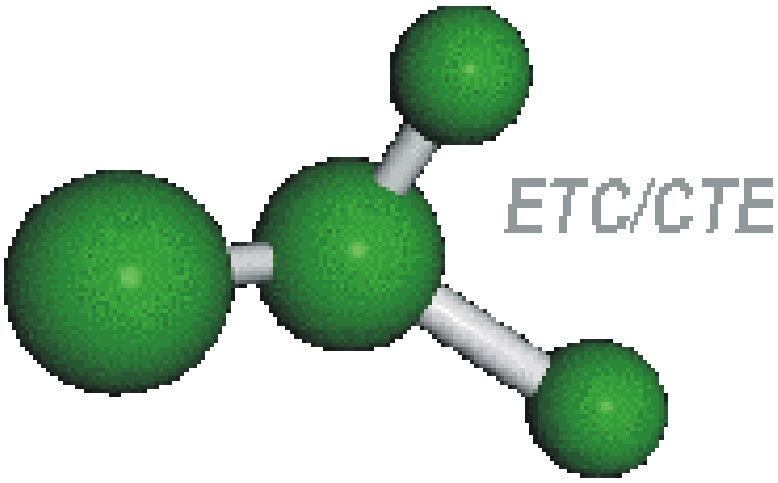
Determination of Level of Quantification for Measuring PCBs in Stack Emission and Ash Samples



Draft 1

AAQD/ERMD
Environmental Technology Centre

January 2001

Environment Canada Environnement Canada

Summary

Environment Canada has included polychlorinated biphenyls (PCBs) in the list of Track 1 substances - toxic substances that are persistent, bioaccumulative and anthropogenic. Virtual elimination from the environment of the Track 1 substances is the main objective of the Toxic Substance Management Policy.

The Level of Quantification (LoQ) described in this report will be the bench mark for achieving virtual elimination for the release of PCBs from combustion facilities. LoQ is defined as the lowest concentration of target compounds that can be quantified confidently. It is determined by assessing the variability (standard deviation) of replicate measurements of analytes at a concentration near the detection limit.

A landfill gas incinerator and a municipal solid waste incinerator fly ash were chosen for this study. Stack gas samples were collected using 12 sampling trains spiked with PCBs and analyzed following the PCB reference method. Analyses of ten replicates of a 5-gram ash sample spiked with PCBs were also performed. LoQ is determined as ten times the standard deviation of the repeated measurements. LoQ for measuring PCBs in stack emission and incineration ash samples is estimated as 2 ng/m^3 (7.5 ng/sample) and 0.8 ng/g (4 ng/sample) respectively. These values are applicable to a single PCB congener-peak.

Abbreviations

Abbreviation	***Meaning***
σ	Standard Deviation of a population
CEPA	Canadian Environmental Protection Act
cfm	Cubic feet per minute
DL	Detection Limit
ETC	Environmental Technology Centre
GC-MS	Gas Chromatography – Mass Spectrometry
HPLC	High Performance Liquid Chromatography
IUPAC	International Union of Pure and Applied Chemistry
LFG	Landfill Gas
LoD	Level of Detection
LoQ	Level of Quantification
MM5	Modified Method 5
MSD	Mass Selective Detector
ng/g	nanogram/gram dry weight at reference conditions(nano = 10^{-9})
ng/m^3	nanogram/cubic meter at reference conditions
ng/mL	nanogram/milliliter
PCBs	Polychlorinated Biphenyls
RSD	Relative Standard Deviation
s	Standard Deviation of a sample
SD	Standard Deviation
TEF	Toxic Equivalency Factor
TEQ	Toxicity Equivalent
TSMP	Toxic Substance Management Policy

Acknowledgements

This report represents the result of team effort. Team members who have made significant contribution to this project in specific areas are:

Sampling: Dominic Cianciarelli[a], Ian Rigden [a] and Stephanie Bourgeau [a]
Sample Workup: Gary Poole[b], Anna Moudrakovskaia[b], Mike Lister[b], Aline Dombroskie[b] and John Coltess[b]
GC-MS Analysis: Mylaine Tardif [b], Monique Lanoy[b] and David Harnish[b]
Coordinator and report writing: Chung Chiu[b]

[a] Emissions Research and Measurement Division, ETC
[b] Analysis and Air Quality Division, ETC

Introduction

Polychlorinated biphenyls (PCBs) were commercially produced between 1929 and 1977. It was estimated that a total of 610,000 tonnes were produced worldwide [(1)] and approximately 40,000 tonnes of PCBs were imported into Canada prior to 1997 [(2)]. Because of its unique properties, PCBs were widely used as dielectric fluids in transformers and large capacitors, as heat transfer fluids, hydraulic fluids, lubrication oils and as additives in pesticides, paints, copying papers, adhesive and plastics [(3)].

All PCBs used in North America were manufactured in the United States under the trade name Aroclor – a series of product with various degrees of chlorination. In theory, there are 209 possible PCB compounds or congeners. Up to 157 congeners have been identified with a weight above 0.01% in commercial Aroclor mixtures [(4)].

Environment Canada has declared PCBs as toxic substances under the Canadian Environmental Protection Act (CEPA). A substance is defined as toxic if it adversely affects the environment and/or may constitute a danger to human life or health.

The Toxic Substance Management Policy (TSMP), published in June 1995, provides a framework for making science-based decisions on the effective management of toxic substances. The key management objectives are:

> a) Virtual elimination from the environment of the Track 1 substances - toxic substances that result predominantly from human activities and that are persistent and bioaccumulative.
>
> b) Management of Track 2 substances - through their entire life cycles, to prevent or minimize their release into the environment.

An assessment report [(2)] published by Environment Canada concluded that PCBs meet all the criteria of Track 1 substances and therefore are destined for virtual elimination.

The Level of Quantification (LoQ) is defined as the lowest concentration that can be accurately measured using sensitive but routine sampling and analytical methods (CEPA section 65.1). Any measurement below the LoQ may not be reliably quantifiable. LoQ should therefore be used as a baseline to assist in establishing a virtual elimination target.

The guideline recommended by the American Chemical Society's Committee on Environmental Improvement [(5)] was adopted for the determination of LoQ. American Society for Testing and Materials has also adopted this guideline as a standard practice since 1998 [(6)]. The calculation for LoQ is:

$$\mathbf{LoQ = 10\sigma}$$

where σ is estimated as *s*, the standard deviation (SD) of replicate measurements of an

analyte at a concentration near the detection limit (DL). For a measurement at the LoQ, the uncertainty is ±30% ($10\sigma \pm 3\sigma$) at the 99% confidence level. The 10σ is to ensure that a measurement greater than this value will certainly be greater than 3σ, the level of detection (LoD). The region between 3σ and 10σ represents an area of less-certain quantification and the region above 10σ is in the region of quantification.

This report summarizes the sampling and analytical process for the determination of LoQ for measuring PCBs in stack emission and incineration ash samples.

Study Design

A stable emission source of combustion with almost non-detectable or very little PCBs is an ideal site for the LoQ study. After conducting a survey on available emission sources with consideration to cost effectiveness, a landfill gas facility at a municipal solid waste disposal site near Ottawa was chosen for testing. A total of 12 test runs were conducted using 12 sets of the modified Method-5 (MM5) sampling train apparatus except that the sampling probe was reused each time after cleaning. Before sampling, each train-filter was spiked with a cocktail of PCB mixture at a level around 5 to 10 times the estimated detection limit. Each day after sampling, the train apparatus was transported to the laboratory for sample recovery. A municipal waste incineration ash with a low level of PCBs was also selected for the study. The PCB mixture spiked onto the train filter and the fly ash contained two groups of target compounds: one group had 20 congeners for total PCB analysis, the other group had 12 dioxin-like PCBs for TEQ analysis. PCB congeners and their spiked levels are listed in **Table 1**.

Sampling

Sampling Site and Location

Sampling was conducted at the Trail Road landfill site incinerator in Nepean, Ontario. Trail Road is the main municipal solid waste disposal site for the Regional Municipality of Ottawa-Carleton. The site qualifies as a sanitary landfill as it has a polyethylene liner both above and below the refuse. Trail Road is divided into four stages with stages I, II & III filled and closed. Stage IV has started to receive waste.

The Trail Road facility opened in late 1980 and all waste, which enters the site, is weighed with detailed records available. The total area of the site, including the 135-hectare buffer zone, is 200 hectares and the thickness of the waste is 20 meters. Stages I, II & III have a landfill gas (LFG) collection and flaring system. Roughly 1200 cfm of LFG with approximately 35-45 % methane is collected and flared in a 10-m high stack at around 1000°C and a residence time of 0.75 sec. The presence of the polyethylene liners above and below the refuse and the high number of gas wells across the surface of the two stages allow almost 100 % collection by the LFG collection system. Eventually stage IV will also

be connected to the gas collection system and Trail Road intends to utilize all the site's LFG to produce electricity.

Sampling was conducted from one 4-inch port approximately 12 feet below the stack exit as shown in **Figure 1**. Samples were collected from a single point at a proportional rate.

Sampling Train Description

Each morning prior to sampling, the filter was spiked with a PCB solution in the laboratory. A 200uL volume of isooctane solution containing 2.5-10 ng/ml of the PCB cocktail was transferred onto the filter and allowed to air dry for approximately 2 minutes. The filter was then sealed and transported to the sampling site.

