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SAMPLING AND ANALYSIS PROGRAM AT THE  
NEW BEDFORD MUNICIPAL  
SEWAGE SLUDGE INCINERATOR

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## SECTION 1

### INTRODUCTION

Due to widespread PCB contamination, resulting primarily from the manufacture of PCB capacitors, the entire New Bedford Harbor area has been classified as a National Priority List (NPL) site under Superfund. To assess the magnitude of the contamination program, the Environmental Protection Agency is currently conducting a comprehensive evaluation of the occurrences, distribution, transport and fate of PCBs, and related organic contaminants within the New Bedford area.<sup>1-6</sup> This report augments these studies by reporting on stack gas PCB emissions from the sewage sludge incinerator installed at the New Bedford Municipal Wastewater Treatment Plant. This work was conducted for EPA's Industrial Environmental Research Laboratory, Research Triangle Park, NC, by GCA/Technology Division under Contract No. 68-02-3168, Work Assignment No. 99.

The New Bedford Municipal Wastewater Treatment Plant has previously been identified as a potential source of both fugitive and stack gas atmospheric PCB emissions.<sup>5,7</sup> One study has estimated that the facility contributes from 90 to 300 kilograms per year (kg/yr) of PCBs in the receiving waterway, Buzzards Bay.<sup>7</sup> The initial source of all of these PCBs is the contaminated industrial discharge lines that feed the treatment plant. While PCBs are no longer used by manufacturing plants in New Bedford, the historical contamination of these discharge lines has resulted in a slow leaching of PCBs into the wastewater and ultimately into the treatment facility. A portion of the PCBs are discharged to Buzzards Bay with the liquid discharge from the plant, while the remainder is concentrated in the sewage sludge. This sludge is subsequently incinerated onsite in the facility's multiple hearth incinerator. Due to the relatively low operating temperature of this incinerator (1,000 to 1,500°F), it is suspected that PCBs in the sludge may not be completely destroyed. A PCB destruction efficiency test was conducted

on the incinerator in 1976.<sup>8</sup> This test series indicated an incinerator PCB destruction efficiency of between 46 and 77 percent. However, these results were considered inconclusive due to uncertainties introduced through the use of chlorinated scrubber water.

Under a previous PCB technical assistance effort (Contract No. 68-02-3168, Work Assignment No. 58), GCA/Technology Division prepared a Sampling and Analysis Plan and a Quality Assurance Project Plan for a proposed PCB destruction efficiency test of the New Bedford sewage sludge incinerator.<sup>9</sup> However, this test series was not conducted due to equipment problems at the plant which precluded the establishment of normal operating conditions. By late 1983, all equipment-related problems at the facility were resolved and Region I personnel requested that a stack test be conducted. Four principal reasons were cited:

1. To establish the PCB destruction efficiency of a conventional multiple hearth sewage sludge incinerator while burning contaminated sludge under actual operating conditions. The New Bedford situation is unique in that the incinerator regularly burns sludge contaminated with PCBs. No additional PCB spiking is required. A test of this incinerator, under standard operating conditions would, therefore, serve to define the typical performance characteristics of multiple hearth incinerators when burning difficult to incinerate hazardous wastes such as PCBs. Little research and emission testing has been conducted to-date on the capabilities of multiple hearth incinerators, and this testing will provide useful information in this area.
2. Define the historical impact of the incinerator on New Bedford ambient air quality. Region I is requiring a capacitor manufacturing company to clean the PCB contaminated sewer lines. Once this activity is completed, the largest known source of PCBs into the municipal treatment plant will have been eliminated. Before this process is completed, a stack test is essential to define what the historical long-term impact of PCB contaminated sludge incineration may have had on ambient air quality in the New Bedford harbor area.
3. Emission data on combustion byproducts (i.e., polychlorinated dibenzo-p-dioxins, polychlorinated dibenzofurans (PCDDs, PCDFs) potentially formed during the sludge incineration process do not presently exist on this unit. In fact, the PCDD/PCDF data base on municipal sludge incineration in general is somewhat limited at this time and this test series may provide valuable data in this area as well.

4. A valid sampling and analysis test plan and a quality assurance protocol of these tests have been completed and are in place. Only slight modifications are needed to adapt these plans to reflect existing agency policy on sampling and analytical approaches when conducting PCB destruction efficiency burns.

To meet these goals, GCA modified its existing sampling and analysis plan and quality assurance project plan to insure that they would:

- Quantify PCB levels in incinerator sludge feed, incinerator ash, precooler/scrubber outlet water and flue gas emissions.
- Quantify polychlorinated dibenzo-p-dioxin (PCDD) and polychlorinated dibenzofuran (PCDF) levels in the incinerator ash, and flue gas emissions.
- Obtain sufficient operating data on the sludge, ash, water, and flue gas feed rates in an attempt at calculation of a PCB materials balance of the sewage sludge incinerator.
- Conduct all sampling and analysis in accordance with recommended protocols, including Quality Assurance/Quality Control criteria.

The modified sampling and analysis plan is found in Appendix A of this report, while the Quality Assurance Project Plan is presented in Appendix B.

Section 2 of this study presents the results and conclusions of the sampling effort. Data are provided on incinerator stack gas emissions of PCBs, polychlorinated dibenzo-p-dioxins (PCDDs), and polychlorinated dibenzofurans (PCDFs). In addition, the PCB concentrations of all influent and effluent streams are quantified. The PCB destruction efficiency of the incinerator is calculated and the particulate emission rate of the facility is presented and compared with the applicable Massachusetts State Regulation. Finally, continuous emission monitoring data for carbon dioxide, carbon monoxide, and oxygen are presented and discussed.

Section 3 presents operating data on the wastewater treatment plant and the multiple hearth incinerator. A description of the facility is given first and followed by specific facility data taken during the incinerator test series. Highlights of these data are discussed.

Section 4 provides the sampling approach that was used during the collection of all sludge, water, ash, and stack gas samples. Discrepancies in certain measured flow rates (e.g., sludge feed) are described in this section.

The analytical approach used to quantify contaminant levels on all collected samples are discussed in Section 5. The techniques employed and the instrumentation utilized are addressed.

Finally, the Quality Assurance/Quality Control (QA/QC) procedures that were employed to track analysis of the collected samples are presented in Section 6. The multimedia samples collected at the treatment plan were spiked with several surrogate compounds, and the recovery efficiency of these surrogates are discussed in this section.

In addition to the S&A Plan and QA Project Plan, the appendices to this report include sample calculations for several data elements cited in the report (Appendix C) and field data sheets and Quality Assurance Calibration Data (Appendix D). These calculations and calibrations are referenced in the body of this document.

