core
TI-UC	Upper Sediment Composite
TI-C	Sediment Core
59	Discrete Reach Sample
60	Discrete Reach Sample
Composite # 17	Composite Reach Sample
Composite # 18	Composite Reach Sample
SS-3	Floodplain Soil Sample
SS-4	Floodplain Soil Sample
SS-10	Floodplain Soil Sample

TABLE 5

Date collected		8/30/01		8/30/01		8/31/01		8/31/01		9/4/01	
		TA-C		TA-UC		TB-C		TB-UC		TC-C	
VOLATILE ORGANICS	**CAS #**	**RESULTS**	**LIMIT**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
Dichlorodifluoromethane	75-71-8	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
Chloromethane	74-87-3	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
Vinyl chloride	75-01-4	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Bromomethane	74-83-9	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
Chloroethane	75-00-3	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
Trichlorofluoromethane	75-69-4	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
Acetone	67-64-1	N.D.	1100	N.D.	1000	N.D.	1200	N.D.	950	N.D.	1200
Diethyl ether	60-29-7	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
1,1-Dichloroethylene	75-35-4	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Methyl iodide	74-88-4	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
Acrylonitrile	107-13-1	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
Methylene chloride	75-09-2	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
Carbon disulfide	75-15-0	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
trans-1,2-Dichloroethylene	156-60-5	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Methyltertbutylether (MTBE)	1634-04-4	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
1,1-Dichloroethane	75-34-3	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
2-Butanone (MEK)	78-93-3	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
cis-1,2-Dichloroethylene	156-59-2	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Chloroform	67-66-3	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Bromochloromethane	74-97-5	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
1,1,1-Trichloroethane	71-55-6	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
1,2-Dichloroethane	107-06-2	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Benzene	71-43-2	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Carbon tetrachloride	56-23-5	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
1,2-Dichloropropane	78-87-5	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Trichloroethylene	79-01-6	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Dibromomethane	74-95-3	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
Bromodichloromethane	75-27-4	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
4-Methyl-2-pentanone (MIBK)	108-10-1	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
cis-1,3-Dichloropropene	10061-01-5	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
trans-1,3-Dichloropropene	10061-02-6	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Toluene	108-88-3	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
1,1,2-Trichloroethane	79-00-5	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
2-Hexanone	591-78-6	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
Dibromochloromethane	124-48-1	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
1,2-Dibromoethane	106-93-4	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Tetrachloroethene	127-18-4	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
Chlorobenzene	108-90-7	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
1,1,1,2-Tetrachloroethane	630-20-6	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
Ethylbenzene	100-41-4	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82

All data in ug/Kg

TABLE 5

		TA-C		TA-UC		TB-C		TB-UC		TC-C	
VOLATILE ORGANICS	**CAS #**	**RESULTS**	**LIMIT**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
m & p-Xylene	108,383,106,423	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
Bromoform	75-25-2	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
Styrene	100-42-5	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
o-Xylene	95-47-6	N.D.	70	N.D.	67	N.D.	83	N.D.	63	N.D.	82
1,1,2,2-Tetrachloroethane	79-34-5	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
1,2,3-Trichloropropane	96-18-4	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
trans-1,4-Dichloro-2-butene	110-57-6	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
Isopropylbenzene	98-82-8	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
Bromobenzene	108-86-1	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
n-Propylbenzene	103-65-1	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
1,3,5-Trimethylbenzene	108-67-8	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
tert-Butylbenzene	98-06-6	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
1,2,4-Trimethylbenzene	95-63-6	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
sec-Butylbenzene	135-98-8	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
1,3-Dichlorobenzene	541-73-1	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
1,4-Dichlorobenzene	106-46-7	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
p-Isopropyl toluene	99-87-6	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
1,2-Dichlorobenzene	95-50-1	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
n-Butylbenzene	104-51-8	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
Hexachloroethane	67-72-1	N.D.	140	N.D.	130	N.D.	170	N.D.	130	N.D.	160
1,2-Dibromo-3-chloropropane	96-12-8	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
1,2,4-Trichlorobenzene	120-82-1	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
Naphthalene	91-20-3	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
1,2,3-Trichlorobenzene	87-61-6	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410
2-Methylnaphthalene	91-57-6	N.D.	350	N.D.	330	N.D.	410	N.D.	320	N.D.	410

All data in ug/Kg

TABLE 5

Date collected		11/15/01		9/4/01		11/15/01		9/5/01		9/5/01	
		TC-C*		TC-UC		TC-UC*		TD-C		TD-UC	
VOLATILE ORGANICS	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
Dichlorodifluoromethane	75-71-8	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
Chloromethane	74-87-3	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
Vinyl chloride	75-01-4	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
Bromomethane	74-83-9	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
Chloroethane	75-00-3	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
Trichlorofluoromethane	75-69-4	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
Acetone	67-64-1	N.D.	1200	N.D.	950	N.D.	990	N.D.	2100	N.D.	1200
Diethyl ether	60-29-7	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
1,1-Dichloroethylene	75-35-4	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
Methyl iodide	74-88-4	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
Acrylonitrile	107-13-1	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
Methylene chloride	75-09-2	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
Carbon disulfide	75-15-0	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
trans-1,2-Dichloroethylene	156-60-5	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
Methyltertbutylether (MTBE)	1634-04-4	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
1,1-Dichloroethane	75-34-3	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
2-Butanone (MEK)	78-93-3	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
cis-1,2-Dichloroethylene	156-59-2	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
Chloroform	67-66-3	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
Bromochloromethane	74-97-5	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
1,1,1-Trichloroethane	71-55-6	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
1,2-Dichloroethane	107-06-2	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
Benzene	71-43-2	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
Carbon tetrachloride	56-23-5	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
1,2-Dichloropropane	78-87-5	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
Trichloroethylene	79-01-6	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
Dibromomethane	74-95-3	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
Bromodichloromethane	75-27-4	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
4-Methyl-2-pentanone (MIBK)	108-10-1	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
cis-1,3-Dichloropropene	10061-01-5	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
trans-1,3-Dichloropropene	10061-02-6	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
Toluene	108-88-3	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
1,1,2-Trichloroethane	79-00-5	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
2-Hexanone	591-78-6	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
Dibromochloromethane	124-48-1	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
1,2-Dibromoethane	106-93-4	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
Tetrachloroethene	127-18-4	N.D.	77	95	64	N.D.	66	N.D.	140	N.D.	77
Chlorobenzene	108-90-7	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
1,1,1,2-Tetrachloroethane	630-20-6	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
Ethylbenzene	100-41-4	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77

All data in ug/Kg

TABLE 5

		TC-C*		TC-UC		TC-UC*		TD-C		TD-UC	
VOLATILE ORGANICS	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
m & p-Xylene	108,383,106,423	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
Bromoform	75-25-2	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
Styrene	100-42-5	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
o-Xylene	95-47-6	N.D.	77	N.D.	64	N.D.	66	N.D.	140	N.D.	77
1,1,2,2-Tetrachloroethane	79-34-5	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
1,2,3-Trichloropropane	96-18-4	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
trans-1,4-Dichloro-2-butene	110-57-6	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
Isopropylbenzene	98-82-8	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
Bromobenzene	108-86-1	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
n-Propylbenzene	103-65-1	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
1,3,5-Trimethylbenzene	108-67-8	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
tert-Butylbenzene	98-06-6	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
1,2,4-Trimethylbenzene	95-63-6	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
sec-Butylbenzene	135-98-8	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
1,3-Dichlorobenzene	541-73-1	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
1,4-Dichlorobenzene	106-46-7	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
p-Isopropyl toluene	99-87-6	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
1,2-Dichlorobenzene	95-50-1	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
n-Butylbenzene	104-51-8	N.D.	380	N.D.	320	N.D.	330	N.D.	710	N.D.	390
Hexachloroethane	67-72-1	N.D.	150	N.D.	130	N.D.	130	N.D.	280	N.D.	150
1,2-Dibromo-3-chloropropane	96-12-8	N.D.	38	N.D.	320	N.D.	330	N.D.	710	N.D.	390
1,2,4-Trichlorobenzene	120-82-1	N.D.	38	N.D.	320	N.D.	330	N.D.	710	N.D.	390
Naphthalene	91-20-3	N.D.	38	N.D.	320	N.D.	330	N.D.	710	N.D.	390
1,2,3-Trichlorobenzene	87-61-6	N.D.	38	N.D.	320	N.D.	330	N.D.	710	N.D.	390
2-Methylnaphthalene	91-57-6	N.D.	38	N.D.	320	N.D.	330	N.D.	710	N.D.	390

All data in ug/Kg

TABLE 5

Date collected		9/5/01		9/6/01		10/19/01		10/19/01		10/19/01	
		TE-C		TE-UC		TF-C		TF-CR		TF-UC	
VOLATILE ORGANICS	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
Dichlorodifluoromethane	75-71-8	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
Chloromethane	74-87-3	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
Vinyl chloride	75-01-4	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Bromomethane	74-83-9	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
Chloroethane	75-00-3	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
Trichlorofluoromethane	75-69-4	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
Acetone	67-64-1	N.D.	1100	N.D.	1000	N.D.	1200	N.D.	1200	N.D.	1000
Diethyl ether	60-29-7	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
1,1-Dichloroethylene	75-35-4	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Methyl iodide	74-88-4	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
Acrylonitrile	107-13-1	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
Methylene chloride	75-09-2	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
Carbon disulfide	75-15-0	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
trans-1,2-Dichloroethylene	156-60-5	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Methyltertbutylether (MTBE)	1634-04-4	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
1,1-Dichloroethane	75-34-3	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
2-Butanone (MEK)	78-93-3	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
cis-1,2-Dichloroethylene	156-59-2	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Chloroform	67-66-3	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Bromochloromethane	74-97-5	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
1,1,1-Trichloroethane	71-55-6	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
1,2-Dichloroethane	107-06-2	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Benzene	71-43-2	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Carbon tetrachloride	56-23-5	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
1,2-Dichloropropane	78-87-5	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Trichloroethylene	79-01-6	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Dibromomethane	74-95-3	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
Bromodichloromethane	75-27-4	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
4-Methyl-2-pentanone (MIBK)	108-10-1	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
cis-1,3-Dichloropropene	10061-01-5	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
trans-1,3-Dichloropropene	10061-02-6	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Toluene	108-88-3	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
1,1,2-Trichloroethane	79-00-5	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
2-Hexanone	591-78-6	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
Dibromochloromethane	124-48-1	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
1,2-Dibromoethane	106-93-4	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Tetrachloroethene	127-18-4	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
Chlorobenzene	108-90-7	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
1,1,1,2-Tetrachloroethane	630-20-6	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
Ethylbenzene	100-41-4	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68

All data in ug/Kg

TABLE 5

		TE-C		TE-UC		TF-C		TF-CR		TF-UC	
VOLATILE ORGANICS	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
m & p-Xylene	108,383,106,423	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
Bromoform	75-25-2	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
Styrene	100-42-5	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
o-Xylene	95-47-6	N.D.	75	N.D.	68	N.D.	78	N.D.	80	N.D.	68
1,1,2,2-Tetrachloroethane	79-34-5	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
1,2,3-Trichloropropane	96-18-4	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
trans-1,4-Dichloro-2-butene	110-57-6	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
Isopropylbenzene	98-82-8	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
Bromobenzene	108-86-1	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
n-Propylbenzene	103-65-1	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
1,3,5-Trimethylbenzene	108-67-8	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
tert-Butylbenzene	98-06-6	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
1,2,4-Trimethylbenzene	95-63-6	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
sec-Butylbenzene	135-98-8	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
1,3-Dichlorobenzene	541-73-1	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
1,4-Dichlorobenzene	106-46-7	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
p-Isopropyl toluene	99-87-6	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
1,2-Dichlorobenzene	95-50-1	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
n-Butylbenzene	104-51-8	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
Hexachloroethane	67-72-1	N.D.	150	N.D.	140	N.D.	160	N.D.	160	N.D.	140
1,2-Dibromo-3-chloropropane	96-12-8	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
1,2,4-Trichlorobenzene	120-82-1	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
Naphthalene	91-20-3	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
1,2,3-Trichlorobenzene	87-61-6	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340
2-Methylnaphthalene	91-57-6	N.D.	370	N.D.	340	N.D.	390	N.D.	400	N.D.	340

TABLE 6

Date Collected		08/30/01		08/30/01		08/31/01		08/31/01		09/04/01		11/15/01	
		TA-C		TA-UC		TB-C		TB-UC		TC-C		TC-C*	
SEMI-VOLATILE	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
N-Nitrosodiphenylamine	156-10-5	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
Phenol	108-95-2	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
Bis(2-chloroethyl)ether	111-44-4	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
2-Chlorophenol	95-57-8	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
1,3-Dichlorobenzene	541-73-1	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
1,4-Dichlorobenzene	106-46-7	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
1,2-Dichlorobenzene	95-50-1	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
2-Methylphenol (o-cresol)	95-48-7	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
Bis(2-chloroisopropyl)ether	108-60-1	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
3/4-Methylphenol (m/p-cresol)	108,394,106,445	N.D.	800	N.D.	820	N.D.	820	N.D.	850	N.D.	810	N.D.	800
N-Nitrosodi-n-propylamine	621-64-7	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Hexachloroethane	67-72-1	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
Nitrobenzene	98-95-3	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Isophorone	78-59-1	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
2-Nitrophenol	88-75-5	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
2,4-Dimethylphenol	105-67-9	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
Bis(2-chloroethoxy)methane	111-91-1	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
2,4-Dichlorophenol	120-83-2	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
1,2,4-Trichlorobenzene	120-82-1	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Naphthalene	91-20-3	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
Hexachlorobutadiene	87-68-3	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
4-Chloro-3-methylphenol	59-50-7	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
2-Methylnaphthalene	91-57-6	N.D.	300	N.D.	310	N.D.	310	N.D.	320	N.D.	310	N.D.	300
Hexachlorocyclopentadiene	77-47-4	N.D.	2400	N.D.	2500	N.D.	2500	N.D.	2600	N.D.	2500	N.D.	2400
2,4,6-Trichlorophenol	88-06-2	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
2,4,5-Trichlorophenol	95-95-4	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
2-Chloronaphthalene	91-58-7	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
2-Nitroaniline	88-74-4	N.D.	2100	N.D.	2100	N.D.	2100	N.D.	2200	N.D.	2100	N.D.	2100
Acenaphthylene	208-96-8	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
Dimethyl phthalate	131-11-3	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
2,6-Dinitrotoluene	606-20-2	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
3-Nitroaniline	99-09-2	N.D.	2100	N.D.	2100	N.D.	2100	N.D.	2200	N.D.	2100	N.D.	2100
Acenaphthene	83-32-9	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
2,4-Dinitrophenol	51-28-5	N.D.	2100	N.D.	2100	N.D.	2100	N.D.	2200	N.D.	2100	N.D.	2100
Dibenzofuran	132-64-9	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
4-Nitrophenol	100-02-7	N.D.	2100	N.D.	2100	N.D.	2100	N.D.	2200	N.D.	2100	N.D.	2100
2,4-Dinitrotoluene	121-14-2	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
Fluorene	86-73-7	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
Diethyl phthalate	84-66-2	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
4-Nitroaniline	100-01-6	N.D.	2100	N.D.	2100	N.D.	2100	N.D.	2200	N.D.	2100	N.D.	2100

All data in ug/Kg

TABLE 6

		TA-C		TA-UC		TB-C		TB-UC		TC-C		TC-C*	
SEMI-VOLATILE	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
2-Methyl-4,6-dinitrophenol	534-52-1	N.D.	2100	N.D.	2100	N.D.	2100	N.D.	2200	N.D.	2100	N.D.	2100
4-Chlorophenyl phenylether	7005-72-3	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
N-Nitrosodimethylamine	62-75-9	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Azobenzene	103-33-3	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
4-Bromophenyl phenylether	101-55-3	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Hexachlorobenzene	118-74-1	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Pentachlorophenol	87-86-5	N.D.	4100	N.D.	4200	N.D.	4200	N.D.	4400	N.D.	4200	N.D.	4100
Phenanthrene	85-01-8	N.D.	120	N.D.	120	N.D.	120	N.D.	130	150	120	N.D.	120
Anthracene	120-12-7	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
Carbazole	86-74-8	N.D.	400	N.D.	410	N.D.	410	N.D.	420	N.D.	410	N.D.	400
Di-n-butyl phthalate	84-74-2	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
Fluoranthene	206-44-0	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
Pyrene	129-00-0	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
Butyl benzyl phthalate	85-68-7	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
Benz(a)anthracene	56-55-3	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
Chrysene	218-01-9	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120
Bis(2-ethylhexyl)phthalate	117-81-7	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Di-n-octyl phthalate	117-84-0	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Benzo(b)fluoranthene	205-99-2	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Benzo(k)fluoranthene	207-08-9	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Benzo(a)pyrene	50-32-8	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Indeno(1,2,3-cd)pyrene	193-39-5	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Dibenz(a,h)anthracene	53-70-3	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240
Benzo(g,h,i)perylene	191-24-2	N.D.	240	N.D.	250	N.D.	250	N.D.	260	N.D.	250	N.D.	240

All data in ug/Kg

TABLE 6

Date Collected		09/04/01		11/15/01		09/05/01		09/05/01		09/05/01		09/06/01	
		TC-UC		TC-UC*		TD-C		TD-UC		TE-C		TE-UC	
SEMI-VOLATILE	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
N-Nitrosodiphenylamine	156-10-5	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
Phenol	108-95-2	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
Bis(2-chloroethyl)ether	111-44-4	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
2-Chlorophenol	95-57-8	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
1,3-Dichlorobenzene	541-73-1	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
1,4-Dichlorobenzene	106-46-7	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
1,2-Dichlorobenzene	95-50-1	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
2-Methylphenol (o-cresol)	95-48-7	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
Bis(2-chloroisopropyl)ether	108-60-1	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
3/4-Methylphenol (m/p-cresol)	108,394,106,445	N.D.	820	N.D.	850	N.D.	840	N.D.	850	N.D.	990	N.D.	860
N-Nitrosodi-n-propylamine	621-64-7	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Hexachloroethane	67-72-1	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
Nitrobenzene	98-95-3	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Isophorone	78-59-1	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
2-Nitrophenol	88-75-5	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
2,4-Dimethylphenol	105-67-9	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
Bis(2-chloroethoxy)methane	111-91-1	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
2,4-Dichlorophenol	120-83-2	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
1,2,4-Trichlorobenzene	120-82-1	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Naphthalene	91-20-3	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
Hexachlorobutadiene	87-68-3	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
4-Chloro-3-methylphenol	59-50-7	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
2-Methylnaphthalene	91-57-6	N.D.	310	N.D.	320	N.D.	320	N.D.	320	N.D.	370	N.D.	320
Hexachlorocyclopentadiene	77-47-4	N.D.	2500	N.D.	2600	N.D.	2500	N.D.	2600	N.D.	3000	N.D.	2600
2,4,6-Trichlorophenol	88-06-2	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
2,4,5-Trichlorophenol	95-95-4	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
2-Chloronaphthalene	91-58-7	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
2-Nitroaniline	88-74-4	N.D.	2100	N.D.	2200	N.D.	2200	N.D.	2200	N.D.	2500	N.D.	2200
Acenaphthylene	208-96-8	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
Dimethyl phthalate	131-11-3	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
2,6-Dinitrotoluene	606-20-2	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
3-Nitroaniline	99-09-2	N.D.	2100	N.D.	2200	N.D.	2200	N.D.	2200	N.D.	2500	N.D.	2200
Acenaphthene	83-32-9	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
2,4-Dinitrophenol	51-28-5	N.D.	2100	N.D.	2200	N.D.	2200	N.D.	2200	N.D.	2500	N.D.	2200
Dibenzofuran	132-64-9	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
4-Nitrophenol	100-02-7	N.D.	2100	N.D.	2200	N.D.	2200	N.D.	2200	N.D.	2500	N.D.	2200
2,4-Dinitrotoluene	121-14-2	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
Fluorene	86-73-7	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
Diethyl phthalate	84-66-2	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
4-Nitroaniline	100-01-6	N.D.	2100	N.D.	2200	N.D.	2200	N.D.	2200	N.D.	2500	N.D.	2200

All data in ug/Kg

TABLE 6

		TC-UC		TC-UC*		TD-C		TD-UC		TE-C		TE-UC	
SEMI-VOLATILE	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
2-Methyl-4,6-dinitrophenol	534-52-1	N.D.	2100	N.D.	2200	N.D.	2200	N.D.	2200	N.D.	2500	N.D.	2200
4-Chlorophenyl phenylether	7005-72-3	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
N-Nitrosodimethylamine	62-75-9	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Azobenzene	103-33-3	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
4-Bromophenyl phenylether	101-55-3	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Hexachlorobenzene	118-74-1	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Pentachlorophenol	87-86-5	N.D.	4200	N.D.	4400	N.D.	4300	N.D.	4400	N.D.	5100	N.D.	4400
Phenanthrene	85-01-8	N.D.	120	N.D.	130	N.D.	130	N.D.	130	230	150	N.D.	130
Anthracene	120-12-7	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
Carbazole	86-74-8	N.D.	410	N.D.	420	N.D.	420	N.D.	420	N.D.	490	N.D.	430
Di-n-butyl phthalate	84-74-2	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
Fluoranthene	206-44-0	N.D.	120	N.D.	130	N.D.	130	N.D.	130	370	150	N.D.	130
Pyrene	129-00-0	N.D.	120	N.D.	130	N.D.	130	N.D.	130	280	150	N.D.	130
Butyl benzyl phthalate	85-68-7	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
Benz(a)anthracene	56-55-3	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130
Chrysene	218-01-9	N.D.	120	N.D.	130	N.D.	130	N.D.	130	160	150	N.D.	130
Bis(2-ethylhexyl)phthalate	117-81-7	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Di-n-octyl phthalate	117-84-0	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Benzo(b)fluoranthene	205-99-2	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Benzo(k)fluoranthene	207-08-9	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Benzo(a)pyrene	50-32-8	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Indeno(1,2,3-cd)pyrene	193-39-5	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Dibenz(a,h)anthracene	53-70-3	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260
Benzo(g,h,i)perylene	191-24-2	N.D.	250	N.D.	260	N.D.	250	N.D.	260	N.D.	300	N.D.	260

TABLE 6

Date Collected		10/19/01		10/19/01		10/19/01		11/01/01		11/02/01		11/02/
		TF-C		TF-CR		TF-UC		TG-UC		TH-C		TH-U
SEMI-VOLATILE	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**
												N.D.
N-Nitrosodiphenylamine	156-10-5	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
Phenol	108-95-2	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
Bis(2-chloroethyl)ether	111-44-4	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
2-Chlorophenol	95-57-8	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
1,3-Dichlorobenzene	541-73-1	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
1,4-Dichlorobenzene	106-46-7	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
1,2-Dichlorobenzene	95-50-1	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
2-Methylphenol (o-cresol)	95-48-7	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
Bis(2-chloroisopropyl)ether	108-60-1	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
3/4-Methylphenol (m/p-cresol)	108,394,106,445	N.D.	940	N.D.	940	N.D.	920	N.D.	870	N.D.	1000	N.D.
N-Nitrosodi-n-propylamine	621-64-7	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Hexachloroethane	67-72-1	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
Nitrobenzene	98-95-3	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Isophorone	78-59-1	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
2-Nitrophenol	88-75-5	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
2,4-Dimethylphenol	105-67-9	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
Bis(2-chloroethoxy)methane	111-91-1	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
2,4-Dichlorophenol	120-83-2	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
1,2,4-Trichlorobenzene	120-82-1	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Naphthalene	91-20-3	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
Hexachlorobutadiene	87-68-3	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
4-Chloro-3-methylphenol	59-50-7	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
2-Methylnaphthalene	91-57-6	N.D.	360	N.D.	360	N.D.	350	N.D.	330	N.D.	380	N.D.
Hexachlorocyclopentadiene	77-47-4	N.D.	2900	N.D.	2900	N.D.	2800	N.D.	2600	N.D.	3100	N.D.
2,4,6-Trichlorophenol	88-06-2	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
2,4,5-Trichlorophenol	95-95-4	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
2-Chloronaphthalene	91-58-7	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
2-Nitroaniline	88-74-4	N.D.	2400	N.D.	2400	N.D.	2400	N.D.	2200	N.D.	2600	N.D.
Acenaphthylene	208-96-8	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
Dimethyl phthalate	131-11-3	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
2,6-Dinitrotoluene	606-20-2	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
3-Nitroaniline	99-09-2	N.D.	2400	N.D.	2400	N.D.	2400	N.D.	2200	N.D.	2600	N.D.
Acenaphthene	83-32-9	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
2,4-Dinitrophenol	51-28-5	N.D.	2400	N.D.	2400	N.D.	2400	N.D.	2200	N.D.	2600	N.D.
Dibenzofuran	132-64-9	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
4-Nitrophenol	100-02-7	N.D.	2400	N.D.	2400	N.D.	2400	N.D.	2200	N.D.	2600	N.D.
2,4-Dinitrotoluene	121-14-2	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
Fluorene	86-73-7	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
Diethyl phthalate	84-66-2	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
4-Nitroaniline	100-01-6	N.D.	2400	N.D.	2400	N.D.	2400	N.D.	2200	N.D.	2600	N.D.

All data in ug/Kg

TABLE 6

		TF-C		TF-CR		TF-UC		TG-UC		TH-C		TH-U
SEMI-VOLATILE	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**
2-Methyl-4,6-dinitrophenol	534-52-1	N.D.	2400	N.D.	2400	N.D.	2400	N.D.	2200	N.D.	2600	N.D.
4-Chlorophenyl phenylether	7005-72-3	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
N-Nitrosodimethylamine	62-75-9	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Azobenzene	103-33-3	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
4-Bromophenyl phenylether	101-55-3	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Hexachlorobenzene	118-74-1	N.D.	290	1300	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Pentachlorophenol	87-86-5	N.D.	4900	N.D.	4900	N.D.	4700	N.D.	4500	N.D.	5200	N.D.
Phenanthrene	85-01-8	210	140	240	140	140	140	N.D.	130	N.D.	150	N.D.
Anthracene	120-12-7	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
Carbazole	86-74-8	N.D.	470	N.D.	470	N.D.	460	N.D.	430	N.D.	510	N.D.
Di-n-butyl phthalate	84-74-2	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
Fluoranthene	206-44-0	330	140	390	140	240	140	220	130	N.D.	150	N.D.
Pyrene	129-00-0	290	140	350	140	230	140	150	130	N.D.	150	N.D.
Butyl benzyl phthalate	85-68-7	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
Benz(a)anthracene	56-55-3	N.D.	140	N.D.	140	N.D.	140	N.D.	130	N.D.	150	N.D.
Chrysene	218-01-9	170	140	210	140	140	140	N.D.	130	N.D.	150	N.D.
Bis(2-ethylhexyl)phthalate	117-81-7	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Di-n-octyl phthalate	117-84-0	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Benzo(b)fluoranthene	205-99-2	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Benzo(k)fluoranthene	207-08-9	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Benzo(a)pyrene	50-32-8	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Indeno(1,2,3-cd)pyrene	193-39-5	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Dibenz(a,h)anthracene	53-70-3	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.
Benzo(g,h,i)perylene	191-24-2	N.D.	290	N.D.	290	N.D.	280	N.D.	260	N.D.	310	N.D.

All data in ug/Kg

TABLE 6

Date Collected		01	11/02/01		11/02/01		11/15/01		11/15/01		12/04/01	
		C	TI-C		TI-UC		SS-1		SS-2		SS-3	
SEMI-VOLATILE	**CAS #**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
		310	N.D	520	N.D	570						
N-Nitrosodiphenylamine	156-10-5	310	N.D	520	N.D	570	N.D.	560	N.D.	480	N.D.	460
Phenol	108-95-2	62	N.D	160	N.D	170	N.D.	560	N.D.	480	N.D.	460
Bis(2-chloroethyl)ether	111-44-4	310	N.D	520	N.D	570	N.D.	170	N.D.	140	N.D.	140
2-Chlorophenol	95-57-8	310	N.D	160	N.D	170	N.D.	560	N.D.	480	N.D.	460
1,3-Dichlorobenzene	541-73-1	310	N.D	160	N.D	170	N.D.	170	N.D.	140	N.D.	140
1,4-Dichlorobenzene	106-46-7	930	N.D	160	N.D	170	N.D.	170	N.D.	140	N.D.	140
1,2-Dichlorobenzene	95-50-1	310	N.D	520	N.D	170	N.D.	170	N.D.	140	N.D.	140
2-Methylphenol (o-cresol)	95-48-7	62	N.D	160	N.D	570	N.D.	560	N.D.	480	N.D.	460
Bis(2-chloroisopropyl)ether	108-60-1	120	N.D	1000	N.D	170	N.D.	170	N.D.	140	N.D.	140
3/4-Methylphenol (m/p-cresol)	108,394,106,445	310	N.D	320	N.D	1100	N.D.	1100	N.D.	960	N.D.	930
N-Nitrosodi-n-propylamine	621-64-7	310	N.D	160	N.D	340	N.D.	340	N.D.	290	N.D.	280
Hexachloroethane	67-72-1	310	N.D	320	N.D	170	N.D.	170	N.D.	140	N.D.	140
Nitrobenzene	98-95-3	62	N.D	160	N.D	340	N.D.	340	N.D.	290	N.D.	280
Isophorone	78-59-1	310	N.D	520	N.D	170	N.D.	170	N.D.	140	N.D.	140
2-Nitrophenol	88-75-5	62	N.D	520	N.D	570	N.D.	560	N.D.	480	N.D.	460
2,4-Dimethylphenol	105-67-9	310	N.D	320	N.D	570	N.D.	560	N.D.	480	N.D.	460
Bis(2-chloroethoxy)methane	111-91-1	62	N.D	520	N.D	340	N.D.	340	N.D.	290	N.D.	280
2,4-Dichlorophenol	120-83-2	62	N.D	320	N.D	570	N.D.	560	N.D.	480	N.D.	460
1,2,4-Trichlorobenzene	120-82-1	120	N.D	160	N.D	340	N.D.	340	N.D.	290	N.D.	280
Naphthalene	91-20-3	62	N.D	320	N.D	170	N.D.	170	N.D.	140	N.D.	140
Hexachlorobutadiene	87-68-3	62	N.D	520	N.D	340	N.D.	340	N.D.	290	N.D.	280
4-Chloro-3-methylphenol	59-50-7	62	N.D	400	N.D	570	N.D.	560	N.D.	480	N.D.	460
2-Methylnaphthalene	91-57-6	62	N.D	3200	N.D	430	N.D.	420	N.D.	360	N.D.	350
Hexachlorocyclopentadiene	77-47-4	62	N.D	520	N.D	3400	N.D.	3400	N.D.	2900	N.D.	2800
2,4,6-Trichlorophenol	88-06-2	62	N.D	520	N.D	570	N.D.	560	N.D.	480	N.D.	460
2,4,5-Trichlorophenol	95-95-4	120	N.D	320	N.D	570	N.D.	560	N.D.	480	N.D.	460
2-Chloronaphthalene	91-58-7	120	N.D	2700	N.D	340	N.D.	340	N.D.	290	N.D.	280
2-Nitroaniline	88-74-4	310	N.D	160	N.D	2900	N.D.	2900	N.D.	2500	N.D.	2400
Acenaphthylene	208-96-8	62	N.D	320	N.D	170	N.D.	170	N.D.	140	N.D.	140
Dimethyl phthalate	131-11-3	62	N.D	520	N.D	340	N.D.	340	N.D.	290	N.D.	280
2,6-Dinitrotoluene	606-20-2	62	N.D	2700	N.D	570	N.D.	560	N.D.	480	N.D.	460
3-Nitroaniline	99-09-2	62	N.D	160	N.D	2900	N.D.	2900	N.D.	2500	N.D.	2400
Acenaphthene	83-32-9	310	N.D	2700	N.D	170	N.D.	170	N.D.	140	N.D.	140
2,4-Dinitrophenol	51-28-5	120	N.D	520	N.D	2900	N.D.	2900	N.D.	2500	N.D.	2400
Dibenzofuran	132-64-9	62	N.D	2700	N.D	570	N.D.	560	N.D.	480	N.D.	460
4-Nitrophenol	100-02-7	62	N.D	520	N.D	2900	N.D.	2900	N.D.	2500	N.D.	2400
2,4-Dinitrotoluene	121-14-2	62	N.D	160	N.D	570	N.D.	560	N.D.	480	N.D.	460
Fluorene	86-73-7	120	N.D	160	N.D	170	N.D.	170	N.D.	140	N.D.	140
Diethyl phthalate	84-66-2	62	N.D	2700	N.D	170	N.D.	170	N.D.	140	N.D.	140
4-Nitroaniline	100-01-6	120	N.D	2700	N.D	2900	N.D.	2900	N.D.	2500	N.D.	2400

All data in ug/Kg

TABLE 6

		C	TI-C		TI-UC		SS-1		SS-2		SS-3	
SEMI-VOLATILE	**CAS #**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
2-Methyl-4,6-dinitrophenol	534-52-1	120	N.D	160	N.D	2900	N.D.	2900	N.D.	2500	N.D.	2400
4-Chlorophenyl phenylether	7005-72-3	62	N.D	320	N.D	340	N.D.	170	N.D.	140	N.D.	140
N-Nitrosodimethylamine	62-75-9	62	N.D	320	N.D	340	N.D.	340	N.D.	290	N.D.	280
Azobenzene	103-33-3	120	N.D	320	N.D	340	N.D.	340	N.D.	290	N.D.	280
4-Bromophenyl phenylether	101-55-3	120	N.D	320	N.D	340	N.D.	340	N.D.	290	N.D.	280
Hexachlorobenzene	118-74-1	120	N.D	5400	N.D	340	N.D.	340	N.D.	290	N.D.	280
Pentachlorophenol	87-86-5	120	N.D	160	N.D	5900	N.D.	5800	N.D.	4900	N.D.	4800
Phenanthrene	85-01-8	120	N.D	160	N.D	170	N.D.	170	N.D.	140	N.D.	140
Anthracene	120-12-7	120	N.D	520	N.D	570	N.D.	170	N.D.	140	N.D.	140
Carbazole	86-74-8	120	N.D	160	N.D	170	N.D.	560	N.D.	480	N.D.	460
Di-n-butyl phthalate	84-74-2	310	N.D	160	N.D	170	N.D.	170	N.D.	140	N.D.	140
Fluoranthene	206-44-0	120	190	160	N.D	170	N.D.	170	170	140	170	140
Pyrene	129-00-0	310	N.D	160	N.D	170	N.D.	170	N.D.	140	N.D.	140
Butyl benzyl phthalate	85-68-7	120	N.D	160	N.D	170	N.D.	170	N.D.	140	N.D.	140
Benz(a)anthracene	56-55-3	120	N.D	160	N.D	170	N.D.	170	N.D.	140	N.D.	140
Chrysene	218-01-9	310	N.D	160	N.D	170	N.D.	170	N.D.	140	N.D.	140
Bis(2-ethylhexyl)phthalate	117-81-7	120	N.D	320	N.D	340	N.D.	340	N.D.	290	N.D.	280
Di-n-octyl phthalate	117-84-0	310	N.D	320	N.D	340	N.D.	340	N.D.	290	N.D.	280
Benzo(b)fluoranthene	205-99-2	120	N.D	320	N.D	340	N.D.	340	N.D.	290	N.D.	280
Benzo(k)fluoranthene	207-08-9	310	N.D	320	N.D	340	N.D.	340	N.D.	290	N.D.	280
Benzo(a)pyrene	50-32-8	310	N.D	320	N.D	340	N.D.	340	N.D.	290	N.D.	280
Indeno(1,2,3-cd)pyrene	193-39-5	310	N.D	320	N.D	340	N.D.	340	N.D.	290	N.D.	280
Dibenz(a,h)anthracene	53-70-3	310	N.D	320	N.D	340	N.D.	340	N.D.	290	N.D.	280
Benzo(g,h,i)perylene	191-24-2	310	N.D	320	N.D	340	N.D.	340	N.D.	290	N.D.	280

All data in ug/Kg

TABLE 6

Date Collected		12/04/01		12/04/01		12/04/01		12/04/01		12/04/01		12/04/01
		SS-4		SS-5		SS-6		SS-7		SS-7R		SS
SEMI-VOLATILE	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**
N-Nitrosodiphenylamine	156-10-5	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
Phenol	108-95-2	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
Bis(2-chloroethyl)ether	111-44-4	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
2-Chlorophenol	95-57-8	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
1,3-Dichlorobenzene	541-73-1	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
1,4-Dichlorobenzene	106-46-7	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
1,2-Dichlorobenzene	95-50-1	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
2-Methylphenol (o-cresol)	95-48-7	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
Bis(2-chloroisopropyl)ether	108-60-1	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
3/4-Methylphenol (m/p-cresol)	108,394,106,445	N.D.	940	N.D.	850	N.D.	790	N.D.	880	N.D.	870	N.D.
N-Nitrosodi-n-propylamine	621-64-7	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Hexachloroethane	67-72-1	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
Nitrobenzene	98-95-3	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Isophorone	78-59-1	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
2-Nitrophenol	88-75-5	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
2,4-Dimethylphenol	105-67-9	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
Bis(2-chloroethoxy)methane	111-91-1	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
2,4-Dichlorophenol	120-83-2	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
1,2,4-Trichlorobenzene	120-82-1	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Naphthalene	91-20-3	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
Hexachlorobutadiene	87-68-3	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
4-Chloro-3-methylphenol	59-50-7	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
2-Methylnaphthalene	91-57-6	N.D.	360	N.D.	320	N.D.	300	N.D.	330	N.D.	330	N.D.
Hexachlorocyclopentadiene	77-47-4	N.D.	2900	N.D.	2600	N.D.	2400	N.D.	2700	N.D.	2600	N.D.
2,4,6-Trichlorophenol	88-06-2	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
2,4,5-Trichlorophenol	95-95-4	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
2-Chloronaphthalene	91-58-7	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
2-Nitroaniline	88-74-4	N.D.	2400	N.D.	2200	N.D.	2000	N.D.	2300	N.D.	2200	N.D.
Acenaphthylene	208-96-8	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
Dimethyl phthalate	131-11-3	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
2,6-Dinitrotoluene	606-20-2	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
3-Nitroaniline	99-09-2	N.D.	2400	N.D.	2200	N.D.	2000	N.D.	2300	N.D.	2200	N.D.
Acenaphthene	83-32-9	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
2,4-Dinitrophenol	51-28-5	N.D.	2400	N.D.	2200	N.D.	2000	N.D.	2300	N.D.	2200	N.D.
Dibenzofuran	132-64-9	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
4-Nitrophenol	100-02-7	N.D.	2400	N.D.	2200	N.D.	2000	N.D.	2300	N.D.	2200	N.D.
2,4-Dinitrotoluene	121-14-2	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
Fluorene	86-73-7	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
Diethyl phthalate	84-66-2	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
4-Nitroaniline	100-01-6	N.D.	2400	N.D.	2200	N.D.	2000	N.D.	2300	N.D.	2200	N.D.

