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Project Summary

Evaluation of PCB Destruction Efficiency in an Industrial Boiler

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According to EPA's final ruling on the disposal of polychlorinated biphenyls (PCBs), waste oils which contain PCBs in the range 50-500 ppm can be fired with fuel oil and burned in a high efficiency industrial boiler. In May 1980, waste oil containing approximately 500 ppm of PCBs was co-fired in accordance with applicable state and federal regulations. This combustion took place in a high efficiency industrial boiler owned and operated by General Motors Corporation at Bay City, MI. This report describes the evaluation program undertaken to determine the PCB destruction efficiency which occurred during the verification burn. Also investigated was the environmental and the workplace impact which occurs during the handling and combustion of PCB-contaminated waste oils.

No PCBs were detected in the stack gas within the detection limits of the sampling and analytical techniques used. The data collected during this verification burn indicate that, by following the equipment and performance requirements stated in EPA's final PCB rule (44, *Federal Register*, 31545, May 31, 1979), at least 99.9 percent destruction efficiency of PCBs can be achieved by high efficiency boilers. Furthermore, monitoring of the downwind ambient air, the workplace environment, and employee blood levels has indicated that PCBs can be destroyed with no measurable adverse effect on either the workplace environment or the external environment.

This Project Summary was developed by EPA's Industrial Environmental Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

Background

In May 1980, waste oil contaminated with polychlorinated biphenyl (PCB) was combusted by Chevrolet Motor Division of General Motors at Bay City, MI, in a high efficiency industrial boiler. The burn was conducted in conformance with the conditions established by the U.S. EPA for PCB destruction in high efficiency boilers (*44*, Federal Register, 31545, May 31, 1979). This final ruling on PCB disposal states that waste oil containing PCBs in the range of 50-500 ppm can be disposed of in a high efficiency boiler if:

- The boiler is rated at a minimum of 50 million Btu/hr.
- With the boiler using natural gas or oil as the primary fuel, the CO concentration in the stack is 50 ppm or less, and the excess oxygen is at least 3 percent when PCBs are being burned.

- The waste does not comprise more than 10 percent (on a volume basis) of the total fuel feed rate.
- The waste fluid is not fed into the boiler unless the boiler is operating at its normal operating temperature (this prohibits feeding these fluids during either start-up or shutdown).
- The owner or operator of the boiler continuously monitors and records the CO concentration and excess oxygen percentage in the stack gas while burning waste fluid.
- The primary fuel feed rate, waste fluid feed rate, and total quantities of both primary fuel and waste fluid fed to the boiler are measured and recorded at regular intervals of no longer than 15 minutes while burning waste fluid.
- The CO concentration and the excess oxygen percentage are checked at least once each hour that waste fluid is burned. (If either measurement falls below the levels specified in this rule, the flow of waste fluid to the boiler shall be stopped immediately.)

The PCB burn was monitored by GCA Corporation (under contract to the U.S. EPA), the Michigan Department of Natural Resources, and General Motors to ensure conduct of the burn in accordance with governmental guidelines and to document the PCB destruction efficiency of the burn.

Operating plans (test plans) were developed by GCA Corporation and General Motors Corporation to ensure definition and coordination of responsibilities during the burn and to ensure the use of acceptable sampling and analysis procedures during and subsequent to the burn. PCB levels in the waste oil feed and in the combustion exhaust gases were measured to establish the PCB destruction efficiency. Combustion parameters (CO, O_2) were monitored to evaluate the boiler performance and to establish compliance with federal regulations governing the burn. PCB levels were also measured in the ambient and in-plant air and in workers' bloodstreams before, during, and after the burn to evaluate the environmental effect of the burn.

This report documents the results and conclusions of the PCB burn at Bay City, MI, and summarizes the procedures used to acquire the data.