## SECTION 2

### RESULTS, DISCUSSION AND CONCLUSIONS

#### POLYCHLORINATED BIPHENYLS

##### Flue Gas Emissions

No PCBs were detected in the flue gas samples collected for each test run by the Modified Method 5 sampling train. These data are presented in Table 1. Sampling was conducted using procedures described in Section 4 of this report. These procedures include the use of a glass fiber filter to collect particulates and particulate phase PCBs, and a sorbent resin to collect gaseous phase PCBs. Analysis of these samples followed techniques presented in Section 5 of the report. These techniques employ a gas chromatograph/mass spectrometer (GC/MS) to identify specific PCB isomer classes in both the particulate and sorbent resin samples. The results of this isomer specific PCB analysis are presented in Table 2. As the data indicate, no PCB positional isomers were detected in any of the flue gas samples at the detection limits of the analytical technique. These detection limits vary with the positional isomer category under investigation and the lower chlorinated isomers (mono-, di-, tri-, chlorobiphenyl) demonstrate the greatest sensitivity. For the relatively small flue gas volume collected during each of the New Bedford test runs (<3 dry standard cubic meters (dscm)), the detection limits are 2.0 micrograms per cubic meter ( $\mu\text{g}/\text{m}^3$ ). In the absence of any measurable quantity of PCB in any positional isomer category, this  $2 \mu\text{g}/\text{m}^3$  level was used to express the maximum level of PCBs being emitted from the incinerator. In addition, this value is subsequently used in PCB destruction efficiency calculations as a measure of stack gas PCB levels.

TABLE 1. STACK EMISSION RATE OF PCB

*File*

Run No.	Test date	Gas volume sampled (dscm) <sup>a</sup>	Total PCB isomers detected (mg) <sup>b</sup>	Stack gas concentration of PCB isomers (mg/m <sup>3</sup> )	Stack flow (dscmm) <sup>c</sup>	PCB isomers out (mg/min)
2	2/1/84	2.76	0.006	<2.0 x 10 <sup>-3</sup>	53.34	0.12
3	2/6/84	2.70	0.006	<2.0 x 10 <sup>-3</sup>	47.16	0.11
4	2/7/84	2.75	0.006	<2.0 x 10 <sup>-3</sup>	47.16	0.11

<sup>a</sup>Dry standard cubic meters.

<sup>b</sup>Based on GC/MS results (<3.0 µg PCB/particulate or resin extract).

<sup>c</sup>Dry standard cubic meters per minute.

TABLE 2. NEW BEDFORD INCINERATOR: POLYCHLORINATED BIPHENYL ISOMERS--GC/MS<sup>a</sup>

Sample	Test Run	GCA Sample No.	Total concentration--positional isomer category <sup>b,c</sup>											
			Mono	Di	Tri	Tetra	Penta	Hexa	Hepta	Octa	Non	Deca	Total	
Stack gas ( $\mu\text{g}/\text{m}^3$ )	2	36239/68 <sup>d</sup>	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	<2
	3	36240/69 <sup>d</sup>	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	<2
	4	36241/70 <sup>d</sup>	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	<2
Sludge feed (mg/kg)	2	36292 <sup>d</sup>	ND	3.3	1.1	ND	ND	ND	ND	ND	ND	ND	ND	4.4
	3	36298 <sup>d</sup>	ND	1.8	1.4	ND	ND	ND	ND	ND	ND	ND	ND	3.2
	4	36305 <sup>d</sup>	ND	ND	2.2	ND	ND	ND	ND	ND	ND	ND	ND	2.2
Hopper ash (mg/kg)	2	36312												
	3	36315	<10	<10	<10	<20	<20	<20	<20	<20	<50	<50	<70	
	4	36318												
Scrubber effluent (mg/l)	2	36333												
	3	36334	<0.4	<0.4	<0.4	<1	<1	<1	<1	<1	<2	<2	<3	
	4	36335												

<sup>a</sup>Analyses conducted using GC/MS protocols provided in Section 5.

<sup>b</sup>The concentration provided is a total concentration for the positional isomers within the PCB category (e.g., the value for Tetra- represents the total concentration of all tetrachlorobiphenyl isomers). Quantitations achieved using response factors established for representative isomers within each PCB category.

<sup>c</sup>Detection limits were determined on the basis of instrument response to standard mixes of PCB isomers representative of each category. Standards, as listed in Section 5, were analyzed at 3 concentration levels to establish calibration curves and detection limits. Detection limits for a 3 m<sup>3</sup> stack sample are as follows: mono-, di-, tri-chlorobiphenyl: 2.0  $\mu\text{g}/\text{m}^3$ ; tetra-, penta-, hexa/hepta-chlorobiphenyl; 5.3  $\mu\text{g}/\text{m}^3$ ; octa-, non-, deca-chlorobiphenyl; 13.3  $\mu\text{g}/\text{m}^3$ .

Detection limits for the sludge feed (dry basis) are as follows: mono-, di-, tri-chlorobiphenyl: 1 mg/kg; tetra-, penta-, hexa-, hepta-, chlorobiphenyl: 2 mg/kg; octa-, nonchlorobiphenyl; 4 mg/kg; deca-chlorobiphenyl; 6 mg/kg.

Detection limits for the hopper ash are as follows: mono-, di-, tri-chlorobiphenyl: 10 mg/kg; tetra-, penta-, hexa-, hepta-chlorobiphenyl: 2.0 mg/kg; octa-, nonchloro-biphenyl: 50 mg/kg; deca-chlorobiphenyl: 70 mg/kg.

Detection limits for the scrubber effluent are as follows: mono-, di-, tri-chlorobiphenyl: 0.4 mg/l; tetra-, penta-, hexa-, hepta-chlorobiphenyl: 1.0 mg/l; octa-, nonchlorobiphenyl: 2.0 mg/l; deca-chlorobiphenyl: 3.0 mg/l.

<sup>d</sup>Composite sample.

## Sludge Feed

Incinerator inlet sludge samples were taken at 30 minute intervals during each test run. These samples were initially analyzed for total PCB (Aroclor mixture) concentration by a gas chromatograph equipped with an electron capture detector (GC/ECD). The results of this analysis is presented in Table 3.

The purpose of this analysis was to identify the type(s) of Aroclor mixtures present in the sludge and to quantify the temporal variation in PCB concentration during the course of the test program. The results of this temporal investigation were to be subsequently used to decide if the samples could be composited for further GC/MS isomer specific quantitation.

The data presented in Table 3 reveal that between 85 and 95 percent of the PCBs detected in the sludge were Aroclor 1242; that the variations in sludge PCB concentration during each test run were not excessive for compositing purposes and that the average PCB content of the sludge steadily decreased from test Run 2 to test Run 4. Based on these results, individual sludge samples were combined to form one composite sludge sample per test run. These three composite samples were subsequently analyzed for individual PCB isomers by GC/MS. The results of this analysis are presented in Table 2.

It should be noted that for all three test runs the average PCB concentration of the sludge obtained by GC/ECD and reported in Table 3 is from 2.3 to 3.3 times higher than the PCB isomer concentration values of the composite samples obtained by GC/MS, and reported in Table 2. It was anticipated that reported values would not be identical because of the inherent differences in the quantitation techniques used.