The MM5 sampling train consisted of a water-cooled probe, heated filter enclosure, leak-free vacuum line, vacuum gauge, flow control valves, vacuum pump and a dry gas meter. Stack gases and orifice pressures were measured using an inclined manometer. Temperatures were monitored in the stack, hot box, impinger train outlet and at the inlet and outlet of the dry gas meter as well as at the Amberlite XAD-2 inlet. Most of the train components as shown in **Figure 2,** were assembled in the ETC laboratory. Leak-checks were conducted at the beginning and at the end of each run or whenever a train joint was opened. Stack gas was withdrawn at a proportional rate over a three-hour period.

Glassware Cleaning and Proofing

Prior to the test program, all train glassware, probe brushes, glass wool and aluminum foil were cleaned with appropriate solvents. The cleanliness of glassware was verified by analyzing the proofing rinses of sampling trains used in the study. A total of twelve complete sets of glassware were prepared. Pre-cleaned and proofed commercial sample storage bottles were used. The XAD-2 was pre-cleaned and analyzed for contamination prior to the study. Details of the cleaning and proofing procedures are given in the reference sampling method EPS 1/RM/2 [(7)].

Sample Recovery

Following the completion of each run, the sampling train was recovered in the ETC laboratory. During the transportation between sampling site and lab, all openings were sealed with pre-cleaned glass plugs, caps or aluminum foil. The sample recovery procedures involved the brushing and rinsing of the train components 3x each with acetone and hexane. Only Teflon wash bottles were used during sample recovery. Amberlite tubes were capped and re-wrapped in clean aluminum foil. Liquid samples were stored in pre-cleaned amber bottles with Teflon lids. Sampling train components including filter, XAD, glycol and solvent rinsing of the probe, filter holder condenser, impingers and all glass connectors were recovered following the reference method [(7)]. All samples were kept refrigerated until analysis. Sampling data relating to gas volume, stack temperature, and concentration of oxygen and carbon dioxide are provided in **Table 2**.

Analysis

Train samples recovered from the front-half and the back-half components were combined as one sample for PCB analysis. The solid portion (filter and XAD-2) of the sample was spiked with a solution containing twelve $^{13}C_{12}$-labelled PCB surrogates followed by Soxhlet extraction with toluene for 16 hours. The aqueous portion (condensate and diluted ethylene glycol) was extracted with dichloromethane. Both extracts were combined with the glassware rinses, changed to hexane and concentrated to a small volume before cleanup.

Approximately five grams of the ash sample was weighed out into a glass fiber thimble and spiked with native PCBs and isotope-labelled surrogates. Spiked samples were allowed to dry for 30 minutes before placing into the Soxhlet extractor. Each ash sample was extracted with 400 mL of toluene for 20 hours. Extracts were changed to hexane and reduced to a small volume before column cleanup.

The concentrated extract was cleaned by passing it through a multi-layer acid/base/silver nitrate/silica column. This cleaned extract was split in half for further cleanup. For total PCB, a half portion of the extract was fractionated on an alumina column by eluting with different solvents. After concentrating the eluent to less than 0.18 mL, a 0.02-mL recovery standard was added and the final volume was adjusted to 0.2 mL before gas chromatography – mass spectrometry (GC-MS) analysis. Environment Canada's Reference Method EPS 1/RM/31[8] with respect to sample extraction, cleanup and GC-MS analysis was followed.

An on-column injection of 2 μL was made into an Agilent 6890 GC interfaced to a 5973 Mass Selective Detector (MSD). The GC was equipped with a 10 m x 0.5 mm deactivated fused silica pre-column and a 30 m DB-XLB (14% diphenyl methyl polysiloxane) analytical column with 0.25 mm ID and 0.25 μm film thickness. This analytical column was directly inserted into the MSD operated at 70 eV electron energy with a source temperature of 230 ^{o}C. GC temperature program was 90 ^{o}C for 2 min., 15 ^{o}C/min to 180 ^{o}C, 3 ^{o}C/min to 240 ^{o}C, 10 ^{o}C/min to 285 ^{o}C and hold for 10 min.

For TEQ analysis, the 12 toxic PCB congeners must be free from the interference of other PCBs. This was accomplished by fractionating the other split portion using high performance liquid chromatography (HPLC) with an ethyldimethylsilylated silica column. The fraction containing target PCBs was concentrated to almost dryness and re-dissolved in 0.05 mL recovery standard before GC-MS analysis. Detailed analytical procedures are described elsewhere. [9]

Results and Discussion

Selected PCB Congeners for Spiking

Each train or ash sample was spiked with 2 ng each of the 20 individual congeners for total PCB analysis. These congeners are the same congeners specified in the Reference Method as calibration standards. They represent some of the major PCB compounds in our environment and therefore are selected for the LoQ study. CEPA defines PCBs as chlorobiphenyls that have the molecular formula $C_{12}H_{10-n}Cl_n$ in which "n" is greater than 2 (i.e., tri- to deca-chlorobiphenlys). TEQ has not yet been included in Canadian PCB regulations. However, the interest in PCB-TEQ monitoring by researchers and other government agencies has increased in recent years. To provide adequate information on TEQ for the LoQ study, samples were also spiked with 0.5 ng each of the 12 dioxin-like PCBs. The spike levels as shown in **Table 1** represent approximately 5 to 10 times the detection limit estimated for each method. This level is to ensure that all analytes can be measured even in the event of poor recovery.

Total PCB Analysis

Approximately 3.7 m^3 of gas sample was collected by into each sampling train. **Tables 3** and **3A** summarize PCB train results in ng/train and ng/m^3 respectively. The mean concentration of congeners varies from 2.27 to 8.23 ng. Results suggest that the level of some congeners present in the gas stream is significant. GC-MS data demonstrate that some non-spiked congeners were also present in train samples. The highest relative standard deviation (RSD) is 30% for PCB-18 – one of the lighter compounds. It seems that variance (s^2) decreases as the number of chlorine substitutions increases. Since tri- (e.g., PCB-18) and tetra-chlorobiphenyls (e.g., PCB-52) are relatively volatile, higher loss of these two groups of compounds during sample workup is anticipated. Surrogate recoveries of tri- through deca-chlorobiphenyls are in a range of 40 to 75%. PCBs were automatically corrected for the surrogate recovery. Standard deviations (SD) for all 20-target congeners were pooled. The pooled SD was then used for LoQ determination. With this method, LoQ was determined on the basis of individual congener peak.

An example of the GC-MS chromatographic profile of PCBs in train samples (#531) is shown in **Figure 3**. Target PCBs were identified by matching retention times with those of the calibration standards and abundance ratio of characteristic ions with those specified in the reference method. The ion masses shown in Figure 3 are the lower intensity ions of the two quantification ions for each homologue. Numbers in brackets are the IUPAC number of PCB congener and x denotes a peak identified as PCB. Many non-target peaks were detected as tri-, tetra-, penta-, hexa-, hepta- and octa- chlorobiphenyls as depicted in the ion chromatogram.

The ash sample has less background PCBs as compared to the train sample as shown in **Tables 4** and **4A**. However, congeners such as PCB-23, PCB-33, PCB-70, PCB-118 and PCB-153 were detected at a higher concentration than the spiked level. Mean congener concentration varies from 0.3 to 0.78 ng/g. Variance and RSD, similar to train sample, is high for tri- and tetra-chlorobiphenyls and low for homologues with higher number of

chlorine substitutions. RSD varies from 5% or lower for octa- nona- and deca-chlorobiphenyls to over 20% for trichlorobiphenyls. As for train data, ash samples have lower recovery for PCB-28 and PCB-52 due to their high volatility. The recovery of PCB-180 surrogate in excess of 100% is likely attributed to matrix interference. Overall, the surrogate recovery is satisfactory.

TEQ Analysis

Of the 209 possible PCB congeners, about a dozen compounds are of most concern because they have been shown to exert a number of common toxic responses similar to those observed for 2,3,7,8-TCDD. The international consensus on toxic PCB congeners and their toxic equivalency factors (TEF) had been established and modified during the past decade. The target PCBs and TEF adopted for this study are based on the most recent publication from World Health Organization [(10)]. PCB-TEQ is the sum of the individual toxic PCB congener's TEQ that were derived from multiplying the congener concentration by the corresponding TEF.

Train sample results are reported in **Tables 5** and **5A** as ng/sample and ng/m^3 respectively. The spike level for all toxic congeners except for two compounds was 0.5 ng per sample. Since PCB-105 and PCB-118 are the common congeners used for both total PCB and TEQ analysis, 2.5 ng of each of these two compounds was added to each sample. Although this level exceeds the desirable range (5 to 10 times the detection limit) for LoQ study, the TEQ contribution from these two compounds accounted for less than 1.5% of PCB TEQ. PCB-126, on the other hand, has the highest impact to PCB-TEQ because of its high TEF value. This congener has the same toxic factor as 2,3,7,8-TCDF. RSD varies between 9 and 31% with an average of 11% for the 12 toxic congeners while the RSD for PCB-126 is also 11%. Surrogate recoveries are mostly in the range of 80 to 90%.