GCA Test Program

GCA conducted stack tests using an EPA Method 5 sampling train (*42*, Federal Register, August 18, 1977), modified specifically for capturing PCBs. It used a second modified train for organic compounds and chlorinated hydrocarbons which could be combustion byproducts. Simultaneously with the stack testing, GCA operated four ambient air monitors—one upwind and three downwind—to verify the presence or absence of significant PCB loadings to ambient air during the verification burn.

Three PCB tests were conducted with the boiler operating under normal load conditions. The usual boiler fuel, No. 6 fuel oil, was co-fired with 10 percent waste oil prepared to contain approximately 500 ppm PCB; hence, the PCB concentration in the fuel being burned was approximately 50 ppm. In order to provide background data on emissions from the boiler, 1 day of testing was also conducted with No. 6 fuel oil only.

General Motors Boiler Operation

The boiler that was used for the PCB burn was a Wickes Type "K" 65-4K-7 package boiler with a 60,000 lb/hr steam generation capacity at 8 gpm of No. 6 fuel oil. For the duration of the verification burn, continuous monitors were installed to measure CO and O_2 as prescribed by the federal regulations (*44*, Federal Register, 31545, May 31, 1979). Provision was made for shutdown of waste fuel feed unless the following criteria were met:

- Flame temperature—normal (~ 2500 °F).
- CO concentration—less than 50 ppm.
- Excess O_2—greater than 3 percent.

An additional requirement was for steady-state operation with 4 gpm minute fuel feed.

Figure 1 is a schematic of the plant layout, indicating the points at which samples were taken during the test program.

Results and Conclusions

PCB Destruction Efficiency

Table 1 shows that at least 99.99 percent destruction efficiency of PCBs was achieved during the test burn. This is significantly greater than the 99.9 percent destruction efficiency expected by EPA's PCB regulation. The destruction could have been greater because the limits were set by the detection sensitivity of the sampling and analytical techniques used. The range in PCB concentrations shown in Table 1 results from the uncertainties inherent in the analytical techniques used to measure PCBs. This is discussed in more detail below, under "Fuel Analysis."

No PCBs were detected in the stack gas within the detection limit of the instruments used. Stack emission rates of PCB are shown in Table 2. Calculation of the destruction efficiency of PCB in the GM industrial boiler is based on a mass balance into and out of the boiler during the verification burn.

Dibenzofuran/Dioxin Emissions

As previously mentioned, a second EPA Method 5 sampling train was used to measure other organic combustion products emitted during the PCB burn. Analyses for chlorinated dibenzofurans and dioxins were undertaken. This train was operated simultaneously with the PCB sampling train during two of three PCB burns and during the burning of No. 6 fuel oil. As with PCB stack samples, all analyses were performed at the GCA laboratory.

The results of the analysis of stack samples for dibenzofurans and dioxins are shown in Table 3. The data are shown as quantities extracted from the particulates and the XAD-2 resin (used to trap organic vapor) with a series of solvents. No dioxin or dibenzofuran was detected in these samples at the limits of detectability.

Ambient PCB Concentrations

The ambient monitoring network was designed to measure PCB concentrations in the ambient air on plant property and in the immediate populated area downwind of the stack during the test burns. Meteorological conditions on the date of each burn were used to position the monitors in areas on the plant property where maximum downwash from the stack effluent was predicted. In this way, PCB background levels, increases in concentrations of PCBs, and PCB concentration in the nearest populated area which could have been exposed to PCBs as a result of the test burn were measured.

Sampling involved high-volume samplers, modified with a series of two precleaned polyurethane foam plugs housed in the sampler throat made from a 16-cm long threaded aluminum tube.