Electron capture detection is sensitive to and cannot discriminate from electronegatively substituted interferences which can be assumed to be present in the complex sewage sludge samples. These interferences, therefore, may have biased the sludge PCB concentration when determined by GC/ECD. However, these interferences do not have an impact on the isomer-specific GC/MS PCB results which are calculated in this report, including PCB destruction efficiency, because all stack emission data for PCBs is based on GC/MS analysis. The GC/ECD analysis presented in Table 3 was used only as a

TABLE 3. PCB CONCENTRATION IN INCINERATOR SLUDGE FEED BY GC/ECD

GCA Control Number	Sample Identification <sup>b</sup>	Concentration, ppm (mg/kg) <sup>a</sup>		
		Aroclor 1242	Aroclor 1254	Total PCB
Run 2	Date: 2/1/84	Time: 13:07 - 16:36		
36292	2a	7.4	0.84	8.2
36293	2b	11	1.3	12
36294	2c	9.4	1.3	11
36295	2d	9.1	0.95	10
36296	2e	8.4	1.1	9.5
36297	2f	8.7	1.3	10
$\bar{X} \pm S_x$	--	9.0 $\pm$ 1.2	1.1 $\pm$ 0.20	10 $\pm$ 1.3
Run 3	Date: 2/6/84	Time: 15:08 - 18:26		
36298	3a	8.5	1.0	9.5
36299	3b	8.0	0.71	8.7
36300	3c	10	0.87	11
36301	3d	7.4	0.78	8.2
36302	3e	5.9	0.83	6.7
36303	3f	11	1.1	12
36304	3g	5.6	0.69	6.3
$\bar{X} \pm S_x$	--	8.1 $\pm$ 2.0	0.85 $\pm$ 0.15	8.9 $\pm$ 2.1

(continued)

TABLE 3 (continued)

GCA Control Number	Sample Identification <sup>b</sup>	Concentration, ppm (mg/kg) <sup>a</sup>		
		Aroclor 1242	Aroclor 1254	Total PCB
Run 4	Date: 2/7/84	Time: 10:19 - 13:42		
36305	4a	5.8	0.97	6.8
36306	4b	6.1	1.1	7.2
36307	4c	5.1	1.1	6.2
36308	4d	5.7	0.84	6.5
36309	4e	6.7	1.0	7.7
36310	4f	8.5	1.3	9.8
36311	4g	6.9	0.83	7.7
$\bar{X} \pm S_x$	--	6.4 $\pm$ 1.1	1.0 $\pm$ 0.16	7.4 $\pm$ 1.2

<sup>a</sup>Reported results have been blank corrected. All results provided on a dry weight basis. Quantitation performed using Webb & McCall technique as detailed in EPA-600/7-79-047.

<sup>b</sup>Grab samples collected from screw conveyor at 1/2 hour intervals during run period noted above.

screening tool to identify major variations in sludge feed PCB concentration and to quantify the specific Aroclors present in the sludge. These goals were successfully met by use of this technique.

The isomeric distribution pattern noted by GC/MS for the sludge samples in Table 2 is consistent with the pattern of an Aroclor 1242 or 1016 mixture. The lower boiling, lower molecular weight chlorobiphenyl groups (di-, and tri-chlorobiphenyl classes) predominate. These patterns, therefore, are also consistent with the GC/ECD screening analysis done on all sludge feed samples reported in Table 3, which identified Aroclor 1242 as the predominant PCB mixture in the sludge.

#### Hopper Ash

Incinerator hopper ash samples were collected and composited using techniques described in Section 4. The composited sample for each run was analyzed for PCB positional isomers by GC/MS. The results of these analyses are reported in Table 2 and indicate that no PCBs of any isomer class were detected. The lowest detection limits for these analyses associated with the lower chlorinated species and are equal to 10 milligrams of PCB per kilogram of ash analyzed (mg/kg).

#### Aqueous Samples

Both inlet and outlet aqueous streams for the flue gas scrubber were analyzed for PCB content. Since the scrubber feed water is fresh New Bedford city water, the scrubber inlet samples were simply screened for total PCB (Aroclor) content using GC/ECD. As anticipated, no Aroclors were detected in any of these inlet samples, at a detection limit of 1 microgram per liter (1  $\mu\text{g}/\text{l}$ ) and no further analysis was conducted on them.

The scrubber effluent discharge represents a potential discharge stream for PCBs not destroyed in the incinerator and was therefore analyzed for PCB positional isomers using GC/MS. The results of this analysis are presented in Table 2. As indicated no PCBs were detected in any of the samples at the levels of detection stated. The lower isomer classes exhibit the greatest sensitivity; 0.4 mg/l.

## Overall PCB Removal Efficiency

The PCB concentrations in all incineration system influent and effluent streams are summarized in Table 4. As previously indicated, each of these streams was analyzed by GC/MS for PCB positional isomers, with the exception of the scrubber inlet which was investigated using GC/ECD.

In addition to the stream PCB concentration, Table 4 presents a PCB mass feed rate or mass emission for each stream. These mass PCB rates were calculated from the stream PCB concentration and the specific stream flow rate. Because PCB levels were below detection limits for all streams except the sludge, both the stream PCB concentration and the stream PCB mass emission rate for most streams are expressed as quantities less than (<) an emission rate calculated at the detection limit. To maintain a consistency in the analytical approach, and to facilitate comparison with the effluent stream PCB concentrations, the sludge sample PCB concentrations are those determined using GC/MS and not GC/ECD.

The total PCB feed rate into the incinerator is calculated using the sludge PCB concentration and the average sludge feed rate for each test run. Because there is no continuous sludge feed measurement guage installed at the facility, the average feed rate was calculated by several methods, and they are described in detail in Section 4. The feed rate data used in Table 4 were based on the grab sample, reverse screw technique which is considered the most representative and accurate of the measurement methods used.

The remaining influent and effluent streams of concern at the incineration facility were sampled and measured according to procedures presented in Section 4. The pre-cooler/scrubber water feed was a constant 365-gallons per minute; the flue gas rate varied from 1665 to 1883 dry standard cubic feet per minute, the ash generation rate varied from 30 to 87 pounds per hour and the scrubber effluent was calculated to be a relatively constant 370 to 371-gallons per minute over the 3 run test series. The scrubber effluent flow rate calculations are presented in Appendix C.

PCBs fed into the incinerator in the sludge can be compared with PCBs being emitted from the incinerator in the flue gas to calculate an incinerator PCB destruction efficiency. These data are presented in Table 5 and indicate that a destruction efficiency of at least 98.5 percent was achieved during

TABLE 4. PCB CONCENTRATIONS IN ALL FEED AND EFFLUENT STREAMS

Run No.	Test date	PCBs in incinerator sludge feed		PCBs in pre-cooler and scrubber water feed		PCBs in incinerator flue gas		PCBs in ash from incinerator		PCBs in scrubber water effluent	
		Concentration $\mu\text{g/g}$ (d,w) <sup>a,b</sup>	Feed rate mg/hr	Feed concentration $\mu\text{g/lc}$	Feed rate mg/hr	Concentration $\mu\text{g/m}^3$	Emission rate mg/hr	Concentration $\mu\text{g/g}^b$	Discharge rate mg/hr	Concentration $\mu\text{g/g}^b$	Discharge rate mg/hr
2	2/1/84	4.3	1,066	<1.0	<82.9	<2.0	<7.2	<0.01	<0.3	<0.4	<33.7
3	2/7/84	3.2	7931	<1.0	<82.9	<2.0	<7.2	<0.01	<0.4	<0.4	<33.7
4	2/8/84	2.2	432	<1.0	<82.9	<2.0	<7.2	<0.01	<0.2	<0.4	<33.7

<sup>a</sup>Dry weight.

<sup>b</sup>Based on GC/MS results, monochloro through trichloro isomers.

<sup>c</sup>Based on GC/ECD results as Aroclor mixture.