Tables 6 and **6A** summarize results of the spiked ash analysis. No background level of any dioxin-like PCBs was observed. Mean concentrations are close to the spiked level of 0.4 ng per sample except for the two common congeners PCB-118 and PCB-105. The RSD appears to be slightly better than train samples and is within a range between 8 and 14%. The RSD for the most toxic congener, PCB-126, is 10%. Surrogate recoveries for measuring TEQ in ash sample are adequate.

Front-Half and Back-Half Train Results

The front-half (filter and solvent rinses of train components located in front of the filter) and the back-half (absorbent and solvent rinses of train components located behind the filter) of the last two trains were analyzed separately to see how much PCBs would be retained on the filter. Results in **Table 7** demonstrate that over 98% of the spiked PCBs have migrated to the back-half by the end of sampling. Only trace amounts of PCB-101 and PCB-194 were detected in the front-half. These two train samples were also analyzed for non-target PCBs. Results show that significant amounts of other PCB congeners,

mainly in tetra- penta- hexa- and hepta-homologues, were detected in the back-half train samples.

Conclusion

Mean concentrations and SD for total PCB analysis for train and ash samples are summarized in **Table 8**. Relative standard deviation and variance (s^2) are high for PCBs that are relatively volatile (i.e., tri- and tetra-chlorobiphenyls) and low for less volatile PCBs that have more chlorine substitutions (i.e., nona- and deca-chlorobiphenyl). The large variation in concentration among congeners is attributed to the background level of PCBs present in the gas and ash samples. Pooled standard deviation (s_p) of the target congeners for train samples is 0.199 ng/m^3 or 0.755 ng/train while s_p for the ash is 0.077 ng/g or 0.385 ng/sample. LoQ for measuring PCBs from an emission source involving MM5 sampling train and GC-MS analysis is therefore estimated as 2 ng/m^3 or 7.5 ng per sample. LoQ for the incineration ash is estimated as 0.8 ng/g or 4 ng per sample.

For TEQ analysis, SD for train and ash samples is 0.0016 ng/m^3 and 0.001 ng/g respectively. It is approximately 0.005 ng/sample for both train and ash samples. The estimated LoQ for measuring PCB-TEQ in stack emissions and ash is 0.016 ng/m^3 and 0.01 ng/g respectively. Since PCB-TEQ measurement is not currently required for PCB regulation under CEPA, these TEQ study results are provided for information only.

References

(1) Durfee, R.L., et al, "*PCBs in the United States – Industrial Use and Environmental Distributions*", USEPA, EPA 560/6-76-005, NTIS No. PB-252 012 (1976).

(2) Environment Canada, *"Scientific Justification, Polychlorinated Biphenyls – Candidate Substance for Management Under Track 1 of the TSMP"*, March 1997.

(3) Erickson, M.D., *"Analytical Chemistry of PCBs, Second Edition"*, CRC Press, 1997.

(4) Frame, G.M., et al, *"Complete PCB Congener Distributions for 17 Aroclor Mixtures Determined by 3 HRGS System Optimized for comprehensive, Quantitative, Congener-Specific Analysis"*, J. High Resol. Chromatogr., vol. 19, 1996.

(5) Keith, L.H., et al, *"Principles of Environmental Analysis"*, Analytical Chemistry, 1983, vol 55, no. 14, p. 2210-2218.

(6) ASTM, *"Standard Practice for Determination of a Pooled Limit of Quantitation"*, D6259-98, July 1998.

(7) Environment Canada, *"Reference Method for Source Testing: Measurement of Releases of Selected Semi-volatile Organic Compounds from Stationary Sources"*, Report EPS 1/RM/2, June 1989.

(8) Environment Canada, *"Reference Method for the Analysis of Polychlorinated Biphenyls (PCBs)"*, Report EPS 1/RM/31, March 1997.

(9) Huang, E.B., et al., "Isolation and Determination of Toxic Congeners of Polychlorinated Biphenyls in Environmental Samples", J. High Resol. Chromatogr., vol. 20, 1997.

(10) Van den Berg, M., et al., Environmental Health Perspectives, 106: 775-792, 1998.