Ambient sampling occurred on the same days as the stack sampling; i.e., 1 day was specified as a background burn (No. 6 fuel oil only), followed by 3 days of burning a combination of No. 6 fuel oil and PCB-contaminated waste oil. The

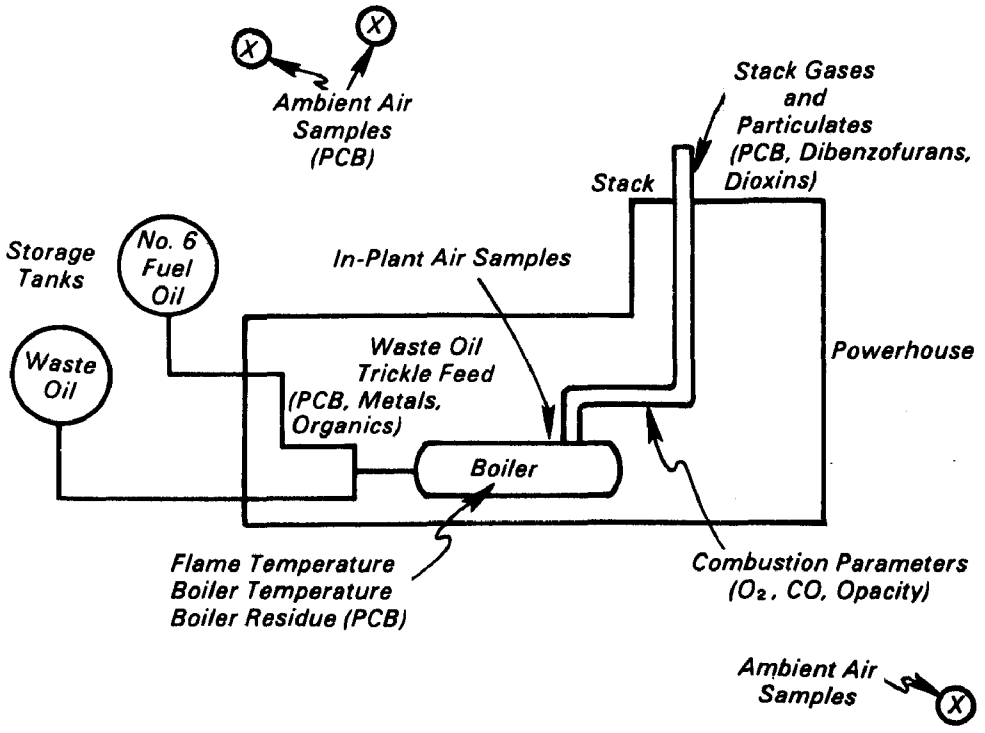

Figure 1. Schematic diagram of plant layout showing points where samples were taken.

Table 1. Destruction Efficiency of PCB

Run No.	PCB in Fuel[a] Concentration Range (mg/kg)	PCB[b] in (mg/min)	PCB out (mg/min)	PCB[c] Destruction Efficiency (%)
PCB-2	34-72	480-1000	$<5.8 \times 10^{-2}$	≥99.99[d]
PCB-3	34-76	480-1100	$<5.6 \times 10^{-2}$	≥99.99[d]
PCB-4	34-76	480-1100	$<5.6 \times 10^{-2}$	≥99.99[d]

Note: Density of 1:10 dilution of waste oil: No. 6 fuel oil is 3.4 kg/gal.
[a]*Based on GM and GCA reported results.*
[b]*Fuel combustion rate: 3.43 kg/gal. x 4 gal/min fuel flow = 14 kg/min.*
[c]*Percent destruction* $= 100 \left[\frac{\text{PCB in} - \text{PCB out}}{\text{PCB in}}\right]$
[d]*Assumes 100 percent sample collection efficiency. A validated test of sample collection efficiency was not conducted as part of this verification burn.*

monitor operation was synchronized with the collection of stack gas samples.

Four runs, each 3 hours long, comprised the 12-hour ambient monitoring sample period. The four runs were: a pre-burn, a post-burn, and two runs during the burning of waste oil. Testing was coordinated so that no waste oil would enter the boiler until the 3-hour pre-burn was complete. The post-burn was not started until a time lag, derived from prevailing wind velocity, sufficient to ensure that effluent from the stack would have passed by the monitors, had occurred.