*Micrograms/gallon*  
*10.9 mg/g = P.P.M.*  
*is in note 8-10 Table 3*

TABLE 5. DESTRUCTION EFFICIENCY OF PCB

Run No.	Test date	Sludge PCB concentration (mg/kg) <sup>a</sup>	PCB in (mg/min) <sup>b</sup>	PCB out (mg/min)	PCB destruction efficiency <sup>c</sup> (%)
2	2/1/84	4.4	17.77	0.12	99.3 <sup>d</sup>
3	2/6/84	3.2	13.23	0.11	99.2 <sup>d</sup>
4	2/7/84	2.2	7.21	0.11	98.5 <sup>d</sup>

<sup>a</sup>Based on GC/MS results.

<sup>b</sup>Based on sludge feed rates as determined by reverse screw method (see Section 4).

<sup>c</sup>Percent destruction =  $100 \times \frac{\text{PCB in} - \text{PCB out}}{\text{PCB in}}$

<sup>d</sup>This assumes 100 percent sample collection efficiency.

each of the test runs. The actual destruction efficiency may be greater than the stated numerical value because the detection limits of the analytical methods were used to estimate the detection of stack emission levels when no PCBs were observed in the flue gas samples. This is a conservative approach to destruction efficiency determinations and provides a minimum destruction efficiency value. The detection limits employed were set at the optimum sensitivity of the sampling and analysis protocols used during the program.

Additional factors which affected these determinations of PCB destruction efficiency included the relatively low PCB concentrations found in the incinerator sludge feed and the relatively small volume of flue gas that was sampled. Higher sludge levels of PCBs, in conjunction with the same stack gas levels would have permitted the calculation of a PCB destruction efficiency greater than those stated in Table 5. The sludge levels noted in Table 5, ranging from 2.2 to 4.3 mg/kg were, in fact, substantially lower than those expected. A 1981 EPA compliance inspection of this incineration facility cited PCB levels of up to 70 ppm in the sludge immediately prior to incineration.<sup>10</sup> In addition, a recent report on PCB sampling and analysis of the New Bedford municipal sewer system reported "hot spot" PCB levels of up to 78,000 mg/kg in the sludge.<sup>3</sup> These elevated levels, however, were not observed during the incinerator test campaign.

Use of the PCB sludge concentrations obtained by GC/ECD would also have increased the calculated PCB destruction efficiency, as these values are consistently higher than those detected by the GC/MS analyses. However, while more sensitive than GC/MS, GC/ECD analyses are less compound specific and consequently it cannot be said with certainty that the compound under investigation is, in fact, PCBs. In addition, since GC/MS was the analytical method used for flue gas analyses, it was essential that PCB concentrations derived by this same technique be used for inlet PCB level determinations. For these reasons, PCB sludge values derived by GC/ECD were not used.

The sample volume collected during each test run of the stack sampling campaign directly affects the lowest stack gas PCB concentration that can be confirmed by the analytical approach. If the amount (mass) of PCB collected in the sampling train is divided by a greater gas volume sampled, the observed PCB concentration would be lower. This also holds true if the PCB detection limit is used to indicate the maximum amount of PCB collected. As indicated

in Table 1, for a mass PCB detection level of 6  $\mu\text{g}$  and a gas volume sampled of approximately 3 cubic meters, the maximum PCB concentration of the stack gas is 2  $\mu\text{g}/\text{m}^3$ . Had a larger volume of stack sample been obtained and no PCBs been detected, then this level would be lower. Unfortunately, the New Bedford incineration system has a relatively small flue gas flow rate compared to boilers and incinerators that have been used for PCB destruction in the past. Consequently, the sample volume collected using isokinetic sampling techniques is proportionately smaller. This sample volume cannot be readily modified and therefore the confirmed PCB destruction efficiency of the incinerator is, in part, limited by this variable.

#### DIOXIN/DIBENZOFURAN EMISSIONS

Incinerator hopper ash and flue gas particulate and resin samples were analyzed for positional isomer classes of polychlorinated dibenzo-p-dioxins. The results of this analysis are reported in Table 6. This analysis was conducted by GC/MS in accordance with the analytical protocols presented in Section 5. No dioxins were detected in any of the samples at the levels of detectability stated. As indicated in the table, these detection limits vary with positional isomer class, and the lowest level of detection stated, 3.0  $\mu\text{g}/\text{m}^3$  was used to define the total concentration level potentially present in the sample.

Incinerator hopper ash and flue gas particulate and resin samples were also analyzed for positional isomer classes of polychlorinated dibenzofurans. The results of this analysis are reported in Table 7. This analysis was conducted by GC/MS in accordance with the analytical protocols presented in Section 5. No furans were detected in any of the samples at the levels of detectability stated. As with PCB and dioxin analysis, these detection limits vary with positional isomer class, and the lowest level of detection stated (3.0  $\mu\text{g}/\text{m}^3$ ) was used to define the maximum concentration level potentially present in the sample.

In light of the public concern over potential airborne dioxin and dibenzofuran emissions from the incinerator, the total dioxin and dibenzofuran data cited in Tables 6 and 7 were reformatted in terms of stack gas concentrations. These data are presented in Table 8. As discussed

TABLE 6. NEW BEDFORD INCINERATOR: DIOXIN ISOMERS--GC/MS<sup>a</sup>

Sample	Test Run	GCA Sample No.	Total concentration--positional isomer category <sup>b,c</sup>									
			Mono	Di	Tri	Tetra	Penta	Hexa	Hepta	Octa	Total	
Stack gas ( $\mu\text{g}/\text{m}^3$ )	2	36239/68 <sup>d</sup>	ND	ND	ND	ND	ND	ND	ND	ND	ND	<3.0
	3	36240/69 <sup>d</sup>	ND	ND	ND	ND	ND	ND	ND	ND	ND	<3.0
	4	36241/70 <sup>d</sup>	ND	ND	ND	ND	ND	ND	ND	ND	ND	<3.0
Hopper ash ( $\mu\text{g}/\text{kg}$ )	2	36312	ND	ND	ND	ND	ND	ND	ND	ND	ND	<48.0
	3	36315	ND	ND	ND	ND	ND	ND	ND	ND	ND	<48.0
	4	36318	ND	ND	ND	ND	ND	ND	ND	ND	ND	<48.0

<sup>a</sup>Analyses conducted using GC/MS protocols provided in Section 5.

<sup>b</sup>The concentration provided is a total concentration for the positional isomers within the PCDD category (e.g., the value for Tetra- represents the total concentration of all tetrachlorodibenzo-p-dioxin isomers). Quantitations achieved using response factors established for representative isomers within each PCDD category.

<sup>c</sup>Detection limits were determined on the basis of instrument response to standard mixes of PCDD isomers representative of each category. Standards, as listed in Section 5, were analyzed at 3 concentration levels to establish calibration curves and detection limits. Stack gas detection limits for a 3 m<sup>3</sup> stack gas sample are as follows: mono-, di-, tri-tetra-chlorodibenzo-p-dioxin: 3.0  $\mu\text{g}/\text{m}^3$ ; penta-, hexa-hepta dibenzo-p-dioxins; 10  $\mu\text{g}/\text{m}^3$ ; octa-, chlorodibenzo-p-dioxin: 100  $\mu\text{g}/\text{m}^3$ .