Figures and Tables

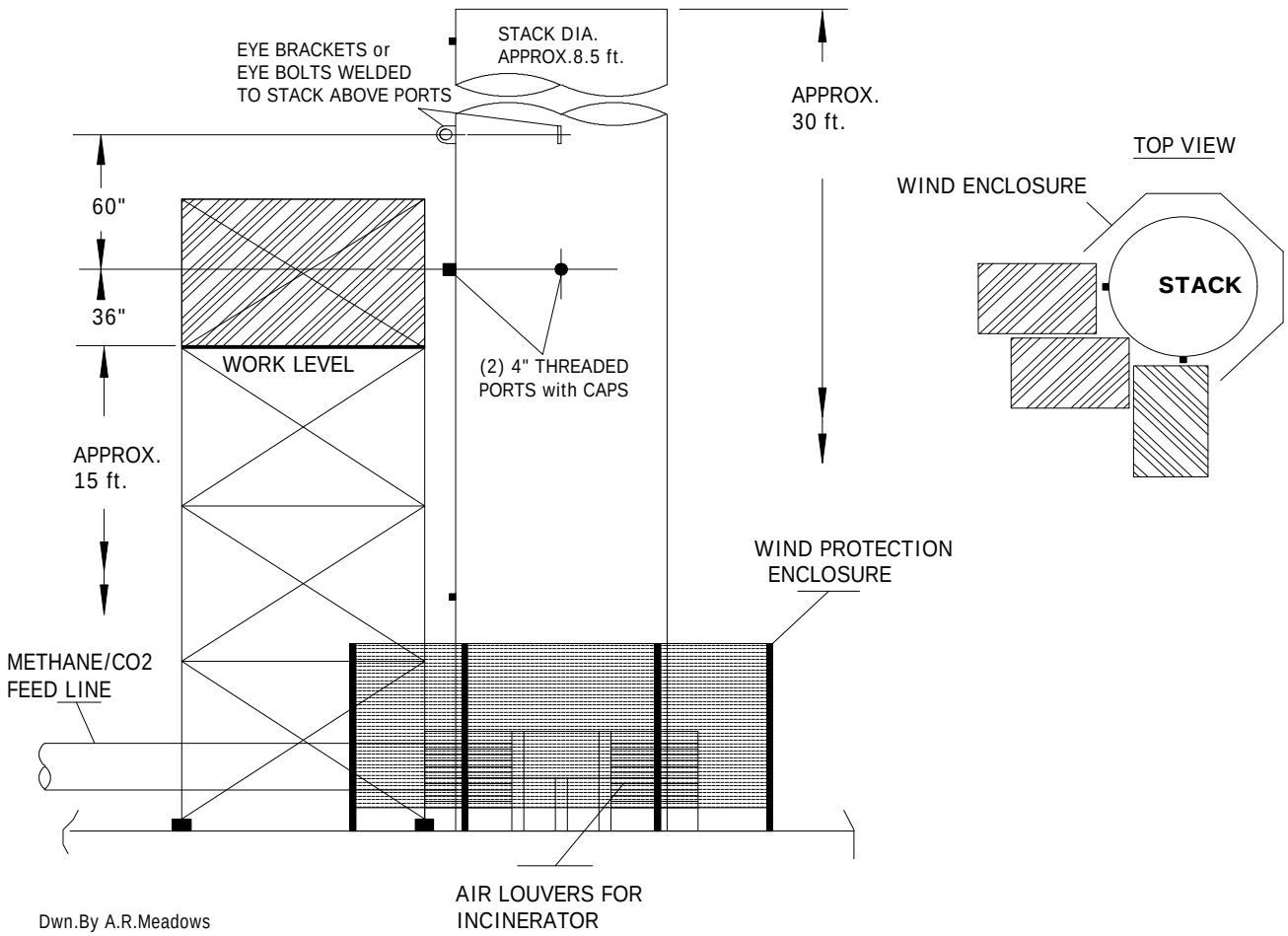


Figure 1 Trail Road Incinerator Sampling Location

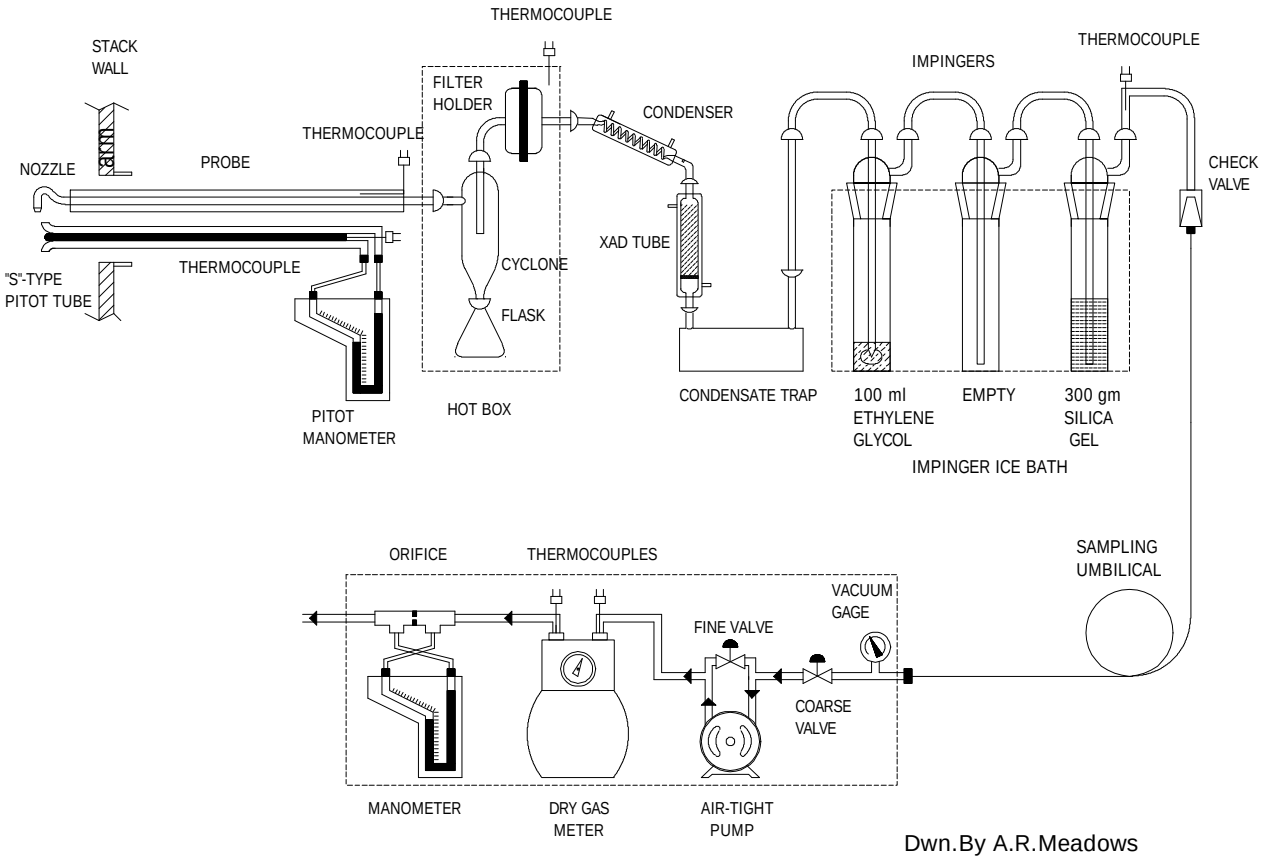


Figure 2 Semi-Volatile Organics Sampling Train

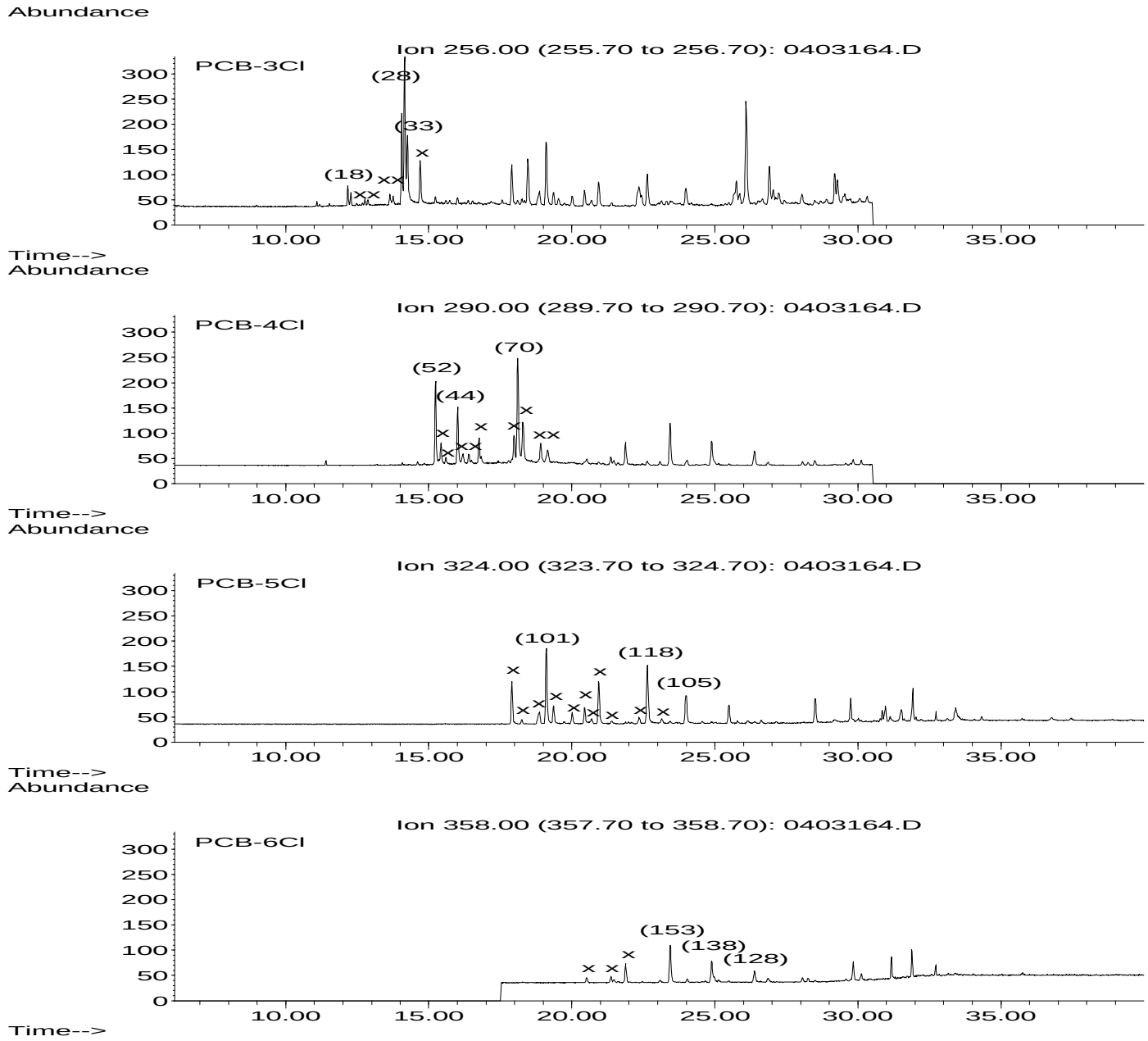


Figure 3 GC-MS Chromatographic Profile of PCBs in Train Sample

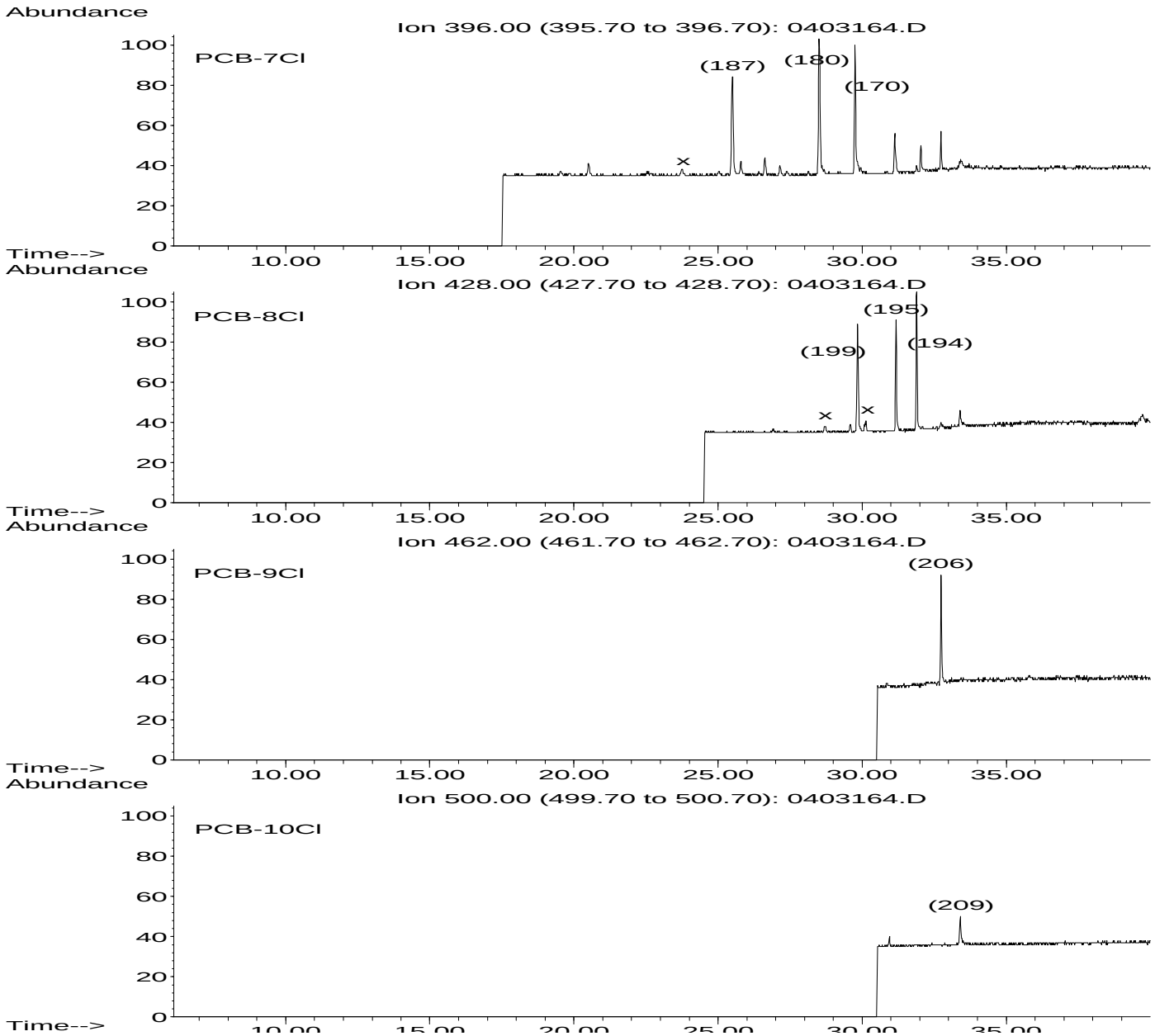


Figure 3 GC-MS Chromatographic Profile of PCBs in Train Sample

Table 1 PCB Spiked for Train and Ash Samples

PCB Congener IUPAC No.	Chlorine Positions	Spiked Level ng	Total PCB Analysis	TEQ Analysis	TEF
PCB-18	2,2',5	2	v		
PCB-28	2,4,4'	2	v		
PCB-33	2',3,4	2	v		
PCB-44	2,2',3,5'	2	v		
PCB-52	2,2',5,5'	2	v		
PCB-70	2,3',4',5	2	v		
PCB-77	3,3',4,4'	0.5		v	0.0001
PCB-81	3,4,4',5	0.5		v	0.0001
PCB-101	2,2',4,5,5'	2	v		
PCB-105*	2,3,3',4,4'	2.5	v	v	0.0001
PCB-114	2,3,4,4',5	0.5		v	0.0005
PCB-118*	2,3',4,4'.5	2.5	v	v	0.0001
PCB-123	2',3,4,4',5	0.5		v	0.0001
PCB-126	3,3',4,4',5	0.5		v	0.1
PCB-128	2,2',3,3'4,4'	2	v		
PCB-138	2,2',3,4,4',5’	2	v		
PCB-153	2,2',4,4',5,5'	2	v		
PCB-156	2,3,3',4,4',5	0.5		v	0.0005
PCB-157	2,3,3',4,4',5'	0.5		v	0.0005
PCB-167	2,3',4,4',5,5'	0.5		v	0.00001
PCB-169	3,3',4,4',5,5'	0.5		v	0.01
PCB-170	2,2',3,3',4,4',5	2	v		
PCB-180	2,2',3,4,4',5,5'	2	v		
PCB-187	2,2',3,4',5,5',6	2	v		
PCB-189	2,3,3',4,4',5,5'	0.5		v	0.0001
PCB-194	2,2',3,3',4,4',5,5'	2	v		
PCB-195	2,2',3,3',4,4',5,6	2	v		
PCB-199	2,2',3,3',4,5,5’,6'	2	v		
PCB-206	2,2',3,3',4,4',5,5',6	2	v		
PCB-209	2,2',3,3',4,4',5,5',6,6'	2	v		

** congener was spiked for both methods; TEF = toxic equivalency factor (Reference 10)*

Table 2 Gas Volume, Stack Temperature, and Concentration of Oxygen and Carbon Dioxide

Run #	Date	Time	Volume Sampled (m^3)	Average Stack Temp. (°C)	O_2 (%)	CO_2 (%)
1	March 22/2000	10:45-13:45	3.776	958	9.65	10.08
2	March 23/2000	10:30-13:30	3.799	990	11.08	8.8
3	March 24/2000	10:02-13:02	3.764	1016	9.45	9.7
4	March 27/2000	10:21-13:21	3.726	1001	10.58	8.58
5	March 29/2000	12:45-15:45	3.705	1007	10.15	8.4
6	March 30/2000	10:14-13:14	3.741	1004	9.54	10.46
7	March 31/2000	9:52-12:52	3.718	998	10.37	9.67
8	April 3/2000	10:12-13:12	3.754	1016	9.25	10.45
9	April 4/2000	10:00-13:00	3.633	1013	10.1	9.7
10	April 5/2000	10:25-13:25	3.733	1001	10.2	8.53
11	April 6/2000	9:55-12:55	3.662	1004	9.65	9.65
12	April 7/2000	10:13-13:13	3.607	889	11.1	9.26

Table 3 PCBs in Train Samples, ng/train

Sample ID	531	532	541	542	548	549	591	592	002	007	011	012	Mean ng	s ng	RSD %	s_i^2	df_i	$df_i \times s_i^2$