All data in ug/Kg

TABLE 6

		SS-4		SS-5		SS-6		SS-7		SS-7R		SS
SEMI-VOLATILE	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**
2-Methyl-4,6-dinitrophenol	534-52-1	N.D.	2400	N.D.	2200	N.D.	2000	N.D.	2300	N.D.	2200	N.D.
4-Chlorophenyl phenylether	7005-72-3	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
N-Nitrosodimethylamine	62-75-9	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Azobenzene	103-33-3	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
4-Bromophenyl phenylether	101-55-3	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Hexachlorobenzene	118-74-1	N.D.	290	280	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Pentachlorophenol	87-86-5	N.D.	4900	N.D.	4400	N.D.	4000	N.D.	4500	N.D.	4500	N.D.
Phenanthrene	85-01-8	N.D.	140	N.D.	130	N.D.	120	N.D.	130	350	130	N.D.
Anthracene	120-12-7	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
Carbazole	86-74-8	N.D.	470	N.D.	420	N.D.	390	N.D.	440	N.D.	430	N.D.
Di-n-butyl phthalate	84-74-2	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
Fluoranthene	206-44-0	N.D.	140	130	130	N.D.	120	170	130	510	130	170
Pyrene	129-00-0	N.D.	140	N.D.	130	N.D.	120	130	130	380	130	N.D.
Butyl benzyl phthalate	85-68-7	N.D.	140	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.
Benz(a)anthracene	56-55-3	N.D.	140	N.D.	130	N.D.	120	N.D.	130	190	130	N.D.
Chrysene	218-01-9	N.D.	140	N.D.	130	N.D.	120	N.D.	130	230	130	N.D.
Bis(2-ethylhexyl)phthalate	117-81-7	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Di-n-octyl phthalate	117-84-0	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Benzo(b)fluoranthene	205-99-2	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Benzo(k)fluoranthene	207-08-9	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Benzo(a)pyrene	50-32-8	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Indeno(1,2,3-cd)pyrene	193-39-5	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Dibenz(a,h)anthracene	53-70-3	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.
Benzo(g,h,i)perylene	191-24-2	N.D.	290	N.D.	260	N.D.	240	N.D.	270	N.D.	260	N.D.

All data in ug/Kg

TABLE 6

Date Collected			12/04/01		12/04/01	
		-8	SS-9		SS-10	
SEMI-VOLATILE	**CAS #**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
N-Nitrosodiphenylamine	156-10-5	420	N.D.	410	N.D.	440
Phenol	108-95-2	420	N.D.	410	N.D.	440
Bis(2-chloroethyl)ether	111-44-4	130	N.D.	120	N.D.	130
2-Chlorophenol	95-57-8	420	N.D.	410	N.D.	440
1,3-Dichlorobenzene	541-73-1	130	N.D.	120	N.D.	130
1,4-Dichlorobenzene	106-46-7	130	N.D.	120	N.D.	130
1,2-Dichlorobenzene	95-50-1	130	N.D.	120	N.D.	130
2-Methylphenol (o-cresol)	95-48-7	420	N.D.	410	N.D.	440
Bis(2-chloroisopropyl)ether	108-60-1	130	N.D.	120	N.D.	130
3/4-Methylphenol (m/p-cresol)	108,394,106,445	850	N.D.	810	N.D.	880
N-Nitrosodi-n-propylamine	621-64-7	260	N.D.	250	N.D.	270
Hexachloroethane	67-72-1	130	N.D.	120	N.D.	130
Nitrobenzene	98-95-3	260	N.D.	250	N.D.	270
Isophorone	78-59-1	130	N.D.	120	N.D.	130
2-Nitrophenol	88-75-5	420	N.D.	410	N.D.	440
2,4-Dimethylphenol	105-67-9	420	N.D.	410	N.D.	440
Bis(2-chloroethoxy)methane	111-91-1	260	N.D.	250	N.D.	270
2,4-Dichlorophenol	120-83-2	420	N.D.	410	N.D.	440
1,2,4-Trichlorobenzene	120-82-1	260	N.D.	250	N.D.	270
Naphthalene	91-20-3	130	N.D.	120	N.D.	130
Hexachlorobutadiene	87-68-3	260	N.D.	250	N.D.	270
4-Chloro-3-methylphenol	59-50-7	420	N.D.	410	N.D.	440
2-Methylnaphthalene	91-57-6	320	N.D.	310	N.D.	330
Hexachlorocyclopentadiene	77-47-4	2600	N.D.	2500	N.D.	2700
2,4,6-Trichlorophenol	88-06-2	420	N.D.	410	N.D.	440
2,4,5-Trichlorophenol	95-95-4	420	N.D.	410	N.D.	440
2-Chloronaphthalene	91-58-7	260	N.D.	250	N.D.	270
2-Nitroaniline	88-74-4	2200	N.D.	2100	N.D.	2300
Acenaphthylene	208-96-8	130	N.D.	120	N.D.	130
Dimethyl phthalate	131-11-3	260	N.D.	250	N.D.	270
2,6-Dinitrotoluene	606-20-2	420	N.D.	410	N.D.	440
3-Nitroaniline	99-09-2	220	N.D.	2100	N.D.	2300
Acenaphthene	83-32-9	130	N.D.	120	N.D.	130
2,4-Dinitrophenol	51-28-5	2200	N.D.	2100	N.D.	2300
Dibenzofuran	132-64-9	420	N.D.	410	N.D.	440
4-Nitrophenol	100-02-7	2200	N.D.	2100	N.D.	2300
2,4-Dinitrotoluene	121-14-2	420	N.D.	410	N.D.	440
Fluorene	86-73-7	130	N.D.	120	N.D.	130
Diethyl phthalate	84-66-2	130	N.D.	120	N.D.	130
4-Nitroaniline	100-01-6	2200	N.D.	2100	N.D.	2300

All data in ug/Kg

TABLE 6

		SS-8	SS-9		SS-10	
SEMI-VOLATILE	**CAS #**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
2-Methyl-4,6-dinitrophenol	534-52-1	2200	N.D.	2100	N.D.	2300
4-Chlorophenyl phenylether	7005-72-3	130	N.D.	120	N.D.	130
N-Nitrosodimethylamine	62-75-9	260	N.D.	250	N.D.	270
Azobenzene	103-33-3	260	N.D.	250	N.D.	270
4-Bromophenyl phenylether	101-55-3	260	N.D.	250	N.D.	270
Hexachlorobenzene	118-74-1	260	N.D.	250	N.D.	270
Pentachlorophenol	87-86-5	4400	N.D.	4200	N.D.	4500
Phenanthrene	85-01-8	130	N.D.	120	N.D.	130
Anthracene	120-12-7	130	N.D.	120	N.D.	130
Carbazole	86-74-8	42	N.D.	410	N.D.	440
Di-n-butyl phthalate	84-74-2	130	N.D.	120	N.D.	130
Fluoranthene	206-44-0	130	N.D.	120	N.D.	130
Pyrene	129-00-0	130	N.D.	120	N.D.	130
Butyl benzyl phthalate	85-68-7	130	N.D.	120	N.D.	130
Benz(a)anthracene	56-55-3	130	N.D.	120	N.D.	130
Chrysene	218-01-9	130	N.D.	120	N.D.	130
Bis(2-ethylhexyl)phthalate	117-81-7	260	N.D.	250	N.D.	270
Di-n-octyl phthalate	117-84-0	260	N.D.	250	N.D.	270
Benzo(b)fluoranthene	205-99-2	260	N.D.	250	N.D.	270
Benzo(k)fluoranthene	207-08-9	260	N.D.	250	N.D.	270
Benzo(a)pyrene	50-32-8	260	N.D.	250	N.D.	270
Indeno(1,2,3-cd)pyrene	193-39-5	260	N.D.	250	N.D.	270
Dibenz(a,h)anthracene	53-70-3	260	N.D.	250	N.D.	270
Benzo(g,h,i)perylene	191-24-2	260	N.D.	250	N.D.	270

All data in ug/Kg

TABLE 7

Date Collected		8/30/01		8/30/01		8/31/01		8/31/01		9/4/01		11/15/01		9/4/01	
		TA-C		TA-UC		TB-C		TB-UC		TC-C		TC-C*		TC-UC	
PESTICIDES AND PCB'S	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
a-BHC	319-84-6	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
b-BHC	319-85-7	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
g-BHC (lindane)	58-89-9	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
d-BHC	319-86-8	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
Heptachlor	76-44-8	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
Aldrin	309-00-2	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
Heptachlor epoxide	1024-57-3	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
g-Chlordane	5103-74-2	N.D.	61	N.D.	62	N.D.	62	N.D.	64	N.D.	62	N.D.	61	N.D.	62
Endosulfan I	959-98-8	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
a-Chlordane	5103-71-9	N.D.	61	N.D.	62	N.D.	62	N.D.	64	N.D.	62	N.D.	61	N.D.	62
Dieldrin	60-57-1	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
4,4'-DDE	72-55-9	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
Endrin	72-20-8	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
Endosulfan II	33213-65-9	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
4,4'-DDD	72-54-8	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
Endrin Aldehyde	7421-93-4	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
Endosulfan Sulfate	1031-07-8	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
4,4'-DDT	50-29-3	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
Endrin Ketone	53494-70-5	N.D.	24	N.D.	25	N.D.	25	N.D.	26	N.D.	25	N.D.	24	N.D.	25
Hexabromobenzene	87-82-1	N.D.	120	N.D.	120	N.D.	120	N.D.	130	N.D.	120	N.D.	120	N.D.	120
Methoxychlor	72-43-5	N.D.	61	N.D.	62	N.D.	62	N.D.	64	N.D.	62	N.D.	61	N.D.	62
Mirex	2385-85-5	N.D.	61	N.D.	62	N.D.	62	N.D.	64	N.D.	62	N.D.	61	N.D.	62
Toxaphene	8001-35-2	N.D.	210	N.D.	210	N.D.	210	N.D.	220	N.D.	210	N.D.	210	N.D.	210
BP-6 (PBB)	59080-40-9	N.D.	300	N.D.	310	N.D.	310	N.D.	320	N.D.	310	N.D.	300	N.D.	310
Aroclor 1016 (PCB)	12674-11-2	N.D.	120	N.D.	210	N.D.	120	N.D.	130	N.D.	120	N.D.	120	N.D.	120
Aroclor 1221 (PCB)	11104-28-2	N.D.	120	N.D.	210	N.D.	120	N.D.	130	N.D.	120	N.D.	120	N.D.	120
Aroclor 1232 (PCB)	11141-16-5	N.D.	120	N.D.	210	N.D.	120	N.D.	130	N.D.	120	N.D.	120	N.D.	120
Aroclor 1242 (PCB)	53469-21-9	N.D.	120	N.D.	210	N.D.	120	N.D.	130	N.D.	120	N.D.	120	N.D.	120
Aroclor 1248 (PCB)	12672-29-6	N.D.	120	N.D.	210	N.D.	120	N.D.	130	N.D.	120	N.D.	120	N.D.	120
Aroclor 1254 (PCB)	11097-69-1	N.D.	120	N.D.	210	N.D.	120	N.D.	130	N.D.	120	N.D.	120	N.D.	120
Aroclor 1260 (PCB)	11096-82-5	N.D.	120	N.D.	210	N.D.	120	N.D.	130	N.D.	120	N.D.	120	N.D.	120
Aroclor 1262 (PCB)	37324-23-5	N.D.	120	N.D.	210	N.D.	120	N.D.	130	N.D.	120	N.D.	120	N.D.	120
Aroclor 1268 (PCB)	11100-14-4	N.D.	120	N.D.	210	N.D.	120	N.D.	130	N.D.	120	N.D.	120	N.D.	120

All data in ug/Kg

TABLE 7

Date Collected		11/15/01		9/5/01		9/5/01		9/5/01		9/6/01		10/19/01		10/19/00	
		TC-UC*		TD-C		TD-UC		TE-C		TE-UC		TF-C		TF-CR	
PESTICIDES AND PCB'S	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
a-BHC	319-84-6	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
b-BHC	319-85-7	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
g-BHC (lindane)	58-89-9	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
d-BHC	319-86-8	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
Heptachlor	76-44-8	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
Aldrin	309-00-2	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
Heptachlor epoxide	1024-57-3	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
g-Chlordane	5103-74-2	N.D.	64	N.D.	63	N.D.	64	N.D.	75	N.D.	65	N.D.	71	N.D.	71
Endosulfan I	959-98-8	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
a-Chlordane	5103-71-9	N.D.	64	N.D.	63	N.D.	64	N.D.	75	N.D.	65	N.D.	71	N.D.	71
Dieldrin	60-57-1	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
4,4'-DDE	72-55-9	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
Endrin	72-20-8	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
Endosulfan II	33213-65-9	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
4,4'-DDD	72-54-8	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
Endrin Aldehyde	7421-93-4	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
Endosulfan Sulfate	1031-07-8	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
4,4'-DDT	50-29-3	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
Endrin Ketone	53494-70-5	N.D.	26	N.D.	25	N.D.	26	N.D.	30	N.D.	26	N.D.	29	N.D.	29
Hexabromobenzene	87-82-1	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130	N.D.	140	N.D.	140
Methoxychlor	72-43-5	N.D.	64	N.D.	63	N.D.	64	N.D.	75	N.D.	65	N.D.	71	N.D.	71
Mirex	2385-85-5	N.D.	64	N.D.	63	N.D.	64	N.D.	75	N.D.	65	N.D.	71	N.D.	71
Toxaphene	8001-35-2	N.D.	220	N.D.	220	N.D.	220	N.D.	250	N.D.	220	N.D.	240	N.D.	240
BP-6 (PBB)	59080-40-9	N.D.	320	N.D.	320	N.D.	320	N.D.	370	N.D.	320	N.D.	360	N.D.	360
Aroclor 1016 (PCB)	12674-11-2	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130	N.D.	140	N.D.	140
Aroclor 1221 (PCB)	11104-28-2	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130	N.D.	140	N.D.	140
Aroclor 1232 (PCB)	11141-16-5	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130	N.D.	140	N.D.	140
Aroclor 1242 (PCB)	53469-21-9	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130	N.D.	140	N.D.	140
Aroclor 1248 (PCB)	12672-29-6	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130	N.D.	140	N.D.	140
Aroclor 1254 (PCB)	11097-69-1	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130	N.D.	140	N.D.	140
Aroclor 1260 (PCB)	11096-82-5	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130	N.D.	140	N.D.	140
Aroclor 1262 (PCB)	37324-23-5	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130	N.D.	140	N.D.	140
Aroclor 1268 (PCB)	11100-14-4	N.D.	130	N.D.	130	N.D.	130	N.D.	150	N.D.	130	N.D.	140	N.D.	140

All data in ug/Kg

TABLE 7

Date Collected		10/19/01		11/1/01		11/2/01		11/2/01		11/2/01		11/2/01		11/15/01	
		TF-UC		TG-UC		TH-C		TH-UC		TI-C		TI-UC		SS-1	
PESTICIDES AND PCB'S	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
a-BHC	319-84-6	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
b-BHC	319-85-7	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
g-BHC (lindane)	58-89-9	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
d-BHC	319-86-8	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
Heptachlor	76-44-8	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
Aldrin	309-00-2	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
Heptachlor epoxide	1024-57-3	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
g-Chlordane	5103-74-2	N.D.	69	N.D	66	N.D.	77	N.D.	64	N.D.	79	N.D.	86	N.D.	85
Endosulfan I	959-98-8	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
a-Chlordane	5103-71-9	N.D.	69	N.D	66	N.D.	77	N.D.	64	N.D.	79	N.D.	86	N.D.	85
Dieldrin	60-57-1	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
4,4'-DDE	72-55-9	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
Endrin	72-20-8	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
Endosulfan II	33213-65-9	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
4,4'-DDD	72-54-8	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	83	340	N.D.	34
Endrin Aldehyde	7421-93-4	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
Endosulfan Sulfate	1031-07-8	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
4,4'-DDT	50-29-3	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	1100	340	N.D.	34
Endrin Ketone	53494-70-5	N.D.	28	N.D	26	N.D.	31	N.D.	26	N.D.	32	N.D.	34	N.D.	34
Hexabromobenzene	87-82-1	N.D.	140	N.D	130	N.D.	150	N.D.	130	N.D.	160	N.D.	170	N.D.	170
Methoxychlor	72-43-5	N.D.	69	N.D	66	N.D.	77	N.D.	64	N.D.	79	N.D.	86	N.D.	85
Mirex	2385-85-5	N.D.	69	N.D	66	N.D.	77	N.D.	64	N.D.	79	N.D.	86	N.D.	85
Toxaphene	8001-35-2	N.D.	240	N.D	330	N.D.	380	N.D.	320	N.D.	400	N.D.	430	N.D.	290
BP-6 (PBB)	59080-40-9	N.D.	350	N.D	130	N.D.	150	N.D.	130	N.D.	160	N.D.	170	N.D.	420
Aroclor 1016 (PCB)	12674-11-2	N.D.	140	N.D	130	N.D.	150	N.D.	130	N.D.	160	N.D.	170	N.D.	170
Aroclor 1221 (PCB)	11104-28-2	N.D.	140	N.D	130	N.D.	150	N.D.	130	N.D.	160	N.D.	170	N.D.	170
Aroclor 1232 (PCB)	11141-16-5	N.D.	140	N.D	130	N.D.	150	N.D.	130	N.D.	160	N.D.	170	N.D.	170
Aroclor 1242 (PCB)	53469-21-9	N.D.	140	N.D	130	N.D.	150	N.D.	130	N.D.	160	N.D.	170	N.D.	170
Aroclor 1248 (PCB)	12672-29-6	N.D.	140	N.D	130	N.D.	150	N.D.	130	N.D.	160	N.D.	170	N.D.	170
Aroclor 1254 (PCB)	11097-69-1	N.D.	140	N.D	130	N.D.	150	N.D.	130	N.D.	160	N.D.	170	N.D.	170
Aroclor 1260 (PCB)	11096-82-5	N.D.	140	N.D	130	N.D.	150	N.D.	130	N.D.	160	N.D.	170	N.D.	170
Aroclor 1262 (PCB)	37324-23-5	N.D.	140	N.D	130	N.D.	150	N.D.	130	N.D.	160	N.D.	170	N.D.	170
Aroclor 1268 (PCB)	11100-14-4	N.D.	140	N.D	220	N.D.	260	N.D.	220	N.D.	270	N.D.	290	N.D.	170

All data in ug/Kg

TABLE 7

Date Collected		11/15/01		12/4/01		12/4/01	
		SS-2		SS-3		SS-4	
PESTICIDES AND PCB'S	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
a-BHC	319-84-6	N.D.	29	N.D.	28	N.D.	29
b-BHC	319-85-7	N.D.	29	N.D.	28	N.D.	29
g-BHC (lindane)	58-89-9	N.D.	29	N.D.	28	N.D.	29
d-BHC	319-86-8	N.D.	29	N.D.	28	N.D.	29
Heptachlor	76-44-8	N.D.	29	N.D.	28	N.D.	29
Aldrin	309-00-2	N.D.	29	N.D.	28	N.D.	29
Heptachlor epoxide	1024-57-3	N.D.	29	N.D.	28	N.D.	29
g-Chlordane	5103-74-2	N.D.	72	N.D.	70	N.D.	71
Endosulfan I	959-98-8	N.D.	29	N.D.	28	N.D.	29
a-Chlordane	5103-71-9	N.D.	72	N.D.	70	N.D.	71
Dieldrin	60-57-1	N.D.	29	N.D.	28	N.D.	29
4,4'-DDE	72-55-9	N.D.	29	N.D.	28	170	29
Endrin	72-20-8	N.D.	29	N.D.	28	N.D.	29
Endosulfan II	33213-65-9	N.D.	29	N.D.	28	N.D.	29
4,4'-DDD	72-54-8	N.D.	29	N.D.	28	31	29
Endrin Aldehyde	7421-93-4	N.D.	29	N.D.	28	N.D.	29
Endosulfan Sulfate	1031-07-8	N.D.	29	N.D.	28	N.D.	29
4,4'-DDT	50-29-3	N.D.	29	N.D.	28	170	29
Endrin Ketone	53494-70-5	N.D.	29	N.D.	28	N.D.	29
Hexabromobenzene	87-82-1	140	140	N.D.	140	170	140
Methoxychlor	72-43-5	N.D.	72	N.D.	70	N.D.	71
Mirex	2385-85-5	N.D.	72	N.D.	70	N.D.	71
Toxaphene	8001-35-2	N.D.	250	N.D.	240	N.D.	240
BP-6 (PBB)	59080-40-9	N.D.	360	N.D.	350	N.D.	360
Aroclor 1016 (PCB)	12674-11-2	N.D.	140	N.D.	140	N.D.	140
Aroclor 1221 (PCB)	11104-28-2	N.D.	140	N.D.	140	N.D.	140
Aroclor 1232 (PCB)	11141-16-5	N.D.	140	N.D.	140	N.D.	140
Aroclor 1242 (PCB)	53469-21-9	N.D.	140	N.D.	140	N.D.	140
Aroclor 1248 (PCB)	12672-29-6	N.D.	140	N.D.	140	N.D.	140
Aroclor 1254 (PCB)	11097-69-1	N.D.	140	N.D.	140	N.D.	140
Aroclor 1260 (PCB)	11096-82-5	N.D.	140	N.D.	140	N.D.	140
Aroclor 1262 (PCB)	37324-23-5	N.D.	140	N.D.	140	N.D.	140
Aroclor 1268 (PCB)	11100-14-4	N.D.	140	N.D.	140	N.D.	140

All data in ug/Kg

TABLE 7

Date Collected		12/4/01		12/4/01		12/4/01		12/4/01		12/4/01		12/4/01	
		SS-5		SS-6		SS-7		SS-7R		SS-8		SS-9	
PESTICIDES AND PCB'S	**CAS #**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**	**RESULTS**	**LIMITS**
a-BHC	319-84-6	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
b-BHC	319-85-7	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
g-BHC (lindane)	58-89-9	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
d-BHC	319-86-8	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
Heptachlor	76-44-8	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
Aldrin	309-00-2	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
Heptachlor epoxide	1024-57-3	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
g-Chlordane	5103-74-2	N.D.	64	N.D.	60	N.D.	67	N.D.	66	N.D.	26	N.D.	62
Endosulfan I	959-98-8	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
a-Chlordane	5103-71-9	N.D.	64	N.D.	60	N.D.	67	N.D.	66	N.D.	64	N.D.	62
Dieldrin	60-57-1	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
4,4'-DDE	72-55-9	39	26	N.D.	24	N.D.	27	N.D.	26	N.D.	64	N.D.	25
Endrin	72-20-8	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
Endosulfan II	33213-65-9	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
4,4'-DDD	72-54-8	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
Endrin Aldehyde	7421-93-4	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
Endosulfan Sulfate	1031-07-8	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
4,4'-DDT	50-29-3	70	26	N.D.	24	62	27	47	26	N.D.	26	N.D.	25
Endrin Ketone	53494-70-5	N.D.	26	N.D.	24	N.D.	27	N.D.	26	N.D.	26	N.D.	25
Hexabromobenzene	87-82-1	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	120
Methoxychlor	72-43-5	N.D.	64	N.D.	60	N.D.	67	N.D.	66	N.D.	64	N.D.	62
Mirex	2385-85-5	N.D.	64	N.D.	60	N.D.	67	N.D.	66	N.D.	64	N.D.	62
Toxaphene	8001-35-2	N.D.	220	N.D.	200	N.D.	230	N.D.	220	N.D.	220	N.D.	210
BP-6 (PBB)	59080-40-9	N.D.	320	N.D.	300	N.D.	330	N.D.	330	N.D.	320	N.D.	310
Aroclor 1016 (PCB)	12674-11-2	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	120
Aroclor 1221 (PCB)	11104-28-2	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	120
Aroclor 1232 (PCB)	11141-16-5	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	120
Aroclor 1242 (PCB)	53469-21-9	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	120
Aroclor 1248 (PCB)	12672-29-6	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	120
Aroclor 1254 (PCB)	11097-69-1	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	120
Aroclor 1260 (PCB)	11096-82-5	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	120
Aroclor 1262 (PCB)	37324-23-5	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	120
Aroclor 1268 (PCB)	11100-14-4	N.D.	130	N.D.	120	N.D.	130	N.D.	130	N.D.	130	N.D.	120

All data in ug/Kg

TABLE 7

Date Collected		12/4/01	
		SS-10	
PESTICIDES AND PCB'S	**CAS #**	**RESULTS**	**LIMITS**
a-BHC	319-84-6	N.D.	27
b-BHC	319-85-7	N.D.	27
g-BHC (lindane)	58-89-9	N.D.	27
d-BHC	319-86-8	N.D.	27
Heptachlor	76-44-8	N.D.	27
Aldrin	309-00-2	N.D.	27
Heptachlor epoxide	1024-57-3	N.D.	27
g-Chlordane	5103-74-2	N.D.	67
Endosulfan I	959-98-8	N.D.	27
a-Chlordane	5103-71-9	N.D.	67
Dieldrin	60-57-1	N.D.	27
4,4'-DDE	72-55-9	N.D.	27
Endrin	72-20-8	N.D.	27
Endosulfan II	33213-65-9	N.D.	27
4,4'-DDD	72-54-8	N.D.	27
Endrin Aldehyde	7421-93-4	N.D.	27
Endosulfan Sulfate	1031-07-8	N.D.	27
4,4'-DDT	50-29-3	N.D.	27
Endrin Ketone	53494-70-5	N.D.	27
Hexabromobenzene	87-82-1	N.D.	130
Methoxychlor	72-43-5	N.D.	67
Mirex	2385-85-5	N.D.	67
Toxaphene	8001-35-2	N.D.	230
BP-6 (PBB)	59080-40-9	N.D.	330
Aroclor 1016 (PCB)	12674-11-2	N.D.	130
Aroclor 1221 (PCB)	11104-28-2	N.D.	130
Aroclor 1232 (PCB)	11141-16-5	N.D.	130
Aroclor 1242 (PCB)	53469-21-9	N.D.	130
Aroclor 1248 (PCB)	12672-29-6	N.D.	130
Aroclor 1254 (PCB)	11097-69-1	N.D.	130
Aroclor 1260 (PCB)	11096-82-5	N.D.	130
Aroclor 1262 (PCB)	37324-23-5	N.D.	130
Aroclor 1268 (PCB)	11100-14-4	N.D.	130

All data in ug/Kg

TABLE 8

Date Collected	9/4/01	9/4/01	9/4/01	9/4/01	9/7/01	11/15/01	9/7/01	11/15/01	9/7/01	9/7/01	9/7/01	9/7/01	10/22/01
	TA-C	**TA-UC**	**TB-C**	**TB-UC**	**TC-C**	**TC-C***	**TC-UC**	**TC-UC***	**TD-C**	**TD-UC**	**TE-C**	**TE-UC**	**TF-C**
METALS	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**
Aluminum in Sediment	795.0	1500.0	1100.0	1230.0									1900.0
Arsenic in Sediment	1.1	1.7	1.1	1.3	1.0	1.2	1.3	1.1	0.8	2.8	1.6	1.4	1.5
Barium in Sediment	7.0	14.0	10.0	12.0	6.0	5.0	5.0	9.0	5.0	38.0	23.0	15.0	18.0
Beryllium in Sediment	< 0.2	< 0.2	< 0.2	< 0.2									< 0.2
Cadmium in Sediment	< 2	< 2	< 2	< 2	< 2		< 2	< 2	< 2	< 2	< 2	< 2	< 2
Calcium in Sediment	9200.0	16700.0	14600.0	15800.0		< 2							22800.0
Chromium in Sediment	3.0	5.0	3.0	4.0	4.0	3.0	3.0	4.0	3.0	13.0	6.0	10.0	5.0
Cobalt in Sediment	< 2	< 2	< 2	< 2		< 2		< 2					< 2
Copper in Sediment	2.0	4.0	3.0	3.0	3.0	3.0	2.0	4.0	3.0	10.0	8.0	5.0	7.0
Iron in Sediment	1880.0	3170.0	2360.0	2670.0		1440.0		2015.0					4040.0
Lead in Sediment	< 5	6.0	<5	<5	< 5	< 5	< 5	< 5	< 5	< 5	7.0	< 5	< 5
Lithium in Sediment	< 2	2.0	<2	<2	<2	< 2	<2	< 2	<2	13.0	4.0	3.0	3.0
Magnesium in Sediment	3030.0	4530.0	3870.0	4510.0									6790.0
Manganese in Sediment	69.0	195.0	100.0	125.0	54.0	48.0	47.0	80.0	51.0	209.0	162.0	115.0	119.0
Mercury in Sediment	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< .05	< 0.05	< .05	< 0.05	< 0.05	< .05	< .05	< .05
Molybdenum in Sediment	< 5	< 5	< 5	< 5									< 5
Nickel in Sediment	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	12.0	< 5	< 5	< 5
Potassium in Sediment	61.0	151.0	91.0	104.0									197.0
Selenium in Sediment	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Silver in Sediment	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25
Sodium in Sediment	< 50	59.0	58.0	75.0									58.0
Solids - Total	80.0	82.5	76.3	78.0	83.0	81.9	81.5	78.1	78.5	70.5	61.8	72.9	70.3
Strontium in Sediment	9.0	18.0	15.0	16.0	10.0		8.0		8.0	42.0	32.0	21.0	24.0
Thallium in Sediment	< 1	< 1	< 1	< 1	< 1		< 1		< 1	< 1	< 1.0	< 1.0	< 1
Titanium in Sediment	68.0	60.0	71.0	77.0									86.0
Vanadium in Sediment	3.0	4.0	3.0	4.0									5.4
Zinc in Sediment	13.0	18.0	13.0	14.0	10.0	9.0	8.0	12.0	10.0	25.0	28.0	18.0	22.0

All data in mg/Kg

TABLE 8

Date Collected	10/22/01	10/22/01	11/1/01	11/2/01	11/2/01	11/2/01	11/2/01	11/15/01	11/15/01	12/4/01	12/4/01	12/4/01	12/4/01
	TF-CR	**TF-UC**	**TG-UC**	**TH-C**	**TH-UC**	**TI-C**	**TI-UC**	**SS-1**	**SS-2**	**SS-3**	**SS-4**	**SS-5**	**SS-6**
METALS	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**
Aluminum in Sediment	2000.0	1520.0	1140.0	1450.0	1050.0	2470.0	2680.0			4300.0	7060.0	5720.0	6190.0
Arsenic in Sediment	1.8	1.5	1.1	1.1	1.1	1.9	2.0	4.7	23.0	2.5	3.5	5.3	3.5
Barium in Sediment	21.0	14.0	12.0	11.0	9.0	28.0	28.0	92.0	62.0	39.0	67.0	45.0	51.0
Beryllium in Sediment	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2			0.2	0.3	0.3	0.3
Cadmium in Sediment	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2
Calcium in Sediment	23700.0	21500.0	18100.0	11000.0	9010.0	38100.0	43500.0			26900.0	42700.0	27600.0	6300.0
Chromium in Sediment	5.0	4.0	3.0	3.0	3.0	6.0	6.0	28.0	21.0	8.0	22.0	15.0	11.0
Cobalt in Sediment	< 2	< 2	< 2	< 2	< 2	3.0	3.0	8.0	5.0	3.0	5.0	4.0	4.0
Copper in Sediment	8.0	5.0	6.0	4.0	2.0	9.0	8.0	36.0	35.0	11.0	40.0	23.0	8.0
Iron in Sediment	4290.0	3530.0	2720.0	2760.0	2100.0	5240.0	5200.0	17900.0	11680.0	7630.0	11260.0	9350.0	9290.0
Lead in Sediment	< 5	< 5	< 5	< 5	< 5	8.0	7.0	35.0	26.0	19.0	20.0	20.0	12.0
Lithium in Sediment	3.0	3.0	2.0	2.4	< 2	4.4	4.6	21.0	12.0	7.0	12.0	10.0	9.0
Magnesium in Sediment	6970.0	6520.0	4820.0	4070.0	3140.0	9880.0	10900.0			12100.0	12600.0	13200.0	3650.0
Manganese in Sediment	125.0	120.0	112.0	68.0	78.0	225.0	219.0	350.0	430.0	354.0	505.0	370.0	234.0
Mercury in Sediment	< .05	< .05	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	0.2	0.2	< .05	0.1	0.1	< .05
Molybdenum in Sediment	< 5	< 5	< 5	< 5	< 5	< 5	< 5			< 5	< 5	< 5	< 5
Nickel in Sediment	< 5	< 5	< 5	< 5	< 5	< 5	< 5	21.0	11.0	7.0	11.0	9.0	8.0
Potassium in Sediment	206.0	162.0	131.0	157.0	113.0	284.0	346.0			766.0	1480.0	833.0	592.0
Selenium in Sediment	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	0.8	0.8	< 0.5	< 0.5	< 0.5	< 0.5
Silver in Sediment	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	0.3	< 0.25	< 0.25	< 0.25	< 2.5
Sodium in Sediment	< 50	< 50	< 50	< 50	< 50	< 50	< 50			< 50	68.0	61.0	< 50
Solids - Total	69.6	71.7	76.1	64.8	78.5	63.0	57.5	59.1	68.9	71.4	70.0	77.8	83.9
Strontium in Sediment	26.0	20.0	17.0	10.0	8.0	44.0	46.0			26.0	71.0	35.0	13.0
Thallium in Sediment	< 1	< 1								< 1.0	< 1.0	< 1.0	< 1.0
Titanium in Sediment	94.0	84.0	62.0	75.0	57.0	78.0	104.0			97.0	81.0	90.0	90.0
Vanadium in Sediment	5.7	4.9	3.7	4.5	3.2	7.0	7.8			10.0	15.0	13.0	14.0
Zinc in Sediment	24.0	19.0	15.0	13.0	10.0	29.0	27.0	119.0	88.0	40.0	69.0	60.0	33.0