Detection limits for a 100 gram ash sample are as follows: mono-, di-, tri-, tetra-chlorodibenzo-p-dioxin: 48  $\mu\text{g}/\text{kg}$ ; penta-, hexa-, hepta-chlorodibenzo-p-dioxin: 160  $\mu\text{g}/\text{kg}$ ; octa-, chlorodibenzo-p-dibenzo-p-dioxin: 240  $\mu\text{g}/\text{kg}$ .

<sup>d</sup>Composite sample.

TABLE 7. NEW BEDFORD INCINERATOR: FURAN ISOMERS--GC/MS<sup>a</sup>

Sample	Test Run	GCA Sample No.	Total concentration--positional isomer category <sup>b,c</sup>									
			Mono	Di	Tri	Tetra	Penta	Hexa	Hepta	Octa	Total	
Stack gas ( $\mu\text{g}/\text{m}^3$ )	2	36239/68 <sup>d</sup>	ND	ND	ND	ND	ND	ND	ND	ND	ND	<3.0
	3	36240/69 <sup>d</sup>	ND	ND	ND	ND	ND	ND	ND	ND	ND	<3.0
	4	36241/70 <sup>d</sup>	ND	ND	ND	ND	ND	ND	ND	ND	ND	<3.0
Hopper ash ( $\mu\text{g}/\text{kg}$ )	2	36312	ND	ND	ND	ND	ND	ND	ND	ND	ND	<48.0
	3	36315	ND	ND	ND	ND	ND	ND	ND	ND	ND	<48.0
	4	36318	ND	ND	ND	ND	ND	ND	ND	ND	ND	<48.0

<sup>a</sup>Analyses conducted using GC/MS protocols provided in Section 5.

<sup>b</sup>The concentration provided is a total concentration for the positional isomers within the PCDF category (e.g., the value for Tetra- represents the total concentration of all tetrachlorodibenzofuran isomers). Quantitations achieved using response factors established for representative isomers within each PCDF category.

<sup>c</sup>Detection limits were determined on the basis of instrument response to standard mixes of PCDF isomers representative of each category. Standards, as listed in Section 5, were analyzed at 3 concentration levels to establish calibration curves and detection limits. Stack gas detection limits for a 3 m<sup>3</sup> stack sample are as follows: mono-, di-, tri-tetra-chlorodibenzofuran: 3.0  $\mu\text{g}/\text{m}^3$ ; penta-, hexa-, hepta-, octa-, chlorodibenzofuran; 10  $\mu\text{g}/\text{m}^3$ .

Detection limits for a 100 gram ash sample are as follows: mono-, di-, tri-, tetra-chlorodibenzofuran: 48  $\mu\text{g}/\text{kg}$ ; penta-, hexa-, hepta-, octa-chlorodibenzofuran: 160  $\mu\text{g}/\text{kg}$ .

<sup>d</sup>Composite sample.

TABLE 8. STACK EMISSIONS DATA FOR TOTAL DIBENZOFURANS AND TOTAL DIBENZO-p-DIOXINS

Run No.	Test date	Gas volume sampled (dscm) <sup>a</sup>	Weight in particulate extract (µg) <sup>b</sup>	Concentration in particulates (µg/m <sup>3</sup> )	Weight in resin extract (µg) <sup>b</sup>	Concentration in resin extract (µg/m <sup>3</sup> )	Instrumental detection limit (ng/µl) extract <sup>b</sup>
2	2/1/84	2.76	<4.5	<1.09	<4.5	<1.09	<3
3	2/6/84	2.70	<4.5	<1.12	<4.5	<1.12	<3
4	2/7/84	2.75	<4.5	<1.10	<4.5	<1.10	<3

<sup>a</sup>Dry standard cubic meters.

<sup>b</sup>Based on the detection limit of the monochloro through tetra chloro isomers.

previously, the stack sampling train was equipped with a glass fiber filter to capture particulates and particulate related emissions and a sorbent resin to capture gaseous phase emissions. The instrumental detection limits for extracts derived from each of these samples is the same, 4.5  $\mu\text{g}$ . In addition, these detection limits are identical for both dioxins and dibenzofurans, as indicated in Tables 6 and 7. Therefore, the concentration data presented in Table 8 describe both dioxin and dibenzofuran emissions. These data indicate that the concentration of dioxins or dibenzofurans in either the particulate or gaseous phases does not exceed 1.12  $\mu\text{g}/\text{m}^3$  in any test run. The actual concentrations may be substantially lower since the stated values are set by the detection limits of the methodology employed and are affected by the relatively small gas volume sampled.

#### OTHER ORGANICS/CHLORIDES

In accordance with the sampling and analysis protocols, the flue gas, ash and scrubber water sample extracts were all analyzed by GC/MS for additional major organic components, particularly chlorinated organics (e.g., chlorinated pesticides, chlorinated benzenes, chlorinated naphthalenes). These analyses were conducted in the total ion mode of the GC/MS. None of these compounds were detected in any of the samples analyzed. The detection level of these analyses were as follows: flue gas 50  $\mu\text{g}/\text{m}^3$ ; aqueous samples 200  $\mu\text{g}/\text{l}$ , ash samples 2000  $\mu\text{g}/\text{kg}$ .

The fourth and fifth impingers of the Modified Method 5 sampling train for each run contained one normal sodium hydroxide solution. These solutions were analyzed for total chloride content with the following results: Run 2--12.4 mg; Run 3--8.7 mg; Run 4--6.8 mg. These levels are equivalent to stack gas hydrogen chloride levels of: 4.62  $\text{mg}/\text{m}^3$  for Run 2; 3.3  $\text{mg}/\text{m}^3$  for Run 3; and 2.54  $\text{mg}/\text{m}^3$  for Run 4. The data sheet for these analyses are presented in Appendix C.

#### PARTICULATE EMISSION RATES

An integral part of the comprehensive test program conducted on the New Bedford incinerator was the establishment of a particulate emission rate for the unit. This incinerator is governed by Massachusetts Department of

Environmental Quality Engineering Regulations for sewage sludge incinerators. This regulation requires that the incinerator demonstrate compliance with a maximum mass particulate emission level of 0.65 grams of particulate per kilogram of sludge charged (dry basis) (0.65 g/kg). The stack gas sample was collected in accordance with EPA Method 5 procedures, as described in Section 4. A summary of the stack sampling data is presented in Table 9 and a summary of particulate emission data associated with each test run is presented in Table 10. All test runs were within the  $\pm 10$  percent isokinetic range allowed by Method 5 procedures.

Based on the data reported in Table 10, the New Bedford sewage sludge incinerator is in compliance with the Massachusetts State standard. The three run particulate emission rate average was 0.595 g/kg.

It is interesting to note that runs 2 and 3 were significantly below the 0.65 g/kg standard, while Run 4 was significantly greater. Run 4, as discussed in Section 3, was characterized by a low sludge feed rate, a watery sludge feed that required constant operator supervision and incinerator operating temperatures substantially lower than those recorded for Run 2. In addition, as the total combustion air flow through the incineration system did not change from run to run, even when the sludge feed rate dropped off, the excess air level for run 4 is much higher than for the other runs. These operating conditions apparently contributed to the higher particulate emission rate recorded for run 4.