3 Cl:																		
PCB-18	1.2	3.4	4.8	4.4	2.9	2.2	3.1	3.3	3.3	3.9	3.6	2.6	3.23	0.96	30	0.924	11	10.16
PCB-28	11.1	7.1	9.5	9.4	7.3	6.6	7.1	9.3	8.6	8.0	9.2	5.6	8.23	1.55	19	2.408	11	26.49
PCB-33	6.1	6.2	7.3	5.6	6.8	4.7	7.4	5.5	6.3	6.7	8.4	5.0	6.33	1.06	17	1.132	11	12.45
4 Cl:																		
PCB-52	8.7	5.7	7.4	7.0	4.1	3.5	4.6	5.9	4.6	5.2	4.7	4.3	5.48	1.54	28	2.368	11	26.04
PCB-44	6.3	3.6	5.0	4.9	3.6	3.1	4.1	4.4	5.1	5.1	4.5	3.2	4.41	0.94	21	0.883	11	9.71
PCB-70	9.6	7.2	8.0	6.3	7.8	7.5	7.6	6.6	8.0	8.3	8.3	6.1	7.61	0.97	13	0.950	11	10.45
5 Cl:																		
PCB-101	5.9	4.5	5.7	4.4	4.0	3.1	3.9	3.5	3.8	4.9	5.0	4.8	4.46	0.85	19	0.723	11	7.95
PCB-118	5.5	4.6	4.9	4.2	5.1	3.8	4.5	3.8	4.6	4.1	4.5	3.8	4.45	0.54	12	0.294	11	3.23
PCB-105	3.0	2.9	3.2	2.9	3.2	2.5	2.8	2.8	3.3	3.0	2.8	2.7	2.93	0.23	8	0.053	11	0.58
6 Cl:																		
PCB-153	5.8	4.8	5.3	4.2	5.2	3.7	5.2	3.9	4.4	5.1	5.1	3.6	4.69	0.71	15	0.508	11	5.59
PCB-138	4.4	5.1	3.8	3.0	3.6	2.9	4.1	3.2	3.5	3.7	3.7	3.2	3.68	0.62	17	0.391	11	4.30
PCB-128	3.1	2.9	2.7	2.8	2.5	1.9	2.6	2.0	2.6	2.3	2.4	2.0	2.48	0.38	15	0.143	11	1.58
7 Cl:																		
PCB-187	2.2	2.1	2.4	2.0	2.2	1.6	2.2	1.7	2.0	2.0	2.2	1.7	2.03	0.25	12	0.060	11	0.66
PCB-180	2.9	3.1	2.3	2.4	2.3	2.0	2.6	2.0	2.5	2.2	2.5	1.9	2.39	0.36	15	0.130	11	1.43
PCB-170	2.7	3.1	2.9	2.3	2.4	1.8	2.4	1.9	2.4	2.2	2.2	2.0	2.36	0.39	17	0.152	11	1.67
8 Cl:																		
PCB-199	2.5	2.7	2.7	2.6	2.5	1.9	2.5	2.3	2.8	2.2	2.4	2.1	2.43	0.27	11	0.072	11	0.79
PCB-195	2.5	2.8	2.2	2.1	2.3	1.9	2.2	2.0	2.5	2.2	2.1	2.2	2.25	0.25	11	0.061	11	0.67
PCB-194	3.1	3.5	3.2	2.4	2.7	2.4	2.5	2.8	2.8	2.9	2.7	2.9	2.83	0.33	12	0.108	11	1.18
9 Cl:																		
PCB-206	2.1	2.3	2.5	2.2	2.3	2.2	2.3	2.2	2.5	2.2	2.3	2.3	2.28	0.12	5	0.014	11	0.16
10 Cl:																		
PCB-209	2.3	2.4	2.5	2.2	2.3	2.0	2.3	2.1	2.7	2.1	2.3	2.0	2.27	0.21	9	0.042	11	0.47
																SUM	220	126
																s_p =	**0.755**	
% Recovery of $^{13}C_{12}$-Surrogates:																		
PCB-28	54	46	58	49	43	38	41	43	42	39	41	41						
PCB-52	56	49	58	51	56	43	46	45	46	42	45	42						
PCB-118	74	72	78	73	73	73	75	69	72	66	74	70						
PCB-153	69	67	73	68	67	68	70	64	67	63	69	65						
PCB-180	65	63	68	64	64	66	67	62	64	60	66	63						
PCB-194	67	65	70	66	67	68	68	64	66	61	68	64						
PCB-206	65	62	67	63	64	65	66	61	64	59	66	61						
PCB-209	62	57	64	61	62	63	63	58	61	56	64	60						