All data in mg/Kg

TABLE 8

Date Collected	12/4/01	12/4/01	12/4/01	12/4/01	12/4/01
	SS-7	**SS-7R**	**SS-8**	**SS-9**	**SS-10**
METALS	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**	**RESULTS**
Aluminum in Sediment	3940.0	3960.0	3180.0	2620.0	3800.0
Arsenic in Sediment	4.0	3.8	2.3	1.8	2.7
Barium in Sediment	38.0	37.0	28.0	20.0	32.0
Beryllium in Sediment	0.2	0.2	< 0.2	< 0.2	0.2
Cadmium in Sediment	< 2	< 2	< 2	< 2	< 2
Calcium in Sediment	28900.0	26900.0	26000.0	14100.0	23500.0
Chromium in Sediment	10.0	10.0	7.0	8.0	7.0
Cobalt in Sediment	3.0	3.0	2.0	2.0	3.0
Copper in Sediment	14.0	14.0	8.0	10.0	8.0
Iron in Sediment	7150.0	7370.0	5470.0	4520.0	6550.0
Lead in Sediment	10.0	11.0	6.0	6.0	6.0
Lithium in Sediment	7.0	7.0	5.0	4.0	6.0
Magnesium in Sediment	9290.0	9610.0	8550.0	5870.0	11100.0
Manganese in Sediment	314.0	320.0	229.0	99.0	287.0
Mercury in Sediment	< .05	< .05	< .05	< .05	< .05
Molybdenum in Sediment	< 5	< 5	< 5	< 5	< 5
Nickel in Sediment	77.0	7.0	5.0	5.0	7.0
Potassium in Sediment	720.0	707.0	417.0	475.0	665.0
Selenium in Sediment	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Silver in Sediment	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25
Sodium in Sediment	106.0	88.0	100.0	70.0	59.0
Solids - Total	74.8	75.6	77.6	81.4	74.9
Strontium in Sediment	60.0	47.0	30.0	19.0	21.0
Thallium in Sediment	< 1.0	< 1.0	< 1.0	< 1.0	< 1.0
Titanium in Sediment	87.0	90.0	102.0	63.0	57.0
Vanadium in Sediment	9.0	9.0	8.0	6.0	9.0
Zinc in Sediment	42.0	43.0	30.0	36.0	35.0

All data in mg/Kg

TABLE 9
Part 1

Part 1																
				TA-C					TA-UC						TB-C	
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		0.96	0.9600	0.9600	0.9600		1.51	1.5100	1.5100	1.5100		2.24	2.2400	2.2400	2.2400
12378-PeCDD	0.5	ND	0.50	0.2500	0.1250	0.0000		0.85	0.4250	0.4250	0.4250		0.92	0.4600	0.4600	0.4600
123478-HxCDD	0.1		6.15	0.6150	0.6150	0.6150		7.83	0.7830	0.7830	0.7830		13.99	1.3990	1.3990	1.3990
123678-HxCDD	0.1	ND	0.50	0.0500	0.0250	0.0000		4.30	0.4300	0.4300	0.4300		4.74	0.4740	0.4740	0.4740
123789-HxCDD	0.1	ND	0.50	0.0500	0.0250	0.0000		0.61	0.0610	0.0610	0.0610		0.53	0.0530	0.0530	0.0530
1234678-HpCDD	0.01		15.47	0.1547	0.1547	0.1547		47.12	0.4712	0.4712	0.4712		59.48	0.5948	0.5948	0.5948
12346789-OCDD	0.001		137.98	0.1380	0.1380	0.1380		418.99	0.4190	0.4190	0.4190		560.61	0.5606	0.5606	0.5606
2378TCDF	0.1		39.04	3.9040	3.9040	3.9040		75.85	7.5850	7.5850	7.5850		159.04	15.9040	15.9040	15.9040
12378-PeCDF	0.05		20.31	1.0155	1.0155	1.0155		34.73	1.7365	1.7365	1.7365		58.59	2.9295	2.9295	2.9295
23478-PeCDF	0.5		12.52	6.2600	6.2600	6.2600		30.90	15.4500	15.4500	15.4500		38.47	19.2350	19.2350	19.2350
123478-HxCDF	0.1		13.09	1.3090	1.3090	1.3090		21.24	2.1240	2.1240	2.1240		35.53	3.5530	3.5530	3.5530
123678-HxCDF	0.1		2.84	0.2840	0.2840	0.2840		4.61	0.4610	0.4610	0.4610		8.48	0.8480	0.8480	0.8480
234678-HxCDF	0.1		1.15	0.1150	0.1150	0.1150		2.97	0.2970	0.2970	0.2970		3.84	0.3840	0.3840	0.3840
123789-HxCDF	0.1	ND	0.50	0.0500	0.0250	0.0000	ND	0.50	0.0500	0.0250	0.0000	ND	0.50	0.0500	0.0063	0.0000
1234678-HpCDF	0.01		25.52	0.2552	0.2552	0.2552		68.01	0.6801	0.6801	0.6801		115.52	1.1552	1.1552	1.1552
1234789-HpCDF	0.01		2.32	0.0232	0.0232	0.0232		5.17	0.0517	0.0517	0.0517		7.20	0.0720	0.0720	0.0720
12346789-OCDF	0.001		23.63	0.0236	0.0236	0.0236		84.48	0.0845	0.0845	0.0845		121.00	0.1210	0.1210	0.1210
nondetects = detection limit			TEQ=	15.4572				TEQ=	32.6190				TEQ=	50.0331		
nondetects = 1/2 d.l.			*teq=*		*15.2572*			*teq=*		*32.594*			*teq=*		*49.9894*	
nondetects = zero			teq=			15.0572		teq=			32.569		teq=			49.9831
Moisture content (%)				16.54					19.96					20.63		
Organic carbon (%)				0.11					0.38					0.23		
Total organic matter (%)				0.19					0.65					0.4		
Sample I.D.			TA-C					TA-UC					TB-C			
tetra-PCDF (TCDF)			39.0					75.9					159.0			
penta-PCDF (PeCDF)			32.8					65.6					97.1			
hexa-PCDF (HxCDF)			17.1					28.8					47.8			
hepta-PCDF (HpCDF)			27.8					73.2					123.0			
octa-PCDF (OCDF)			23.6					84.5					121.0			
tetra-PCDD (TCDD)			1.0					1.5					2.2			
penta-PCDD (PeCDD)		ND	0.5					0.9					0.9			
hexa-PCDD (HxCDD)			6.2					12.7					19.3			
hepta-PCDD (HpCDD)			15.5					47.1					59.5			
octa-PCDD (OCDD)			138.0					419.0					561.0			

Summary concentrations of the homolog groups [pg/g dry weight]
Concentrations of all compound (except of OCDF) are calculated based on internal standard calibration
Recalculation for internal standards accounts for the recoveries
* OCDF concentrations were calculated based on external standard and they were not recalculated for recoveries since no specific internal standard (no 13C-OCDF) was available
if required, OCDF concentrations can be normalized for 13C-OCDD recoveries
ND = non detected, the detection limits will be specified on individual sample bases

TABLE 9
Part 1

Part 1																
				TB-UC					TC-C					TC-UC		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		1.20	1.2000	1.2000	1.2000		0.92	0.9200	0.9200	0.9200		1.60	1.600	1.600	1.6000
12378-PeCDD	0.5	ND	0.50	0.2500	0.1250	0.0000	ND	0.50	0.2500	0.1250	0.0000		0.54	0.270	0.270	0.2700
123478-HxCDD	0.1		10.41	1.0410	1.0410	1.0410		13.32	1.3320	1.3320	1.3320		10.54	1.054	1.054	1.0540
123678-HxCDD	0.1		3.44	0.3440	0.3440	0.3440		1.43	0.1430	0.1430	0.1430	ND	0.50	0.050	0.025	0.0000
123789-HxCDD	0.1	ND	0.50	0.0500	0.0250	0.0000	ND	0.50	0.0500	0.0250	0.0000	ND	0.50	0.050	0.025	0.0000
1234678-HpCDD	0.01		53.56	0.5356	0.5356	0.5356		31.74	0.3174	0.3174	0.3174		23.64	0.236	0.236	0.2364
12346789-OCDD	0.001		483.51	0.4835	0.4835	0.4835		208.19	0.2082	0.2082	0.2082		186.10	0.186	0.186	0.1861
2378TCDF	0.1		547.68	54.7680	54.7680	54.7680		33.80	3.3800	3.3800	3.3800		162.20	16.220	16.220	16.2200
12378-PeCDF	0.05		198.27	9.9135	9.9135	9.9135		14.27	0.7135	0.7135	0.7135		96.44	4.822	4.822	4.8220
23478-PeCDF	0.5		130.15	65.0750	65.0750	65.0750		8.26	4.1300	4.1300	4.1300		52.75	26.375	26.375	26.3750
123478-HxCDF	0.1		90.04	9.0040	9.0040	9.0040		10.01	1.0010	1.0010	1.0010		54.51	5.451	5.451	5.4510
123678-HxCDF	0.1		23.34	2.3340	2.3340	2.3340		1.54	0.1540	0.1540	0.1540		12.66	1.266	1.266	1.2660
234678-HxCDF	0.1		10.21	1.0210	1.0210	1.0210	ND	0.50	0.0500	0.0250	0.0000		4.85	0.485	0.485	0.4850
123789-HxCDF	0.1		0.87	0.0870	0.0870	0.0870	ND	0.50	0.0500	0.0250	0.0000	ND	0.50	0.050	0.025	0.0000
1234678-HpCDF	0.01		91.15	0.9115	0.9115	0.9115		43.12	0.4312	0.4312	0.4312		60.70	0.607	0.607	0.6070
1234789-HpCDF	0.01		8.37	0.0837	0.0837	0.0837		4.05	0.0405	0.0405	0.0405		69.57	0.696	0.696	0.6957
12346789-OCDF	0.001		88.81	0.0888	0.0888	0.0888		55.54	0.0555	0.0555	0.0555		39.12	0.039	0.039	0.0391
nondetects = detection limit			TEQ=	147.1906				TEQ=	13.2263				TEQ=	59.4573		
nondetects = 1/2 d.l.			*teq=*		*147.0406*			*teq=*		*13.0263*			*teq=*		*59.3823*	
nondetects = zero			teq=			146.8906		teq=			12.8263		teq=			59.3073
Moisture content (%)				18.59					18.64					20.77		
Organic carbon (%)				0.26					0.18					0.33		
Total organic matter (%)				0.45					0.31					0.57		
Sample I.D.			TB-UC					TC-C					TC-UC			
tetra-PCDF (TCDF)			548.0					33.8					162.0			
penta-PCDF (PeCDF)			328.0					22.5					149.0			
hexa-PCDF (HxCDF)			124.0					11.6					72.0			
hepta-PCDF (HpCDF)			99.5					47.2					130.0			
octa-PCDF (OCDF)			88.8					55.5					39.1			
tetra-PCDD (TCDD)			1.2					0.9					1.6			
penta-PCDD (PeCDD)		ND	0.5				ND	0.5					0.5			
hexa-PCDD (HxCDD)			13.9					14.8					10.5			
hepta-PCDD (HpCDD)			53.6					31.7					23.6			
octa-PCDD (OCDD)			484.0					208.0					186.0			
Summary concentrations of the homolog gı																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba:																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 1

Part 1																
				TD-C					TD-UC					TE-C		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		1.56	1.560	1.560	1.560		0.97	0.970	0.970	0.9700		1.95	1.950	1.950	1.9500
12378-PeCDD	0.5		1.03	0.515	0.515	0.515		0.60	0.300	0.300	0.3000		0.74	0.370	0.370	0.3700
123478-HxCDD	0.1		32.32	3.232	3.232	3.232		1.44	0.144	0.144	0.1440		63.47	6.347	6.347	6.3470
123678-HxCDD	0.1		2.06	0.206	0.206	0.206	ND	1.20	0.120	0.060	0.0000		14.68	1.468	1.468	1.4680
123789-HxCDD	0.1		2.11	0.211	0.211	0.211	ND	1.20	0.120	0.060	0.0000		3.34	0.334	0.334	0.3340
1234678-HpCDD	0.01		131.46	1.315	1.315	1.315		25.25	0.253	0.253	0.2525		207.34	2.073	2.073	2.0734
12346789-OCDD	0.001		911.49	0.911	0.911	0.911		287.56	0.288	0.288	0.2876		2139.70	2.140	2.140	2.1397
2378TCDF	0.1		3084.98	308.498	308.498	308.498		267.47	26.747	26.747	26.7470		2577.95	257.795	257.795	257.7950
12378-PeCDF	0.05		4891.86	244.593	244.593	244.593		139.34	6.967	6.967	6.9670		1496.96	74.848	74.848	74.8480
23478-PeCDF	0.5		1660.30	830.150	830.150	830.150		91.78	45.890	45.890	45.8900		980.42	490.210	490.210	490.2100
123478-HxCDF	0.1		5020.21	502.021	502.021	502.021		49.60	4.960	4.960	4.9600		663.00	66.300	66.300	66.3000
123678-HxCDF	0.1		1014.95	101.495	101.495	101.495		10.06	1.006	1.006	1.0060		177.19	17.719	17.719	17.7190
234678-HxCDF	0.1		219.77	21.977	21.977	21.977		4.91	0.491	0.491	0.4910		80.29	8.029	8.029	8.0290
123789-HxCDF	0.1		63.91	6.391	6.391	6.391		1.10	0.110	0.110	0.1100		5.22	0.522	0.522	0.5220
1234678-HpCDF	0.01		844.04	8.440	8.440	8.440		53.23	0.532	0.532	0.5323		436.54	4.365	4.365	4.3654
1234789-HpCDF	0.01		346.97	3.470	3.470	3.470		7.37	0.074	0.074	0.0737		52.29	0.523	0.523	0.5229
12346789-OCDF	0.001		594.49	0.594	0.594	0.594		38.01	0.038	0.038	0.0380		485.02	0.485	0.485	0.4850
nondetects = detection limit			TEQ=	2035.5797				TEQ=	89.0091				TEQ=	935.4784		
nondetects = 1/2 d.l.			*teq=*		*2035.5797*			*teq=*		*88.8891*			*teq=*		*935.4784*	
nondetects = zero			teq=			2035.5797		teq=			88.7691		teq=			935.4784
Moisture content (%)				17.82					24.44					33.27		
Organic carbon (%)				0.12					0.33					1.27		
Total organic matter (%)				0.21					0.57					2.19		
Sample I.D.			TD-C					TD-UC					TE-C			
tetra-PCDF (TCDF)			3085.0					267.0					2578.0			
penta-PCDF (PeCDF)			6552.0					231.0					2477.0			
hexa-PCDF (HxCDF)			6319.0					65.7					926.0			
hepta-PCDF (HpCDF)			1191.0					60.6					489.0			
octa-PCDF (OCDF)			594.0					38.0					485.0			
tetra-PCDD (TCDD)			1.6					1.0					1.9			
penta-PCDD (PeCDD)			1.0					0.6					0.7			
hexa-PCDD (HxCDD)			36.5					1.4					81.5			
hepta-PCDD (HpCDD)			131.0					25.2					207.0			
octa-PCDD (OCDD)			911.0					288.0					2140.0			
Summary concentrations of the homolog gı																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba:																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 1

Part 1																
				TE-UC					TF-C					TF-CR		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		2.94	2.9400	2.9400	2.9400		2.15	2.150	2.1500	2.1500		2.97	2.970	2.9700	2.9700
12378-PeCDD	0.5		2.16	1.0800	1.0800	1.0800		1.28	0.640	0.6400	0.6400		1.20	0.600	0.6000	0.6000
123478-HxCDD	0.1		44.45	4.4450	4.4450	4.4450		48.48	4.848	4.8480	4.8480		84.18	8.418	8.4180	8.4180
123678-HxCDD	0.1		36.72	3.6720	3.6720	3.6720		12.96	1.296	1.2960	1.2960		12.62	1.262	1.2620	1.2620
123789-HxCDD	0.1		5.53	0.5530	0.5530	0.5530		3.09	0.309	0.3090	0.3090		2.62	0.262	0.2620	0.2620
1234678-HpCDD	0.01		715.59	7.1559	7.1559	7.1559		302.17	3.022	3.0217	3.0217		270.92	2.709	2.7092	2.7092
12346789-OCDD	0.001		4904.67	4.9047	4.9047	4.9047		3880.96	3.881	3.8810	3.8810		2763.04	2.763	2.7630	2.7630
2378TCDF	0.1		423.68	42.3680	42.3680	42.3680		1085.39	108.539	108.5390	108.5390		1243.66	124.366	124.3660	124.3660
12378-PeCDF	0.05		218.95	10.9475	10.9475	10.9475		590.27	29.514	29.5135	29.5135		614.32	30.716	30.7160	30.7160
23478-PeCDF	0.5		134.41	67.2050	67.2050	67.2050		313.37	156.685	156.6850	156.6850		367.97	183.985	183.9850	183.9850
123478-HxCDF	0.1		144.23	14.4230	14.4230	14.4230		318.40	31.840	31.8400	31.8400		294.10	29.410	29.4100	29.4100
123678-HxCDF	0.1		23.23	2.3230	2.3230	2.3230		74.01	7.401	7.4010	7.4010		72.19	7.219	7.2190	7.2190
234678-HxCDF	0.1		8.30	0.8300	0.8300	0.8300		25.94	2.594	2.5940	2.5940		25.92	2.592	2.5920	2.5920
123789-HxCDF	0.1		1.83	0.1830	0.1830	0.1830		6.50	0.650	0.6500	0.6500		5.72	0.572	0.5720	0.5720
1234678-HpCDF	0.01		625.86	6.2586	6.2586	6.2586		359.70	3.597	3.5970	3.5970		611.81	6.118	6.1181	6.1181
1234789-HpCDF	0.01		47.51	0.4751	0.4751	0.4751		41.18	0.412	0.4118	0.4118		39.96	0.400	0.3996	0.3996
12346789-OCDF	0.001		2134.61	2.1346	2.1346	2.1346		617.51	0.618	0.6175	0.6175		775.25	0.775	0.7753	0.7753
nondetects = detection limit			TEQ=	171.8984				TEQ=	357.9945				TEQ=	405.1372		
nondetects = 1/2 d.l.			*teq=*		*171.8984*			*teq=*		*357.9945*			*teq=*		*405.1372*	
nondetects = zero			teq=			171.8984		teq=			357.9945		teq=			405.1372
Moisture content (%)				24.67					25.96					26.8		
Organic carbon (%)				0.71					0.65					0.62		
Total organic matter (%)				1.22					1.12					1.07		
Sample I.D.			TE-UC					TF-C					TF-CR			
tetra-PCDF (TCDF)			424.0					1085.0					1244.0			
penta-PCDF (PeCDF)			353.0					904.0					982.0			
hexa-PCDF (HxCDF)			178.0					425.0					398.0			
hepta-PCDF (HpCDF)			673.0					401.0					652.0			
octa-PCDF (OCDF)			2135.0					618.0					775.0			
tetra-PCDD (TCDD)			2.9					2.2					3.0			
penta-PCDD (PeCDD)			2.2					1.3					1.2			
hexa-PCDD (HxCDD)			86.7					64.5					99.4			
hepta-PCDD (HpCDD)			716.0					302.0					271.0			
octa-PCDD (OCDD)			4905.0					3881.0					2763.0			
Summary concentrations of the homolog gı																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba:																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 1

Part 1																
				TF-UC					TG-UC					TH-C		
Analyte	TEF		sampled	toxic eq.	*nondetect*	nondetect		sampled	toxic eq.	*nondetect*	nondetect		sampled	toxic eq.	*nondetect*	nondetect
			pg/g	pg/g	*1/2 d.l.*	zero		pg/g	pg/g	*1/2 d.l.*	zero		pg/g	pg/g	*1/2 d.l.*	zero
2378-TCDD	1		2.56	2.560	2.5600	2.5600	ND	0.50	0.500	0.2500	0.0000	ND	0.500	0.500	0.2500	0.0000
12378-PeCDD	0.5	ND	0.50	0.250	0.1250	0.0000	ND	0.50	0.250	0.1250	0.0000	ND	0.500	0.250	0.1250	0.0000
123478-HxCDD	0.1		24.71	2.471	2.4710	2.4710		0.92	0.092	0.0920	0.0920	ND	0.500	0.050	0.0250	0.0000
123678-HxCDD	0.1		9.13	0.913	0.9130	0.9130		5.85	0.585	0.5850	0.5850	ND	0.500	0.050	0.0250	0.0000
123789-HxCDD	0.1		4.61	0.461	0.4610	0.4610	ND	0.50	0.050	0.0250	0.0000	ND	0.500	0.050	0.0250	0.0000
1234678-HpCDD	0.01		121.15	1.212	1.2115	1.2115		31.80	0.318	0.3180	0.3180		6.382	0.064	0.0638	0.0638
12346789-OCDD	0.001		1099.11	1.099	1.0991	1.0991		224.25	0.224	0.2243	0.2243		52.715	0.053	0.0527	0.0527
2378TCDF	0.1		1718.24	171.824	171.8240	171.8240		8.96	0.896	0.8960	0.8960		2.790	0.279	0.2790	0.2790
12378-PeCDF	0.05		959.43	47.972	47.9715	47.9715		5.28	0.264	0.2640	0.2640		1.346	0.067	0.0673	0.0673
23478-PeCDF	0.5		507.40	253.700	253.7000	253.7000		2.52	1.260	1.2600	1.2600	ND	0.500	0.250	0.1250	0.0000
123478-HxCDF	0.1		428.50	42.850	42.8500	42.8500		6.20	0.620	0.6200	0.6200		2.430	0.243	0.2430	0.2430
123678-HxCDF	0.1		90.64	9.064	9.0640	9.0640		0.78	0.078	0.0780	0.0780	ND	0.500	0.050	0.0250	0.0000
234678-HxCDF	0.1		33.59	3.359	3.3590	3.3590	ND	0.50	0.050	0.0250	0.0000	ND	0.500	0.050	0.0250	0.0000
123789-HxCDF	0.1		3.59	0.359	0.3590	0.3590	ND	0.50	0.050	0.0250	0.0000	ND	0.500	0.050	0.0250	0.0000
1234678-HpCDF	0.01		234.05	2.341	2.3405	2.3405		30.42	0.304	0.3042	0.3042		3.730	0.037	0.0373	0.0373
1234789-HpCDF	0.01		27.71	0.277	0.2771	0.2771		2.59	0.026	0.0259	0.0259		0.640	0.006	0.0064	0.0064
12346789-OCDF	0.001		356.53	0.357	0.3565	0.3565		31.31	0.031	0.0313	0.0313		2.953	0.003	0.0030	0.0030
nondetects = detection limit			TEQ=	541.0672				TEQ=	5.5987				TEQ=	2.0525		
nondetects = 1/2 d.l.			*teq=*		*540.9422*			*teq=*		*5.1487*			*teq=*		*1.4025*	
nondetects = zero			teq=			540.8172		teq=			4.6987		teq=			0.7525
Moisture content (%)				23.9					25.18					27.01		
Organic carbon (%)				0.33					0.26					0.41		
Total organic matter (%)				0.57					0.45					0.71		
Sample I.D.			TF-UC					TG-UC					TH-C			
tetra-PCDF (TCDF)			1718.0					9.0					2.8			
penta-PCDF (PeCDF)			1467.0					7.8					1.3			
hexa-PCDF (HxCDF)			556.0					7.0					2.4			
hepta-PCDF (HpCDF)			262.0					33.0					4.4			
octa-PCDF (OCDF)			357.0					31.3					3.0			
tetra-PCDD (TCDD)			2.6				ND	0.5				ND	0.5			
penta-PCDD (PeCDD)		ND	0.5				ND	0.5				ND	0.5			
hexa-PCDD (HxCDD)			38.4					6.8				ND	0.5			
hepta-PCDD (HpCDD)			121.0					31.8					6.4			
octa-PCDD (OCDD)			1099.0					224.0					52.7			
Summary concentrations of the homolog gı																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba:																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 1

Part 1																
				TH-UC					TI-C						TI-UC	
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1	ND	0.50	0.500	0.2500	0.0000		1.23	1.230	1.2300	1.2300	ND	0.50	0.500	0.2500	0.0000
12378-PeCDD	0.5	ND	0.50	0.250	0.1250	0.0000	ND	0.50	0.250	0.1250	0.0000	ND	0.50	0.250	0.1250	0.0000
123478-HxCDD	0.1	ND	0.50	0.050	0.0250	0.0000	ND	0.50	0.050	0.0250	0.0000	ND	0.50	0.050	0.0250	0.0000
123678-HxCDD	0.1	ND	0.50	0.050	0.0250	0.0000	ND	0.50	0.050	0.0250	0.0000	ND	0.50	0.050	0.0250	0.0000
123789-HxCDD	0.1	ND	0.50	0.050	0.0250	0.0000		0.50	0.050	0.0500	0.0500	ND	0.60	0.060	0.0300	0.0000
1234678-HpCDD	0.01		3.25	0.033	0.0325	0.0325		20.56	0.206	0.2056	0.2056		11.17	0.112	0.1117	0.1117
12346789-OCDD	0.001		31.55	0.032	0.0316	0.0316		178.72	0.179	0.1787	0.1787		82.66	0.083	0.0827	0.0827
2378TCDF	0.1		1.01	0.101	0.1010	0.1010		7.18	0.718	0.7180	0.7180		2.42	0.242	0.2420	0.2420
12378-PeCDF	0.05		0.73	0.037	0.0365	0.0365		2.22	0.111	0.1110	0.1110		1.99	0.100	0.0995	0.0995
23478-PeCDF	0.5	ND	0.50	0.250	0.1250	0.0000		2.87	1.435	1.4350	1.4350		1.49	0.745	0.7450	0.7450
123478-HxCDF	0.1		1.42	0.142	0.1420	0.1420		3.16	0.316	0.3160	0.3160		1.16	0.116	0.1160	0.1160
123678-HxCDF	0.1	ND	0.50	0.050	0.0250	0.0000		1.21	0.121	0.1210	0.1210	ND	0.50	0.050	0.0250	0.0000
234678-HxCDF	0.1	ND	0.50	0.050	0.0250	0.0000	ND	0.50	0.050	0.0250	0.0000	ND	0.50	0.050	0.0250	0.0000
123789-HxCDF	0.1	ND	0.50	0.050	0.0250	0.0000	ND	0.50	0.050	0.0250	0.0000	ND	0.50	0.050	0.0250	0.0000
1234678-HpCDF	0.01		2.90	0.029	0.0290	0.0290		17.90	0.179	0.1790	0.1790		6.32	0.063	0.0632	0.0632
1234789-HpCDF	0.01		0.53	0.005	0.0053	0.0053		1.02	0.010	0.0102	0.0102		0.82	0.008	0.0082	0.0082
12346789-OCDF	0.001		2.61	0.003	0.0026	0.0026		11.28	0.011	0.0113	0.0113		4.18	0.004	0.0042	0.0042
nondetects = detection limit			TEQ=	1.6805				TEQ=	5.0158				TEQ=	2.5324		
nondetects = 1/2 d.l.			*teq=*		*1.0305*			*teq=*		*4.7908*			*teq=*		*2.0024*	
nondetects = zero			teq=			0.3805		teq=			4.5658		teq=			1.4724
Moisture content (%)				20.93					36.61					34.14		
Organic carbon (%)				0.49					1.28					0.87		
Total organic matter (%)				0.85					2.21					1.5		
Sample I.D.			TH-UC					TI-C					TI-UC			
tetra-PCDF (TCDF)			1.0					7.2					2.4			
penta-PCDF (PeCDF)			0.7					5.1					3.5			
hexa-PCDF (HxCDF)			1.4					4.4					1.2			
hepta-PCDF (HpCDF)			3.4					18.9					7.1			
octa-PCDF (OCDF)			2.6					11.3					4.2			
tetra-PCDD (TCDD)		ND	0.5					1.2				ND	0.5			
penta-PCDD (PeCDD)		ND	0.5				ND	0.5				ND	0.5			
hexa-PCDD (HxCDD)		ND	0.5					0.5				ND	0.5			
hepta-PCDD (HpCDD)			3.3					20.6					11.2			
octa-PCDD (OCDD)			31.5					179.0					82.7			
Summary concentrations of the homolog gı																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba:																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 2

Part 2 (continued)																
				C1					C2					C-3		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		1.52	1.520	1.5200	1.5200		2.27	2.270	2.2700	2.2700		3.13	3.130	3.1300	3.1300
12378-PeCDD	0.5	ND	0.50	0.250	0.1250	0.0000	ND	0.50	0.250	0.1250	0.0000		0.98	0.490	0.4900	0.4900
123478-HxCDD	0.1		45.03	4.503	4.5030	4.5030		17.28	1.728	1.7280	1.7280		63.20	6.320	6.3200	6.3200
123678-HxCDD	0.1		4.86	0.486	0.4860	0.4860		6.07	0.607	0.6070	0.6070		13.87	1.387	1.3870	1.3870
123789-HxCDD	0.1	ND	0.50	0.050	0.0250	0.0000		1.14	0.114	0.1140	0.1140		3.44	0.344	0.3440	0.3440
1234678-HpCDD	0.01		78.44	0.784	0.7844	0.7844		60.90	0.609	0.6090	0.6090		141.34	1.413	1.4134	1.4134
12346789-OCDD	0.001		659.09	0.659	0.6591	0.6591		476.34	0.476	0.4763	0.4763		1100.78	1.101	1.1008	1.1008
2378TCDF	0.1		625.06	62.506	62.5060	62.5060		3600.50	360.050	360.0500	360.0500		747.63	74.763	74.7630	74.7630
12378-PeCDF	0.05		353.71	17.686	17.6855	17.6855		1145.95	57.298	57.2975	57.2975		411.60	20.580	20.5800	20.5800
23478-PeCDF	0.5		211.16	105.580	105.5800	105.5800		812.82	406.410	406.4100	406.4100		235.92	117.960	117.9600	117.9600
123478-HxCDF	0.1		160.78	16.078	16.0780	16.0780		359.52	35.952	35.9520	35.9520		163.82	16.382	16.3820	16.3820
123678-HxCDF	0.1		32.84	3.284	3.2840	3.2840		93.16	9.316	9.3160	9.3160		30.18	3.018	3.0180	3.0180
234678-HxCDF	0.1		11.68	1.168	1.1680	1.1680		43.55	4.355	4.3550	4.3550		11.48	1.148	1.1480	1.1480
123789-HxCDF	0.1		2.54	0.254	0.2540	0.2540		8.64	0.864	0.8640	0.8640		3.01	0.301	0.3010	0.3010
1234678-HpCDF	0.01		254.43	2.544	2.5443	2.5443		147.40	1.474	1.4740	1.4740		351.97	3.520	3.5197	3.5197
1234789-HpCDF	0.01		13.89	0.139	0.1389	0.1389		19.50	0.195	0.1950	0.1950		28.17	0.282	0.2817	0.2817
12346789-OCDF	0.001		155.26	0.155	0.1553	0.1553		198.13	0.198	0.1981	0.1981		267.76	0.268	0.2678	0.2678
nondetects = detection limit			TEQ=	217.65				TEQ=	882.17				TEQ=	252.41		
nondetects = 1/2 d.l.			*teq=*		*217.50*			*teq=*		*882.04*			*teq=*		*252.41*	
nondetects = zero			teq=			217.35		teq=			881.92		teq=			252.41
Moisture content (%)				21.74					22.27					27.11		
Organic carbon (%)				0.37					0.36					0.94		
Total organic matter (%)				0.64					0.61					1.62		
Sample I.D.			C-1					C-2					C-3			
tetra-PCDF (TCDF)			625					3601					748			
penta-PCDF (PeCDF)			565					1959					648			
hexa-PCDF (HxCDF)			208					505					208			
hepta-PCDF (HpCDF)			268					167					380			
octa-PCDF (OCDF)			155					198					268			
tetra-PCDD (TCDD)			1.5					2.3					3.1			
penta-PCDD (PeCDD)		ND	0.5				ND	0.5					1			
hexa-PCDD (HxCDD)			49.9					24.5					80.5			
hepta-PCDD (HpCDD)			78.4					60.9					141			
octa-PCDD (OCDD)			659					476					1101			

Summary concentrations of the homolog groups [pg/g dry weight]
Concentrations of all compound (except of OCDF) are calculated based on internal standard calibration
Recalculation for internal standards accounts for the recoveries
* OCDF concentrations were calculated based on external standard and they were not recalculated for recoveries since no specific internal standard (no 13C-OCDF) was available
if required, OCDF concentrations can be normalized for 13C-OCDD recoveries
ND = non detected, the detection limits will be specified on individual sample bases

TABLE 9
Part 2

Part 2 (continued)