Ambient samples were analyzed at the GCA laboratory. Results showed that ambient concentrations of PCBs at the downwind test sites were less than the upwind concentrations by factors of 200 to 400 percent. This indicates emissions from the test burn did not increase the ambient air loading of PCBs significantly. The ambient tests results are shown in Table 4.

Breathing Zone and In-Plant PCB Monitoring

During the verification burn and the 1-day background burn, 42 tests were conducted to measure employee workplace exposure to PCB. The tests were conducted by the GM Industrial Hygiene Department using modified sampling and analysis techniques recommended for the isomers of PCB as specified in Physical and Chemical Analytical Method-Number 253 of the NIOSH Manual. A total of 16 tests were made in the breathing zone of the boiler operators. The remaining 26 were general conditions tests at two sites in the powerhouse. Samples were analyzed for PCB by GM's Industrial Hygiene Department. All tests showed nondetectable levels of PCB which indicates that concentrations were less than 3 and 12 $\mu g/m^3$ for Aroclor 1242 and 1254, respectively. These concentrations are considerably lower than current OSHA 8-hour workplace exposure standards for PCB.

Boiler Residue PCB Analysis

Before and after the PCB verification burn, scrapings from various points inside the boiler were taken to determine PCB residue. These residual scrapings were analyzed by the GM Industrial Hygiene Department to determine the presence of PCBs.

Results of the scraping analysis indicate no significant level of PCBs in the boiler. The residual level of PCBs was less than 1 ppm.

PCB Blood Chemistry Levels

GM powerhouse operators were tested to measure blood PCB levels before and after the verification burn. Preburn levels ranged from 7 to 36 ppb with an average of 18.0 ppb. Post-burn levels were 5.4 to 40 ppb with an average of 18.6 ppb. Within the limits of accuracy of this testing, the pre- and post-burn samples showed identical blood levels of PCB.

A comparison study was performed on 39 office workers who presumably would have no work association with

Table 2. *Stack Emission Rate of PCB*

Run No.	Test Day	Gas Volume Sampled (DSCM)[b]	Aroclor 1242 Detected (mg)[c]	Stack Gas Concentration of Aroclor 1242 (mg/m)	Stack Flow (DSCMM)[d]	PCB (mg/min)
PCB-1[a]	1	4.898	<0.001	$<2.0 \times 10^{-4}$	274	$<5.6 \times 10^{-2}$
PCB-2	2	5.380	<0.001	$<1.9 \times 10^{-4}$	307	$<5.8 \times 10^{-2}$
PCB-3	3	5.785	<0.001	$<1.7 \times 10^{-4}$	328	$<5.6 \times 10^{-2}$
PCB-4	3	5.964	<0.001	$<1.7 \times 10^{-4}$	346	$<5.6 \times 10^{-2}$
PCB-5	4	3.207	NA[e]	NA[e]	264	-

[a] *A run to measure background levels of PCBs in the stack gas; no PCB contaminated waste oil was burned.*
[b] *Dry standard cubic meters.*
[c] *Based on GC/ECD results (<1.0 µg Aroclor 1242/extract). (Gas chromatograph with electron capture detector.)*
[d] *Dry standard cubic meters/min.*
[e] *NA – Sample extract was inadvertently spilled during analysis. This loss was not integral to calculating destruction efficiency with confidence because of redundant sampling.*

Table 3. *Stack Emissions Data for Dibenzofurans and Dioxins*

Run No.	Test Day	Gas Volume Sampled (DSCM)	Weight in Particulate Extract (mg)	Concentration in Particulates (mg/m^3)[b]	Weight in Resin Extract (mg)	Concentration in Resin Extract (mg/m^3)
M5-1[a]	1	4.808	$<1.0 \times 10^{-3}$	$<2.1 \times 10^{-4}$	$<8.6 \times 10^{-4}$	$<1.8 \times 10^{-4}$
M5-2	2	5.446	$<5.0 \times 10^{-4}$	$<9.2 \times 10^{-5}$	$<8.5 \times 10^{-4}$	$<1.6 \times 10^{-4}$
M5-3	4[c]	3.290	$<5.0 \times 10^{-3}$	$<1.5 \times 10^{-4}$	$<8.5 \times 10^{-4}$	$<2.6 \times 10^{-4}$