Table 3A PCBs in Train Samples, ng/m^3

Sample ID	531	532	541	542	548	549	591	592	002	007	011	012	Mean	s	RSD	s_i^2	df_i	$df_i \times s_i^2$
Sample size	3.776	3.799	3.764	3.726	3.705	3.741	3.718	3.754	3.633	3.733	3.662	3.607	ng/m^3	ng/m^3	%			
3 Cl:																		
PCB-18	0.32	0.89	1.28	1.18	0.78	0.59	0.83	0.88	0.91	1.04	0.98	0.72	0.867	0.256	30	0.066	11	0.722
PCB-28	2.94	1.87	2.52	2.52	1.97	1.76	1.91	2.48	2.37	2.14	2.51	1.55	2.213	0.406	18	0.165	11	1.816
PCB-33	1.62	1.63	1.94	1.50	1.84	1.26	1.99	1.47	1.73	1.79	2.29	1.39	1.704	0.290	17	0.084	11	0.924
4 Cl:																		
PCB-52	2.30	1.50	1.97	1.88	1.11	0.94	1.24	1.57	1.27	1.39	1.28	1.19	1.470	0.399	27	0.159	11	1.751
PCB-44	1.67	0.95	1.33	1.32	0.97	0.83	1.10	1.17	1.40	1.37	1.23	0.89	1.185	0.248	21	0.062	11	0.678
PCB-70	2.54	1.90	2.13	1.69	2.11	2.00	2.04	1.76	2.20	2.22	2.27	1.69	2.046	0.256	13	0.065	11	0.720
5 Cl:																		
PCB-101	1.56	1.18	1.51	1.18	1.08	0.83	1.05	0.93	1.05	1.31	1.37	1.33	1.199	0.226	19	0.051	11	0.560
PCB-118	1.46	1.21	1.30	1.13	1.38	1.02	1.21	1.01	1.27	1.10	1.23	1.05	1.197	0.141	12	0.020	11	0.219
PCB-105	0.79	0.76	0.85	0.78	0.86	0.67	0.75	0.75	0.91	0.80	0.76	0.75	0.787	0.064	8	0.004	11	0.044
6 Cl:																		
PCB-153	1.54	1.26	1.41	1.13	1.40	0.99	1.40	1.04	1.21	1.37	1.39	1.00	1.261	0.185	15	0.034	11	0.378
PCB-138	1.17	1.34	1.01	0.81	0.97	0.78	1.10	0.85	0.96	0.99	1.01	0.89	0.990	0.159	16	0.025	11	0.279
PCB-128	0.82	0.76	0.72	0.75	0.67	0.51	0.70	0.53	0.72	0.62	0.66	0.55	0.667	0.098	15	0.010	11	0.105
7 Cl:																		
PCB-187	0.58	0.55	0.64	0.54	0.59	0.43	0.59	0.45	0.55	0.54	0.60	0.47	0.545	0.064	12	0.004	11	0.046
PCB-180	0.77	0.82	0.61	0.64	0.62	0.53	0.70	0.53	0.69	0.59	0.68	0.53	0.643	0.093	14	0.009	11	0.094
PCB-170	0.72	0.82	0.77	0.62	0.65	0.48	0.65	0.51	0.66	0.59	0.60	0.55	0.634	0.099	16	0.010	11	0.109
8 Cl:								0.00										
PCB-199	0.66	0.71	0.72	0.70	0.67	0.51	0.67	0.61	0.77	0.59	0.66	0.58	0.654	0.071	11	0.005	11	0.056
PCB-195	0.66	0.74	0.58	0.56	0.62	0.51	0.59	0.53	0.69	0.59	0.57	0.61	0.605	0.065	11	0.004	11	0.046
PCB-194	0.82	0.92	0.85	0.64	0.73	0.64	0.67	0.75	0.77	0.78	0.74	0.75	0.755	0.083	11	0.007	11	0.075
9 Cl:																		
PCB-206	0.56	0.61	0.66	0.59	0.62	0.59	0.62	0.59	0.69	0.59	0.63	0.64	0.614	0.037	6	0.001	11	0.015
10 Cl:																		
PCB-209	0.61	0.63	0.66	0.59	0.62	0.53	0.62	0.56	0.74	0.56	0.63	0.55	0.610	0.057	9	0.003	11	0.036
																SUM	220	8.672
																s_p =	**0.199**	

% Recovery of $^{13}C_{12}$-Surrogates:

PCB-28	54	46	58	49	43	38	41	43	42	39	41	41
PCB-52	56	49	58	51	56	43	46	45	46	42	45	42
PCB-118	74	72	78	73	73	73	75	69	72	66	74	70
PCB-153	69	67	73	68	67	68	70	64	67	63	69	65
PCB-180	65	63	68	64	64	66	67	62	64	60	66	63
PCB-194	67	65	70	66	67	68	68	64	66	61	68	64
PCB-206	65	62	67	63	64	65	66	61	64	59	66	61
PCB-209	62	57	64	61	62	63	63	58	61	56	64	60

Table 4 PCBs in Ash Samples, ng/sample

Sample ID	316	317	318	319	320	321	322	323	324	325	Mean	s	RSD	s_i^2	df_i	$df_i \times s_i^2$
Sample size	5.01	4.99	4.98	5.00	5.00	4.99	4.99	5.01	5.02	5.03	ng	ng	%			
3 Cl:																
PCB-18	2.079	1.861	1.733	3.210	1.585	1.906	1.981	2.129	1.908	2.334	2.073	0.450	22	0.2029	9	1.826
PCB-28	2.675	2.475	2.565	4.390	4.390	3.268	3.169	3.412	2.425	4.371	3.314	0.812	24	0.6589	9	5.930
PCB-33	3.236	2.989	2.923	5.315	5.315	3.608	3.623	3.467	3.087	3.964	3.753	0.884	24	0.7807	9	7.026
4 Cl:																
PCB-52	2.425	2.685	2.455	3.855	2.585	2.695	2.899	2.881	2.696	3.028	2.820	0.411	15	0.1687	9	1.518
PCB-44	1.769	2.041	2.012	3.120	2.025	2.061	2.186	1.999	1.928	2.284	2.142	0.370	17	0.1369	9	1.232
PCB-70	3.277	3.533	3.725	4.775	3.625	3.802	4.012	4.098	3.660	4.361	3.887	0.438	11	0.1917	9	1.725
5 Cl:																
PCB-101	1.909	2.011	2.908	3.425	1.955	2.280	2.340	2.791	2.189	2.445	2.425	0.484	20	0.2345	9	2.110
PCB-118	3.231	3.159	3.874	3.690	3.175	3.308	3.353	3.617	3.188	3.466	3.406	0.248	7	0.0615	9	0.553
PCB-105	2.540	2.515	2.759	2.890	2.595	2.730	2.660	2.771	2.686	2.651	2.680	0.114	4	0.0129	9	0.116
6 Cl:																
PCB-153	2.610	2.695	4.333	3.585	2.660	2.834	2.814	3.557	2.686	2.978	3.075	0.567	18	0.3215	9	2.894
PCB-138	2.204	2.236	3.257	2.735	2.320	2.365	2.445	2.851	2.334	2.374	2.512	0.334	13	0.1115	9	1.004
PCB-128	1.944	1.851	1.972	1.900	1.890	1.961	1.941	1.939	2.003	2.118	1.952	0.073	4	0.0053	9	0.048
7 Cl:																
PCB-187	1.398	1.422	1.678	1.535	1.440	1.537	1.442	1.553	1.466	1.584	1.506	0.087	6	0.0076	9	0.069
PCB-180	1.613	1.687	1.907	1.730	1.605	1.717	1.687	1.738	1.647	1.801	1.713	0.091	5	0.0082	9	0.074
PCB-170	1.794	1.876	1.942	2.280	1.825	2.091	2.141	1.919	1.842	2.113	1.982	0.163	8	0.0266	9	0.239
8 Cl:																
PCB-199	2.024	2.216	2.146	2.190	2.165	2.226	2.290	2.189	2.169	2.168	2.178	0.068	3	0.0046	9	0.042
PCB-195	2.019	2.066	1.987	2.055	2.060	2.106	2.236	1.909	2.169	1.987	2.059	0.094	5	0.0089	9	0.080
PCB-194	2.410	2.480	2.505	2.580	2.615	2.495	2.555	2.420	2.354	2.455	2.487	0.081	3	0.0066	9	0.059
9 Cl:																
PCB-206	2.234	2.176	2.221	2.350	2.125	2.260	2.265	2.239	2.128	2.188	2.219	0.068	3	0.0046	9	0.042
10 Cl:																
PCB-209	2.134	2.265	2.221	2.170	2.420	2.176	2.126	2.234	2.179	2.455	2.238	0.114	5	0.0129	9	0.117
														SUM	180	26.703
														s_p =	**0.385**	

% Recovery of $^{13}C_{12}$-Surrogates:

PCB-28	71	61	76	75	54	74	65	73	74	66
PCB-52	72	67	80	82	62	79	71	80	78	71
PCB-118	102	100	102	107	97	107	103	109	106	104
PCB-153	102	99	100	104	97	105	103	106	105	104
PCB-180	136	131	128	134	126	136	136	137	133	133
PCB-194	106	101	100	103	98	106	106	108	104	105
PCB-206	104	99	97	100	96	102	103	105	101	102
PCB-209	101	95	94	96	92	99	99	100	97	99