#### CONTINUOUS EMISSION MONITORING DATA

Concurrent with the stack samples, continuous emission monitoring data for carbon monoxide, carbon dioxide and oxygen were taken during each test run. These data were first reduced to 3 minute averages for each fixed gas sample, then to 15 minute averages, and finally to one composite average for each run. The 3 minute and 15 minute average data for each gas are included in Appendix C. Furthermore, the 3 minute averages for each gas are graphically presented in Section 4. The composite values for each run are presented in Table 11.

As indicated by this table, the average carbon dioxide concentration consistently decreased from run 2 to run 4, while the average oxygen content increased over those runs. The carbon monoxide level was relatively low for

TABLE 9. STACK TEST DATA SUMMARY

Run Number	*****	2	3	4
Date of Run	*****	1 Feb 84	6 Feb 84	7 Feb 84
Clock Time: Initial	*****	13:07	15:08	10:19
Clock Time: Final	*****	16:36	18:26	13:42
Avg. Stack Temperature	Degrees F	53.0	50.0	50.0
Avg. Square Delta P	Inches H <sub>2</sub> O	0.08	0.07	0.07
Nozzle Diameter	Inches	0.622	0.622	0.622
Barometric Pressure	In. Hg	30.05	29.85	29.86
Sampling Time	Min.	192.0	192.0	192.0
Sample Volume	Cubic feet	103.332	100.315	101.205
Avg. Meter Temp.	Degrees F	96.0	93.0	89.0
Avg. Delta H	In. H <sub>2</sub> O	0.99	0.9	0.9
DGM Calib. Factor (Y)	*****	0.997	0.997	0.997
Water Collected	Milliliters	30.5	11.9	30.0
CO <sub>2</sub>	Percent	9.5	7.0	3.9
O <sub>2</sub>	Percent	7.0	11.5	13.8
CO	Percent	0.0	0.0	0.0
N <sub>2</sub>	Percent	83.5	81.5	82.3
Stack Area	Square inches	1017.4	1017.4	1017.4
Static Pressure	Inches Hg	0.0	0.0	0.0
Pitot Coefficient	*****	0.84	0.84	0.84
Sample Volume Dry	DSCF	97.544	95.480	97.062
Water at Std.	SCF	1.438	0.561	1.415
Moisture	Percent	1.5	0.6	1.4
Mole Fraction Dry Gas	*****	0.99	0.99	0.99
Molecular Wt. Dry	Lb/lb mole	29.80	29.58	29.18
Excess Air	Percent	46.53	114.82	174.08
Molecular Wt. Wet	Lb/lb mole	29.63	29.51	29.02
Stack Gas Pressure	Inches Hg	30.05	29.85	29.86
Stack Velocity	AFPM	261.7	229.5	231.4
Volumetric Flowrate, Dry ST	DSCFM	1883.4	1665.1	1665.2
Volumetric Flowrate, Actual	ACFM	1848.8	1621.6	1635.1
Isokinetic Ratio	Percent	90.3	100.0	101.7

TABLE 10. PARTICULATE EMISSION RATES<sup>a</sup>

Run Number	2	3	4	Average
Run Date	1 Feb 84	6 Feb 84	7 Feb 84	
<b>Front Half</b>				
Part. Collected	95.00	70.63	124.74	96.79
Particulate Loading	0.0150	0.0114	0.0198	0.0154
Loading @ Stk Conditions	0.0153	0.0116	0.0202	0.0157
Emission Rate (Conc. method)	0.2421	0.1626	0.2825	0.2291
Emission Rate (Area method)	0.2187	0.1626	0.2871	0.2228
<b>Back Half</b>				
Part. Collected	17.36	16.80	51.80	28.65
Part. Loading	0.0027	0.0027	0.0082	0.0046
Loading & Stk Conditions	0.0028	0.0028	0.0084	0.0047
Emission Rate (Conc. method)	0.0442	0.0387	0.1173	0.0667
Emission Rate (Area method)	0.0400	0.0387	0.1192	0.0660
<b>Total</b>				
Total Collected	112.36	87.43	176.54	125.44
Particulate Loading	0.0177	0.0141	0.0280	0.0200
Loading @ Stk Conditions	0.0182	0.0144	0.0287	0.0204
Emission Rate (Conc. method)	0.2864	0.2013	0.3998	0.2958
Emission Rate (Area method)	0.2586	0.2013	0.4064	0.2888
Emission Rate (Average)	0.2725	0.2013	0.4031	0.2923
Emission Rate (Average)	123.6095	91.2916	182.8406	132.5806
F - Factor	9487.9	9487.9	9487.9	9487.9
Btu F Factor Method	0.0362	0.0425	0.1118	0.0635
<b>Feed Rate - Rev Screw</b>				
Emission - Rev Screw	248.0	256.2	196.7	233.6
Emission - Allowable	0.498	0.356	0.930	0.595
Emission - Allowable	0.650	0.650	0.650	0.650

TABLE 11. CONTINUOUS EMISSION MONITORING DATA

Run No.	Average carbon monoxide concentration (ppm)	Average carbon dioxide concentration (%)	Average oxygen concentration (%)	Average combustion efficiency <sup>a</sup>
2	449	9.5	7.0	99.5
3	1385	7.0	11.5	98.0
4	1029	3.9	13.8	97.4

a: Calculated as follows:  $\text{Combustion efficiency} = \frac{\% \text{CO}_2}{\% \text{CO}_2 + \% \text{CO}} \times 100$

where: % CO<sub>2</sub> = carbon dioxide concentration

% CO = carbon monoxide concentration

run 2, increased to a maximum for run 3 and then decreased for run 4. These variations are due to several factors. Auxiliary fuel (natural gas) use was at a maximum during run 2 and then decreased to one-half this level for runs 3 and 4. Subsequently, the average hearth temperatures were at a 3 run maximum during run 2 and this contributed to low carbon monoxide levels during this test run.

The sludge feed rate decreased sharply during run 4 and this resulted in the high oxygen and low carbon dioxide values noted for this run. Run 3 exhibited a sludge feed rate similar to that of run 2, but a lower auxiliary fuel rate, and these factors contributed to the median carbon dioxide and oxygen values for this run.

One additional analysis that can be conducted using the continuous emission monitoring data concerns combustion efficiency. This indicator is cited in the PCB regulations, for Annex I incinerators (40 CFR 761.40(a)) and is computed as follows:

$$\text{Combustion efficiency} = \frac{C \text{ CO}_2}{(C \text{ CO}_2 + C \text{ CO})} \times 100$$

where

C CO<sub>2</sub> = concentration of carbon dioxide

C CO = concentration of carbon monoxide

The Annex I incinerator criteria require a combustion efficiency of at least 99.9 percent be maintained throughout any PCB destruction test. The New Bedford incinerator is not classified as an Annex I PCB incinerator and is not required to demonstrate compliance with this combustion efficiency requirement. Nevertheless, it is noteworthy to examine the combustion efficiency values calculated for the three test runs on the New Bedford unit. An average combustion efficiency for each run is presented in Table 11. In addition, combustion efficiency values can be calculated from the 3 minute CO<sub>2</sub> and CO readings. A graphical plot of these 3 minute combustion efficiency figures versus elapsed sampling for each run series to highlight changes in combustion efficiency that occurred during the course of each run, and gives us greater insight into the sewage sludge combustion process. This plot is presented in Figure 1.