Analyte	TEF		C-4 sampled pg/g	C-4 toxic eq. pg/g	C-4 *nondetect 1/2 d.l.*	C-4 nondetect zero		C-5 sampled pg/g	C-5 toxic eq. pg/g	C-5 *nondetect 1/2 d.l.*	C-5 nondetect zero		C-6 sampled pg/g	C-6 toxic eq. pg/g	C-6 *nondetect 1/2 d.l.*	C-6 nondetect zero
2378-TCDD	1		1.54	1.540	1.5400	1.5400		1.57	1.570	1.5700	1.5700		1.74	1.740	1.7400	1.7400
12378-PeCDD	0.5	ND	0.50	0.250	0.1250	0.0000		4.45	2.225	2.2250	2.2250		0.93	0.465	0.4650	0.4650
123478-HxCDD	0.1		53.25	5.325	5.3250	5.3250		23.59	2.359	2.3590	2.3590		32.64	3.264	3.2640	3.2640
123678-HxCDD	0.1		14.47	1.447	1.4470	1.4470		20.19	2.019	2.0190	2.0190		8.70	0.870	0.8700	0.8700
123789-HxCDD	0.1		3.29	0.329	0.3290	0.3290		5.66	0.566	0.5660	0.5660		2.09	0.209	0.2090	0.2090
1234678-HpCDD	0.01		149.46	1.495	1.4946	1.4946		95.30	0.953	0.9530	0.9530		95.18	0.952	0.9518	0.9518
12346789-OCDD	0.001		1281.52	1.282	1.2815	1.2815		715.05	0.715	0.7151	0.7151		974.11	0.974	0.9741	0.9741
2378TCDF	0.1		5449.06	544.906	544.9060	544.9060		1025.88	102.588	102.5880	102.5880		1716.96	171.696	171.6960	171.6960
12378-PeCDF	0.05		2261.76	113.088	113.0880	113.0880		1064.92	53.246	53.2460	53.2460		509.79	25.490	25.4895	25.4895
23478-PeCDF	0.5		1617.75	808.875	808.8750	808.8750		540.82	270.410	270.4100	270.4100		378.61	189.305	189.3050	189.3050
123478-HxCDF	0.1		895.98	89.598	89.5980	89.5980		551.68	55.168	55.1680	55.1680		220.90	22.090	22.0900	22.0900
123678-HxCDF	0.1		160.95	16.095	16.0950	16.0950		95.57	9.557	9.5570	9.5570		44.01	4.401	4.4010	4.4010
234678-HxCDF	0.1		97.50	9.750	9.7500	9.7500		50.15	5.015	5.0150	5.0150		28.92	2.892	2.8920	2.8920
123789-HxCDF	0.1		5.74	0.574	0.5740	0.5740		6.83	0.683	0.6830	0.6830		3.14	0.314	0.3140	0.3140
1234678-HpCDF	0.01		388.22	3.882	3.8822	3.8822		255.26	2.553	2.5526	2.5526		229.01	2.290	2.2901	2.2901
1234789-HpCDF	0.01		45.92	0.459	0.4592	0.4592		39.16	0.392	0.3916	0.3916		15.55	0.156	0.1555	0.1555
12346789-OCDF	0.001		450.79	0.451	0.4508	0.4508		139.79	0.140	0.1398	0.1398		182.65	0.183	0.1827	0.1827
nondetects = detection limit			TEQ=	1599.35				TEQ=	510.16				TEQ=	427.29		
nondetects = 1/2 d.l.			*teq=*		*1599.22*			*teq=*		*510.16*			*teq=*		*427.29*	
nondetects = zero			teq=			1599.10		teq=			510.16		teq=			427.29
Moisture content (%)			22.1					24.59					23.93			
Organic carbon (%)			0.66					0.38					0.49			
Total organic matter (%)			1.13					0.66					0.84			
Sample I.D.			C-4					C-5					C-6			
tetra-PCDF (TCDF)			5449					1026					1717			
penta-PCDF (PeCDF)			3880					1606					888			
hexa-PCDF (HxCDF)			1160					704					297			
hepta-PCDF (HpCDF)			434					294					245			
octa-PCDF (OCDF)			451					140					183			
tetra-PCDD (TCDD)			1.5					1.6					1.7			
penta-PCDD (PeCDD)		ND	0.5					4.5					0.9			
hexa-PCDD (HxCDD)			71					49.4					43.4			
hepta-PCDD (HpCDD)			149					95.3					95.2			
octa-PCDD (OCDD)			1282					715					974			

Summary concentrations of the homolog gr
Concentrations of all compound (except of
Recalculation for internal standards accoun
* OCDF concentrations were calculated ba
if required, OCDF concentrations can be n
ND = non detected, the detection limits wil

TABLE 9
Part 2

Part 2 (continued)																
				C-7					C-8					C-9		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		4.67	4.6700	4.6700	4.6700	ND	1.40	1.4000	0.7000	0.0000		2.99	2.9900	2.9900	2.9900
12378-PeCDD	0.5		4.27	2.1350	2.1350	2.1350		2.21	1.1050	1.1050	1.1050	ND	0.75	0.3750	0.1875	0.0000
123478-HxCDD	0.1		103.43	10.3430	10.3430	10.3430		64.35	6.4350	6.4350	6.4350		30.09	3.0090	3.0090	3.0090
123678-HxCDD	0.1		26.09	2.6090	2.6090	2.6090		3.25	0.3250	0.3250	0.3250		19.33	1.9330	1.9330	1.9330
123789-HxCDD	0.1		7.11	0.7110	0.7110	0.7110		2.29	0.2290	0.2290	0.2290		2.20	0.2200	0.2200	0.2200
1234678-HpCDD	0.01		271.62	2.7162	2.7162	2.7162		234.30	2.3430	2.3430	2.3430		186.48	1.8648	1.8648	1.8648
12346789-OCDD	0.001		2943.76	2.9438	2.9438	2.9438		1645.38	1.6454	1.6454	1.6454		1540.70	1.5407	1.5407	1.5407
2378TCDF	0.1		1045.95	104.5950	104.5950	104.5950		333.59	33.3590	33.3590	33.3590		1024.03	102.4030	102.4030	102.4030
12378-PeCDF	0.05		665.06	33.2530	33.2530	33.2530		239.80	11.9900	11.9900	11.9900		673.81	33.6905	33.6905	33.6905
23478-PeCDF	0.5		362.66	181.3300	181.3300	181.3300		141.93	70.9650	70.9650	70.9650		356.56	178.2800	178.2800	178.2800
123478-HxCDF	0.1		392.42	39.2420	39.2420	39.2420		127.71	12.7710	12.7710	12.7710		243.52	24.3520	24.3520	24.3520
123678-HxCDF	0.1		84.80	8.4800	8.4800	8.4800		34.49	3.4490	3.4490	3.4490		54.00	5.4000	5.4000	5.4000
234678-HxCDF	0.1		41.46	4.1460	4.1460	4.1460		2.69	0.2690	0.2690	0.2690		20.47	2.0470	2.0470	2.0470
123789-HxCDF	0.1		3.67	0.3670	0.3670	0.3670		2.10	0.2100	0.2100	0.2100		31.81	3.1810	3.1810	3.1810
1234678-HpCDF	0.01		601.20	6.0120	6.0120	6.0120		364.28	3.6428	3.6428	3.6428		211.18	2.1118	2.1118	2.1118
1234789-HpCDF	0.01		33.21	0.3321	0.3321	0.3321		25.62	0.2562	0.2562	0.2562		26.44	0.2644	0.2644	0.2644
12346789-OCDF	0.001		389.61	0.3896	0.3896	0.3896		291.78	0.2918	0.2918	0.2918		401.07	0.4011	0.4011	0.4011
nondetects = detection limit			TEQ=	404.27				TEQ=	150.69				TEQ=	364.06		
nondetects = 1/2 d.l.			*teq=*		*404.27*			*teq=*		*149.99*			*teq=*		*363.88*	
nondetects = zero			teq=			404.27		teq=			149.29		teq=			363.69
Moisture content (%)			25.05					34.35					24.86			
Organic carbon (%)			0.43					1.3					0.51			
Total organic matter (%)			0.74					1.24					0.88			
Sample I.D.			C-7					C-8					C-9			
tetra-PCDF (TCDF)			1046					334					1024			
penta-PCDF (PeCDF)			1028					382					1030			
hexa-PCDF (HxCDF)			522					167					350			
hepta-PCDF (HpCDF)			634					390					238			
octa-PCDF (OCDF)			390					292					401			
tetra-PCDD (TCDD)			4.7				ND	1.4					3			
penta-PCDD (PeCDD)			4.3					2.2				ND	0.75			
hexa-PCDD (HxCDD)			137					69.9					51.6			
hepta-PCDD (HpCDD)			272					234					186			
octa-PCDD (OCDD)			2944					1645					1541			
Summary concentrations of the homolog gı																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba:																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 2

Part 2 (continued)																
				C-10					C-11					C-12		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		8.00	8.0000	8.0000	8.0000	ND	1.00	1.0000	0.5000	0.0000	ND	1.00	1.000	0.500	0.0000
12378-PeCDD	0.5		1.50	0.7500	0.7500	0.7500		0.59	0.2950	0.2950	0.2950	ND	1.00	0.500	0.250	0.0000
123478-HxCDD	0.1		82.69	8.2690	8.2690	8.2690		41.84	4.1840	4.1840	4.1840		31.65	3.165	3.165	3.1650
123678-HxCDD	0.1		10.62	1.0620	1.0620	1.0620		6.82	0.6820	0.6820	0.6820		10.46	1.046	1.046	1.0460
123789-HxCDD	0.1		2.82	0.2820	0.2820	0.2820	ND	1.20	0.1200	0.0600	0.0000	ND	1.10	0.110	0.055	0.0000
1234678-HpCDD	0.01		293.86	2.9386	2.9386	2.9386		65.03	0.6503	0.6503	0.6503		218.37	2.184	2.184	2.1837
12346789-OCDD	0.001		4383.43	4.3834	4.3834	4.3834		854.33	0.8543	0.8543	0.8543		2337.80	2.338	2.338	2.3378
2378TCDF	0.1		1972.76	197.2760	197.2760	197.2760		1376.10	137.6100	137.6100	137.6100		725.61	72.561	72.561	72.5610
12378-PeCDF	0.05		1304.55	65.2275	65.2275	65.2275		792.08	39.6040	39.6040	39.6040		371.88	18.594	18.594	18.5940
23478-PeCDF	0.5		664.49	332.2450	332.2450	332.2450		470.51	235.2550	235.2550	235.2550		242.30	121.150	121.150	121.1500
123478-HxCDF	0.1		597.68	59.7680	59.7680	59.7680		391.52	39.1520	39.1520	39.1520		170.73	17.073	17.073	17.0730
123678-HxCDF	0.1		126.67	12.6670	12.6670	12.6670		106.53	10.6530	10.6530	10.6530		40.24	4.024	4.024	4.0240
234678-HxCDF	0.1		50.19	5.0190	5.0190	5.0190		33.47	3.3470	3.3470	3.3470		15.82	1.582	1.582	1.5820
123789-HxCDF	0.1		1.68	0.1680	0.1680	0.1680		1.52	0.1520	0.1520	0.1520	ND	1.00	0.100	0.050	0.0000
1234678-HpCDF	0.01		670.77	6.7077	6.7077	6.7077		287.08	2.8708	2.8708	2.8708		275.84	2.758	2.758	2.7584
1234789-HpCDF	0.01		53.22	0.5322	0.5322	0.5322		26.62	0.2662	0.2662	0.2662		23.43	0.234	0.234	0.2343
12346789-OCDF	0.001		434.54	0.4345	0.4345	0.4345		113.81	0.1138	0.1138	0.1138		300.27	0.300	0.300	0.3003
nondetects = detection limit			TEQ=	705.73				TEQ=	476.81				TEQ=	248.72		
nondetects = 1/2 d.l.			*teq=*		*705.73*			*teq=*		*476.25*			*teq=*		*247.86*	
nondetects = zero			teq=			705.73		teq=			475.69		teq=			247.01
Moisture content (%)			24.55					28.37					23.18			
Organic carbon (%)			0.45					0.82					0.44			
Total organic matter (%)			0.78					1.42					0.76			
Sample I.D.			C-10					C-11					C-12			
tetra-PCDF (TCDF)			1973					1376					726			
penta-PCDF (PeCDF)			1969					1263					614			
hexa-PCDF (HxCDF)			776					533					227			
hepta-PCDF (HpCDF)			724					314					299			
octa-PCDF (OCDF)			435					114					300			
tetra-PCDD (TCDD)			8				ND	1				ND	1			
penta-PCDD (PeCDD)			1.5					0.6				ND	1			
hexa-PCDD (HxCDD)			96.1					48.7					42.1			
hepta-PCDD (HpCDD)			294					65					218			
octa-PCDD (OCDD)			4383					854					2338			
Summary concentrations of the homolog gr																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 2

Part 2 (continued)

			C-13				C-14				C-15			
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero	sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero	sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		0.54	0.540	0.540	0.540	0.55	0.550	0.550	0.5500	2.40	2.400	2.400	2.4000
12378-PeCDD	0.5	ND	1.00	0.500	0.250	0.000	2.29	1.145	1.145	1.1450	1.44	0.720	0.720	0.7200
123478-HxCDD	0.1		21.87	2.187	2.187	2.187	32.09	3.209	3.209	3.2090	49.78	4.978	4.978	4.9780
123678-HxCDD	0.1		3.19	0.319	0.319	0.319	15.76	1.576	1.576	1.5760	18.47	1.847	1.847	1.8470
123789-HxCDD	0.1	ND	1.00	0.100	0.050	0.000	ND 1.00	0.100	0.050	0.0000	1.33	0.133	0.133	0.1330
1234678-HpCDD	0.01		116.71	1.167	1.167	1.167	277.48	2.775	2.775	2.7748	252.82	2.528	2.528	2.5282
12346789-OCDD	0.001		1293.06	1.293	1.293	1.293	3260.86	3.261	3.261	3.2609	3902.75	3.903	3.903	3.9028
2378TCDF	0.1		279.45	27.945	27.945	27.945	637.85	63.785	63.785	63.7850	2734.66	273.466	273.466	273.4660
12378-PeCDF	0.05		175.84	8.792	8.792	8.792	313.39	15.670	15.670	15.6695	1798.75	89.938	89.938	89.9375
23478-PeCDF	0.5		82.92	41.460	41.460	41.460	172.21	86.105	86.105	86.1050	961.52	480.760	480.760	480.7600
123478-HxCDF	0.1		94.83	9.483	9.483	9.483	148.70	14.870	14.870	14.8700	715.08	71.508	71.508	71.5080
123678-HxCDF	0.1		17.40	1.740	1.740	1.740	28.18	2.818	2.818	2.8180	155.24	15.524	15.524	15.5240
234678-HxCDF	0.1		7.58	0.758	0.758	0.758	13.99	1.399	1.399	1.3990	56.28	5.628	5.628	5.6280
123789-HxCDF	0.1		0.90	0.090	0.090	0.090	1.99	0.199	0.199	0.1990	19.94	1.994	1.994	1.9940
1234678-HpCDF	0.01		156.34	1.563	1.563	1.563	290.16	2.902	2.902	2.9016	538.14	5.381	5.381	5.3814
1234789-HpCDF	0.01		19.87	0.199	0.199	0.199	28.79	0.288	0.288	0.2879	55.97	0.560	0.560	0.5597
12346789-OCDF	0.001		254.60	0.255	0.255	0.255	434.54	0.435	0.435	0.4345	564.28	0.564	0.564	0.5643
nondetects = detection limit			TEQ=	98.39			TEQ=	201.09			TEQ=	961.83		
nondetects = 1/2 d.l.			*teq=*		*98.09*		*teq=*		*201.04*		*teq=*		*961.83*	
nondetects = zero			teq=			97.79	teq=			200.99	teq=			961.83
Moisture content (%)			29.29				30.35				29.66			
Organic carbon (%)			0.87				1.04				0.62			
Total organic matter (%)			1.5				1.79				1.07			
Sample I.D.			C-13				C-14				C-15			
tetra-PCDF (TCDF)			279				638				2735			
penta-PCDF (PeCDF)			259				486				2760			
hexa-PCDF (HxCDF)			121				193				947			
hepta-PCDF (HpCDF)			176				319				594			
octa-PCDF (OCDF)			255				435				564			
tetra-PCDD (TCDD)			0.5				0.5				2.4			
penta-PCDD (PeCDD)		ND	1				2.3				1.4			
hexa-PCDD (HxCDD)			25.1				48				70			
hepta-PCDD (HpCDD)			117				277				253			
octa-PCDD (OCDD)			1293				3261				3903			

Summary concentrations of the homolog gr
Concentrations of all compound (except of
Recalculation for internal standards accoun
* OCDF concentrations were calculated ba
if required, OCDF concentrations can be n
ND = non detected, the detection limits wil

TABLE 9
Part 2

Part 2 (continued)																
				C-16					C-17					C-18		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1	ND	1.00	1.0000	0.5000	0.0000	ND	1.00	1.000	0.5000	0.0000	ND	1.00	1.000	0.5000	0.0000
12378-PeCDD	0.5		3.24	1.6200	1.6200	1.6200		1.28	0.640	0.6400	0.6400	ND	1.00	0.500	0.2500	0.0000
123478-HxCDD	0.1		2.42	0.2420	0.2420	0.2420	ND	1.50	0.150	0.0750	0.0000	ND	1.30	0.130	0.0650	0.0000
123678-HxCDD	0.1		14.60	1.4600	1.4600	1.4600	ND	1.50	0.150	0.0750	0.0000	ND	1.30	0.130	0.0650	0.0000
123789-HxCDD	0.1		3.43	0.3430	0.3430	0.3430		1.49	0.149	0.1490	0.1490	ND	1.30	0.130	0.0650	0.0000
1234678-HpCDD	0.01		499.92	4.9992	4.9992	4.9992		9.76	0.098	0.0976	0.0976		8.31	0.083	0.0831	0.0831
12346789-OCDD	0.001		7068.38	7.0684	7.0684	7.0684		104.60	0.105	0.1046	0.1046		83.25	0.083	0.0833	0.0833
2378TCDF	0.1		27.98	2.7980	2.7980	2.7980		4.38	0.438	0.4380	0.4380		3.11	0.311	0.3110	0.3110
12378-PeCDF	0.05		13.07	0.6535	0.6535	0.6535		3.39	0.170	0.1695	0.1695	ND	1.70	0.085	0.0425	0.0000
23478-PeCDF	0.5		5.82	2.9100	2.9100	2.9100		1.14	0.570	0.5700	0.5700	ND	1.70	0.850	0.4250	0.0000
123478-HxCDF	0.1		22.98	2.2980	2.2980	2.2980		1.41	0.141	0.1410	0.1410	ND	1.00	0.100	0.0500	0.0000
123678-HxCDF	0.1		5.54	0.5540	0.5540	0.5540	ND	1.30	0.130	0.0650	0.0000		13.20	1.320	1.3200	1.3200
234678-HxCDF	0.1	ND	1.30	0.1300	0.0650	0.0000	ND	1.00	0.100	0.0500	0.0000	ND	1.00	0.100	0.0500	0.0000
123789-HxCDF	0.1	ND	1.00	0.1000	0.0500	0.0000	ND	1.00	0.100	0.0500	0.0000	ND	1.00	0.100	0.0500	0.0000
1234678-HpCDF	0.01		263.91	2.6391	2.6391	2.6391		9.16	0.092	0.0916	0.0916		5.93	0.059	0.0593	0.0593
1234789-HpCDF	0.01		18.78	0.1878	0.1878	0.1878	ND	1.95	0.020	0.0098	0.0000	ND	2.65	0.027	0.0133	0.0000
12346789-OCDF	0.001		675.27	0.6753	0.6753	0.6753		7.55	0.008	0.0076	0.0076		4.93	0.005	0.0049	0.0049
nondetects = detection limit			TEQ=	29.68				TEQ=	4.06				TEQ=	5.01		
nondetects = 1/2 d.l.			*teq=*		*29.06*			*teq=*		*3.23*			*teq=*		*3.44*	
nondetects = zero			teq=			28.45		teq=			2.41		teq=			1.86
Moisture content (%)			30.26					28.54					30.65			
Organic carbon (%)			0.58					0.61					0.95			
Total organic matter (%)			1.00					1.05					1.65			
Sample I.D.			C-16					C-17					C-18			
tetra-PCDF (TCDF)			28					4.4					3.1			
penta-PCDF (PeCDF)			18.9					4.5					0			
hexa-PCDF (HxCDF)			28.5					1.4					13.2			
hepta-PCDF (HpCDF)			283					9.2					5.9			
octa-PCDF (OCDF)			675					7.5					4.9			
tetra-PCDD (TCDD)		ND	1				ND	1				ND	1			
penta-PCDD (PeCDD)			3.2					1.3				ND	1			
hexa-PCDD (HxCDD)			20.4					1.5				ND	1.3			
hepta-PCDD (HpCDD)			500					9.8					8.3			
octa-PCDD (OCDD)			7068					104.6					83.3			
Summary concentrations of the homolog gr																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 2

Part 2 (continued)																
				C-19					13						14	
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1	ND	1.00	1.000	0.5000	0.0000		3.55	3.550	3.5500	3.5500		6.98	6.980	6.9800	6.9800
12378-PeCDD	0.5	ND	1.00	0.500	0.2500	0.0000		1.03	0.515	0.5150	0.5150	ND	1.00	0.500	0.2500	0.0000
123478-HxCDD	0.1	ND	1.00	0.100	0.0500	0.0500		50.84	5.084	5.0840	5.0840		69.79	6.979	6.9790	6.9790
123678-HxCDD	0.1		2.06	0.206	0.2060	0.2060		26.61	2.661	2.6610	2.6610		5.89	0.589	0.5890	0.5890
123789-HxCDD	0.1	ND	1.15	0.115	0.0575	0.0000	ND	1.40	0.140	0.0700	0.0000		1.69	0.169	0.1690	0.1690
1234678-HpCDD	0.01		15.98	0.160	0.1598	0.1598		1269.61	12.696	12.6961	12.6961		211.08	2.111	2.1108	2.1108
12346789-OCDD	0.001		121.31	0.121	0.1213	0.1213		18577.63	18.578	18.5776	18.5776		1909.95	1.910	1.9100	1.9100
2378TCDF	0.1		36.21	3.621	3.6210	3.6210		3024.20	302.420	302.4200	302.4200		2839.23	283.923	283.9230	283.9230
12378-PeCDF	0.05		31.34	1.567	1.5670	1.5670		1958.51	97.926	97.9255	97.9255		1362.21	68.111	68.1105	68.1105
23478-PeCDF	0.5		14.61	7.305	7.3050	7.3050		1156.55	578.275	578.2750	578.2750		862.09	431.045	431.0450	431.0450
123478-HxCDF	0.1		20.08	2.008	2.0080	2.0080		815.44	81.544	81.5440	81.5440		618.89	61.889	61.8890	61.8890
123678-HxCDF	0.1	ND	1.35	0.135	0.0675	0.0000		213.41	21.341	21.3410	21.3410		137.71	13.771	13.7710	13.7710
234678-HxCDF	0.1	ND	1.35	0.135	0.0675	0.0000		88.29	8.829	8.8290	8.8290		59.10	5.910	5.9100	5.9100
123789-HxCDF	0.1	ND	1.00	0.100	0.0500	0.0000	ND	1.36	0.136	0.0680	0.0000		7.51	0.751	0.7510	0.7510
1234678-HpCDF	0.01		12.22	0.122	0.1222	0.1222		909.54	9.095	9.0954	9.0954		525.50	5.255	5.2550	5.2550
1234789-HpCDF	0.01	ND	2.15	0.022	0.0108	0.0000		81.57	0.816	0.8157	0.8157		49.93	0.499	0.4993	0.4993
12346789-OCDF	0.001		9.02	0.009	0.0090	0.0090		2288.67	2.289	2.2887	2.2887		315.35	0.315	0.3154	0.3154
nondetects = detection limit			TEQ=	17.23				TEQ=	1145.89				TEQ=	890.71		
nondetects = 1/2 d.l.			*teq=*		*16.17*			*teq=*		*1145.76*			*teq=*		*890.46*	
nondetects = zero			teq=			15.17		teq=			1145.62		teq=			890.21
Moisture content (%)			29.12					27.54					24.33			
Organic carbon (%)			0.58					0.94					1.17			
Total organic matter (%)			1.00					1.63					2.01			
Sample I.D.			C-19					13					14			
tetra-PCDF (TCDF)			36.2					3024					2839			
penta-PCDF (PeCDF)			45.9					3115					2224			
hexa-PCDF (HxCDF)			20.1					1117					823			
hepta-PCDF (HpCDF)			12.2					991					575			
octa-PCDF (OCDF)			9					2289					315			
tetra-PCDD (TCDD)		ND	1					3.6					7			
penta-PCDD (PeCDD)		ND	1					1				ND	1			
hexa-PCDD (HxCDD)			2.1					77.4					77.4			
hepta-PCDD (HpCDD)			16					1270					211			
octa-PCDD (OCDD)			121					18578					1910			
Summary concentrations of the homolog gr																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 2

Part 2 (continued)																
				15					16					23		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		2.33	2.330	2.3300	2.3300		2.53	2.530	2.5300	2.5300		15.54	15.540	15.5400	15.5400
12378-PeCDD	0.5		2.10	1.050	1.0500	1.0500	ND	1.00	0.500	0.2500	0.0000		12.38	6.190	6.1900	6.1900
123478-HxCDD	0.1		51.09	5.109	5.1090	5.1090		41.24	4.124	4.1240	4.1240		121.86	12.186	12.1860	12.1860
123678-HxCDD	0.1		28.82	2.882	2.8820	2.8820		3.77	0.377	0.3770	0.3770		52.62	5.262	5.2620	5.2620
123789-HxCDD	0.1		1.85	0.185	0.1850	0.0000		1.99	0.199	0.1990	0.1990		15.85	1.585	1.5850	1.5850
1234678-HpCDD	0.01		480.00	4.800	4.8000	4.8000		142.76	1.428	1.4276	1.4276		882.86	8.829	8.8286	8.8286
12346789-OCDD	0.001		3460.10	3.460	3.4601	3.4601		1306.73	1.307	1.3067	1.3067		9539.41	9.539	9.5394	9.5394
2378TCDF	0.1		2117.93	211.793	211.7930	211.7930		5673.44	567.344	567.3440	567.3440		691.92	69.192	69.1920	69.1920
12378-PeCDF	0.05		1531.82	76.591	76.5910	76.5910		2131.69	106.585	106.5845	106.5845		538.39	26.920	26.9195	26.9195
23478-PeCDF	0.5		886.75	443.375	443.3750	443.3750		1413.24	706.620	706.6200	706.6200		329.69	164.845	164.8450	164.8450
123478-HxCDF	0.1		950.83	95.083	95.0830	95.0830		1000.74	100.074	100.0740	100.0740		279.69	27.969	27.9690	27.9690
123678-HxCDF	0.1		209.74	20.974	20.9740	20.9740		204.43	20.443	20.4430	20.4430		48.34	4.834	4.8340	4.8340
234678-HxCDF	0.1		85.80	8.580	8.5800	8.5800		100.29	10.029	10.0290	10.0290		30.70	3.070	3.0700	3.0700
123789-HxCDF	0.1		1.70	0.170	0.1700	0.1700		2.03	0.203	0.2030	0.0000	ND	1.95	0.195	0.0975	0.0000
1234678-HpCDF	0.01		554.31	5.543	5.5431	5.5431		485.14	4.851	4.8514	4.8514		1236.97	12.370	12.3697	12.3697
1234789-HpCDF	0.01		60.62	0.606	0.6062	0.6062		61.23	0.612	0.6123	0.6123		77.28	0.773	0.7728	0.7728
12346789-OCDF	0.001		578.04	0.578	0.5780	0.5780		344.26	0.344	0.3443	0.3443		863.59	0.864	0.8636	0.8636
nondetects = detection limit			TEQ=	883.11				TEQ=	1527.57				TEQ=	370.16		
nondetects = 1/2 d.l.			*teq=*		*883.11*			*teq=*		*1527.32*			*teq=*		*370.06*	
nondetects = zero			teq=			882.92		teq=			1526.87		teq=			369.97
Moisture content (%)			23.15					27.22					59.75			
Organic carbon (%)			0.38					0.46					4.05			
Total organic matter (%)			0.66					0.8					6.98			
Sample I.D.			15					16					23			
tetra-PCDF (TCDF)			2118					5673.0					692			
penta-PCDF (PeCDF)			2419					3545.0					868			
hexa-PCDF (HxCDF)			1248					1307.0					359			
hepta-PCDF (HpCDF)			615					546.0					1314			
octa-PCDF (OCDF)			578					344.0					864			
tetra-PCDD (TCDD)			2.3					2.5					15.5			
penta-PCDD (PeCDD)			2.1				ND	1.0					12.4			
hexa-PCDD (HxCDD)			81.8					47.0					190			
hepta-PCDD (HpCDD)			480					143.0					883			
octa-PCDD (OCDD)			3460					1307.0					9539			
Summary concentrations of the homolog gı																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba:																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 2

Part 2 (continued)																
				37					38					39		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		5.13	5.130	5.1300	5.1300	ND	1.00	1.000	0.5000	0.0000	ND	1.00	1.000	0.5000	0.0000
12378-PeCDD	0.5	ND	1.00	0.500	0.2500	0.0000	ND	1.00	0.500	0.2500	0.0000	ND	1.00	0.500	0.2500	0.0000
123478-HxCDD	0.1		90.60	9.060	9.0600	9.0600		13.22	1.322	1.3220	1.3220		8.46	0.846	0.8460	0.8460
123678-HxCDD	0.1		18.20	1.820	1.8200	1.8200		10.01	1.001	1.0010	1.0010		2.16	0.216	0.2160	0.2160
123789-HxCDD	0.1		2.71	0.271	0.2710	0.2710		10.35	1.035	1.0350	1.0350		1.30	0.130	0.1300	0.1300
1234678-HpCDD	0.01		168.61	1.686	1.6861	1.6861		91.38	0.914	0.9138	0.9138		47.13	0.471	0.4713	0.4713
12346789-OCDD	0.001		2017.35	2.017	2.0174	2.0174		563.92	0.564	0.5639	0.5639		377.90	0.378	0.3779	0.3779
2378TCDF	0.1		2327.82	232.782	232.7820	232.7820		559.37	55.937	55.9370	55.9370		281.15	28.115	28.1150	28.1150
12378-PeCDF	0.05		1781.65	89.083	89.0825	89.0825		493.93	24.697	24.6965	24.6965		314.81	15.741	15.7405	15.7405
23478-PeCDF	0.5		1043.81	521.905	521.9050	521.9050		273.21	136.605	136.6050	136.6050		135.13	67.565	67.5650	67.5650
123478-HxCDF	0.1		1046.97	104.697	104.6970	104.6970		233.31	23.331	23.3310	23.3310		151.64	15.164	15.1640	15.1640
123678-HxCDF	0.1		247.59	24.759	24.7590	24.7590		46.51	4.651	4.6510	4.6510		29.45	2.945	2.9450	2.9450
234678-HxCDF	0.1		105.88	10.588	10.5880	10.5880		16.51	1.651	1.6510	1.6510		12.39	1.239	1.2390	1.2390
123789-HxCDF	0.1	ND	1.72	0.172	0.0860	0.0000	ND	1.95	0.195	0.0975	0.0000	ND	1.00	0.100	0.0500	0.0000
1234678-HpCDF	0.01		581.90	5.819	5.8190	5.8190		121.12	1.211	1.2112	1.2112		87.65	0.877	0.8765	0.8765
1234789-HpCDF	0.01		163.43	1.634	1.6343	1.6343		23.47	0.235	0.2347	0.2347		11.83	0.118	0.1183	0.1183
12346789-OCDF	0.001		188.70	0.189	0.1887	0.1887		71.01	0.071	0.0710	0.0710		42.99	0.043	0.0430	0.0430
nondetects = detection limit			TEQ=	1012.11				TEQ=	254.92				TEQ=	135.45		
nondetects = 1/2 d.l.			*teq=*		*1011.78*			*teq=*		*254.07*			*teq=*		*134.65*	
nondetects = zero			teq=			1011.44		teq=			253.22		teq=			133.85
Moisture content (%)			35.03					23.82					20.63			
Organic carbon (%)			1.63					0.29					0.11			
Total organic matter (%)			2.81					0.51					0.19			
Sample I.D.			37					38					39			
tetra-PCDF (TCDF)			2328.0					559.0					281.0			
penta-PCDF (PeCDF)			2825.0					767.0					450.0			
hexa-PCDF (HxCDF)			1400.0					296.0					193.0			
hepta-PCDF (HpCDF)			745.0					145.0					99.5			
octa-PCDF (OCDF)			189.0					71.0					43.0			
tetra-PCDD (TCDD)			5.1				ND	1.0				ND	1.0			
penta-PCDD (PeCDD)		ND	1.0				ND	1.0				ND	1.0			
hexa-PCDD (HxCDD)			112.0					33.6					11.9			
hepta-PCDD (HpCDD)			169.0					91.4					47.1			
octa-PCDD (OCDD)			2017.0					563.9					377.9			

Summary concentrations of the homolog gr
Concentrations of all compound (except of
Recalculation for internal standards accoun
* OCDF concentrations were calculated ba
if required, OCDF concentrations can be n
ND = non detected, the detection limits wil

TABLE 9
Part 2

Part 2 (continued)																
				52					53					54		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1	ND	1.10	1.100	0.5500	0.0000		20.37	20.370	20.3700	20.3700	ND	1.10	1.100	0.5500	0.0000
12378-PeCDD	0.5	ND	1.00	0.500	0.2500	0.0000	ND	1.00	0.500	0.2500	0.0000		0.89	0.445	0.4450	0.4450
123478-HxCDD	0.1		13.58	1.358	1.3580	1.3580		29.87	2.987	2.9870	2.9870		40.51	4.051	4.0510	4.0510
123678-HxCDD	0.1		1.39	0.139	0.1390	0.1390		7.42	0.742	0.7420	0.7420		10.37	1.037	1.0370	1.0370
123789-HxCDD	0.1	ND	1.30	0.130	0.0650	0.0000	ND	1.70	0.170	0.0850	0.0000		3.08	0.308	0.3080	0.3080
1234678-HpCDD	0.01		26.23	0.262	0.2623	0.2623		126.69	1.267	1.2669	1.2669		139.40	1.394	1.3940	1.3940
12346789-OCDD	0.001		288.55	0.289	0.2886	0.2886		1354.62	1.355	1.3546	1.3546		1375.84	1.376	1.3758	1.3758
2378TCDF	0.1		61.80	6.180	6.1800	6.1800		1880.57	188.057	188.0570	188.0570		1060.58	106.058	106.0580	106.0580
12378-PeCDF	0.05		32.71	1.636	1.6355	1.6355		1395.81	69.791	69.7905	69.7905		298.69	14.935	14.9345	14.9345
23478-PeCDF	0.5		17.49	8.745	8.7450	8.7450		991.73	495.865	495.8650	495.8650		223.09	111.545	111.5450	111.5450
123478-HxCDF	0.1		12.97	1.297	1.2970	1.2970		595.09	59.509	59.5090	59.5090		122.99	12.299	12.2990	12.2990
123678-HxCDF	0.1		3.80	0.380	0.3800	0.3800		143.57	14.357	14.3570	14.3570		28.36	2.836	2.8360	2.8360
234678-HxCDF	0.1	ND	1.00	0.100	0.0500	0.0000		92.61	9.261	9.2610	9.2610		15.16	1.516	1.5160	1.5160
123789-HxCDF	0.1	ND	1.00	0.100	0.0500	0.0000		7.06	0.706	0.7060	0.7060	ND	1.00	0.100	0.0500	0.0000
1234678-HpCDF	0.01		33.05	0.331	0.3305	0.3305		281.26	2.813	2.8126	2.8126		279.48	2.795	2.7948	2.7948
1234789-HpCDF	0.01		4.35	0.044	0.0435	0.0435		43.24	0.432	0.4324	0.4324		19.21	0.192	0.1921	0.1921
12346789-OCDF	0.001		21.94	0.022	0.0219	0.0219		201.11	0.201	0.2011	0.2011		238.67	0.239	0.2387	0.2387
nondetects = detection limit			TEQ=	22.61				TEQ=	868.38				TEQ=	262.22		
nondetects = 1/2 d.l.			*teq=*		*21.65*			*teq=*		*868.05*			*teq=*		*261.62*	
nondetects = zero			teq=			20.68		teq=			867.71		teq=			261.02
Moisture content (%)			21.77					27.84					22.02			
Organic carbon (%)			0.31					0.52					0.29			
Total organic matter (%)			0.54					0.89					0.49			
Sample I.D.			52					53					54			
tetra-PCDF (TCDF)			61.8					1881					1061			
penta-PCDF (PeCDF)			50.2					2388					522			
hexa-PCDF (HxCDF)			16.8					838					167			
hepta-PCDF (HpCDF)			37.4					325					299			
octa-PCDF (OCDF)			21.9					201					239			
tetra-PCDD (TCDD)		ND	1.1					20.4				ND	1.1			
penta-PCDD (PeCDD)		ND	1.0				ND	1.00					0.9			
hexa-PCDD (HxCDD)			15.0					37.3					54			
hepta-PCDD (HpCDD)			26.2					127					139			
octa-PCDD (OCDD)			288.6					1355					1376			