Table 4A PCBs in Ash Samples, ng/g

Sample ID	316	317	318	319	320	321	322	323	324	325	Mean	s	RSD	s_i^2	df_i	$df_i \times s_i^2$
Sample size	5.01	4.99	4.98	5.00	5.00	4.99	4.99	5.01	5.02	5.03	ng/g	ng/g	%			
3 Cl:																
PCB-18	0.415	0.373	0.348	0.642	0.317	0.382	0.397	0.425	0.38	0.464	0.414	0.090	22	0.0081	9	0.073
PCB-28	0.534	0.496	0.515	0.878	0.878	0.655	0.635	0.681	0.483	0.869	0.662	0.162	24	0.0261	9	0.235
PCB-33	0.646	0.599	0.587	1.063	1.063	0.723	0.726	0.692	0.615	0.788	0.750	0.177	24	0.0312	9	0.280
4 Cl:																
PCB-52	0.484	0.538	0.493	0.771	0.517	0.540	0.581	0.575	0.537	0.602	0.564	0.082	15	0.0067	9	0.060
PCB-44	0.353	0.409	0.404	0.624	0.405	0.413	0.438	0.399	0.384	0.454	0.428	0.074	17	0.0055	9	0.049
PCB-70	0.654	0.708	0.748	0.955	0.725	0.762	0.804	0.818	0.729	0.867	0.777	0.087	11	0.0076	9	0.068
5 Cl:																
PCB-101	0.381	0.403	0.584	0.685	0.391	0.457	0.469	0.557	0.436	0.486	0.485	0.097	20	0.0094	9	0.085
PCB-118	0.645	0.633	0.778	0.738	0.635	0.663	0.672	0.722	0.635	0.689	0.681	0.050	7	0.0025	9	0.023
PCB-105	0.507	0.504	0.554	0.578	0.519	0.547	0.533	0.553	0.535	0.527	0.536	0.023	4	0.0005	9	0.005
6 Cl:																
PCB-153	0.521	0.540	0.870	0.717	0.532	0.568	0.564	0.710	0.535	0.592	0.615	0.114	19	0.0131	9	0.118
PCB-138	0.440	0.448	0.654	0.547	0.464	0.474	0.490	0.569	0.465	0.472	0.502	0.068	13	0.0046	9	0.041
PCB-128	0.388	0.371	0.396	0.380	0.378	0.393	0.389	0.387	0.399	0.421	0.390	0.014	4	0.0002	9	0.002
7 Cl:																
PCB-187	0.279	0.285	0.337	0.307	0.288	0.308	0.289	0.310	0.292	0.315	0.301	0.018	6	0.0003	9	0.003
PCB-180	0.322	0.338	0.383	0.346	0.321	0.344	0.338	0.347	0.328	0.358	0.343	0.018	5	0.0003	9	0.003
PCB-170	0.358	0.376	0.390	0.456	0.365	0.419	0.429	0.383	0.367	0.420	0.396	0.033	8	0.0011	9	0.010
8 Cl:																
PCB-199	0.404	0.444	0.431	0.438	0.433	0.446	0.459	0.437	0.432	0.431	0.436	0.014	3	0.0002	9	0.002
PCB-195	0.403	0.414	0.399	0.411	0.412	0.422	0.448	0.381	0.432	0.395	0.412	0.019	5	0.0004	9	0.003
PCB-194	0.481	0.497	0.503	0.516	0.523	0.500	0.512	0.483	0.469	0.488	0.497	0.017	3	0.0003	9	0.003
9 Cl:																
PCB-206	0.446	0.436	0.446	0.470	0.425	0.453	0.454	0.447	0.424	0.435	0.444	0.014	3	0.0002	9	0.002
10 Cl:																
PCB-209	0.426	0.454	0.446	0.434	0.484	0.436	0.426	0.446	0.434	0.488	0.447	0.022	5	0.0005	9	0.004
														SUM	180	1.068
														s_p =	**0.077**	

% Recovery of $^{13}C_{12}$-Surrogates:

PCB-28	71	61	76	75	54	74	65	73	74	66
PCB-52	72	67	80	82	62	79	71	80	78	71
PCB-118	102	100	102	107	97	107	103	109	106	104
PCB-153	102	99	100	104	97	105	103	106	105	104
PCB-180	136	131	128	134	126	136	136	137	133	133
PCB-194	106	101	100	103	98	106	106	108	104	105
PCB-206	104	99	97	100	96	102	103	105	101	102
PCB-209	101	95	94	96	92	99	99	100	97	99

Table 5 Dioxin-Like PCBs in Train Samples, ng/train

Sample ID	TEF	531	532	541	542	548	549	591	592	002	007	011	012	Mean ng	s ng	RSD %
PCB-81	0.0001	0.459	0.451	0.444	0.435	0.439	0.335	0.422	0.435	0.549	0.388	0.463	0.300	0.427	0.064	15
PCB-77	0.0001	0.836	0.749	0.755	0.761	0.775	0.659	0.702	0.724	0.797	0.679	0.702	0.460	0.717	0.095	13
PCB-123	0.0001	0.644	0.561	0.636	0.564	0.631	0.464	0.590	0.518	0.661	0.570	0.626	0.430	0.575	0.073	13
PCB-118	0.0001	5.205	4.265	4.755	4.046	4.651	3.579	3.932	3.552	4.435	3.948	4.232	3.369	4.164	0.541	13
PCB-114	0.0005	0.527	0.473	0.496	0.500	0.484	0.373	0.447	0.936	0.587	0.447	0.479	0.296	0.504	0.154	31
PCB-105	0.0001	3.092	2.831	3.005	2.755	3.025	2.415	2.646	5.169	3.289	2.580	2.751	2.031	2.966	0.770	26
PCB-126	0.1	0.521	0.507	0.486	0.471	0.496	0.435	0.537	0.441	0.558	0.442	0.507	0.377	0.482	0.051	11
PCB-167	0.00001	0.533	0.548	0.557	0.527	0.537	0.457	0.538	0.958	0.618	0.437	0.541	0.394	0.554	0.141	25
PCB-156	0.0005	0.614	0.619	0.526	0.651	0.609	0.544	0.674	0.509	0.606	0.543	0.534	0.444	0.573	0.067	12
PCB-157	0.0005	0.456	0.479	0.479	0.450	0.474	0.414	0.459	0.434	0.533	0.428	0.459	0.365	0.453	0.041	9
PCB-169	0.01	0.601	0.532	0.497	0.509	0.570	0.445	0.535	0.445	0.581	0.463	0.528	0.392	0.508	0.063	12
PCB-189	0.0001	0.457	0.501	0.468	0.450	0.478	0.400	0.479	0.418	0.565	0.443	0.470	0.348	0.456	0.054	12
PCB TEQ		**0.0599**	**0.0577**	**0.0553**	**0.0539**	**0.0571**	**0.0494**	**0.0607**	**0.0506**	**0.0635**	**0.0504**	**0.0577**	**0.0429**	**0.0549**	**0.0058**	**11**

% Recovery of $^{13}C_{12}$-Surrogate:

PCB-77	86	85	85	92	86	85	83	84	88	86	82	69
PCB-126	87	89	85	93	89	88	86	88	91	87	85	74
PCB-156	90	92	89	96	92	93	90	93	96	90	90	77
PCB-169	86	88	84	91	88	87	85	88	91	86	85	73
PCB-180	94	91	92	96	92	94	92	92	95	89	92	78

Table 5A Dioxin-Like PCBs in Train Samples, ng/m3

Sample ID		531	532	541	542	548	549	591	592	002	007	011	012	Mean	s	RSD
Results units		3.776	3.779	3.764	3.726	3.705	3.741	3.718	3.754	3.633	3.733	3.662	3.607	ng/m^3	ng/m^3	%
	TEF															
PCB-81	0.0001	0.122	0.119	0.118	0.117	0.118	0.089	0.113	0.116	0.151	0.104	0.127	0.083	0.115	0.017	15
PCB-77	0.0001	0.221	0.198	0.201	0.204	0.209	0.176	0.189	0.193	0.219	0.182	0.192	0.128	0.193	0.025	13
PCB-123	0.0001	0.171	0.148	0.169	0.151	0.170	0.124	0.159	0.138	0.182	0.153	0.171	0.119	0.155	0.020	13
PCB-118	0.0001	1.378	1.129	1.263	1.086	1.255	0.957	1.058	0.946	1.221	1.057	1.156	0.934	1.120	0.141	13
PCB-114	0.0005	0.140	0.125	0.132	0.134	0.131	0.100	0.120	0.249	0.162	0.120	0.131	0.082	0.135	0.041	30
PCB-105	0.0001	0.819	0.749	0.798	0.739	0.817	0.646	0.712	1.377	0.905	0.691	0.751	0.563	0.797	0.203	25
PCB-126	0.1	0.138	0.134	0.129	0.126	0.134	0.116	0.144	0.118	0.154	0.118	0.139	0.105	0.130	0.014	11
PCB-167	0.00001	0.141	0.145	0.148	0.141	0.145	0.122	0.145	0.255	0.170	0.117	0.148	0.109	0.149	0.037	25
PCB-156	0.0005	0.162	0.164	0.140	0.175	0.165	0.145	0.181	0.135	0.167	0.145	0.146	0.123	0.154	0.017	11
PCB-157	0.0005	0.121	0.127	0.127	0.121	0.128	0.111	0.123	0.116	0.147	0.115	0.125	0.101	0.122	0.011	9
PCB-169	0.01	0.159	0.141	0.132	0.137	0.154	0.119	0.144	0.118	0.160	0.124	0.144	0.109	0.137	0.017	12
PCB-189	0.0001	0.121	0.133	0.124	0.121	0.129	0.107	0.129	0.111	0.155	0.119	0.128	0.097	0.123	0.015	12
PCB TEQ		**0.0159**	**0.0153**	**0.0147**	**0.0145**	**0.0154**	**0.0132**	**0.0163**	**0.0135**	**0.0175**	**0.0135**	**0.0158**	**0.0119**	**0.0148**	**0.0016**	**11**