[a] *Run to measure background levels of dibenzofuran and dioxins in the stack gas: no PCB-contaminated waste oil was burned.*
[b] *M5-1 particulate is 50 percent aliquot; M5-2 and M5-3 are 100 percent.*
[c] *No dibenzofuran or dioxin data were taken on day 3. On day 3, two trains were run simultaneously for PCB sampling to test reproducibility. Dioxin and dibenzofuran sampling was resumed on day 4.*

Table 4. *Results of Ambient PCB Analysis (µg/m^3)*[a]

Site Identification	Test Day 1	2	3	4
Upwind-Site 1[b]	0.19	0.35	0.39	0.12
Downwind-(preburn)-Site 2[c]	1.4	0.16	0.043	0.063
Downwind-(burn)-Site 2[d]	0.43	0.084	0.019	0.13
Downwind-(post-burn)-Site 2[c]	0.050	0.24	0.019	Not Sampled
Downwind-(preburn)-Site 3[c]	0.50	0.092	0.017	0.024
Downwind-(burn)-Site 3[d]	0.29	0.082	0.055	0.026
Downwind-(post-burn)-Site 3[c]	0.015	0.015	0.019	Not Sampled
Populated Area-Site 4[b]	0.063	0.067	0.028	0.051

[a] *Aroclor 1242.*
[b] *Twelve-hour sampling period.*
[c] *Three-hour sampling period.*
[d] *Six-hour sampling period.*

PCB. The study showed that: those who eat in excess of 8 oz of Great Lakes fish per week have in their blood a PCB range of 4 to 71 ppb and an average of 23.5 ppb. Those who average less than 8 oz of Great Lakes fish per week have a range of 3.4 to 50.3 ppb and an average of 18.03 ppb.

The powerhouse employees showed blood levels comparable to office employees with the least exposure to environmental PCB. Even the highest individual blood levels in any of the groups analyzed still remained low, relative to danger levels and statewide levels.

Fuel Analysis

Fuel samples were analyzed for PCB by GM and GCA laboratories, using a gas chromatograph with an electron capture detector (GC/ECD). The PCB concentra-

tions reported by each laboratory are listed in Table 5. The variation shown in Table 5 is not unusual with interlaboratory analyses of a complex mixture such as PCBs. An audit was performed at the GM and GCA laboratories to verify that procedures used on fuel samples were analytically sound. The results of the audit verified the comparability of the GCA and GM analyses.

The range in concentration, as reported above, was further investigated by an independent EPA laboratory which analyzed split samples from GCA's spiked waste oil and GM's spiked waste oil. The EPA laboratory reported values of 614 and 640 mg/kg for aliquots of the GM and GCA samples, respectively. These values are intermediate to the GCA and GM values and within the range of expected agreement for analysis of a complex mixture containing PCBs.

Boiler Operating Conditions

The plant where the tests were performed has a powerhouse with three high efficiency boilers which supply steam, compressed air, and surface cooling water for the manufacturing plant. The three boilers in the powerhouse are Wickes Type "K" 65-4K-7 package boilers each with a 60,000 lb/hr steam generation capacity at 8 gal/min of No. 6 fuel oil feed rate. The No. 3 boiler that was used for the PCB-spiked waste oil burn was installed in 1965. For the duration of the verification burn, continuous monitors were installed to measure CO and O_2 as prescribed by federal regulations (*44*, Federal Register, 31545, May 31, 1979). Also, CO_2 and total hydrocarbons (THC) were continuously monitored at the stack. These instruments monitored flue gas from the stack above boiler No. 3. Provision was made for shutdown of waste fuel feed unless the following criteria, required by federal and state authorities, were met:

- Boiler flame temperature—normal (~2500 °F).
- CO concentration—less than 50 ppm.
- Excess O_2—greater than 3 percent.
- Opacity—less than 5 percent.