Summary concentrations of the homolog gr
Concentrations of all compound (except of
Recalculation for internal standards accoun
* OCDF concentrations were calculated ba
if required, OCDF concentrations can be n
ND = non detected, the detection limits wil

TABLE 9
Part 2

Part 2 (continued)																
				55						59					60	
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		4.34	4.340	4.3400	4.3400	ND	1.00	1.000	0.5000	0.0000	ND	1.10	1.100	0.5500	0.0000
12378-PeCDD	0.5		2.18	1.090	1.0900	1.0900	ND	1.00	0.500	0.2500	0.0000	ND	1.20	0.600	0.3000	0.0000
123478-HxCDD	0.1		155.02	15.502	15.5020	15.5020	ND	1.30	0.130	0.0650	0.0000	ND	1.30	0.130	0.0650	0.0000
123678-HxCDD	0.1		40.51	4.051	4.0510	4.0510	ND	1.30	0.130	0.0650	0.0000	ND	1.30	0.130	0.0650	0.0000
123789-HxCDD	0.1		1.44	0.144	0.1440	0.1440	ND	1.30	0.130	0.0650	0.0000	ND	1.30	0.130	0.0650	0.0000
1234678-HpCDD	0.01		531.96	5.320	5.3196	5.3196		17.41	0.174	0.1741	0.1741		8.40	0.084	0.0840	0.0840
12346789-OCDD	0.001		6670.02	6.670	6.6700	6.6700		142.08	0.142	0.1421	0.1421		63.71	0.064	0.0637	0.0637
2378TCDF	0.1		5518.05	551.805	551.8050	551.8050		6.52	0.652	0.6520	0.6520		8.23	0.823	0.8230	0.8230
12378-PeCDF	0.05		3949.58	197.479	197.4790	197.4790		4.53	0.227	0.2265	0.2265		6.13	0.307	0.3065	0.3065
23478-PeCDF	0.5		2029.73	1014.865	1014.8650	1014.8650	ND	1.40	0.700	0.3500	0.0000		1.78	0.890	0.8900	0.8900
123478-HxCDF	0.1		2035.24	203.524	203.5240	203.5240		2.12	0.212	0.2120	0.2120		3.75	0.375	0.3750	0.3750
123678-HxCDF	0.1		376.46	37.646	37.6460	37.6460	ND	1.45	0.145	0.0725	0.0000	ND	1.60	0.160	0.0800	0.0000
234678-HxCDF	0.1		148.05	14.805	14.8050	14.8050	ND	1.00	0.100	0.0500	0.0000	ND	1.60	0.160	0.0800	0.0000
123789-HxCDF	0.1		25.80	2.580	2.5800	2.5800	ND	1.00	0.100	0.0500	0.0000	ND	2.00	0.200	0.1000	0.0000
1234678-HpCDF	0.01		1359.68	13.597	13.5968	13.5968		7.53	0.075	0.0753	0.0753		5.08	0.051	0.0508	0.0508
1234789-HpCDF	0.01		139.72	1.397	1.3972	1.3972	ND	1.00	0.010	0.0050	0.0000	ND	2.50	0.025	0.0125	0.0000
12346789-OCDF	0.001		1522.50	1.523	1.5225	1.5225		6.10	0.006	0.0061	0.0061		5.62	0.006	0.0056	0.0056
nondetects = detection limit			TEQ=	2076.34				TEQ=	4.43				TEQ=	5.23		
nondetects = 1/2 d.l.			*teq=*		*2076.34*			*teq=*		*2.96*			*teq=*		*3.92*	
nondetects = zero			teq=			2076.34		teq=			1.49		teq=			2.60
Moisture content (%)			32.94					26.34					21.01			
Organic carbon (%)			0.49					0.23					0.6			
Total organic matter (%)			0.85					0.4					1.04			
Sample I.D.			55					59					60			
tetra-PCDF (TCDF)			5518					6.5					8.2			
penta-PCDF (PeCDF)			5979					4.5					7.9			
hexa-PCDF (HxCDF)			2586					2.1					3.8			
hepta-PCDF (HpCDF)			1499					7.5					5.1			
octa-PCDF (OCDF)			1523					6.1					5.6			
tetra-PCDD (TCDD)			4.3				ND	1.0				ND	1.1			
penta-PCDD (PeCDD)			2.2				ND	1.0				ND	1.2			
hexa-PCDD (HxCDD)			197				ND	1.3				ND	1.3			
hepta-PCDD (HpCDD)			532					17.4					8.4			
octa-PCDD (OCDD)			6670					142.2					63.7			
Summary concentrations of the homolog gr																
Concentrations of all compound (except of																
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ND = non detected, the detection limits wil																

TABLE 9
Part 2

Part 2 (continued)																
				SS-1					SS-2					SS-3		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		15.31	15.310	15.3100	15.3100		33.38	33.380	33.3800	33.3800		1.52	1.520	1.5200	1.5200
12378-PeCDD	0.5		11.89	5.945	5.9450	5.9450		17.97	8.985	8.9850	8.9850	ND	1.00	0.500	0.2500	0.0000
123478-HxCDD	0.1		99.18	9.918	9.9180	9.9180		208.10	20.810	20.8100	20.8100	ND	1.30	0.130	0.0650	0.0000
123678-HxCDD	0.1		58.69	5.869	5.8690	5.8690		105.33	10.533	10.5330	10.5330	ND	1.30	0.130	0.0650	0.0000
123789-HxCDD	0.1		23.36	2.336	2.3360	2.3360		31.82	3.182	3.1820	3.1820	ND	1.30	0.130	0.0650	0.0000
1234678-HpCDD	0.01		1016.20	10.162	10.1620	10.1620		2735.37	27.354	27.3537	27.3537		36.32	0.363	0.3632	0.3632
12346789-OCDD	0.001		11122.56	11.123	11.1226	11.1226		26560.18	26.560	26.5602	26.5602		369.60	0.370	0.3696	0.3696
2378TCDF	0.1		950.14	95.014	95.0140	95.0140		2443.05	244.305	244.3050	244.3050		13.64	1.364	1.3640	1.3640
12378-PeCDF	0.05		584.12	29.206	29.2060	29.2060		1430.97	71.549	71.5485	71.5485		9.07	0.454	0.4535	0.4535
23478-PeCDF	0.5		356.00	178.000	178.0000	178.0000		888.76	444.380	444.3800	444.3800		6.94	3.470	3.4700	3.4700
123478-HxCDF	0.1		482.45	48.245	48.2450	48.2450		1059.99	105.999	105.9990	105.9990		6.06	0.606	0.6060	0.6060
123678-HxCDF	0.1		73.48	7.348	7.3480	7.3480		186.34	18.634	18.6340	18.6340		1.29	0.129	0.1290	0.1290
234678-HxCDF	0.1		51.16	5.116	5.1160	5.1160		97.83	9.783	9.7830	9.7830	ND	1.00	0.100	0.0500	0.0000
123789-HxCDF	0.1		3.95	0.395	0.3950	0.3950		2.69	0.269	0.2690	0.2690	ND	1.00	0.100	0.0500	0.0000
1234678-HpCDF	0.01		1973.82	19.738	19.7382	19.7382		4060.38	40.604	40.6038	40.6038		15.66	0.157	0.1566	0.1566
1234789-HpCDF	0.01		76.70	0.767	0.7670	0.7670		204.74	2.047	2.0474	2.0474	ND	1.90	0.019	0.0095	0.0000
12346789-OCDF	0.001		2066.98	2.067	2.0670	2.0670		6795.69	6.796	6.7957	6.7957		11.90	0.012	0.0119	0.0119
nondetects = detection limit			TEQ=	446.56				TEQ=	1075.17				TEQ=	9.55		
nondetects = 1/2 d.l.			*teq=*		*446.56*			*teq=*		*1075.17*			*teq=*		*9.00*	
nondetects = zero			teq=			446.56		teq=			1075.17		teq=			8.44
Moisture content (%)			41.17					30.88					28.32			
Organic carbon (%)			6.46					5.06					4.00			
Total organic matter (%)			11.14					8.72					6.89			
Sample I.D.			SS-1					SS-2					SS-3			
tetra-PCDF (TCDF)			950					2443					13.6			
penta-PCDF (PeCDF)			940					2320					16			
hexa-PCDF (HxCDF)			611					1347					7.3			
hepta-PCDF (HpCDF)			2051					4265					15.7			
octa-PCDF (OCDF)			2067					6796					11.9			
tetra-PCDD (TCDD)			15.3					33.4					1.5			
penta-PCDD (PeCDD)			11.9					18				ND	1.0			
hexa-PCDD (HxCDD)			181					345				ND	1.3			
hepta-PCDD (HpCDD)			1016					2735					36.3			
octa-PCDD (OCDD)			11123					26560					369.6			
Summary concentrations of the homolog gr																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 2

Part 2 (continued)																
				SS-4					SS-5					SS-6		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		1.55	1.550	1.5500	1.5500		40.99	40.990	40.9900	40.9900		2.25	2.250	2.2500	2.2500
12378-PeCDD	0.5	ND	1.00	0.500	0.2500	0.0000		27.75	13.875	13.8750	13.8750		1.21	0.605	0.6050	0.6050
123478-HxCDD	0.1	ND	1.00	0.100	0.0500	0.0000		287.88	28.788	28.7880	28.7880		10.11	1.011	1.0110	1.0110
123678-HxCDD	0.1		2.09	0.209	0.2090	0.2090		142.84	14.284	14.2840	14.2840		22.40	2.240	2.2400	2.2400
123789-HxCDD	0.1	ND	1.50	0.150	0.0750	0.0000		53.02	5.302	5.3020	5.3020		5.82	0.582	0.5820	0.5820
1234678-HpCDD	0.01		61.29	0.613	0.6129	0.6129		2998.75	29.988	29.9875	29.9875		221.05	2.211	2.2105	2.2105
12346789-OCDD	0.001		380.13	0.380	0.3801	0.3801		34835.28	34.835	34.8353	34.8353		2462.16	2.462	2.4622	2.4622
2378TCDF	0.1		1.52	0.152	0.1520	0.1520		1315.76	131.576	131.5760	131.5760		831.82	83.182	83.1820	83.1820
12378-PeCDF	0.05		1.44	0.072	0.0720	0.0720		699.72	34.986	34.9860	34.9860		429.69	21.485	21.4845	21.4845
23478-PeCDF	0.5	ND	1.00	0.500	0.2500	0.0000		456.51	228.255	228.2550	228.2550		294.11	147.055	147.0550	147.0550
123478-HxCDF	0.1		7.49	0.749	0.7490	0.7490		619.14	61.914	61.9140	61.9140		216.36	21.636	21.6360	21.6360
123678-HxCDF	0.1	ND	1.00	0.100	0.0500	0.0000		115.82	11.582	11.5820	11.5820		45.67	4.567	4.5670	4.5670
234678-HxCDF	0.1	ND	1.00	0.100	0.0500	0.0000		68.36	6.836	6.8360	6.8360		27.18	2.718	2.7180	2.7180
123789-HxCDF	0.1	ND	1.00	0.100	0.0500	0.0000		1.70	0.170	0.1700	0.1700	ND	1.40	0.140	0.0700	0.0000
1234678-HpCDF	0.01		38.05	0.381	0.3805	0.3805		4131.74	41.317	41.3174	41.3174		297.60	2.976	2.9760	2.9760
1234789-HpCDF	0.01	ND	2.25	0.023	0.0113	0.0000		243.70	2.437	2.4370	2.4370		21.94	0.219	0.2194	0.2194
12346789-OCDF	0.001		18.83	0.019	0.0188	0.0188		7565.38	7.565	7.5654	7.5654		227.08	0.227	0.2271	0.2271
nondetects = detection limit			TEQ=	5.70				TEQ=	694.70				TEQ=	295.57		
nondetects = 1/2 d.l.			*teq=*		*4.91*			*teq=*		*694.70*			*teq=*		*295.50*	
nondetects = zero			teq=			4.12		teq=			694.70		teq=			295.43
Moisture content (%)			29.84					21.51					16.46			
Organic carbon (%)			4.36					2.38					1.30			
Total organic matter (%)			7.52					4.10					2.24			
Sample I.D.			SS-4					SS-5					SS-6			
tetra-PCDF (TCDF)			1.5					1316					832			
penta-PCDF (PeCDF)			1.4					1156					724			
hexa-PCDF (HxCDF)			7.5					805					289			
hepta-PCDF (HpCDF)			38.0					4375					320			
octa-PCDF (OCDF)			18.8					7565					227			
tetra-PCDD (TCDD)			1.5					41					2.2			
penta-PCDD (PeCDD)		ND	1.0					27.8					1.2			
hexa-PCDD (HxCDD)			2.1					484					38.3			
hepta-PCDD (HpCDD)			61.3					2999					221			
octa-PCDD (OCDD)			380.1					34835					2462			
Summary concentrations of the homolog gr																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 2

Part 2 (continued)																
				SS-7					SS-7r					SS-8		
Analyte	TEF		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero		sampled pg/g	toxic eq. pg/g	*nondetect 1/2 d.l.*	nondetect zero
2378-TCDD	1		5.71	5.710	5.7100	5.7100		7.80	7.800	7.8000	7.8000		4.25	4.250	4.2500	4.2500
12378-PeCDD	0.5		5.08	2.540	2.5400	2.5400		4.17	2.085	2.0850	2.0850		3.71	1.855	1.8550	1.8550
123478-HxCDD	0.1		77.78	7.778	7.7780	7.7780		86.94	8.694	8.6940	8.6940		51.30	5.130	5.1300	5.1300
123678-HxCDD	0.1		85.83	8.583	8.5830	8.5830		68.48	6.848	6.8480	6.8480		19.15	1.915	1.9150	1.9150
123789-HxCDD	0.1		39.06	3.906	3.9060	3.9060		20.32	2.032	2.0320	2.0320		4.93	0.493	0.4930	0.4930
1234678-HpCDD	0.01		1209.01	12.090	12.0901	12.0901		917.28	9.173	9.1728	9.1728		422.36	4.224	4.2236	4.2236
12346789-OCDD	0.001		9453.71	9.454	9.4537	9.4537		11577.69	11.578	11.5777	11.5777		3765.17	3.765	3.7652	3.7652
2378TCDF	0.1		3920.61	392.061	392.0610	392.0610		3274.44	327.444	327.4440	327.4440		2637.92	263.792	263.7920	263.7920
12378-PeCDF	0.05		1917.22	95.861	95.8610	95.8610		1997.95	99.898	99.8975	99.8975		1579.22	78.961	78.9610	78.9610
23478-PeCDF	0.5		1137.82	568.910	568.9100	568.9100		1214.53	607.265	607.2650	607.2650		888.32	444.160	444.1600	444.1600
123478-HxCDF	0.1		1042.46	104.246	104.2460	104.2460		1068.01	106.801	106.8010	106.8010		865.35	86.535	86.5350	86.5350
123678-HxCDF	0.1		254.61	25.461	25.4610	25.4610		197.43	19.743	19.7430	19.7430		150.76	15.076	15.0760	15.0760
234678-HxCDF	0.1		106.63	10.663	10.6630	10.6630		83.06	8.306	8.3060	8.3060		59.84	5.984	5.9840	5.9840
123789-HxCDF	0.1		3.94	0.394	0.3940	0.3940		3.07	0.307	0.3070	0.3070		4.08	0.408	0.4080	0.4080
1234678-HpCDF	0.01		1131.51	11.315	11.3151	11.3151		1251.47	12.515	12.5147	12.5147		727.99	7.280	7.2799	7.2799
1234789-HpCDF	0.01		129.21	1.292	1.2921	1.2921		112.18	1.122	1.1218	1.1218		153.80	1.538	1.5380	1.5380
12346789-OCDF	0.001		859.23	0.859	0.8592	0.8592		2231.17	2.231	2.2312	2.2312		1364.57	1.365	1.3646	1.3646
nondetects = detection limit			TEQ=	1261.12				TEQ=	1233.84				TEQ=	926.73		
nondetects = 1/2 d.l.			*teq=*		*1261.12*			*teq=*		*1233.84*			*teq=*		*926.73*	
nondetects = zero			teq=			1261.12		teq=			1233.84		teq=			926.73
Moisture content (%)			24.18					24.11					24.07			
Organic carbon (%)			3.38					3.21					1.19			
Total organic matter (%)			5.82					5.54					2.05			
Sample I.D.			SS-7					SS-7r					SS-8			
tetra-PCDF (TCDF)			3921					3274					2638			
penta-PCDF (PeCDF)			3055					3212					2468			
hexa-PCDF (HxCDF)			1408					1352					1080			
hepta-PCDF (HpCDF)			1261					1364					882			
octa-PCDF (OCDF)			859					2231					1365			
tetra-PCDD (TCDD)			5.7					7.8					4.2			
penta-PCDD (PeCDD)			5.1					4.2					3.7			
hexa-PCDD (HxCDD)			203					176					75.4			
hepta-PCDD (HpCDD)			1209					917					422			
octa-PCDD (OCDD)			9454					11578					3765			
Summary concentrations of the homolog gr																
Concentrations of all compound (except of																
Recalculation for internal standards accoun																
* OCDF concentrations were calculated ba																
if required, OCDF concentrations can be n																
ND = non detected, the detection limits wil																

TABLE 9
Part 2

Part 2 (continued)											
				SS-9					SS-10		
Analyte	TEF		sampled	toxic eq.	*nondetect*	nondetect		sampled	toxic eq.	*nondetect*	nondetect
			pg/g	pg/g	*1/2 d.l.*	zero		pg/g	pg/g	*1/2 d.l.*	zero
2378-TCDD	1		4.58	4.580	4.5800	4.5800	ND	1.00	1.000	0.5000	0.0000
12378-PeCDD	0.5		4.39	2.195	2.1950	2.1950		0.72	0.360	0.3600	0.3600
123478-HxCDD	0.1		71.64	7.164	7.1640	7.1640	ND	1.20	0.120	0.0600	0.0000
123678-HxCDD	0.1		33.44	3.344	3.3440	3.3440		1.50	0.150	0.1500	0.1500
123789-HxCDD	0.1		7.17	0.717	0.7170	0.7170		1.37	0.137	0.1370	0.1370
1234678-HpCDD	0.01		574.20	5.742	5.7420	5.7420		11.73	0.117	0.1173	0.1173
12346789-OCDD	0.001		6320.70	6.321	6.3207	6.3207		103.53	0.104	0.1035	0.1035
2378TCDF	0.1		3406.24	340.624	340.6240	340.6240		1.00	0.100	0.1000	0.1000
12378-PeCDF	0.05		3084.30	154.215	154.2150	154.2150		1.21	0.061	0.0605	0.0605
23478-PeCDF	0.5		1460.63	730.315	730.3150	730.3150		1.13	0.565	0.5650	0.5650
123478-HxCDF	0.1		1749.01	174.901	174.9010	174.9010	ND	1.00	0.100	0.0500	0.0000
123678-HxCDF	0.1		318.54	31.854	31.8540	31.8540	ND	1.00	0.100	0.0500	0.0000
234678-HxCDF	0.1		116.26	11.626	11.6260	11.6260	ND	1.00	0.100	0.0500	0.0000
123789-HxCDF	0.1		13.30	1.330	1.3300	1.3300	ND	1.00	0.100	0.0500	0.0000
1234678-HpCDF	0.01		1243.62	12.436	12.4362	12.4362		3.91	0.039	0.0391	0.0391
1234789-HpCDF	0.01		125.09	1.251	1.2509	1.2509	ND	2.00	0.020	0.0100	0.0000
12346789-OCDF	0.001		1782.44	1.782	1.7824	1.7824	ND	3.40	0.003	0.0034	0.0000
nondetects = detection limit			TEQ=	1490.40				TEQ=	3.18		
nondetects = 1/2 d.l.			*teq=*		*1490.40*			*teq=*		*2.41*	
nondetects = zero			teq=			1490.40		teq=			1.63
Moisture content (%)			18.58					25.38			
Organic carbon (%)			2.24					3.56			
Total organic matter (%)			3.85					6.14			
Sample I.D.			SS-9					SS-10			
tetra-PCDF (TCDF)			3406					1.0			
penta-PCDF (PeCDF)			4545					2.3			
hexa-PCDF (HxCDF)			2197				ND	1.0			
hepta-PCDF (HpCDF)			1369					3.9			
octa-PCDF (OCDF)			1782				ND	3.4			
tetra-PCDD (TCDD)			4.6				ND	1.0			
penta-PCDD (PeCDD)			4.4					0.7			
hexa-PCDD (HxCDD)			112					2.9			
hepta-PCDD (HpCDD)			574					11.7			
octa-PCDD (OCDD)			6321					104.0			
Summary concentrations of the homolog gı											
Concentrations of all compound (except of											
Recalculation for internal standards accoun											
* OCDF concentrations were calculated ba:											
if required, OCDF concentrations can be n											
ND = non detected, the detection limits wil											

TABLE 9
Part 3

DATA FLAGS

In order to assist with data interpretation, data qualifier flags are used on the final reports. The most commonly used flags are:

ND = analyte not detected. Value is the detection limit.

B = analyte has been detected in the laboratory method blank as well as in an associated field sample.

E = indicates a concentration based on an analyte to internal standard ratio which exceeds the range of the calibration curve. Values which are outside the calibration curve are estimates only.

I = indicates labeled standards have been interfered with on the GC column by coeluting, interferent peaks.

J = indicates a concentration based on an analyte to internal standard ration which is below the calibration curve. Values outside the calibration curve are estimates only.

PR = indicates that a GC peak is poorly resolved. The concentrations or amounts reported for such peaks are most likely overestimated.

Q = indicates the presence of QC ion instabilities caused by quantitative interferences.

S = indicates that the response of a specific PCDD/PCDF isomer has exceeded the normal dynamic range of the mass spectrometer detection system. The corresponding signal is saturated and the reported analyte concentration is a 'minimum estimate'. Results for saturated analytes are reported as greater than the upper calibration limit.

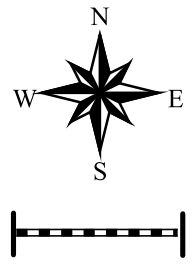
U = indicates that a specific isomer cannot be resolved from a large, coleluting interferent GC peak. The specific isomer is reported as not detected as a valid concentration cannot be determined. The calculated detection limit, therefore, should be considered an underestimated value.

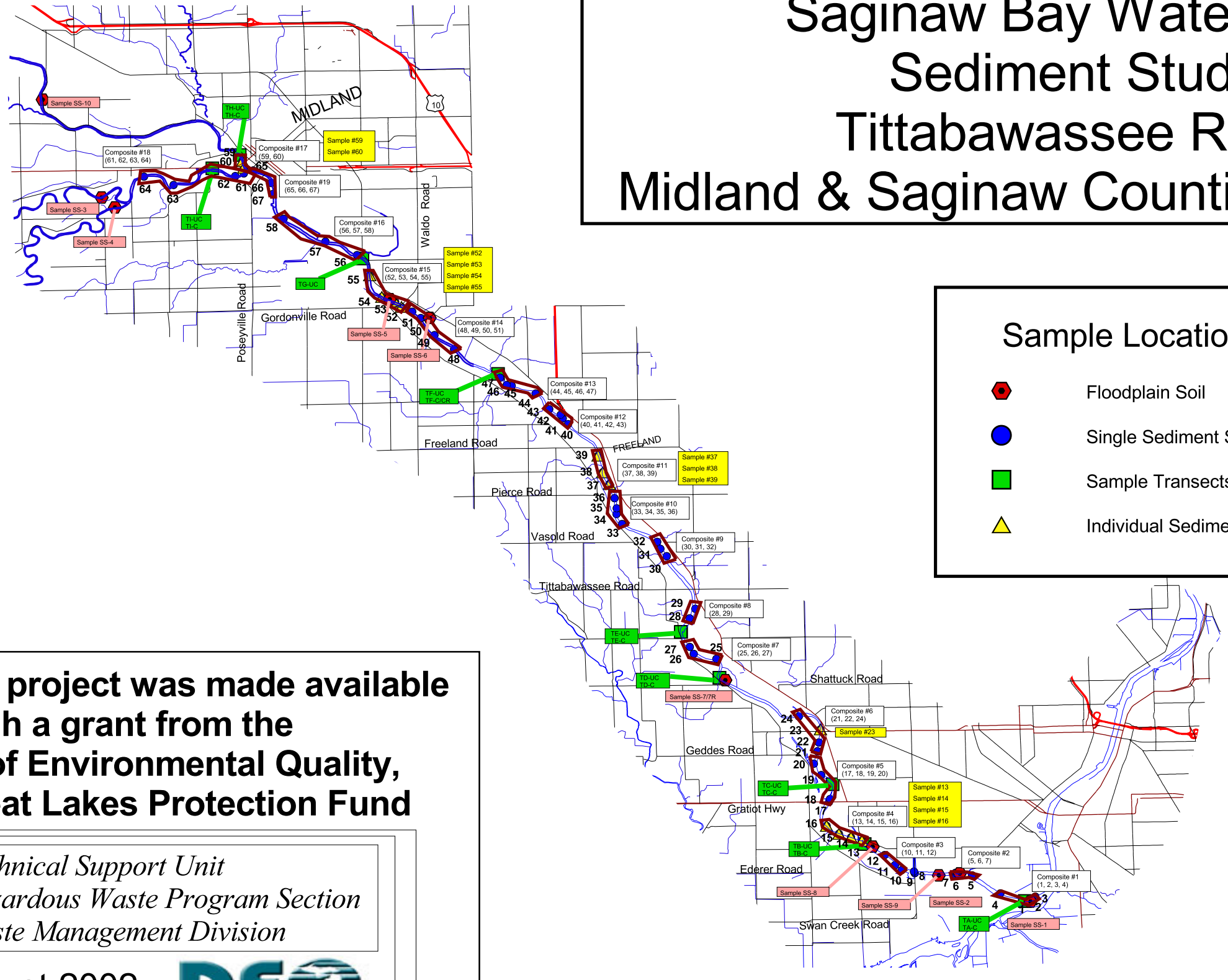
V = indicates that, although the percent recovery of a labeled standard may be below a specific QC limit, the signal-to-noise ratio of the peak is greater than ten-to-one. The standard is considered reliably quantifiable. All quantitations derived from the standard are considered valid as well.

X = indicates that a polychlorodibenzofuran (PCDF) peak has eluted at the same time as the associated diphenyl ether (DPE) and that the DPE peak intensity is at least ten percent of the total PCDF peak intensity. Total PCDF values are flagged "X" if the total DPE contribution to the total PCDF value is greater than ten percent.

FIGURES

Saginaw Bay Watershed
Sediment Study
Tittabawassee River
Midland & Saginaw Counties, Michigan





Sample Locations

- Floodplain Soil
- Single Sediment Samples
- Sample Transects of River
- Individual Sediment Samples

Funding for this project was made available through a grant from the Department of Environmental Quality, Michigan Great Lakes Protection Fund

WMD

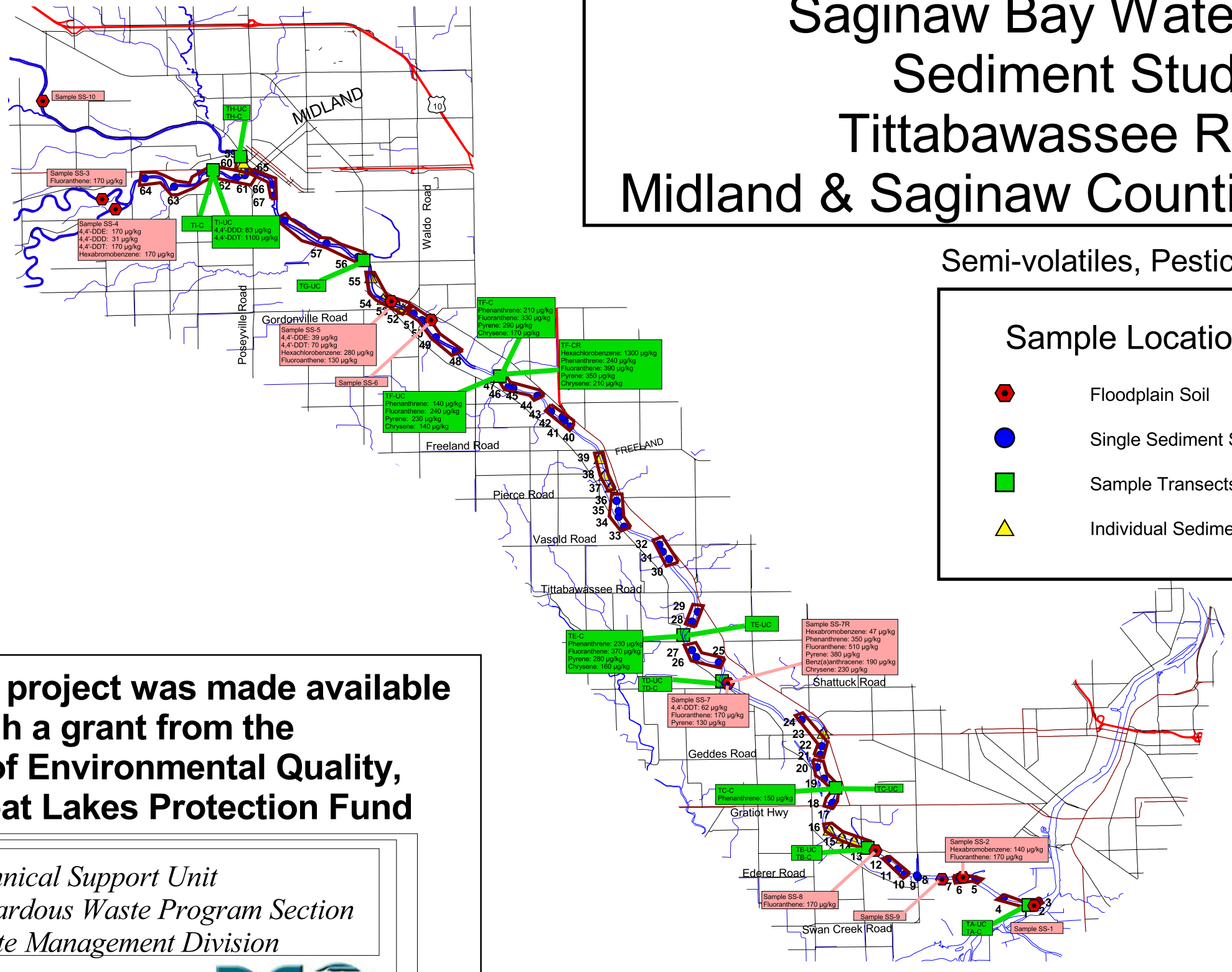
Technical Support Unit
Hazardous Waste Program Section
Waste Management Division

August 2002

DEQ

Figure 3

Saginaw Bay Watershed
Sediment Study
Tittabawassee River
Midland & Saginaw Counties, Michigan



Semi-volatiles, Pesticides and PCBs

Sample Locations

- Floodplain Soil
- Single Sediment Samples
- Sample Transects of River
- Individual Sediment Samples

Funding for this project was made available through a grant from the Department of Environmental Quality, Michigan Great Lakes Protection Fund

WMD

Technical Support Unit
Hazardous Waste Program Section
Waste Management Division

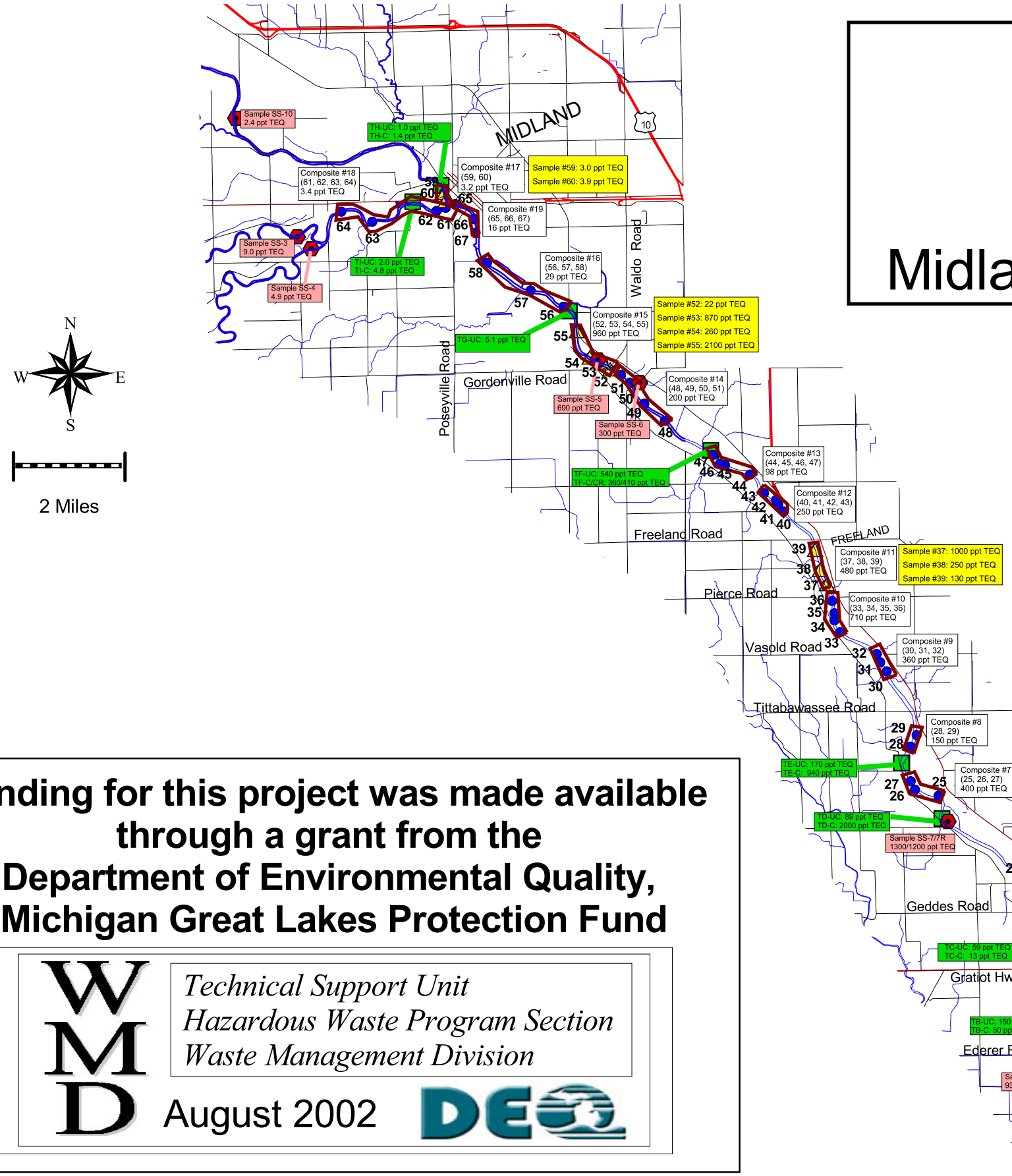
August 2002

DEQ

Figure 7

Saginaw Bay Watershed
Sediment Study
Tittabawassee River
Midland & Saginaw Counties, Michigan