# COMBUSTION EFFICIENCY

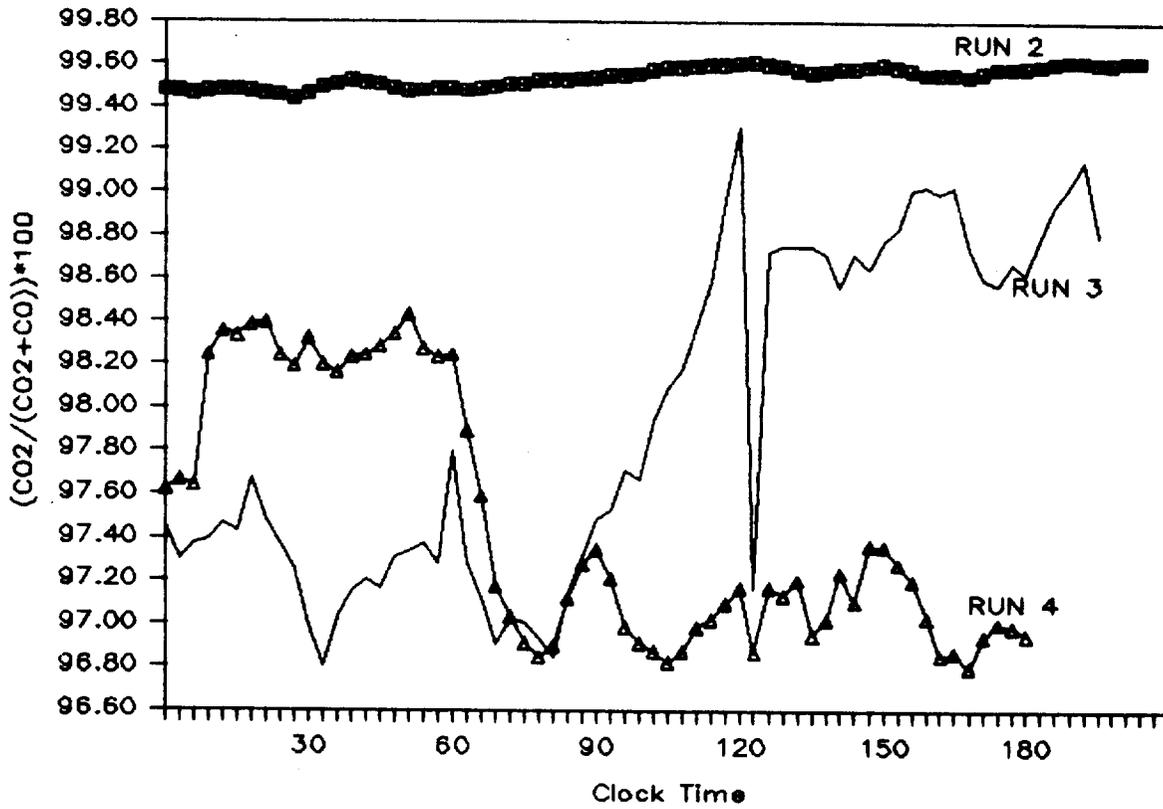


Figure 1. Combustion efficiency versus elapsed time for each test run.

As Figure 1 indicates, at no time during any of the three test runs did the combustion efficiency ever reach 99.9 percent. The three runs, collectively, exhibited a wide variation in combustion efficiencies with one run (run 2) showing little variation, one run (run 3) demonstrating an increasing combustion efficiency throughout the course of the run and the third run (run 4) indicating a dramatic drop off in combustion efficiency.

The relatively high hearth temperatures recorded for run 2 contributed to the relatively low carbon monoxide concentrations for this run. These concentrations, in turn, contributed to the relatively high and generally constant combustion efficiency noted for this test period. One interesting and noteworthy aspect of this run 2 combustion efficiency plot is that no noticeable change occurred in combustion efficiency during the last half hour of the run. During this time sludge feed was halted and the incinerator is allowed to "burn-out." This aspect of plant operation is discussed in more detail in Section 3 of this report. However, based on a total cessation in sludge feed into the unit during this last half hour and resulting in a rapid increase in hearth temperatures, one might expect some noticeable change in combustion efficiency. This condition was not observed.

The combustion efficiency readings for run 3 increased steadily throughout the duration of the run. This increase resulted from a steady decrease in carbon monoxide values recorded for the run. These carbon monoxide readings are graphically presented in Section 4 of this report. During the course of run 3 there was a slight increase in the temperatures for the three uppermost hearths. In addition, as discussed in Section 3, there was a dramatic drop off in sludge feed rates from the start to the completion of this run. These two factors apparently contributed to the rising combustion efficiency noted in Figure 1.

The combustion efficiency plot for run 4 demonstrates an opposite trend, with combustion efficiency decreasing over the course of the test. Moreover, an especially pronounced drop in combustion efficiency occurred 60 to 75 minutes into the run, as shown in Figure 1. The reasons for both this overall and the sudden drop in combustion efficiency are less definitive. Incinerator hearth temperatures, as presented in Section 3, were relatively low throughout this run and did not vary substantially from the start to the end of the test. The average sludge feed rate was at its lowest level of the

test program during this test run. Moreover, as with run 3, the sludge feed rate declined significantly during the course of the run, based on sludge measurements made at the start and completion of each run and as reported in Section 3. The role that these parameters changed in combustion efficiency determinations is uncertain. However, one change in plant operation that was noted by plant personnel during the run was the physical condition of the sludge. Plant operating data presented in Section 3 notes that there were problems with watery ("lousy") feed throughout the duration of the run and that at one point the sludge feed to the incinerator was bypassed to thicken the sludge. This bypassing occurred at roughly the same time as the dramatic decrease in combustion efficiency noted for this run in Figure 1. This watery feed condition, therefore, probably contributed to the change in combustion efficiency values. Even after sludge feed to the incinerator was continued, the combustion efficiency did not appreciably increase.

#### CONCLUSIONS

The data collected during this test program have indicated that the New Bedford municipal sewage sludge incinerator is not emitting PCBs, polychlorinated dibenzo-p-dioxins, and polychlorinated dibenzofurans to the environment. None of these compounds were found in the flue gas, the scrubber water effluent or the hopper ash within the detection limits for the techniques used in these measurements. The PCB destruction efficiency of the incinerator for all three test runs exceeded 98.5 percent, and may in fact be greater. The actual efficiency determination was limited only by the sensitivity of the analytical methodology employed.

During the test program the incinerator was operated according to established standard operating procedures. No attempt was made to increase hearth temperatures or in any other way optimize incineration system performance. Operating data collected and analyzed for the test program reveal that "typical" incineration performance encompasses a broad range of operating characteristics. Combustion temperatures, the physical condition and feed rate of the sludge and stack gas levels of oxygen, carbon dioxide and carbon monoxide all exhibited a significant variation over the course of the

test program. While an analysis of these data indicate operating conditions that may lead to excessive stack particulate emission rates, none of the variations in operation resulted in detectable levels of PCBs, PCDD or PCDF.

Because of the broad range of operating conditions experienced by this incinerator during this test program, incineration system operation is assumed to have been representative of long-term performance. Consequently, the particulate, PCB, PCDD and PCDF emission data reported are also presumed to be indicative of the historical facility emissions.