An additional requirement was for maintaining steady-state operation with 4 gal/min fuel feed rate. Steady-state operation was maintained during testing by setting boiler No. 3 at base load and compensating for fluctuating steam demand with another boiler.

Steady-state operation of the boiler was the responsibility of GM personnel, but the flue gas parameters and composition were monitored by GCA. Any changes were noted and relayed to GM personnel so that action could be taken if necessary. Table 6 summarizes boiler parameters maintained during the burn.

Oxygen concentrations were determined using a Beckman Model 741 Paramagnetic O_2 Analyzer with a measuring range of 0 to 10 percent O_2 full-scale. The analyzer was calibrated at 0 percent O_2 with ultrapure nitrogen and with 8.03 percent O_2 certified calibration gas before and after each test.

CO_2 concentrations were determined using an Infrared Industries Model 702 NDIR Carbon Dioxide Analyzer with a measuring range of 0 to 30 percent CO_2 full-scale. The analyzer was calibrated at 0 percent CO_2 with ultrapure nitrogen and with 7.99 percent CO_2 certified calibration gas before and after each test period.

CO concentrations were determined using a Beckman Model 65 NDIR CO Analyzer with a measuring range of 0 to 50 ppm CO full-scale and calibrated at 0 ppm with ultrapure nitrogen and with 39.9 ppm certified calibration gas before and after each test period.

THC concentrations were determined using a Beckman Model 108-A Flame Ionization Hydrocarbon Analyzer with a measuring range of 0 to 50 ppm full-scale. It was calibrated at 0 ppm with ultrapure nitrogen and with 40.1 ppm methane certified calibration gas before and after each test period.

Table 6 shows that O_2 and CO concentrations were well within federal requirements during testing and that good combustion efficiency, as indicated by the low CO levels, was achieved.

Other physical parameters were also measured in this testing program:

- Flame temperature: 2500 to 2700 °F.
- Furnace temperature (i.e., backwall of boiler): 1500 to 1700 °F.
- Smoke density (i.e., in-stack opacity): 0 to 2 percent.

Table 5. PCB Concentrations in Fuel

Sample Identification	GM Results (mg/kg)	GCA Results (mg/kg)
Spiked Waste Oil	496	750
Fuel Feed (PCB Burn No. 1)	34	72
Fuel Feed (PCB Burn No. 2)	34	76
Fuel Feed (PCB Burn No. 3)	34	88

Conclusions

The data collected during this verification burn indicate that, by following the equipment and performance requirements stated in EPA's final PCB Rule (*44*, Federal Register, 31545, May 31, 1979), at least 99.9 percent PCB destruction efficiency can be achieved in high efficiency boilers.

Furthermore, monitoring of the downwind ambient air, the workplace environment, and employee blood levels indicates that PCB destruction burns can be conducted with no measurable adverse effect on either the workplace environment or the external environment.

Sampling Procedures

PCB Train

The PCB sampling protocol in the combustion flue gas included 3 days of boiler testing at normal-load operation while burning a No. 6 fuel mixed with approximately 10 percent waste oil (500 ppm PCB in the waste oil). The resulting oil fired contained approximately 50 ppm PCB. The mixed fuel was fed to the boiler after a normal burn temperature had been established. During test planning, it was calculated that a minimum sampling period of 6 hours would be required to yield approximately 10 μg of PCB for analysis, based on an assumed destruction efficiency of 99.9 percent.

The sampling train used for PCBs was an EPA Method 5 train modified as described in "Measurement of PCB Emissions from Combustion Sources," EPA-600/7-79-047 (NTIS No. PB 293-360), February 1979. This train utilizes a florisil adsorbent tube and distilled water impingers to trap PCB.