% Recovery of $^{13}C_{12}$-Surrogate:

PCB-77	86	85	85	92	86	85	83	84	88	86	82	69
PCB-126	87	89	85	93	89	88	86	88	91	87	85	74
PCB-156	90	92	89	96	92	93	90	93	96	90	90	77
PCB-169	86	88	84	91	88	87	85	88	91	86	85	73
PCB-180	94	91	92	96	92	94	92	92	95	89	92	78

Table 6 Dioxin-Like PCBs in Ash Sample, ng/sample

Sample ID		316	317	318	319	320	321	322	323	324	325	Mean	s	RSD
Sample size		5.01	4.99	4.98	5.00	5.00	4.99	4.99	5.01	5.02	5.03	ng	ng	%
	TEF													
PCB-81	0.0001	0.357	0.354	0.433	0.369	0.402	0.368	0.320	0.372	0.393	0.407	0.377	0.032	9
PCB-77	0.0001	0.409	0.450	0.560	0.479	0.476	0.475	0.395	0.462	0.510	0.518	0.473	0.049	10
PCB-123	0.0001	0.369	0.355	0.493	0.441	0.392	0.411	0.321	0.386	0.424	0.414	0.401	0.048	12
PCB-118	0.0001	1.867	2.092	3.004	2.503	2.156	2.421	2.065	2.443	2.613	2.322	2.348	0.326	14
PCB-114	0.0005	0.321	0.336	0.413	0.343	0.360	0.360	0.303	0.317	0.382	0.369	0.351	0.033	9
PCB-105	0.0001	1.694	1.814	2.332	1.914	1.846	2.044	1.728	1.898	2.147	1.977	1.939	0.195	10
PCB-126	0.1	0.436	0.454	0.537	0.459	0.466	0.522	0.404	0.432	0.532	0.484	0.473	0.045	10
PCB-167	0.00001	0.386	0.399	0.516	0.406	0.429	0.431	0.378	0.417	0.490	0.418	0.427	0.044	10
PCB-156	0.0005	0.393	0.400	0.494	0.419	0.402	0.432	0.391	0.446	0.475	0.458	0.431	0.036	8
PCB-157	0.0005	0.334	0.367	0.423	0.362	0.370	0.395	0.320	0.356	0.410	0.403	0.374	0.033	9
PCB-169	0.01	0.367	0.402	0.483	0.417	0.425	0.430	0.360	0.389	0.479	0.417	0.417	0.041	10
PCB-189	0.0001	0.427	0.442	0.525	0.455	0.456	0.442	0.421	0.421	0.499	0.563	0.465	0.048	10
PCB TEQ		**0.0483**	**0.0505**	**0.0600**	**0.0512**	**0.0520**	**0.0577**	**0.0450**	**0.0483**	**0.0593**	**0.0538**	**0.0526**	**0.0050**	**10**

% Recovery of $^{13}C_{12}$-Surrogate:

PCB-77	67	76	90	79	79	75	68	75	88	81
PCB-126	69	76	91	78	78	77	69	73	92	80
PCB-156	67	77	90	77	79	83	76	80	96	84
PCB-169	65	75	89	74	76	72	67	70	90	74
PCB-180	66	75	87	80	77	87	75	78	97	78

Table 6A Dioxin-Like PCBs in Ash Samples, ng/g

Sample ID		316	317	318	319	320	321	322	323	324	325	Mean	s	RSD
Sample size		5.01	4.99	4.98	5.00	5.00	4.99	4.99	5.01	5.02	5.03	ng/g	ng/g	%
	TEF													
PCB-81	0.0001	0.071	0.071	0.087	0.074	0.080	0.074	0.064	0.074	0.078	0.081	0.075	0.006	8
PCB-77	0.0001	0.082	0.090	0.112	0.096	0.095	0.095	0.079	0.092	0.102	0.103	0.095	0.010	10
PCB-123	0.0001	0.074	0.071	0.099	0.088	0.078	0.082	0.064	0.077	0.085	0.082	0.080	0.010	12
PCB-118	0.0001	0.373	0.419	0.603	0.501	0.431	0.485	0.414	0.488	0.520	0.462	0.470	0.065	14
PCB-114	0.0005	0.064	0.067	0.083	0.069	0.072	0.072	0.061	0.063	0.076	0.073	0.070	0.007	10
PCB-105	0.0001	0.338	0.364	0.468	0.383	0.369	0.410	0.346	0.379	0.428	0.393	0.388	0.039	10
PCB-126	0.1	0.087	0.091	0.108	0.092	0.093	0.105	0.081	0.086	0.106	0.096	0.094	0.009	10
PCB-167	0.00001	0.077	0.080	0.104	0.081	0.086	0.086	0.076	0.083	0.098	0.083	0.085	0.009	10
PCB-156	0.0005	0.078	0.080	0.099	0.084	0.080	0.087	0.078	0.089	0.095	0.091	0.086	0.007	8
PCB-157	0.0005	0.067	0.074	0.085	0.072	0.074	0.079	0.064	0.071	0.082	0.080	0.075	0.007	9
PCB-169	0.01	0.073	0.081	0.097	0.083	0.085	0.086	0.072	0.078	0.095	0.083	0.083	0.008	10
PCB-189	0.0001	0.085	0.089	0.105	0.091	0.091	0.089	0.084	0.084	0.099	0.112	0.093	0.010	10
PCB TEQ		**0.0096**	**0.0101**	**0.0120**	**0.0102**	**0.0104**	**0.0116**	**0.0090**	**0.0096**	**0.0118**	**0.0107**	**0.0105**	**0.0010**	**10**

% Recovery of $^{13}C_{12}$-Surrogate:

PCB-77	67	76	90	79	79	75	68	75	88	81
PCB-126	69	76	91	78	78	77	69	73	92	80
PCB-156	67	77	90	77	79	83	76	80	96	84
PCB-169	65	75	89	74	76	72	67	70	90	74
PCB-180	66	75	87	80	77	87	75	78	97	78

Table 7 PCB Distribution in Front-Half and Back-Half Train Samples

Sample ID	011			012		
Sample type	Front-Half	Back-Half		Front-Half	Back-Half	
Results units	ng	ng	Other PCBs	ng	ng	Other PCBs
Trichloro			nd			1.5 (1)
PCB-18	nd	3.6		nd	2.6	
PCB-28	nd	9.2		nd	5.6	
PCB-33	nd	8.4		nd	5.0	
Tetrachloro			5.3 (2)			3.8 (2)
PCB-52	nd	4.7		nd	4.3	
PCB-44	nd	4.5		nd	3.2	
PCB-70	nd	8.3		nd	6.1	
Pentachloro			6.9 (5)			6.8 (5)
PCB-101	0.5	4.5		0.7	4.1	
PCB-118	nd	4.5		nd	3.8	
PCB-105	nd	2.8		nd	2.7	
Hexachloro			6.3 (5)			3.3 (4)
PCB-153	nd	5.1		nd	3.6	
PCB-138	nd	3.7		nd	3.2	
PCB-128	nd	2.4		nd	2.0	
Heptachloro			4.1 (4)			2.8 (2)
PCB-187	nd	2.2		nd	1.7	
PCB-180	nd	2.5		nd	1.9	
PCB-170	nd	2.2		nd	2.0	
Octachloro			0.4 (1)			0.4 (1)
PCB-199	nd	2.4		nd	2.1	
PCB-195	nd	2.1		nd	2.2	
PCB-194	0.3	2.4		0.5	2.4	
Nonachloro			nd			nd
PCB-206	nd	2.3		nd	2.3	
Decachloro						
PCB-209	nd	2.3		nd	2.0	
Total	0.8	80.1		1.1	62.8	

nd = not detected or below the detection limit 0.2-0.3 ng; value in bracket is the number of other isomers detected

Table 8 Summary of PCBs in Train and Ash Samples

Sample-Method	Concentration	Number of Replicates	Mean Concentration	s	RSD, %	s_p
Train-congener	ng/train	12	2.0-7.6	0.12-1.55	5-30	0.755
Train-congener	ng/m^3	12	0.61-2.21	0.037-0.41	6-30	0.199
Ash-congener	ng/sample	10	1.71-3.89	0.068-0.884	3-24	0.385
Ash-congener	ng/g	10	0.3-0.78	0.01-0.18	3-24	0.077
Train-TEQ	ng/train	12	0.0549	0.0058	11	
Train-TEQ	ng/m^3	12	0.0148	0.0016	11	
Ash-TEQ	ng/sample	10	0.0526	0.005	10	
Ash-TEQ	ng/g	10	0.0105	0.0010	10	

s = standard deviation; s_p = pooled standard deviation