Dioxins and Furans
TEQ Concentration in ppt



Sample Locations

- Floodplain Soil
- Single Sediment Samples
- Sample Transects of River
- Individual Sediment Samples (analyzed separately)

Funding for this project was made available through a grant from the Department of Environmental Quality, Michigan Great Lakes Protection Fund

WMD

Technical Support Unit
Hazardous Waste Program Section
Waste Management Division

August 2002

DEQ

Figure 8

Figure 9

TITTABAWASSEE RIVER STUDY Transect Analyses

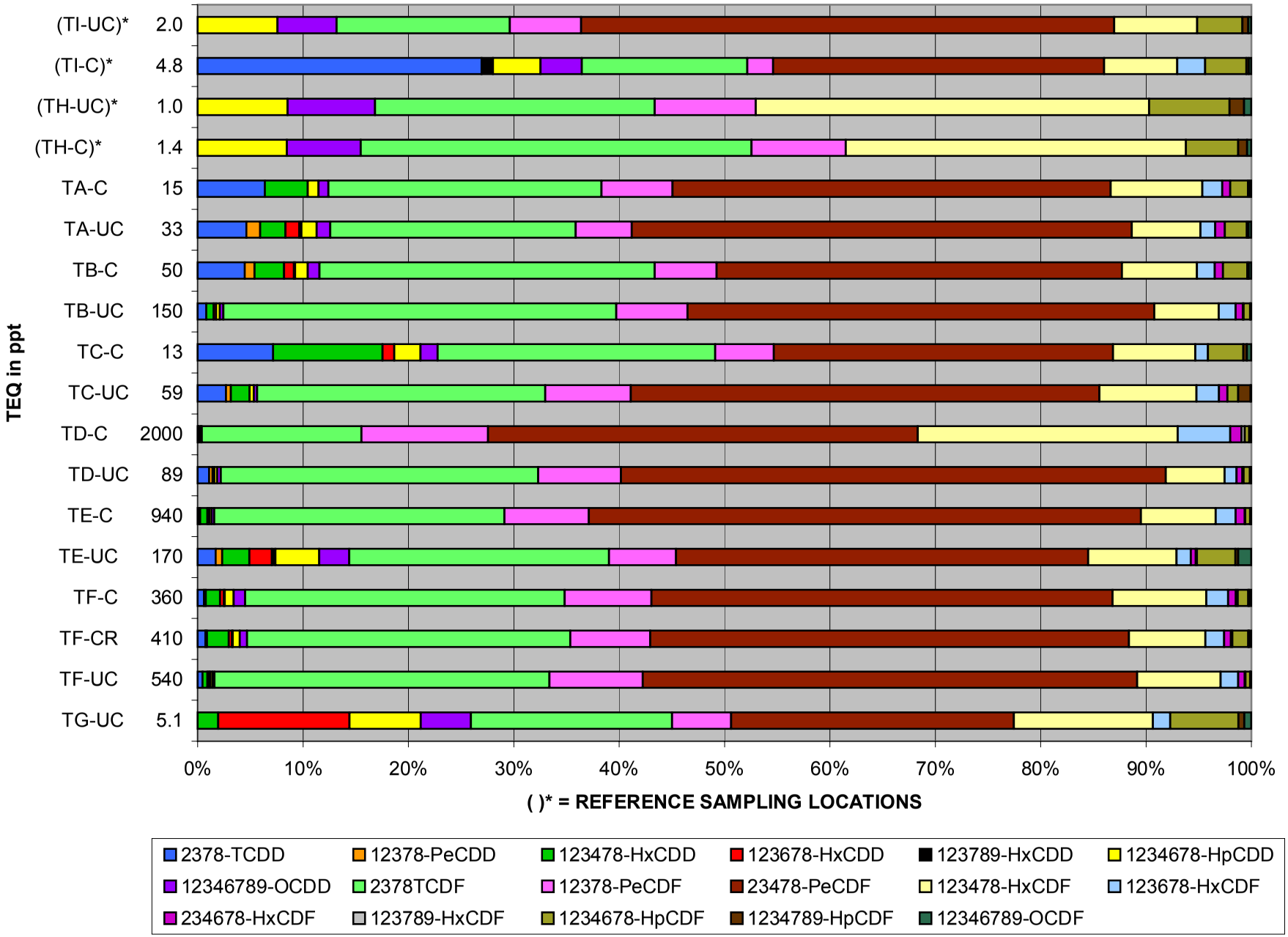


Figure 10

TITTABAWASSEE RIVER STUDY Composite Analyses

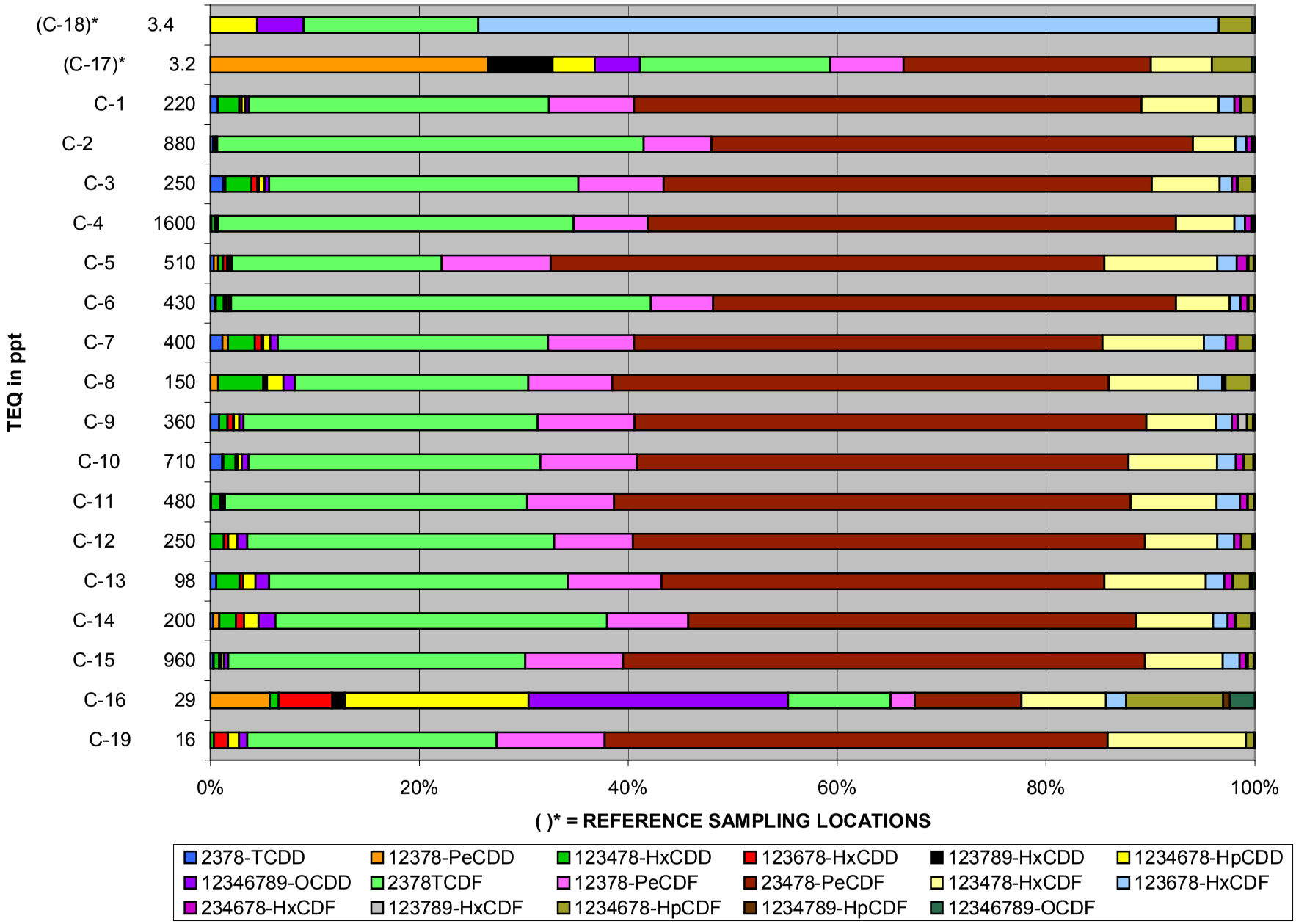


Figure 11

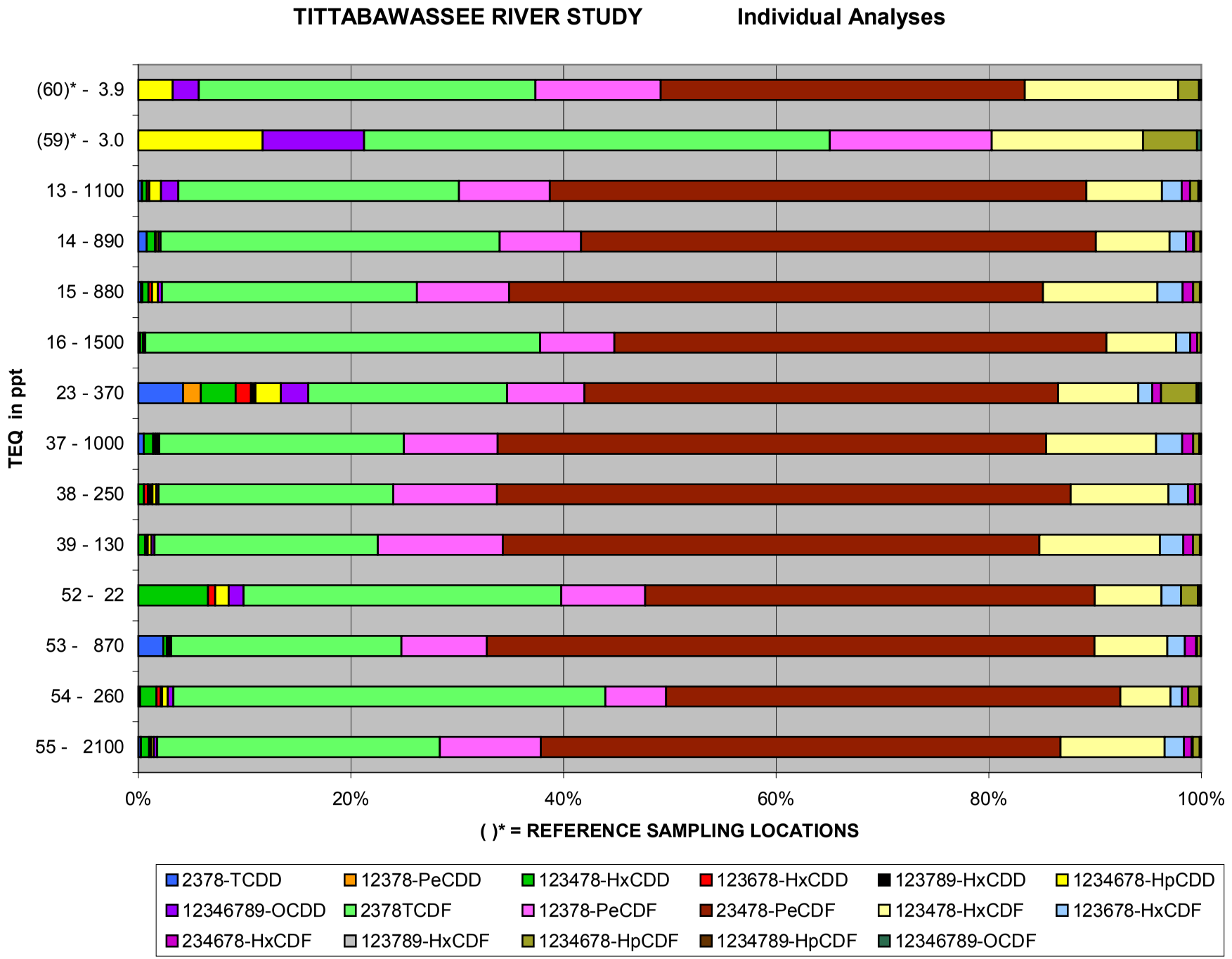
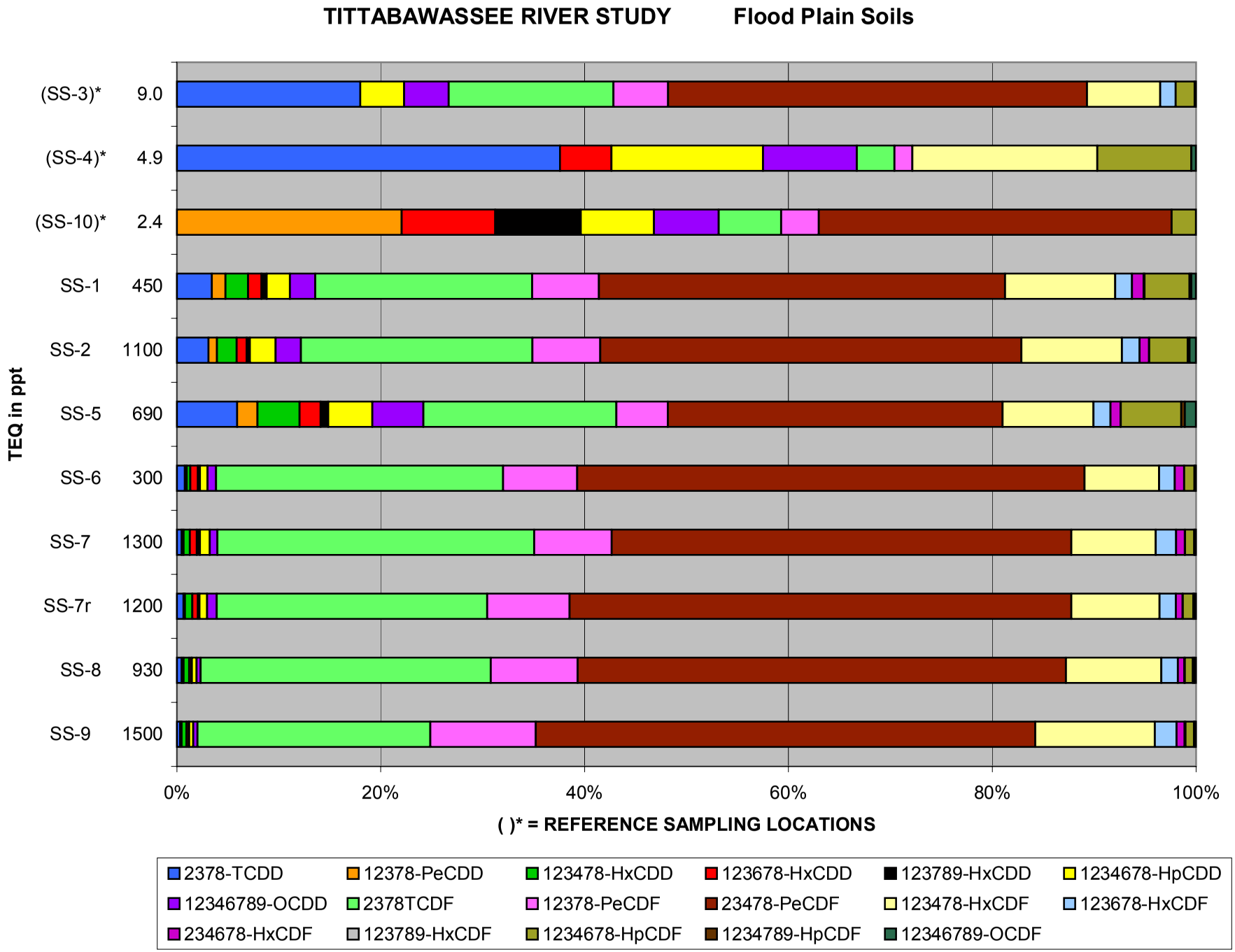

TITTABAWASSEE RIVER STUDY
Individual Analyses
(60)* - 3.9
(59)* - 3.0
13 - 1100
14 - 890
15 - 880
16 - 1500
23 - 370
37 - 1000
38 - 250
39 - 130
52 - 22
53 - 870
54 - 260
55 - 2100
TEQ in ppt
0%
20%
40%
60%
80%
100%
( )* = REFERENCE SAMPLING LOCATIONS
2378-TCDD
12378-PeCDD
123478-HxCDD
123678-HxCDD
123789-HxCDD
1234678-HpCDD
12346789-OCDD
2378TCDF
12378-PeCDF
23478-PeCDF
123478-HxCDF
123678-HxCDF
234678-HxCDF
123789-HxCDF
1234678-HpCDF
1234789-HpCDF
12346789-OCDF

Figure 12


TITTABAWASSEE RIVER STUDY Flood Plain Soils
(SS-3)* 9.0
(SS-4)* 4.9
(SS-10)* 2.4
SS-1 450
SS-2 1100
SS-5 690
SS-6 300
SS-7 1300
SS-7r 1200
SS-8 930
SS-9 1500
TEQ in ppt
0%
20%
40%
60%
80%
100%
( )* = REFERENCE SAMPLING LOCATIONS
2378-TCDD
12378-PeCDD
123478-HxCDD
123678-HxCDD
123789-HxCDD
1234678-HpCDD
12346789-OCDD
2378TCDF
12378-PeCDF
23478-PeCDF
123478-HxCDF
123678-HxCDF
234678-HxCDF
123789-HxCDF
1234678-HpCDF
1234789-HpCDF
12346789-OCDF

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APPENDICES

APPENDIX 1:

Original Funding Proposal
And
Budget

Baseline Characterization of Saginaw Bay Watershed Sediments
Scope of Work – Michigan Great Lakes Protection Fund
October 19, 2000

Persistent bioaccumulative compounds have historically been released from industrial entities in the Saginaw Bay watershed. Currently, there is a lack of detailed information regarding the presence, location, and concentration of these compounds in the sediments and on the flood plain along certain portions of rivers in the Saginaw Bay watershed (in particular, the Tittabawassee River). Dioxins and furans, polychlorinated and polybrominated biphenyl compounds (PCBs and PBBs), pesticides, herbicides, and other persistent bioaccumulative compounds are the primary compounds of concern in this study. The study will collect and analyze samples of sediments and floodplain soils beginning upstream of Midland, Michigan on the Pine, Chippewa, and Tittabawassee Rivers and continuing downstream from Midland on the Tittabawassee River into the upper Saginaw River. These samples would be analyzed for volatile and semivolatile organic compounds, pesticides, herbicides, PCBs, PBBs, dioxins, furans, and heavy metals.

The project will be implemented in four stages: 1) A detailed literature review to inventory and summarize all of the existing and available data on sediment quality in the proposed study area. The first phase of the project will also involve the development of a detailed sampling and analysis plan for the collection of additional sediment samples; 2) The identification and mapping of proposed sampling locations and the actual collection of the sediment samples. 3) The compilation and organization of both historical and new data into a comprehensive and useable baseline assessment of sediment quality in these three tributaries. The sediment quality data will be linked to other ongoing measurements of environmental quality, such as caged fish studies and surface water quality measurements. 4) The dissemination of the project results to stakeholder groups.

The project is expected to take one and one quarter years to complete in accordance with the following schedule:

Project Start	December 2000
Review Existing Data/Identify Data Gaps	February 2001
Selection of Sampling Locations	April 2001
Sediment Sampling	August 2001
Sample Analysis	October 2001
Data Evaluation	December 2001
Completion of Report and Recommendations	March 2002

The information collected during this study will establish a year 2001 baseline level of contamination within the subject Saginaw Bay watershed tributaries and can be used as a benchmark by which future improvements or degradations in sediment and water quality can be measured. This project will supplement existing data and serve as a screening level evaluation of sediment quality to determine if contaminants are present at levels of environmental concern and if they are a significant source of ongoing releases to Lake Huron. The data from this study will be used to complement and supplement the ongoing Strategic Environmental Quality Monitoring Program for Michigan's Surface Water (MDEQ, 1997) that is being implemented by the Michigan Department of Environmental Quality (MDEQ) Surface Water Quality Division and the U.S. Fish and Wildlife Service. In addition, the analytical results from the study could be used to form the basis for a request for corrective action, if necessary, from regulated facilities within the watershed.

Prepared by: Allan B. Taylor and John McCabe, Technical Support Unit, Waste Management Division, Michigan Department of Environmental Quality

Michigan Great Lakes Protection Fund Budget:
Baseline Chemical Characterization of Saginaw Bay Watershed Sediments

	Funds Requested From MGLPF	Matching Resources	Total
A. Personnel Salary:			
A.1a. Principal Investigators	0	8,000	8,000
A.1b. Senior Associate	6,500	0	6,500
A.1c. Student Intern	17,000	0	17,000
A.1d. Secretarial-Clerical	0	1,200	1,200
Total	23,500	9,200	32,700
B. Fringe Benefits	2,275	3,220	5,495
C. Total A plus B	25,775	12,420	38,195
D. Travel	3,000	0	3,000
E. Analytical Costs	60,000	60,000	120,000
F. Totals	**88,775**	**72,420**	**161,195**

- Note that the "Matching Resources" are based on estimates of time and materials that the Principle Investigators (PIs) anticipate on utilizing for this project. These expenditures will not be tracked through the MAIN system, however, matching expenditures will be tracked and reported separately. 75% of this funding is provided through the Federal RCRA Grant.

Item A.1a. The Co Principal Investigators are not requesting Salary funding from MGLPF. The $8000.00 Matching Funds reflects the anticipated PI oversight salary based on $25.00/hour for 320 hours.

Item A.1b. This line item reflects the anticipated salary cost for the support of the MDEQ Surface Water Quality Division experts. The $6500.00 requested funds reflects a salary of $25.00/hour for 260 hours of technical support.

Item A.1c. This line item reflects the salary of a graduate level Student Intern who will be compensated at a rate of $12.00 –$14.00 hour per State of Michigan guidelines. The $17,000.00 is based on an estimated 1300 hours of work by the Student Intern.

Item A.1d. This line item reflects a match for office professional support. The $1200.00 is based on a salary of $15.00/hour for 80 hours.

Item B. These line items reflect the fringe benefit costs at an estimated rate of 35% of salary. The Student Intern does not receive fringe benefits.

Item D. Travel costs are estimated for the presentation of the results of the study at appropriate forums. Travel costs are not estimated for the collection of the sediment samples but are anticipated to be minimal. The State of Michigan Department of Management and Budget rates would be used for any travel costs.

Item E. This line item reflects the anticipated cost of an outside laboratory for analysis of dioxins and furans and other compounds that the MDEQ Environmental Laboratory may be unable to conduct analyses on. Currently, MDEQ – Waste Management Division maintains a contract with Triangle Laboratories in Durham, North Carolina. Dioxin and furan analysis per sample is approximately $750.00, however, it is not known if the same rate can be obtained when the WMD contract is renewed. The market rate for dioxin and furan analysis varies from $700.00 to over $2000.00 per sample. The current contract also includes all bottles, some shipping, coolers, packing materials, etc. Until the sediment deposits are mapped in the Study Area it will not be known exactly how many samples will be collected. Our best estimate at this time is that approximately 50 sampling locations will be identified. Approximately 10 quality assurance/quality control samples such as duplicates, trip blanks, field blanks, etc will also need to be collected. The PI's would like to retain the flexibility to analyze other media from the study area such as caged fish or surface water to support the sediment quality data. The $60,000.00 is based on 60 samples at $1000.00 a sample that includes shipping of the samples to the contract laboratory. A match of 60,000 is included to reflect analyses that will be performed by the Michigan Department of Environmental Quality Environmental Laboratory. As noted above this work will not be tracked through MAIN, but will be tracked and reported separately.

APPENDIX 2:

Literature Search Results

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APPENDIX 3:

Sampling and Analysis Plan

SAMPLING AND ANALYSIS PLAN

For the Great Lakes Protection Fund Grant Project
"Baseline Characterization of Saginaw Bay Watershed Sediments"

- Purpose of sampling: To gather information about the distribution of certain chemical contaminants in tributaries to the Saginaw Bay. This information will provide a baseline against which future changes in sediments and soils can be compared if the sampling protocol is repeated. It is anticipated that this study will be replicated in the future in order to determine if there are changes in these parameters over time and space. The media sampled to gather this information will be river sediments and floodplain soils.

Sampling Design

- Two different strategies will be used to select sediment sampling locations because of holding and turnaround time issues; one for samples going to the MDEQ lab (for volatile organic compounds, semivolatile organic compounds, pesticides, PCBs and metals) and one for samples going to the MSU ATL (for dioxin and furan analysis).
- Samples going to the MDEQ lab will be taken from cross channel transects at approximately ten previously determined depositional areas. Between two and five samples will be obtained across each transect, the number dependent on width and apparent homogeneity of the depositional area. Fine grained sediment will be preferentially sampled over sand and gravel. The decision as to the number of samples taken at each transect will be made in the field. Each sample, except those taken for VOC analysis will be composite of adjacent discrete samples taken with a Petite Ponar dredge. VOC samples will be discrete samples, immediately adjacent to the composite samples, taken with a dredge. The top two to four inches of sediment will be collected. In addition to the surficial sediment samples, a composite core sample will be taken at each transect by compositing the four to twelve inch section of a two inch diameter acetate liner core sample. The composite core samples will serve as an inventory of deeper sediment repositories of contamination. The approximate locations of ten sampling transects have been identified (Fig. 1) and their positions recorded using GIS technology. Actual transect locations will be selected in the field on the day of sampling.
- Samples going to MSU-ATL, for analysis of dioxins and dioxin-like compounds will be collected as composites from transects (as above) and as samples taken at locations along reaches of river (a reach defined as a length of river characterized by flow, erosional or depositional homogeneity). Between two and ten samples will be taken on each reach, dependent on the length of the reach and the distribution of fine grained sediments. Location of the reach samples will be determined in the field and recorded using GIS technology. Individual reach samples will be collected by compositing 4

adjacent Petite Ponar dredge samples at each sample location. An aliquot of each sample within a given reach will then be used to create a composite sample in the laboratory to represent the concentration of dioxins and furans within that reach. Based upon analytical results for each reach composite, the samples from individual locations in a reach may or may not be analyzed further. Compositing samples within a reach, and then conducting further analysis of discrete samples based on composite results, provides a more comprehensive picture of where in the system dioxin and dioxin like compounds are located. This approach is available because of the long sample holding times for these analyses.

- Floodplain soil sampling will be done with hand augers or stainless steel scoops. Floodplain depositional areas will be identified in the field and individual sampling locations recorded using GIS technology. The top eight inches of agricultural soils and the top two inches of non-agricultural soils will be sampled with a hand auger or stainless steel scoop. Agricultural soils are defined as those in which flood deposits have been redistributed within the soil column by plowing or tilling. Discrete samples from four adjacent locations will be composited for all analyses except VOCs. A discrete sample will be taken, immediately adjacent to the four composited, for VOCs. Ten composite samples of floodplain soils will be collected.
- Additional core analysis may be done using samples collected with a "vibracore" sampler at the confluence of the Titabawassee and Saginaw Rivers.

Dredge Sample Collection Methodology

Dredge samples will be collected using a Petite Ponar dredge. The Petite Ponar dredge will be decontaminated prior to use by being scraped with a stiff bristled brush and rinsed with distilled water. The dredge will be dropped at each sample location, either from a boat or from a standing position (where the river is wadeable). In the case of wadeable sample locations, the sampler will take care not to walk through the sampling location or otherwise agitate the sediments prior to sampling. The dredge will be used to collect the top two inches (approximately) of sediments at each sampling location. In the case of composite samples (transects) the dredge will be decontaminated after collecting all the discrete samples used to make a composite. In the case of reach samples, the dredge will be decontaminated after each individual sample is taken. Field decontamination will consist of scraping the dredge with a stiff bristled brush to remove any sediment adhering to the dredge and then rinsing the dredge in the river.

Sample Compositing

The individual samples collected at each transect (with the exception of the sample collected of VOC analysis) will be composited for analysis. As each

dredge sample is collected on a transect, it will be placed in a precleaned aluminum cake pan. Successive dredge samples from that transect will be added to the pan. When all dredge samples on a transect have been collected, the collected sediment in the pan will be thoroughly mixed, using a precleaned stainless steel scoop. The mixed sediment will then be placed into the appropriate sample containers and, if necessary preserved.

Each sediment sample collected using 2 inch diameter acetate liner cores will be placed in its own precleaned aluminum cake pan and thoroughly mixed, using a precleaned stainless steel scoop. The mixed sediment will then be placed into the appropriate sample containers and, if necessary preserved.

Sample Collection for Volatile Organic Compounds

VOC samples will be collected from the first dredge sample taken at each transect. VOC samples will be collected and preserved according to the procedures in U.S. EPA SW-846 Method 5035. Waste Management Division's Operational Memorandum Gen-14, detailing the procedures for using this method, is attached.

Soil Sample Collection

Floodplain Soils will be collected following the attached "Soil Sampling and Analysis Plan."

Sample Handling and Preservation

Sample containers, preservatives and holding times for those samples collected for analysis by the MDEQ Environmental Laboratory (VOCs, semivolatiles, pesticides, PCBs and metals) are listed in Table 1. Samples to be analyzed for dioxins, furans and dioxin-like toxicity by MSU-ATL will be collected in pre-cleaned, wide mouth 500 ml glass jars with teflon lined caps supplied by MSU-ATL. All samples will be placed in a cooler and maintained at 4 degrees Celsius immediately after collection and until delivery to the laboratory.

Chain of Custody

All collected samples will be handled under chain of custody procedures. Sample coolers containing collected samples will remain in the legal custody of the sampling crew until delivery to the respective laboratory. Transfer of custody from the sampling crew to the laboratory will be accompanied by a signed chain of custody form. Chain of custody forms for the MDEQ Environmental Laboratory and MSU-ATL are attached.

Sample Analysis

Samples delivered to the MDEQ Environmental Laboratory will be analyzed using the methods and detection limits in Table 2. Samples delivered to MSU-ATL will be analyzed using the methods in the attached H4IIE LUC bioassay SOP.

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

INTEROFFICE COMMUNICATION

OPERATIONAL MEMO GEN-14
Revision 3

November , 1999

TO: All Waste Management Division Staff

FROM: Jim Sygo, Chief, Waste Management Division

SUBJECT: Volatile Organic Compound (VOC) Sediment Sampling Method 5035 Field Sampling Procedure

The following is the updated Waste Management Division (WMD) soil/sediment sampling procedure to be used for the United States Environmental Protection Agencyís (U.S. EPA) SW-846, Method 5035. This field procedure will take effect as equipment for the previous field procedure (effective on April 30, 1998) is used up. Facilities within the treatment, storage, and disposal facility universe of the Hazardous Waste Program have been previously notified regarding implementation of sampling and laboratory procedures for Method 5035. Training regarding the collection of soil VOCs has been initiated by the Environmental Response Division at several District and Lansing locations. Staff who still need to be trained should contact other field sampling staff who have been trained or John McCabe for a demonstration of the new field procedure.

Plastic syringes with caps, 40 ml Volatile organic analytes (VOA) vials, teflon methanol pouches, and green CH_3OH (methanol) labels will be available from Doug Wood, Drinking Water and Radiological Protection Division (DWRPD), at the Filley Street facility. Mr. McCabe will be responsible for the ordering and distribution for the other supplies (i.e. weights, scales, cutting tools, and clips) needed for the field procedure. Extra supplies will also be kept at the Filley Street facility in the WMD storage area. Contact Mr. McCabe if the supplies are running low. The 40 ml VOA vials are weighed by the manufacturer prior to use and the vial weights are recorded on a label using methanol semi-resistant red ink. Please do not add any other labels (exception is the green hazardous label for the methanol/soil sample jar), tape, etc. Please make sure you remove excess soil from the exterior of the vial and lid threads, otherwise, you will affect the weight and seal of the vial. The lab MUST receive the sample within four days of collection. Total holding time for the sample is 14 days.

This procedure is intended to diminish the volatilization of contaminants, so the less disturbance to the soil matrix and more quickly the sample from the ground is placed in

methanol, the more accurate the analytical results will be. Therefore, the sample should be prepared and the procedure followed as quickly as safety and accuracy allow.

EQUIPMENT REQUIREMENTS

-Methanol 10 ml Pouches
-Plastic 10 ml Syringes with caps
-10 Gram Weight
-Scale (Spring Loaded or Electronic)
-Steel-Toed Boots
-Decontamination Material
-40 ml Glass VOA Vials (labeled)
-Scissors or other Cutting Tool
-Safety Goggles
-Gloves
-Hard Hat

I. TRIP BLANK

1. Wear your safety goggles and appropriate gloves.

2. Pour methanol from a pouch into an empty 40 ml vial at the beginning of the sampling event. This will serve as a trip blank to check on cross contamination of methanol preserved samples. In specific instances where very low detection limits are desired, an aqueous trip blank can be used in place of the methanol trip blank. Staff at the Michigan Department of Environmental Quality Environmental Laboratory should be consulted to determine if an aqueous trip blank is appropriate.

3. Label the trip blank.

4. A pure methanol sample (trip blank) or an aqueous trip blank must accompany each batch of samples (each cooler) for each site and each day that samples are collected.

II. FIELD PROCEDURE

1. Wear your safety goggles and appropriate gloves.

2. Decontaminate the field scale and calibrate it with the 10 gram weight provided. Use the scale and weigh an empty syringe. Make sure the cap is on the syringe when weighing. Write the weight of the empty syringe in your field notebook. (If you do not have a 10 gram weight for calibration, you can use an American nickel, which weighs approximately 5 grams. Even though a 5 gram nickel may not provide exact calibration for the soil sample, you should still be

able to calibrate your scale with 5 grams and be within the 1 gram +/- tolerance range for the 10 grams soil/sediment sample.)

3. Remove the cap from the syringe. Take the split spoon sample, hand auger sample, or sample from whatever collection device you are using, and insert the open end of the syringe into a fresh face of undisturbed soil (if possible).

4. Push the syringe into the soil and fill it to the point where you believe that you have 10 grams of soil (you may wish to practice filling and weighing the syringe with similar soil from the same sampling location prior to taking your actual sample in order to get an idea of how much soil in required for a 10 gram sample).

5. Take your index finger, thumb, or other instrument and push the soil deeper into the syringe. Note that any material (gloves, instruments, etc.) touching the sample must be decontaminated and clean. Attempt to obtain an area at the opening of the syringe clear of soil. This will assist you in minimizing the amount of contaminants that will adhere to the scale clip.

6. Replace the cap on the syringe immediately after filling the syringe with sample. This will minimize loss of volatile compounds to the atmosphere.

7. Weigh the soil-filled syringe with the field scale and write the weight in your field notebook.

8. Subtract the weight of the syringe from the total weight of syringe and soil (soil must weigh 10 grams +/- 1 gram tolerance for a 9 to 11 gram range, **WITHOUT** the weight of the syringe). If you do not have 10 +/- 1 grams of soil, you MUST repeat steps four through seven until you have a total of 10 +/- 1 grams of soil. For most soils, the same volume of soil will yield approximately the same weight of soil. If you have too much soil, you must discharge soil from the syringe until you fall within the 9 to 11 gram range. Remember to replace the cap prior to each weighing of the syringe.

9. Write the soil weight in your field notebook. You do not need to provide the weight of the sample to the lab, the lab will reweigh the sample upon receipt.

10. Remove the cap from the 40 ml vial.

11. Remove the cap from the end of the syringe, insert the open end of the syringe into the vial, push the plunger, and discharge the soil.