Method 5 Train

The sampling train used to collect non-PCB organics and HCl was a standard EPA Method 5 particulate train modified by the addition of a condenser fitted with an XAD-2 adsorbent column. Flue gas particulate was collected on a 4-in. glass fiber filter. Organic emissions not collected on the particulate filter were trapped by the XAD-2 adsorbent. Moisture from the flue gas was condensed in the train, for the measurement of organics and HCl. The train included an im-

Table 6. *Flue Gas Analyses for O_2, CO, CO_2, THC*

Date Sampled	O_2 (%)			CO_2 (%)			CO (ppm)			THC (ppm)		
	Min	Max	Ave	Min	Max	Ave	Min	Max	Ave	Min	Max	Ave
Day 1	5.6	6.7	6.3	11.2	13.4	12.3	12.3	16.7	14.2	1.3	3.1	2.2
Day 2	5.3	7.3	6.1	12.0	21.3	14.1	6.8	9.7	8.7	0.2	0.8	0.4
Day 3	3.9	8.0	5.6	8.3	12.6	11.3	8.0	11.0	9.6	0.7	2.4	1.4
Day 4	5.3	>10.23[a]	6.5	6.7	13.12	11.3	7.2	12.4	8.0	0.5	0.8	0.8

[a]*Reading was off-scale for the O_2 meter used.*

pinger containing 8 percent Na_2CO_3/H_2O as a trapping solution for HCl emissions.

The sampling and velocity traverse for each train was along two diameters of the stack. A total of 44 points were sampled to provide a representative sample of the flue gas composition. Total sampling time was 308 minutes; 7 minutes per point on test days 1 through 3. The expected flow rate was approximately 0.6 ft^3/min. Sampling was isokinetic (± 10 percent) with readings of flue gas parameters recorded at every sampling point during the traverse.

The PCB train and Method 5 train were run simultaneously except on the second day of waste oil burning. On that day duplicate PCB trains were operated to provide replicate measurements of PCB concentration. The replicate measurements (if PCB had been detected), would indicate the precision of stack collection of PCBs. The Method 5 particulate train from that day was therefore replaced by the second PCB train.

Ambient Air Sampling Procedures

High-volume sampling of ambient air was performed with samplers modified as described in "A Method for Sampling and Analysis of Polychlorinated Biphenyls (PCBs) in Ambient Air" (EPA-600/4-78-048; NTIS No. PB 288-410). Airborne PCB was collected on a series of two precleaned polyurethane foam plugs housed in the throat of a monitor. Flow rate through the sampler was between 10 and 30 ft^3/min.

Monitors were placed each morning during the test burn, based on weather forecasts, on-site wind conditions, and predicted plume dispersions from air quality models. Downwind sites 2 and 3 were located for maximum predicted plume downwash.

Polyurethane foam plugs and HiVol filters were collected from ambient monitors and returned to the laboratory in precleaned amber glass jars with Teflon-lined caps. The samples were combined as necessary and soxhlet-extracted overnight with hexane. The extracts were transferred to roundbottom flasks and the volume reduced via rotary evaporation for analysis by GC/ECD using the conditions listed in Table 7.

Analytical Chemistry Procedures for PCB

The contents of each florisil adsorbent tube were soxhlet-extracted with 200 ml of hexane for at least 4 hours. The apparatus was allowed to cool and the contents held for combination with the impinger extract and train rinse.

Table 7. *GC/ECD Parameters for PCB Analysis*

Instrument:	*Hewlett-Packard Model 5840A with a Ni^{63} electron capture detector and a Model 7671A automatic liquid sampler*
Column:	*6 ft x 2 mm (I.D.) 1.5% OV-17/1.95%*
	QF-1 on 100/200 mesh Chromosorb WHP.
Temperatures:	*Column - 175°C*
	Injector - 270°C
	Detector - 300°C
Flow Rate:	*50 cc/min argon/methane*
Detection Limit:	*0.1 µg/ml Aroclor 1242 (in hexane)*

The impinger waters from each train were returned to the laboratory as one combined sample. Each aqueous sample was transferred to a separatory funnel with acetone and hexane rinses of the sample container. The sample was extracted with three 100 ml portions of hexane which were then combined with the florisil adsorbent tube extract and the concentrate of the hexane and acetone rinses. This combined extract was dried with a sodium sulfate column and rotary-evaporated (40 °C) to less than 10 ml.

This extract was transferred to a 50 ml separatory funnel and partitioned against concentrated sulfuric acid. Further cleanup of the organic extract was not necessary since all sample extracts were colorless.

The extract was then aliquotted for the three quantitative procedures for PCB: (a) gas chromatography with electron capture detection (GC/ECD); (b) gas chromatography coupled with selected ion monitoring mass spectrometry (GC/MS-SIM); and (c) perchlorination to decachlorobiphenyl (DCB) (using the method described in EPA-600/4-78-048) with GC/ECD. The instrumental conditions used for the three quantitation techniques are summarized in Tables 7, 8, and 9, respectively.

The replicate analyses for PCB outlined in this protocol were designed to confirm the GC/ECD quantitation of PCB in stack gas. Since no PCB was detected, replicate procedures merely provided additional evidence that PCBs, if present in the stack gas, were less than 0.2 $\mu g/m^3$.

Instrumentation was calibrated using an Aroclor 1242 solution distributed by Applied Science Labs, Inc., State College, PA. Calibration standards of chlorinated biphenyl isomers were supplied by RFR, Inc., Hope, RI.

Table 8. GC/MS Parameters for PCB Analysis

Instrument:	*Hewlett Packard 5985*
	Gas Chromatograph
Column:	*6 ft x 2 mm (I.D.) Pyrex packed with 1% SP2250 on 80/100 mesh Supelcoport*
Injection Temperature:	*270°C*
Temperature Program:	• *Isothermal for 4.0 min at 160°C*
	• *Linear program from 160° to 270° at 10°C/min*
	• *Isothermal for 15 min*
	Mass Spectrometer
Emission:	*300 μA*
Integration Time:	*250 msec/amu**
Electron Energy:	*70 eV*
Operating Condition:	*Selected Ion Monitor*
Detection Limit:	*1.0 μg/ml chlorinated biphenyl isomer (in hexane)*

(*) Atomic mass unit.

Table 9. GC/ECD Parameters for DCB Analysis

Instrument:	*Perkin-Elmer Model 3920 with a Ni^{63} electron capture detector*
Integrator:	*Hewlett-Packard Model 3380S*
Column:	*6 ft x 2 mm (I.D.) 3% SE 30 on 100/120 mesh Gas Chrom Q*
Temperatures:	*Column - 240°C*
	Injector - 270°C
	Detector - 300°C
Flow Rate:	*50 cc/min argon/methane*
Retention Time:	*3.5 min*
Detection Time:	*0.02 μg/ml as Aroclor 1242 (in hexane)*

Metric Conversion

To Convert:	To:	Multiply By:
BTU	kJ	1.06
°F	°C	5/9(°F-32)
ft	m	0.30
ft^3	liters	28.32
gal.	liters	3.79
in.	cm	2.54
lb	kg	0.45
oz	g	28.35

Glossary

PCBs (polychlorinated biphenyls): Mixtures of compounds and isomers in which the chlorine content ranges from 18 to 70 percent by weight.

DCB (decachlorobiphenyl): The single end product of perchlorination of PCBs, used for confirmation of PCB presence.

GC/ECD (gas chromatograph with electron capture detector): A sensitive instrument for detecting trace amounts of PCB.

GC/MS (gas chromatograph interfaced with mass spectrometer): An instrument used for confirmation of PCB presence.

O_2 (oxygen).

CO (carbon monoxide).

CO_2 (carbon dioxide).

THC (total hydrocarbons).

*USGOVERNMENT PRINTING OFFICE:1982-559-092-435