12. Immediately take the methanol pouch and open it by cutting one end off with scissors. At all times you must be wearing your safety goggles.

13. Pour the methanol into the 40 ml vial over the soil.* If any methanol is spilled outside of the vial, you MUST discard the sample and take another. Loss of methanol can cause erroneously high results. Care should be taken to ensure that all the methanol is introduced into the vial.

14. Place cap TIGHTLY on the 40 ml vial and gently shake it for ten seconds.

15. Completely fill out the sample label on the 40 ml vial.

16. Place the prepared soil sample in an upright position on ice in a cooler. (It is recommended that each sample vial be placed in a zip lock bag in case of leakage).**

17. Use the plastic syringe to collect an additional soil sample from the same source as the first. Fill the syringe approximately 3/4 full. Cap the syringe with the plastic cap and attach a sample identifying label. This sample is for dry weight/total solids analysis.*** It is very important that the dry weight soil sample come from the same soil type as the methanol sample, i.e. sand to sand/clay to clay, etc.

18. Decontaminate the clip on your scale using standard decontamination procedures. Dispose of all waste materials appropriately.

NOTE: Sediment/high moisture content samples are collected the same as soil samples by following this protocol. You will want to exhibit caution in weighing the sample to prevent spillage of the sample.

NOTE: Labs may use a variety of tracking mechanisms for vial weights (taring), so only use the vials supplied by the lab performing the analysis.

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* You can opt to place the methanol into the 40 ml vial prior to the soil. There is the possibility of splash, so take precautions when emptying soil into the methanol. All other steps of the soil sampling procedure remain the same.

** Please note that, any sample collected by following this procedure, which is not sent to a lab, is considered hazardous waste and must be disposed of properly.

*** The amount of moisture will affect your sample results. The higher the moisture content, the higher the detection limit will be.

NOTE: If you are collecting samples under a court order, i.e. warrant, it is recommended you take two methanol samples in case of leakage/breakage/incorrect weight <9 or >11 grams.

CAUTION: Methanol is a poison. Handle appropriately. If ingested, contact a physician immediately.

If you have any questions regarding these field procedures and equipment, please contact Mr. McCabe, the WMD Lab Coordinator, at 517-335-4789, or GroupWise him at MCCABEJ.

cc: Paul Little, U.S. EPA
Hak Cho, U.S. EPA
Bob Avery, DWRPD
Doug Wood, DWRPD

SOIL SAMPLING AND ANALYSIS PLAN FOR THE SAGINAW BAY WATERSHED FLOODPLAIN SAMPLING

CLEANING OF SAMPLING EQUIPMENT .

Each stainless steel sampling spoon and aluminum cake pan will be cleaned prior to using them for sample collection.. Cleaning will consist of alconox solution wash, potable water rinse, methanol rinse, followed by a deionized water rinse. The equipment will then be placed in a clean sealed plastic bag.. All decontamination procedures will be completed at the shop before the sampling event and again after sampling has been completed. At least one individual grab sample of the final rinsate water will be collected during the initial pre-cleaning of a stainless steel spoon and an aluminum cake pan and these samples will be submitted to MSU-ATL for dioxin/furan analysis to verify that the equipment was satisfactorily cleaned. No decontamination procedures will take place in the field. The aluminum cake pans will not be reused. They will be disposed following their one-time use. New sampling gloves will be used each time a sample is collected and they will be disposed immediately after their use.

SAMPLING EQUIPMENT:

-Stainless steel sampling spoons
-Disposable aluminum cake pans-approximately 8 inches square by 2 inches high
-Laboratory cleaned and supplied 8 ounce brown glass jars with teflon lids (from MSU-ATL)
-4 ounce brown glass jars containing 20 grams of toluene washed sand for trip blanks (from MSU-ATL)
-Coolers and blue ice
-Plastic bags with twist ties to wrap samples individually
-Large plastic garbage bags to lay on the ground for sample collection
-Chain-of-custody forms and custody seals
-Foot long ruler and flagging stakes to mark the sampling area
-Disposable latex gloves
-Camera and film

SAMPLING PROCEDURES:

1. Once the sampling location has been selected, the samplers will mark off a one foot square using a tape or a ruler for measurement and flagging stakes to mark the corners of the area.

2. Every attempt will be made to find sampling locations that represent undisturbed ground and locations that are free from vegetation. If vegetation is present, the samplers will clear the vegetation from the sampling area using grass clippers. Vegetation should be clipped away as close to the ground as possible. A large plastic garbage bag or plastic

sheeting will be placed on the ground next to the sampling location and sample compositing will be performed over top of this isolated work area.

3. The sampling crew team leader will note in the written sampling log the general soil type present at the location.

4. If vegetation is present at the sampling location, the sampling crew will use a stainless steel spoon to carefully peel the grass and roots from the sampling site. The samplers will then use the stainless steel spoon to collect free dirt from the bottom of the grass/root mass and place it in to the aluminum cake **pan.** The samplers will then proceed to collect soil from the upper 0 to 8 inches within the marked sampling area for agricultural soils and from 0 to two inches for non agriculltural soils. The soil that is collected will be placed in the disposable aluminum cake pan with the dirt from the grass/root mass and the composite will be thoroughly mixed to homogenize the sample. A new pair of disposable latex gloves must be worn at each sample location. The gloves are to be properly discarded immediately following sample collection at each individual location.

5. The samplers will use the same stainless steel spoon used to collect and homogenize the sample to remove the soil from the aluminum pan and place it in a brown amber wide mouth 8 ounce jar provided by MSU-ATL. The samplers will fill out the labels affixed to the jar prior to placing the sample in to the jar. A copy of the label that is affixed to each jar is attached. The jar will be filled to capacity, a teflon lid will be used to seal the jar, and a completed and signed chain-of custody seal will be placed over the lid with the seal adhesive extending over the lid edges for security purposes.

6. Once the sample is collected, any dirt will be wiped from the outside of the jar with a paper towel and the jar will be placed in to a plastic bag where it will be individually wrapped. .The sample will then be wrapped in bubble wrap to protect it from breakage and it will be placed in an iced cooler for storage. The sample jars, when filled, must be kept in the cooler and out of the sunlight. The cooler must be kept under chain-of custody at all times. Once the cooler is filled, a completed and signed chain-of-custody seal shall be placed over the latch on the cooler.

7. Once all the soil that is needed has been placed in the jar, the sampling crew will dump any remaining soil back into the sampling hole, smooth out the area, and pack the grass/root mass back in to place. Care must be taken to return the sampling area back to its original condition.

8. Details on specific sampling locations with rationale for the selection of each sampling point will be carefully documented prior to the time of sample collection. The documentation of each sampling location will be maintained in the team leader's site log along with information about the site. A photograph will be taken at each sampling location to document the spot where the sample was collected and to show the general regional area where the sample was located. GIS technology will be used to precisely

locate the position of the sample for future replication of sampling.

9. Log book entries, sample labels, and field record sheets identifying sampling locations, collection date, collection time, and team leader's initials will be filled out by the sampling team leader. The team leader will be the person responsible for completing and signing the chain-of-custody seals that are placed on the sample jars and the cooler lid. The team leader will also be **responsible** for assuring that the chain-of-custody forms that will accompany the samples are properly completed and signed.

10. No more than 5 days following sample collection, samples will be transported to MSU-ATL. Samples will be stored (if needed) at the MDPH cold room until sample shipment. The room is dark, locked and has restricted access.

11. All composite pans, spoons, and other sampling devices will be either decontaminated per the WMD decon protocol identified in this plan or it will be recycled/disposed by the sampling team when the sampling event is finished.

FIELD QUALITY ASSURANCE

The field quality assurance plan will consist of the following:

1. 1 trip blank for each sample cooler. The trip blank will consist of one 4 ounce jar containing 20 grams of washed sand shipped to MSU-ATL.

2. . From 1-3 field blanks will be submitted to MSU-ATL for analysis. The field blanks will consist of a 4 ounce brown glass jar filled with 20 grams of washed sand shipped to the WMD by MSU-ATL. When collected, each field blank will be collected simultaneously with a regular soil sample. The field blank bottle will be opened and left open while the regular soil sample is being collected and it will be capped when sampling at the regular location is finished. The field blank sample will then be wrapped, etc. in the same way as the regular soil samples described in this plan. They will then be placed in to the cooler and shipped to MSU-ATL along with the soil samples that have been collected and they will be analyzed for dioxins and furans.

3. 1 temperature blank for each sample cooler. This will ensure that the temperature of the cooler was maintained at 4 degrees C.

4. Each sampling team shall collect a minimum of one replicate sample for every ten samples collected during a one day period. If the sampling team does not collect ten samples during a sampling episode, one replicate shall be collected on that day's sampling.

5. At least one equipment blank, respectively, will be collected from the final rinsate water generated during initial equipment pre-cleaning of both the stainless steel spoons and the aluminum cake pans.

CHAIN OF CUSTODY

An example chain of custody form has been attached.

ANALYTICAL METHOD AND DETECTION LIMIT

An SOP for the H4II-E LUC bioassay for dioxin like toxicity is attached.

Michigan State University
National Food Safety and Toxicology Center
Aquatic Toxicology Laboratory

STANDARD OPERATING PROCEDURE

H4IIE-luc Bioassay For The Detection Of Ah Receptor Agonists

Version 1 September 14, 1998

Alan Blankenship, Dan Villeneuve, J. Thomas Sanderson,
Sarah Cholger, Katherine Kemler, and John P. Giesy

Supported through:

National Food Safety and Toxicology Center,
Institute for Environmental Toxicology,
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APPROVAL PAGE

Revisions to an existing SOP, addition of an SOP change form, or preparation of a new SOP must be reviewed, approved, and signed by the following:

Authored By: _____ Date: _____

Supervisor Review By: _____ Date: _____

Reviewed By: _____ Date: _____
(QA Coordinator)

Reviewed By: _____ Date: _____
(CALS Program Manager)

DEFINITIONS AND ACRONYMS

AhR	Aryl hydrocarbon receptor
ATL	Aquatic Toxicology Laboratory (Michigan Sate University)
DCCFBS	Fetal bovine serum that has been charcoal-stripped
EC50	conentration of test agent that causes 50% of maximal response
FBS	Fetal bovine serum
H4IIE-luc	rat hepatoma cells stably transfected with an AhR-controlled luciferase reporter gene construct
PBS	Phosphate-buffered saline
RLU	Relative luminescent units
TCDD	2,3,7,8-tetrachlorodibenzo-*p*-dioxin

TABLE OF CONTENTS

1.0. PURPOSE

The H4IIE-luciferase induction assay is an *in vitro* technique for the identification of aryl hydrocarbon (Ah) receptor-active compounds. The technique uses rat hepatoma cells (H4IIE-luc) stably transfected with an AhR-controlled luciferase reporter gene construct were developed at Michigan State University by Dr. Jac Aarts (Univ. of Wageningen, The Netherlands; Sanderson et al., 1996). The assay is also referred to as the chemical activated luciferase gene expression (CALUX) system (Murk et al., 1996). These cells express firefly luciferase in response to Ah receptor agonists. Luciferase activity is measured conveniently and with high sensitivity as light emission using a plate-scanning luminometer. Luciferase induction potential is assessed by comparison of the response to that of 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD), the most potent agonist for the mammalian Ah receptor.

2.0 SCOPE AND APPLICATION

Ah receptor agonists include polyhalogenated aromatic hydrocarbons (PHAHs), such as polychlorinated biphenyls (PCBs), dibenzo-*p*-dioxins (PCDDs) and dibenzofurans (PCDFs) which are persistent environmental contaminants found in all parts of the world. A number of these compounds cause a variety of adverse effects in laboratory studies on rats and mice (reviewed by Poland and Knutson 1982). These include hepatotoxicity, certain types of cancer, thymic atrophy and other immunotoxicities, a wasting syndrome, reproductive toxicities, terata and the induction of enzymes and porphyrins. A number of these toxicities have also been observed in wildlife in areas with elevated levels of PHAHs, particularly in fish-eating birds in the Great Lakes (reviewed by Gilbertson *et al*. 1991; Giesy *et al*. 1994a; 1994b).

Interest exists in assessing the risk posed by these PHAHs to fish and wildlife, which may also reflect the risk to humans. One aspect of risk assessment is the use of bioanalytical assays to detect and determine the toxicity of complex mixtures of these chemicals in extracts of environmental compartments such as soil, water and biota. Quantitative instrumental analysis of complex mixtures of these compounds is a difficult and expensive task. Furthermore, demonstrating the presence of one or many of these compounds in samples provides only limited information on their biological potency, particularly when present in a complex mixture with many potential interactions.

In order to develop a suitable bioassay, an understanding of the mechanism of action for the compounds is required. In the case of a number of PHAHs, considerable knowledge of the mechanism by which they cause their toxicities has been acquired. As previously mentioned, PHAHs are persistent agonists for the Ah receptor (Poland and Knutson 1982). Binding of agonist to receptor results in an activated receptor-ligand complex that translocates to the nucleus. Here it interacts with specific sequences on the DNA, termed dioxin-responsive elements (DREs; also called XREs or AhREs), in order to alter gene transcription (reviewed by Whitlock 1990; Okey *et al*. 1994). A rapid and sensitive response that is under direct

regulation of the Ah receptor is the induction of cytochrome P-450 1A isoenzymes (CYP1A1 and 1A2) and their associated ethoxyresorufin O-deethylase (EROD) activity (Nebert and Gonzalez 1987). Good correlations exist between the Ah receptor-binding affinity of persistent PHAHs and their EROD-inducing potency *in vitro*, and, dependent on the endpoint, their toxic potential *in vivo* (Safe 1986; 1990). 2,3,7,8-Tetrachlorodibenzo-*p*-dioxin (TCDD), in particular, binds tightly to the Ah receptor, and is a potent inducer of EROD activity and highly toxic. For the above reasons, the capacity of single compounds or complex environmental mixtures to induce EROD activity is considered to be a reasonable measure of their toxic potential. This mechanistic knowledge has been applied *in vivo* in biological monitoring of fish and birds, and *in vitro* in the use of bioassays for the screening of environmental extracts for Ah-active components. The H4IIE rat hepatoma cell bioassay (Tillitt *et al*. 1991), is widely used for this purpose. In this assay, EROD-inducing potencies (ED_{50} values) of single compounds and environmental samples are determined from complete dose-response curves and compared to that of TCDD in order to express the biological potency of the tested samples in TCDD-equivalents (TCDD-EQs). The bioassay integrates potential non-additive interactions among Ah receptor agonist and between Ah receptor agonists and other compounds by measuring a final receptor-mediated response (Giesy *et al*. 1994a).

For the same purpose as the currently used wild-type H4IIE bioassay (H4IIE-wt), a recombinant H4IIE cell line (H4IIE-luc) has been developed that exhibits Ah receptor-mediated luciferase expression (Aarts *et al*. 1995). This cell line has been stably transfected with a luciferase reporter gene under transcriptional control of several DREs from mouse (Aarts *et al*. 1993; Denison *et al*. 1993). These DRE sequences are highly conserved among species, unlike the Ah receptor which can exhibit greatly different ligand-binding properties among species. A preliminary report using luciferase-transfected mouse Hepa1c1c7 hepatoma cells indicated that these cells are more sensitive to Ah receptor agonists than the wild-type cells (Aarts *et al*. 1993; Sanderson *et al*., 1996). It has been suggested that luciferase-transfected cell lines would have more favorable properties than their respective wild-types, such as greater selectivity, sensitivity and dynamic range. This has been postulated because the Ah receptor-mediated expression of luciferase, being foreign to the cell, is probably not affected by post-transcriptional and -translational events which influence CYP1A1 expression. Furthermore, the recombinant cells would not be dependent on a functional CYP1A1 gene or protein for responsiveness, although the Ah receptor-mediated pathway would still need to be present. Another theoretical consideration is that luciferase is assayed on the basis of light production for which extremely sensitive detectors exist; also, the turnover number or molecular activity of luciferase is so high that it allows the detection of very few molecules of the enzyme, relative to CYP1A1. Further, recombinant cells are readily amenable to further improvements in responsiveness by genetic engineering of the reporter gene construct.

The threshold dose (*i.e*., detection limit) and ED_{50} (*i.e*., effective dose to elicit 50% of the maximal response) for luciferase induction in H4IIE-luc cells were approximately 0.1 and

1.2 pg/well, respectively, as determined from 41 separate standard TCDD curves analyzed in 1997 (Figure 1). Coefficients of variation (standard deviation/mean x 100) for the assay were under 10% at all concentrations tested. For a sample size of 20 g tissue and a final extract volume of 0.25 ml, the H4IIE-luc assay will detect 1 part per trillion (ppt; pg/g, wet weight) TCDD-equivalents. With a sample size of 5 g tissue, 4 pg/g (wet weight) TCDD-equivalents will be detected.

3.0. SAFETY CONSIDERATIONS

TCDD and many related compounds have been found to be carcinogenic. In addition, the ethidium homodimer used in the cell viability assay is a powerful mutagen. Care should be taken to minimize exposure. According to institutional guidelines (refer to the Safety Manual for the Aquatic Toxicology Laboratory at Michigan State University), medium should be collected in a liquid trap for disposal as hazardous waste.

4.0. EQUIPMENT, MATERIALS, AND REAGENTS

4.1. Maintenance, preparation and use of H4IIE-luc cells

The assay uses a stably transfected cell line developed by Dr. Jac Aarts at Michigan State University (Sanderson et al., 1996). Briefly, rat hepatoma cells [American Type Culture Collection (ATCC) catalog #CRL 1548)] were stably-transfected with an inducible reporter plasmid, pGudLuc1.1. This plasmid contains the firefly luciferase gene under PHAH-inducible control of four DREs. Exposure of these cells to Ah receptor-active chemicals results in induction of luciferase activity in a time-, dose-, and AhR-dependent manner.

4.1.1. Maintain adherant cells in continuous culture in 100 mm tissue culture plates (Corning #25020-100, Cambridge, MA; 1-800-492-1110), 75 cm^2 flasks (Corning #25113-75) or any appropriate vessel at a maximum density of 80-90% confluence in 10% Full Medium (See **Media Preparation**). Incubator conditions are 37 C, 5% CO_2 humidified atmosphere.

4.1.2. Subculture cells 1:6 every week (depending upon density) maintaining a minimum cell density of 15-25%.

4.2. Instruments

4.2.1. Dynatech ML3000 Luminometer
(Dynatech Technical Support: (800) 336-4543; Chantilly, VA)

4.2.2. Cytofluor 2300/2350 Fluorescence Measurement System
(Millipore Technical Support: (800) 645-5476; Bedford, MA)

***NOTE**: All users of the Dynatech ML3000 Luminometer and the Cytofluor 2300 must read and be familiar with the Operators Manual before using the instrument. A working knowledge of Microsoft Windows is also necessary. For both the luminometer and fluorescence measurement system, instrument use is recorded in a log book (located next to the instrument). For each use, the person, date, number of samples, type of samples, and results of proficiency standards are recorded. In addition, any abnormal operation of the instrument is recorded. Calibration is performed by analyzing a positive control or sample with known activity each day that the assay is run. If the positive control exceeds ± 20% of the on-going average (as determined by a comparison to a proficiency curve maintained in the instrument log book), the positive control will be rerun. If exceedance is confirmed with the second analysis, the manufacturer will be contacted as a corrective action so that the instrument can be recalibrated.*

4.2.3. Pipets

a. Eppendorf Repeat Pipetter (Brinkmann Instruments #22 26 000-6; Westbury, NY) with sterile, Biopur 12.5 ml combitips (Brinkmann Instruments #22 49 520-8) for dispensing cells into the 96-well plate and for changing media. Calibration checked weekly by weight of water check and the results are entered in calibration log book. Adjustments made by MSU Biochemistry Instrument Shop when accuracy exceeds manufacturerís specifications of ± 0.3%.

b. Rainin Pipetman Pipets (0.5 - 10 µl capacity, Rainin #P-10; 20 - 200 µl, Rainin #P-200 ; 100 - 1000 µl, Rainin #P-1000; Woburn, MA) for making sample dilutions, dosing cells, etc. Calibration checked weekly by weight of water check and the results are entered in calibration log book. Adjustments made by MSU Biochemistry Instrument Shop when accuracy exceeds manufacturerís specifications of ± 2.5% for P-10, 1.0 % for P-200, and 3.0% for P-1000.

c. Brinkmann Eppendorf Multichannel Pipetter 30-300 µl (Brinkmann Instruments #22 45 120-1) for washing cells and adding reagents for viability and Luclite reagents. Calibration checked weekly by weight of water check and the results are entered in calibration log book.

Adjustments made by MSU Biochemistry Instrument Shop when accuracy exceeds manufacturerís specifications of ± 1.5%.

d. Drummond Pipet-Aid Filler/Dispenser (Drummond #4-000-111-TC, available from Fisher #13-681-15D) for dispensing volumes greater than 1 ml, such as for changing media on cells.

NOTE: *For all pipettors, except the Drummond Pipet-Aid, calibration test results are recorded weekly, along with the name of the person and any abnormal operation of the instrument.*

4.3. Supplies and Biochemicals

4.3.1. 96-Well ViewPlates™ (Packard Instruments #6005181; Meriden, CT); white 96-well plates, sterile, tissue culture treated, with lids and self-adhesive sticker for bottom of plates

Cost: $297/50 plates;
Ordering: (800) 856-0734
Technical support: (800) 323-1891

4.3.2. Cell viability assay reagents; sold either as a kit from Molecular Probes (#L-3224; Eugene, OR) or as individual components:

Calcein AM (Molecular Probes #C-3100);
MW = 994.87; made up as 4000x (2 mM) stock (50 µg/12.56 µl DMSO)

Ethidium homodimer I (Molecular Probes #E 1169)
MW = 857; made up as 2000x (1 mM) stock in DMSO

Ordering: (800) 438-2209
Technical support: (541) 465-8353

4.3.3. LucLite™ Kit (Packard Instruments # 6016911 - 1000 assay kit; Meriden, CT). Make fresh on same day as assay. Dissolve one bottle of lyophilized reagent with 10ml buffer (supplied) for every 133 assays (individual wells) to be analyzed.

Cost: $420/1333 assays using 75 µl/assay (or 1000 assays if using the manufacturerís suggested volume of 100 µl/assay. Preliminary studies showed equivalent responses at both 75 µl and 100 µl).
Ordering: (800) 856-0734
Technical support: (800) 323-1891

4.3.4. 10x Trypsin-EDTA solution (Sigma #T-4174, St. Louis, MO) for dissociation of cells from plates. From a 10x concentrated solution, a 1x working solution is prepared using sterile PBS as the diluent.

4.3.5. Dulbeccoís phosphate-buffered saline (PBS) with Ca^{2+} and Mg^{2+}

First, make up 10 L of Ca^{2+}-free and Mg^{2+}- free Dulbeccoís PBS:

2.0 g	KCl
2.0 g	KH_2PO_4
80.0 g	NaCl
11.5 g	Na_2HPO_4
10.0 L	H_2O[a]

[a]distilled/deionized or Nanopure biological grade

To each 1 L of PBS, add 0.1 g anhydrous $CaCl_2$ and 0.1 g $MgCl_2 \bullet 6H_20$
Keep at room temperature (good for at least 2 - 4 weeks).

4.4. Media Preparation

4.4.1. 10% Full Medium

Dulbeccoís Modified Eagleís Medium (Sigma #D-2902) **without phenol red** (known to be estrogenic and with unknown influence on H4IIE cells), and sodium bicarbonate. Prepare as instructed by the manufacturer, adjust the pH to ~7.3, and then add :

10% fetal bovine serum (Hyclone defined FBS # SH30070.03;
Logan, UT; 1-800-492-5663)

4.4.2. 10% DCCFBS Medium

Prepared as for the full medium, except the fetal bovine serum is replaced with dextran/charcoal-stripped fetal bovine serum (available from Hyclone #SH30068.03).

5.0 METHOD, PROCEDURES, AND REQUIREMENTS

5.1. Sample Preparation

Two types of samples may be assayed: pure compounds and mixtures derived from environmental or tissue samples. For pure compounds, sample preparation consists of dissolving the material in an appropriate solvent. The solvent of choice is isooctane (because of its low toxicity and for direct comparison to TCDD, which is dissolved in isooctane). However, if the material is insoluble in isooctane, the following solvents can be tried:, ethanol, acetone, *p*-dioxane, acetonitrile, and methanol. A stock solution should be prepared at 5 mM for compounds of unknown activity and stored in amber glass vials at -20^{0}C. However, it may be necessary to test concentrations near the limit of solubility of the compound. Ideally, a volume of 1-2 ml at the highest possible concentration should be obtained for standards and samples. This volume is sufficient for splitting samples for instrumental analysis and for preparation of serial dilutions.

Samples derived from environmental matrices such as tissue, water, or sediment should be extracted and concentrated according to appropriate protocols (in accordance with SOP #211 - Extraction and Analysis of PCBs and Non-ortho Coplanar PCBs in Biological Matrices). The volume of sample should be recorded before re-dissolution in the assay solvent so that a dilution or concentration factor can be calculated. To prepare dilutions, 0.5 ml of isooctane (assuming that this is the solvent of choice) is added to five appropriately labeled 2 ml amber GC glass vials with teflon lined caps. Then 0.5 ml of the 1x extract (original extract that is undiluted) is added to the first vial (labeled 0.5x), the lid enclosure tightened, and then the sample is vortexed well. Then 0.5 ml of the 0.5x stock is added to the next vial (labeled 0.25x), the lid enclosure tightened, and then the sample is vortexed well. These steps are repeated until all dilutions are prepared. The sample dilutions will be 100, 50, 25, 12.5, 6, and 3% of the extract. If less than 1 ml of original 1x extract is available, the above mentioned volumes should be proportionately reduced. After dissolution in the assay solvent, the samples should be stored at -20^{0}C.

5.2. Standards Preparation

A large range of standards should be prepared to deliver a final concentration of **TCDD** between 0.03 - 100 pg/well in a volume of 1.25 µl (*i.e.*, make 200x stock solutions). Generally, 6 concentrations will achieve a full dose response curve (final = 0.1, 0.3, 1, 3, 10, 30 pg/well) for TCDD. Ideally, standards should be dissolved in the same solvent as the samples, but this is not always possible. In this case, be sure to conduct assays with both solvent controls and compare them to blanks (see Dosing Cells). A stock of TCDD in isooctane is maintained in Room 181, URCF, in a 1 liter volumetric flask. The concentration is written on the flask and is tested for purity and accuracy by GC/MS and GC/ECD by comparison to commercially available certified standards. To make dilutions,

prepare the following stock concentrations (assuming a stock concentration of 10000 ng/ml):

final mass. in wells (pg/1.25 ul)	stock concentration (ng/ml)
12500	10000
10000	8000
1000	800
100	80
The following six concentrations are used for the bioassay:	
30	24
10	8
3	2.4
1	0.8
0.3	0.24
0.1	0.08

***NOTE**: Adherence to these procedures will insure consistent response of cells. Timing and cell density are critical. An assay may be completed in 5 days.*

5.3. Plating Cells (Day 1)

Prior to confluence, write down the passage number of the cells, aspirate media from cell culture dishes, wash the cells with sterile PBS without Ca^{2+} and Mg^{2+}, and trypsinize the cells with 3 ml of 1x sterile trysin-EDTA solution for 5 minutes at 37^0C. Add trysinized cell suspension to 27 ml of ì10% Full Mediumî and determine the number of cells/ml with a hemacytometer (for more information on cell counting, refer to ìCell Culture: A Manual of Basic Techniquesî, by Dr. Ian Freshney, 1996) . Dilute the cell solution to a concentration of 60,000 cells/ml with media. Add 0.25 ml of cell suspension to each well (15,000 cells) of a 96-well ViewPlate™ using an Eppendorf™ repeat pipettor. Care must be taken that the cell suspension is uniform each time that the pipettor is refilled. This is done by gently inverting the tube or bottle of cell suspension end-over-end several times immediately prior to refilling the pipettor. If the outer 36 wells are not being used for the experiment (recommended), fill them with either sterile media or PBS to maintain humidity consistently across the plate. Use of the outer wells is not recommended because of an edge effect caused by inconsistent growth of cells in these wells.

***NOTE**: cell number per well is one of the largest contributors to variation in the data - so take plenty of time to do this step properly. Cell passage number should be noted to monitor long-term changes in the responsiveness and growth characteristics of the cells.*

5.4. Dosing Cells (Day 2)

Dose cells 24 hours after plating and continue exposures for 3 days. First, examine cells to ensure consistent plating. Aspirate media (attach p-10 tips to the suction line to minimize cell scraping), and replace with 0.25 ml of DCCFBS media that is prewarmed to 37^0C.

***NOTE**: the use of cold media should be avoided because it may ì shockî or stress the cells and it may cause precipitation of test agents that have poor water solubility).*

A typical plate design is shown in Figure 2. Use at least three replicates per treatment. Note that with TCDD, only two replicates are used for a typical plate design. Each control and TCDD concentration are averaged for all plates within a given experiment. Use a negative control with no treatment (blank) and a solvent control treated with pure solvent only. Use at least five concentrations of each compound or extracts tested. Prepare 200x chemical stocks in the appropriate solvent (see Sample Preparation). In general, it is best to dose cells directly in the well; however, for water miscible solvents, the test agent can be added to a sterile glass vial containing 2 ml of media, mixed, and then added to each well. Each well shall receive 1.25 µl of sample or standard or solvent control as noted in the plate design below.

Earlier versions of this assay determined that cross-talk can occur when high activity samples are directly adjacent to low activity samples. Therefore, as a corrective action, the plate design shown in Figure 2 was developed. Note that two blank columns border samples, so that there is no cross-talking between samples and TCDD and between samples and controls.

Row/Col	1	2	3	4	5	6	7	8	9	10	11	12
A												
B		0.1 pg TCDD	0.1 pg TCDD			C1 Conc. 1	C1 Conc. 1	C1 Conc. 1			solvent control	
C		0.3 pg TCDD	0.3 pg TCDD			C1 Conc. 2	C1 Conc. 2	C1 Conc. 2			solvent control	
D		1 pg TCDD	1 pg TCDD			C1 Conc. 3	C1 Conc. 3	C1 Conc. 3			solvent control	
E		3 pg TCDD	3 pg TCDD			C1 Conc. 4	C1 Conc. 4	C1 Conc. 4			blank	
F		10 pg TCDD	10 pg TCDD			C1 Conc.5	C1 Conc.5	C1 Conc.5			blank	
G		30 pg TCDD	30 pg TCDD			C1 Conc. 6	C1 Conc. 6	C1 Conc. 6			blank	
H												

Figure 2. A typical plate design for determination of Ah receptor agonist activity in H4IIE-luc cells. Note that blanks, solvent controls, and a standard curve for TCDD can be analyzed on the same plate with one test sample (labeled C1, at six different concentrations). Two blank columns are recommended between samples and either TCDD or controls to prevent any possible cross-contamination.

5.5. Conducting the Bioassay (Day 5)

5.5.1. Preparation steps (prior to assay)

a. Inspect plates visually with and without microscope - check degree of confluence, homogeneity from well-to-well, and any signs of cytotoxicity or altered morphology

b. Set up cytofluor for viability assay:

set 1: excitation = B emission = B sensitivity = 3
set 2: excitation = C emission = E sensitivity = 4

***NOTE**: sensitivity can be adjusted if values are too high (9999) or low (not different from blanks)*

c. Preparation of viability assay reagent (refer to Supplies and Biochemicals Section):

Each plate will need 1.8 ml or 3 ml, depending on whether 36 or 60 wells are used per plate, respectively plus a little extra (2 ml). Dilute the appropriate amounts of calcein and ethidium with the appropriate volume of media without FBS as shown below:

Number of plates	Volume of viability assay reagent needed [total volume (ml); calcein stock (µl); Ethidium stock (µl)]	
	using 36 wells	using 60 wells
1	3.8 ml; 0.95 µl; 1.9 µl	5 ml; 1.25 µl; 2.5 µl
2	5.6 ml; 1.4 µl; 2.8 µl	8 ml; 2 µl; 4 µl
3	7.4 ml; 1.85 µl; 3.7 µl	11 ml; 2.75 µl; 5.5 µl
4	9.2 ml; 2.3 µl; 4.6 µl	14 ml; 3.5 µl; 7 µl
5	11 ml; 2.75 µl; 5.5 µl	17 ml; 4.25 µl; 8.5 µl
6	12.8 ml; 3.2 µl; 6.4 µl	20 ml; 5 µl; 10 µl
7	14.6 ml; 3.65 µl; 7.3 µl	23 ml; 5.75 µl; 11.5 µl
8	16.4 ml; 4.1 µl; 8.2 µl	26 ml; 6.5 µl; 13 µl

***CAUTION**: ethidium homodimer is a powerful mutagen - handle with care and throw contaminated tips, etc., into biohazard bags*

d. Set up luminometer:

Mode = Cycle;	**Pause** = 2 sec;
Data = All;	**Mix** = Off;
Gain = High;	**Temp**. = 30^0C;
Cycle = 1-3;	**A/D reads** = 20 (number of times that each well is analyzed per cycle)

e. Set up vacuum aspirator (attach p-10 tips to each suction line to minimize scraping surface area and only use the plate washer to aspirate and **not** to dispense PBS)

f. Prepare LucLite substrate solution and luciferase positive control (must be used on the same day as prepared)

1) Reconstitute lyophilized substrate solution by adding 10 ml of Assay buffer solution A (provided with kit) to one vial of lyophilized substrate (each vial is enough for 133 assays if 75 µl is used). Agitate gently until a homogeneous solution is formed (a slight turbidity is acceptable). Equilibrate to room temperature before use.

***NOTE**: if more than one vial is reconstituted, combine and mix to prevent any variation from the substrate between plates.*

2) Reconstitute the lyophilized luciferase positive control with 200 µl of distilled (or nanopure water). Each vial contains sufficient luciferase for 20 controls.

5.5.2. Cell Viability Assay Procedure (process one plate at a time)

a. remove plate from incubator and aspirate media, then rinse 1 time with PBS

b. Add 50 µl of PBS with Ca^{2+} and Mg^{2+} to all wells using a 8-channel pipet

c. Add 50 µl of viability assay reagent to all wells using a 8-channel pipet

d. Incubate at room temperature for 10 minutes and then scan plate in the Cytofluor instrument

e. Export/print data (check that values are appropriate, otherwise adjust sensitivity and rescan). Password protect data files with a project-specific password. Data analysis are discussed in Section E along with an example raw data file and a Microsoft Excel spreadsheet version of a sample data analysis.

f. Aspirate viability reagent/PBS, and rinse 1 time with PBS using vacuum aspirator

g. Seal the bottom of the ViewPlates with self-adhesive TopSeal (or tape very well). This increases the signal detection in the luminometer.

h. Add 75 µl of PBS with Ca^{2+} and Mg^{2+} (or media without serum or phenol red) to all wells using an 8-channel pipet

i. Working under SUBDUED light conditions, add 75 µl/well of reconstituted LucLite substrate solution, cover with foil, and agitate gently.

j. Wait twenty minutes before counting the plate to allow full signal generation.

k. Measure luminescence on the Dynatech ML3000 luminometer

l. Save, print, and copy data to disk. Password protect data files with a project-specific password. Data analysis are discussed in Section E along with an example raw data file and a Microsoft Excel spreadsheet version of a sample data analysis.

5.6. Protein Determination

Protein determination is readily accomplished in the same plates with same cell lysates after measuring luminescence. A fluorescamine-based protein assay (Sanderson *et al*., 1996) or Micro-BCA Assay (available from Pierce (800) 874-3723) can be used. Degree of confluency of wells was verified microscopically, and in most cases this made normalization to protein unnecessary; therefore, luciferase activity is reported as either relative light units (RLU) or percent of solvent control.

5.7. Data Analysis

5.7.1. Viability Data

Average the three measurements for calcein AM and ethidium. Divide the average calcein AM fluorescence for each sample by its ethidium homodimer fluorescence to obtain a live to dead ratio. Graph the average calcein AM fluorescence and standard deviation for the negative control, solvent control, and each concentration tested. Examine the calcein AM data visually. If the blank has greater viability than the other treatments, the solvent may be toxic to the cells. If viability decreases with increasing concentration of the test substance, the test substance may be toxic to the cells. In either of these cases, the luciferase data must be regarded with great suspicion. If the solvent is toxic, try a different solvent or a lower concentration of solvent. If the test substance is toxic, try extracting the toxic component (*e.g.*, removing sulfur compounds from sediment extracts), or conclude that cytotoxicity is likely to preclude any dioxin-like effects.

5.7.2. Luciferase Data

Calculate averages and standard deviations for each treatment. Graph the change in response and its standard deviation against concentration. Relative potencies should be calculated through conversion of the data to probit values or any other appropriate transformation that linearizes the sigmoidal dose-response curve. For a complete description and decision tree for data analysis methods, refer to the Standard Operating Procedures entitled, ìEstimation of Relative Potencies Based on In Vitro Bioassay Resultsî. To convert data to probit values, the concentration producing maximal induction is set at 100% and the responses of all other concentrations are converted to a percentage of this maximal level. A lookup table function can be used in a spreadsheet program such as Excel or Lotus 1,2,3 to convert percentages to probit values (contact the Aquatic Toxicology Laboratory for a copy of this table on disk).

6.0. RECORDS, DOCUMENTATION, AND QC REQUIREMENTS

6.1 Records and Documentation

The primary analyst shall document any anomalies and/or deviation from the specified method in a bound, serially numbered, laboratory notebook with tear-out carbon copies. All electronic files and hardcopies will be kept at the Aquatic Toxicology Laboratory at Michigan State University and a duplicate copy will be kept in the Archive Room of Dr. John Giesy (Dept. of Zoology, Michigan State University). This information will also be recorded in the data package and listed in the Case Narrative Form (in accordance with SOP 802). The primary analyst will sign and date any forms as the analyst.

The technical reviewer will record any problems noted during the technical review. The technical reviewer will return the items to the analyst for corrections prior to inclusion into the data package. The technical reviewer will sign and date all forms as the reviewer.

6.2 QC Requirements and Data Quality Objectives

The threshold dose and EC_{50} for luciferase induction in H4IIE-luc cells were approximately 0.1 and 1.2 pg/well, respectively, as determined from 41 separate standard TCDD curves analyzed in 1997 (Figure 1). Coefficients of variation (standard deviation/mean x 100) for the assay were under 10% at all concentrations tested for any single day of experiments. A proficiency curve is maintained for the EC_{50} and threshold doses for TCDD standard curves (every plate has a standard curve). If for a particular plate, the EC_{50} or threshold for the TCDD standard exceeds $\pm$ 20 % of the proficiency curve, then the sample on the plate in question will be reanalyzed. For a sample size of 20 g tissue and a final extract volume of 0.25 ml, the H4IIE-luc assay will detect 1 part per trillion (ppt; pg/g, wet weight) TCDD-equivalents. With a sample size of 5 g tissue, 4 pg/g (wet weight) TCDD-equivalents will be detected.

7.0. RESPONSIBILITIES

The primary analyst will complete the analysis as specified in this SOP and provide documentation of raw data and any anomalies and provide data to the data analyst who will perform data calculations in accordance with SOP 202.

The technical reviewer will determine if data quality objectives were met, notify the analyst if any problems were found.

8.0. REFERENCES

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ATTACHMENTS

Attachment 201-1: Figure 1 - TCDD Dose-response Curve with H4IIE-luc Cells
Attachment 201-2: Figure 3 - Viability Assay Sample Data Set (Raw Data)
Attachment 201-3: Figure 4 - Viability Assay Sample Data Set (Calculated Data)
Attachment 201-4: Figure 5 - Luciferase Assay Sample Data Set (Raw Data)

Attachment 201-5: Figure 6 - Luciferase Assay Sample Data Set (Calculated Data)

APPENDIX 4:

Standard Operating Procedure
For
Instrumental Analysis of PCDDs and PCDFs

Michigan State University
National Food Safety and Toxicology Center
Aquatic Toxicology Laboratory

STANDARD OPERATING PROCEDURE

Extraction and Analysis of 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxins (PCDDs) and dibenzofurans (PCDFs) in Sediments Using High Resolution Gas Chromatography- High resolution Mass Spectrometry

Version 1 March, 2001

Kurunthachalam Kannan and John P. Giesy

Supported through:
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APPROVAL PAGE

Revisions to an existing SOP, addition of a SOP change form, or preparation of a new SOP must be reviewed, approved, and signed by the following:

Authored By: Kurunthachalam Kannan and John P. Giesy Date: 03/21/01

Supervisor Review By: _____ Date: _____

Reviewed By: _____ Date: _____
(QA Coordinator)

DEFINITIONS AND ACRONYMS

PCDDs	Polychlorinated dibenzo-*p*-dioxins
PCDFs	Polychlorinated dibenzofurans
TCDD	Tetrachlorodibenzo-*p*-dioxin
ATL	Aquatic Toxicology Laboratory (Michigan State University)
HRGC/HRMS	High resolution gas chromatograph/ High resolution Mass spectrometer

1.0 SCOPE AND APPLICATION

This method describes the homogenization, extraction and clean-up procedure of sediment samples for measuring tetra- through octa-chlorinated PCDDs and PCDFs. The method is similar to the U.S. Environmental Protection Agency method 8290 with some modifications and improvements to accommodate sample and laboratory needs. The analytical method calls for the use of high resolution gas chromatography and high-resolution mass spectrometry (HRGC-HRMS) on purified sample extracts. Although the seventeen 2,3,7,8-substituted congeners of PCDDs and PCDFs are the target analytes (Table 1), non-2,3,7,8-substituted can be quantified when appropriate standards are available. The sensitivity of this method is dependent upon the level of interferences within a given sample. The method detection limit for 2,3,7,8-substituted congeners is generally in the range of 0.1 to 1 pg/g, on a dry weight basis. This method is designed to use by analysts who are experienced with residue analysis and skilled in HRGC-HRMS. Because of the extreme toxicity of many compounds, the analyst must take adequate precautions to prevent exposure to materials known or believed to contain PCDDs/DFs. While the sample extraction and clean up procedures are performed at Aquatic Toxicology Laboratory, Michigan State University, high resolution instrumental analysis is performed at Yokohama National University, Japan. Adequate Quality Assurance and Quality Control protocols are employed to verify the integrity of data quality.

2.0. SUMMARY OF METHOD

Sediment samples are ground with a mortar and a pestle, then sieved through 0.34 mm mesh. If sediments are freeze-dried, 15 to 20 g of sediment is homogenized with granular sodium sulfate and Soxhlet extracted for 16 h using 400 mL toluene. If wet sediments are used, approximately 40-50 g wet sediment is homogenized with anhydrous sodium sulfate and extracted as mentioned above. The extract is concentrated to 10 mL and known amounts of ^{13}C-labeled PCDDs/DFs (seventeen 2,3,7,8 congeners) are added as internal standards. The solvent is transferred to hexane, and treated with concentrated sulfuric acid. The extract is then passed through 2 g silica gel packed in a glass column and eluted with 130 ml of 10% dichloromethane in hexane. The extract is treated with activated copper to remove sulfur (although this may not be necessary for MS analysis, removal of sulfur improves detection limits). The extract is then passed through a carbon column packed with 1 g of activated carbon-impregnated silica gel. First fraction eluted with 20 mL of 25% dichloromethane in hexane is discarded. The second fraction eluted with 250 mL of toluene contained 2,3,7,8-substituted PCDDs and PCDFs. PCDDs and PCDFs are analyzed using a HRGC-HRMS. A Hewlett-Packard 6890 GC connected to a Autospec Ultima (VG) was used. PCDD and PCDF congeners were separated on a DB-5 capillary column coated at 0.25 μm (60 m x 0.25 mm i.d.). The mass spectrometer is operated at an EI energy of 40 eV and the ion current was at 600 μA. PCDD/DF congeners were monitored by SIM at the two most intensive ions at the molecular ion

cluster. Concentrations of certain PCDD/DF congeners, particularly TCDD and TCDF congeners are confirmed by using DB-17 (60 m x 0.25 mm i.d., 0.25 μm film thickness) column.

3.0 SAFETY CONSIDERATIONS

All safety considerations will be in accordance with MSU-ORCBS procedures and with the requirements of the MSU ATL Safety Manual. These requirements include:

Personnel protective equipment (PPE) consisting of lab coats, safety glasses, and latex gloves will be worn at all times when handling samples.

All equipment used to cut and homogenize samples represent potential hazards. All personnel using such equipment must be aware of these hazards and must operate equipment according to the manufacturerís safety procedures.

Organic solvents are used for rinsing equipment, glassware cleaning, and in the extraction phase of the method. These solvents represent a potential hazard to personnel in the laboratory. Care must be taken to minimize exposure in accordance to institutional guidelines (refer to the Safety Manual for the Aquatic Toxicology Laboratory at Michigan State University).

4.0 EQUIPMENT, MATERIALS, AND REAGENTS

Reuse of glassware should be minimized to avoid contamination. All glassware that is reused must be scruplously cleaned as soon as possible after use according to the following procedure: Rinse glassware with the last solven used in it. Wash with hot detergent water, then rinse with copious amounts of tap water and then with nonapure water (or orgnic-free reagent water). Rinse with high purity acetone and hexane and store it inverted or capped with solvent rinsed aluminum foil in a clean environment.

4.1 Equipment and Materials

The following equipment is used while performing this method. Equivalent equipment is acceptable.

4.1.1 Homogenization

- Balance (sensitivity to 0.100 g)
- Pestle and mortar
- Freezer @ -20^{o} C (For sample storage)
- Organic solvent wash bottles
- Alconox detergent

- Glass sample jars (I-Chem)
- Spatula

4.1.2 Extraction and Concentration

- Soxhlet apparatus (Soxhlet body and condensors)
- 500 mL round bottom flasks
- Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar.
- Teflon boiling chips, washed with methylene chloride prior to use. Note: Teflon boiling chips may not work in the presence of any water phase.
- Nitrogen Evaporator, Organomation
- Rotovap with adopters
- Eppendorf or equivalent pipettes

4.1.3 Sample Clean-up

- Glass columns (11 mm i.d X 30 cm long): custom made to accommodate column reservoirs
- Beakers
- 250 ml collection jars
- long glass rod
- glass fiber filter, rinsed with methylene chloride, dried and stored in a clean beaker
- disposable glass pipets

Note: Prior to using glassware, rinse 3 times with acetone and 3 times with hexane.

4.4.4. Other Equipment

- Syringes
- Aluminum tray
- Rotary Evaporator
- Homogenizer

4.2 Reagents

- ASTM Type I Water or equivalent.
- Toluene, high purity
- Acetone, high purity
- Hexane, high purity

- Methylene chloride, high purity
- 13C-labelled 2,3,7,8-substituted PCDD/DF congeners
- Copper granules
- Concentrated Sulfuric acid
- Sodium sulfate, anhydrous, extracted with methylene choride and dried.
- Silica gel (60/100 mesh)
- Alumina
- Activated carbon
- PCDD/DF congener standards, as appropriate

4.3 Standards Preparation

4.3.1 Prepare PCDD/DF standards for the derivation of a standard curve and/or use as a matrix spike.

4.3.2 Prepare PCDD/DF standards, as appropriate. Multicomponent standards are acceptable (for example, one working standard solution containing seventeen 2,3,7,8-substituted PCDDs/DFs at 100 ng/ml each).

4.3.3 Dilute the stock solution with appropriate solvent for a working standard solution of approximately 1 µg/ml.

4.3.4 Dilute the stock solution with appropriate solvent for a working standard 2 of approximately 500 ng/ml.

4.3.5 Dilute the stock solution with appropriate solvent for a working standard 3 solution of approximately 100 ng/ml.

Note: The final concentration of the standards is based on the final sample volume of 1.0 ml. This value does not represent the amount of sample extracted.

4.4 Surrogate Stock Standard Preparation

4.4.1 13C-labelled PCDD/DF congeners.

4.4.2 Bring to volume with appropriate solvent for a surrogate stock of approximately 1 µg/ml.

4.4.3 Prepare a surrogate standard. Transfer approximately 1.0 ml of surrogate stock to a 10ml volumetric flask and bring to volume with µg/ml for a working standard of 100 ng/ml. Record actual volume transferred.

4.5 Standard Storage and Use Conditions

Store spiking solutions at 4°C in sealed volumetric flasks.

Note: Analysts must allow all spiking solutions to equilibrate to room temperature before use.

Note: Each time a vial containing small volumes of solutions is warmed to room temperature and opened, a small volume of solvent in the vial headspace evaporates, significantly affecting concentration. Solutions should be stored with the smallest possible headspace, and opening vials should be minimized. Also, the solution must be at room temperature before use.

5.0 SAMPLE HANDLING AND HOMOGENIZATION

5.1 Sample Collection, Preservation and Handling

In the laboratory, all samples must be kept frozen (-20°C) until grinding and/or sample extraction. A sample track sheet is used to locate samples, processing date and so on.

5.2 Sample Homogenization and Extraction

5.2.1 Sediment

5.2.1.1 Preparation & Homogenization

Sediment samples must be removed from the freezer and should be allowed to bring to room temperature. If sediments are freeze-dried, 20 g sediment is transferred to a clean aluminum foil using a solvent cleaned spatula. The sediment is mixed thoroughly with the spatula to aliquot a representative sample. The weight of the sample is recorded on a sample log to the nearest 0.01 g. If the sample is wet, 40 g sediment is recommended. Remove, pebbles, roots, twigs etc, if any. Grind/homogenize sediments with anhydrous sodium sulfate in a homogenizer or using pestle and mortar until the mixture is completely dry and freely flowing. The amount of sodium sulfate needed to homogenize the samples to dryness varies depending on the moisture content of the sediment. Generally a ratio of 1:6 sediment:sodium sulfate is adequate. If the dried sample is not of free flowing consistency, more sodium sulfate must be used. Sodium sulfate may contain phthalates and other impurities. Therefore, bake sodium sulfate at 450°C for 3 h or Soxhlet extract with methylene chloride for 24 h, prior to use.

Note: Exercise adequate caution while using homogenizer. Careless handling will harm your fingers.

- The homogenized mixture is transferred to a Soxhlet extractor.
- In a round bottom flask, measure 400 mL toluene. Add solvent cleaned teflon coated boiling chips. Connected the round bottom flask with Soxhlet apparatus and make sure that the connection is intact.
- Open water to the condenser, Turn the Rheostat switches on, and allow the setting at 5. The solvent must cycle completely through the system at least 5 times per hour. Watch for the first two or three cycles to complete.

Note: Donít leave the place immediately after turning the Soxhlet on. Stay for the first few cycles to complete and make sure that the extraction cycle is complete and the condenser is cool enough.

- Extract for 16 h.
- After 16 h, let cool. If the extract contains residues of soil, it has to be filtered through a glass fiber filter into a 500 mL round bottmo flask for evaporation of the toluene.
- Concentrate that toluene extract to near dryness on a rotary evaporator at 40°C.
- Remove the flask from the water bath and allow to cool for 5 minutes. Transfer the residue to a 25 mL tube using hexane and rinse the flask with two additional portions of hexane and add the rinses to the funnel. Evaporate to 10 mL under a gentle stream of nitrogen.

5.2.1.2 Equipment Cleaning

If an homogenizer is used to homogenize sediment samples, between each sample, the homogenizer is to be cleaned. For safety reasons due to the design of the equipment, it is not possible to rinse the body of the grinder with organic solvent. However, all removable parts (e.g. grinding plate and blades, and worm feed) are removed and washed with an Alconox detergent and then flushed with copious quantities of tap water. The parts are then rinsed 3 times with methanol and then 3 times with Milli-Q water. All sample handling equipment (e.g. trays, mixing spoons) are also washed by this procedure.

5.3 Sulfuric Acid Treatment

- Add 10 mL of concentrated sulfuric acid to the extract.
- Shake gently for 2 min.
- Transfer the extract to a another test tube and add 10 mL of hexane-washed water and shake gently to remove acid residues in the extract.

- Transfer the organic extract to a tube. If necessary, organic extract may be dried by pouring it through a filter funnel containing anhydrous sodium sulfate on a glass wool plug and collect it in a 50 mL round bottom flask.

5.4 Silica Gel Column Clean-up

- Pack a gravity column fitted with glass stopcock or teflon stopcock.
- Insert a glass wool plug into the bottom of the glass column (11 mm X 30 cm).
- Add about 5 cm equivalent of hexane and drain the hexane.

Note: Silica gel (60/100 mesh) is activated at 130°C for 3 h prior to use. The activated silica gel is immediately transferred quantitatively (2 g) into a beaker containing hexane.

- Using hexane, transfer 2 g activated silica gel slurry into the column.
- Open stopper to let hexane drain out.
- Rinse side walls of the glass column with hexane to settle the silica gel.
- Tap the sides of the column gently to ensure that the silica gel is compact and that there is no air-bubble/channeling.

Note: Donít let silica gel become exposed to air while and after packing.

- Add 2 g of sodium sulfate to top of silica gel.
- Drain excess hexane, but donít let sodium sulfate exposed to air.

5.4.1. Fractionation

- Transfer the 10 mL sediment extract onto the silica gel (sorbent) bed and drain. Rinse the extract vial or tube with hexane three times and transfer to the column.
- Add 200 mL of hexane to reservoir and elute from top of sodium sulfate.
- Collect the eluate into a flat-round bottom flask.
- Concentrate the extract to 5 mL on a rotary evaporator (40°C water bath temperature).

5.5 Alumina Column Clean-up

- Depending upon the nature of contamination, alumina column clean up is necessary.
- Pack a gravity column (11 mm X 30 cm) fitted with a teflon stopcock with neutral alumina as follows:

- Insert a glass wool plug into the bottom of the column.
- Add a four g layer of sodium sulfate. Add a 4 g layer of neutral alumina. Tap the top of the column gently. Neutral alumina need not be activated or cleaned before use, but it should be stored in a sealed dessicator. Add a 4 g layer of anhydrous sodium sulfate to cover the alumina. Elute with 10 mL of hexane and discard the hexane. Check the column for channeling. If channeling is observed, discard the column. Donít tap the wet column.
- Transfer the extract from the silica gel column on to the top of the alumina column. Rinse the rotary evporator flask or the tube containing the extract twice with 2 mL of hexane and add the rinses to the top of the alumina column.
- Add 20 mL hexane to the alumina column and elute until the hexane level is just below the top of the sodium sulfate. Do not discard the eluted hexane, but collect it in a separate flask and store it for later use, as it may be useful in determining where the labeled analytes are being lost if the recoveries are not satisfactory.
- Add 25 mL of 60% methylene chloride in hexane (v/v) to the alumina and collect the eluate in a round bottom flask and concentrate to 5 mL.

5.6 Carbon Column Clean-up

Activated carbon impregnated silica gel from Wako Chemical Company, Tokyo, Japan, is used. The carbon need not be activated or cleaned.

- Pack a gravity column (11 mm X 30 cm) fitted with a teflon stopcock with activated carbon impregnated with silica gel as follows:
- Insert a glass wool plug into the bottom of the column.
- Add a 1 g layer of sodium sulfate. Add a 1 g layer of activated carbon impregnated silica gel. Tap the top of the column gently. Add a 1 g layer of anhydrous sodium sulfate to cover the carbon. Elute with 150 mL of hexane and discard the hexane. This fraction contains ortho-substituted PCBs. The second fraction, which is eluted with 200 mL of toluene, that contains PCDDs and PCDFs.
- Concentrate the toluene fraction to 1 mL on a rotary evaporator by using a water bath at 50°C. Carefully transfer the concentrate into a 1 mL minivial and reduce the volume to about 100 µL using a gentle stream of nitrogen.

5.7. Chromatographic and Mass Spectrometric Conditions

A Hewlett-Packard 6890 GC connected to a Autospec Ultima (VG) is used. PCDD and PCDF congeners were separated on a DB-5 capillary column coated at 0.25 μm (60 m x 0.25 mm i.d.). The column oven temperature was programmed from 160°C (3 min) to 200°C at a rate of 40°C/min with a 2 min hold time, and to 310°C at 2°C/min, which was held for 1 min. Injector and transfer line/ion source temperatures were held at 280 and 250°C, respectively. The GC oven temperature programming may vary, depending upon the samples, but the same temperature program is followed samples for a given project. This would ensure comparability. The mass spectrometer was operated at an EI energy of 40 eV and the ion current was at 600 μA. PCDD/DF congeners were monitored by SIM at the two most intensive ions at the molecular ion cluster (Table 2). It is important to maintain the same set of ions for both calibration and sample extract analyses.

The recommended mass spectrometer tuning conditions are based on the groups of monitored ions. By using a PFK molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF). By using peak matching conditions and the aforementioned PFK reference peak, verify the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low- and high-mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors.

5.8. Data Acquisition

Total cycle time for data acquisition must be ≤1 second. The total cycle time includes the sum of all the dwell times and voltage reset times.

Acquire SIM data for all the ions in the five descriptors. The theoretical ratios of two selected molecular ions should be within the limits as prescribed in Table 3 for valid identification of PCDD/DF peaks.

6.0 QUALITY CONTROLS

For each set of 20 samples, there will be a minimum quality control that includes a solvent blank, duplicate method blanks, duplicate matrix spike

6.1 Method Blank

The method blank is extracted with the samples to monitor for any interference that may have been introduced to the sample during sample preparation. Blanks include:

6.1.1 A sodium sulfate extract is used as a solvent blank. This extract should pass through all the steps the samples have gone trhough. For every 15 samples, 1 method blank is necessary.

6.1.2 Extract two samples following the procedure and use as matrix blanks.

6.2 Surrogate Spike

6.2.1 All samples, blanks and matrix spike samples will be fortified with 13C-labelled PCDD/DFs at or immediately after solvent extraction.

6.2.2 Surrogates spike samples are needed to monitor the quantitative transfer of the organic compounds of interest throughout sample preparation to the HRMS detector.

6.3 Matrix Spike/Matrix Spike Duplicate

6.3.1 Prepare and analyze matrix spike and matrix spike duplicate samples to determine the accuracy of the extraction for each matrix that is to be evaluated. The spiked compounds are used to monitor sample matrix effects that could interfere with the analytes of interest.

6.3.2 Prepare each spike using a sample chosen by the analyst. This is usually a control for samples received from a laboratory toxicity test. If environmental samples, the sample should be from a reference site.

6.3.3 Expected concentrations should fall in the mid-range of the initial calibration curve. Additional spikes may be included and may fall in the low-range of the initial calibration curve.

6.3.4 Prepare one matrix spike and matrix spike duplicate per 15 samples, with a minimum of two matrix spikes per batch.

6.4 Performance Evaluation Samples

Including among the samples in all batches may be samples containing known amounts of unlabelled 2,3,7,8-substituted PCDDs/DFs or other PCDD/DF congeners.

6.5 Performance Check Solutions

At the beginning of each 24-hr period during which samples are to be analyzed, an aliquot of the GC column performance check solution and a calibration solution may be injected to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration and to establish the PCDD/DF retention time windows. A mass resolution check shall also be performed to demonstrate adequate mass resolution using an appropriate reference compounds (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed.

To validate positive sample data, the routine or continuing calibration and the mass resolution check must be performed also at the end of each 12 or 24-hr period during

which samples are analyzed. Furthermore, an HRGC-HRMS method blank run must be recorded following a calibration run and the first sample run.

Deviations from criteria specified for the GC performance check or for the mass resolution check invalidate all positive sample data collected between the analyses of the performance check solution and the extracts from those positive samples shall be reanalyzed. If the continuing calibration check performed at the end of a 12 hour period fails by no more than 25% RPD for the 17 unlabelled compounds and 35% RPD for the 9 labeled compounds reference compounds, use the mean response factors from the two daily routine calibration runs to compute the analyte concentrations instead of the response factors obtained from the initial calibration.

7. 0 Method Performance

This method has been used for a variety of biological and sediment samples in the past (see Kannan et al., 2001a, b; ES&T 2001, 35, 441-447; ET&C- Detroit River sediments- in press). Detection limits in the range of 0.1 to 1 pg/g, were achieved for 2,3,7,8-substituted congeners. The recoveries of ^{13}C-labelled PCDDs/DFs, varied from 77 to 95%.

Table 1. Target 2,3,7,8-substituted PCDDs/DFs and their CAS numbers

No.	Compound	CAS No
1	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6
2	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	40321-76-4
3	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	57653-85-7
4	1,2,3,4,7,8- Hexachlorodibenzo-p-dioxin (HxCDD)	39227-28-6
5	1,2,3,7,8,9- Hexachlorodibenzo-p-dioxin (HxCDD)	19408-74-3
6	1,2,3,4,6,7,8- Heptachlorodibenzo-p-dioxin (HpCDD)	35822-39-4
7	Octachlorodibenzo-p-dioxin (OCDD)	3268-87-9
8	2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9
9	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6
10	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4
11	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9
12	1,2,3,7,8,9- Hexachlorodibenzofuran (HxCDF)	72918-21-9
13	1,2,3,4,7,8- Hexachlorodibenzofuran (HxCDF)	70648-26-9
14	2,3,4,6,7,8- Hexachlorodibenzofuran (HxCDF)	60851-34-5
15	1,2,3,4,6,7,8- Heptachlorodibenzofuran (HpCDF)	67562-39-4
16	1,2,3,4,7,8,9- Heptachlorodibenzofuran (HpCDF)	55673-89-7
17	Octachlorodibenzofuran (OCDF)	39001-02-0

Table 2. Ions monitored for HRGC-HRMS analysis of PCDDs/DFs

Accurate mass	Ion	Analyte
303.9016	M	TCDF
305.8987	M+2	TCDF
315.9419	M	13C-TCDF
317.9390	M+2	13C-TCDF
318.9792	Lock	PFK lock mass
319.8965	M	TCDD
321.8936	M+2	TCDD
331.9368	M	13C-TCDD
333.9339	M+2	13C-TCDD
339.8598	M+2	PeCDF
341.8569	M+4	PeCDF
351.9000	M+2	13C-PeCDF
353.8970	M+4	13C-PeCDF
353.8576	M	PeCDD
355.8546	M+2	PeCDD
367.8949	M+2	13C-PeCDD
369.8919	M+4	13C-PeCDD
330.9792	Lock	PFK lock mass
373.8209	M+2	HxCDF
375.8179	M+4	HxCDF
383.8639	M	13C-HxCDF
385.8610	M+2	13C-HxCDF
389.8156	M+2	HxCDD
391.8124	M+4	HxCDD
401.8561	M+2	13C-HxCDD
403.8531	M+4	13C-HxCDD
380.9760	Lock	PFK lock mass
407.7818	M+2	HpCDF
409.7788	M+4	HpCDF
417.8250	M	13C-HpCDF
419.8220	M+2	13C-HpCDF
423.7767	M+2	HpCDD
425.7737	M+4	HpCDD
435.8169	M+2	13C-HpCDD
437.8140	M+4	13C-HpCDF
430.9728	Lock	PFK lock mass
441.7428	M+2	OCDF
443.7399	M+4	OCDF
457.7377	M+2	OCDD
459.7348	M+4	OCDD

469.7780	M+2	13C-OCDD
471.7750	M+4	13C-OCDD
442.9728	Lock	PFK lock mass

Table 3. Theoretical ion abundance ratios and their control limits for PCDDs/DFs

No. of Cl	Ion	Theoretical ratio	Control limit	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02
6 (a)	M/M+4	0.51	0.43	0.59
7 (b)	M/M+4	0.44	0.37	0.51

(a) only for 13C-HxCDF
(b) only for 13C-HpCDF

APPENDIX 5:

Dioxin-Like Activity in Soil and Sediments from the Tittabawassee River Watershed: Mass Balance Calculations

Dioxin-Like Acitivity in Soil and Sediments from the Tittabawassee River Watershed: Mass Balance Calculations

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Mass balance calculations were based on comparison of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin equivalents determined by H4IIE-luc bioassay (TCDD-EQs) and those calculated from instrumental analysis of concentrations of PCDD/Fs multiplied by specific relative potencies (TEQs). The bioassay was performed as described in the Standard Operating Procedure 201 of Aquatic Toxicology Laboratory, Michigan State University: H4IIE-luc Bioassay for the Detection of Ah Receptor Agonists.

The following set of experiments was performed to assess the dioxin-like activity of the sediment and soil samples from the Tittabawassee River Basin:

- Experiment 1 - calibration dose-response check to assess cellís responsiveness
- Experiment 2 - screening of all extracts at 2 dilutions
- Experiment 3 - full dose-response curves for the 19 composites of sediment samples
- Experiment 4 - full dose-response curves for the most active sediment composite samples after acidic treatment (C2, C4, C5, C7, C10, C11, C13, C14, C15)
- Experiment 5 - full dose-response curves for 11 soil samples
- Experiment 6 - full dose-response curves for 18 individual sediment samples (transect samples)
- Experiment 7 - full dose-response curves for acid treated soil samples
- Experiment 8 - full dose-response curves for 14 individual sediment samples (individual samples from selected composites)

Results from bioassays: calculation of TCDD-EQs

Dioxin-like activity of 62 sediment/soil samples (6 dilutions each) was tested using *in vitro* bioassays. The 11 soil samples and 9 most active sediment composite samples were also tested after acidic treatment of the extract (acid treatment removes compounds like PAHs which are also active in the Ah-R based bioassays). All sample extracts elicited significant (different from solvent control) induction of dioxin-like activity. Dioxin equivalents were determined from the dose-response relationship of the sample relative to the dose-response curve of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD) standard. The results of bioassay and the mass balance analysis are presented in MS Excel file ì mass balance.xlsî . Part 1 presents the tables with results from individual experiments, they are summarized in part 2 and part 3 (table 1 and 2). The 2,3,7,8-tetrachlorodibenzo-*p*-dioxin equivalents (TCDD-EQs) were derived at three different levels of response (REP-20 as relative potency at 20 % maximal standard response; REP-50 as relative potency at 50 % maximal standard response; REP-80 as relative potency at 80 % maximal standard response). However, not all the samples reached 50 % or 80% maximal standard induction (efficacy), thus the TCDD-EQs derived at these levels of response (REP-50, REP-80) would be estimates. The maximal level of induction for each sample is shown in the tables as efficacy in % of maximal standard (TCDD) induction. All samples (except of SS10 after acidic treatment) reached 20 % of the maximal standard induction, thus the TCDD-EQ derived at REP-20 level was used as the most suitable value for all the samples for the mass balance calculations.

Instrumental analyses: calculations of TEQs

The 2,3,7,8-tetrachlorodibenzo-*p*-dioxin equivalents were calculated based on the results of chemical analysis (TEQs) as the sum of concentrations of individual PCDD/Fs congeners multiplied by their specific relative potencies. The relative potencies were based on a recent study, where specific REP were derived for the different levels of response in a bioassay (REP-20, REP-50, REP-80, Brown et al., 2001, Organohalogen Compounds 53:211-214). Table 3 shows the TEQs at two different levels of response: EC_{20} and EC_{50}. For mass balance, we used the TEQs based on the specific potencies at 20% maximal standard response (EC_{20}), and by this way the bioassay- and analytic-based dioxin equivalents could be directly compared at the same level of response. The calculation of TEQs from the analytical results as well as the specific relative potencies are shown in file ìTittabawassee TEQ calculation.xlsî.

The tables with two significant digits-numbers should always be used for presenting the results, since that is the precision of bioassay and/or the analytical techniques.

Interpreting the mass-balance results

Tables 4 and 5 show the comparison of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin equivalents derived from bioassay (TCDD-EQs) and calculated from the analytical results (TEQs). In table 4 all results are from the raw extracts, in table 5 are the results for samples that were treated with sulfuric acid (C2, C4, C5, C7, C10, C11, C13, C14, C15 and SS1-SS10) along with the results from the raw extracts for all the other samples (same as in table 4). Acidic treatment removes the non-persistent compounds (such as PAHs) that could contribute to total dioxin-like activity. The acid treated extracts were tested on bioassay to confirm the magnitude of the activity caused by persistent compounds such as chlorinated dioxins and furans and coplanar PCBs relative to non-persistent chemicals. Figures 1 to 3 show that the correlation between TCDD-EQs and TEQs was significant. The slope of the regression is close to 1 documenting that the analyzed PCDD/Fs are responsible for most of the dioxin-like activity found in the samples. Figure 1 presents results from the raw extracts, figure 2 the combination of results from the acid-treated samples along with the results from the raw extracts for all the other samples and figure 3 shows the results from acid treated samples only. The correlation is better for the acid treated samples (Figure 3) compared with the non-treated ones (Figure 1), suggesting that there might be some contribution from the non-persistent compounds to the dioxin-like activity in some samples.
Detailed comparison of the TCDD-EQs and TEQs is presented in figures 4 to10. The results show very good agreement between bioassay and analytically derived dioxin equivalents. This documents that PCDD/Fs are responsible for majority of the dioxin-like activity in most samples. Due to the different sources of variation in bioassay measurements less than two-fold difference is not considered significant. From the samples with greater activities C2, C4, C13, C14, SS1 and SS2 (Fig.4 or Fig.6 and 8) had more than 2 fold greater bioassay TCDD-EQs than TEQs based on PCDD/Fs concentrations, suggesting contribution of some unidentified compounds to the total

dioxin-like activity. However the difference between TCDD-EQs and TEQs were much less after treatment of these samples with sulfuric acid (Fig. 9and 10), suggesting that acid labile compounds were the contributors to TCDD-EQs. The relative contribution of unidentified compounds to the total dioxin-like activity has been more pronounced for the samples with very low PCDD/Fs concentrations (such as TG-UC, TH-C, TH-UC, TI-C, TI-UC, C16, C17, C18, C19, 52, 59, 60, SS3, SS4 and SS10, Fig. 4, 5, 6, 7, 8). PCDD/Fs contribute little to total dioxin-like activity in these samples, but also the total TCDD-EQs in these samples is much less compared to other samples. The comparison of some of these samples before and after acidic treatment (SS3, SS4, SS10, Fig.8 and 10) document that the TCDD-EQs were much less after acidic treatment again suggesting the contribution of non-persistent compounds to dioxin-like activity in these samples.
The contribution of the unidentified compounds is more obvious in these samples since there is very little PCDD/Fs present. The contribution of TCDD-EQs by unknown compounds is not significant in the samples that contain greater PCDD/Fs concentrations.

Conclusions of bioassays:

- in all samples a significant (different from solvent control) dioxin-like activity was found.

for most studied samples PCDD/Fs account for most of the dioxin-like activity.

- non-persistent, acid labile compounds (e.g., PAHs) contributed to greater TCDD-EQs in highly-active samples C2, C4, C13, C14, SS1 and SS2

- unidentified compounds contributed to total TCDD-EQs in samples with very low overall dioxin-like activity and PCDD/Fs concentrations: TG-UC, TH-C, TH-UC, TI-C, TI-UC, C16, C17, C18, C19, 52, 59, 60, SS3, SS4 and SS10 (these compounds were shown to be non-persistent in SS3, SS4 and SS10)