## SECTION 3

### PLANT DESCRIPTION

#### FACILITY

The New Bedford Municipal Wastewater Treatment Plant is a 30 million gallon per day primary treatment facility designed to handle both municipal and industrial wastewater. Opened in 1973, the facility is operated on a continuous basis, 24 hours per day 365 days per year and employs 28 people. Figure 2 presents a diagram of the major processing operations at the treatment plant.

Incoming wastewater is directed to four clarifiers each measuring 105 feet in diameter, with a 14 foot center height and a 10 foot sidewall depth. Solids separate from the water by gravity in the clarifiers and this water is directed to two chlorine detention tanks where it is chlorinated and discharged to the ocean. Settled sludge is then pumped via four sludge pumps to two hydrogriters where grit and other inorganic solids are separated from the sludge. The grit is subsequently landfilled onsite. The sludge then continues to two gravity sludge thickeners, each of which is 30 feet in diameter and has a wall depth of 10 feet. The liquid removed at these thickeners is returned to the primary clarifier inlet line. The sludge solids continue onto the macerators which grind up the sludge to a uniform consistency. It is then pumped to centrifuges for final thickening. Immediately prior to the centrifuges, a polymer is added to increase solids retention. This polymer is produced by the Carlisle Company and is added at a rate of 2 to 5 percent of the sludge feed. The addition of polymer has been practiced at the treatment plant for only the past 4 to 5 years according to plant personnel. Its use results in a sludge composition leaving the centrifuges of from 25 to 30 percent solids, as opposed to 18 to 20 percent solids when the polymer is not used. An analysis of the sludge entering and

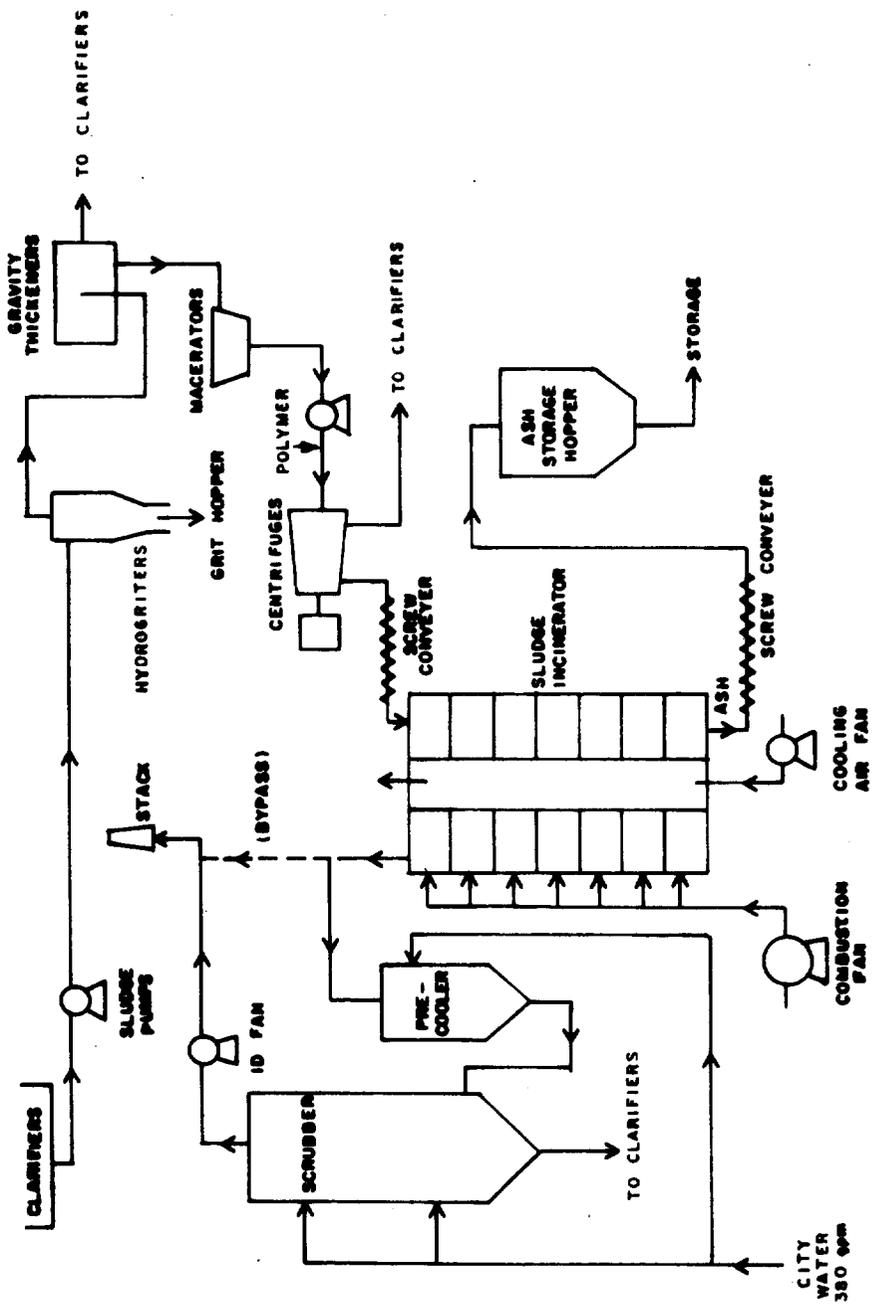


Figure 2. Schematic flow diagram of sludge processing operation. 8

exiting the centrifuges is conducted on a daily basis by facility lab personnel. The most recent analysis indicates that the sludge solids content entering the centrifuges is 11 to 12 percent, while the exiting these units is 28 to 30 percent. The volatiles content of the sludge solids leaving the centrifuges is 75 to 80 percent.

A screw conveyor next transports the thickened sludge from the centrifuges to the multiple hearth sludge incinerator where it is burned. The sludge enters the top of the incinerator and is gradually dried and volatilized as it passes down through the seven stages of the unit. Ash is removed from the bottom hearth and is landfilled onsite. Flue gases generated by sludge combustion exit through the top of the incinerator. They pass sequentially through a precooler and then an impingement scrubber before exiting to the atmosphere through a 3-foot stack located above the incinerator.

Plant operations are directed from a control room located adjacent to the incinerator. Here, key process parameters including incinerator hearth temperature, incinerator draft, scrubber inlet and outlet temperatures, and scrubber differential pressure are continuously monitored and recorded. In addition, automatic alarms are installed for such process controls as burner flame failure, high incinerator draft, high stack oxygen, and high and low incinerator temperature.

### Process

The process of concern with respect to ambient emissions of PCBs at the facility is the Multiple Hearth Sewage Sludge Incinerator and associated Flue Gas Scrubber. The BSP-Envirotech Incinerator Measures 14 ft 3 in. in diameter, and contains seven hearths numbered consecutively from top to bottom. The incinerator has a rated capacity of 1500 pounds per hour of dry sludge feed and is normally operated 24 hours per day, 5 days per week, although this schedule is adjusted to account for fluctuations in sludge production and/or scheduled and nonscheduled equipment maintenance. Figure 3 presents a cross section of a typical Multiple Hearth Incinerator.

Sludge at 28 to 30 percent solids is fed to Hearth No. 2 from centrifuges by screw conveyor. Once inside the incinerator the sludge is raked in a spiral pattern across each hearth by rotating rabble arms. This action turns

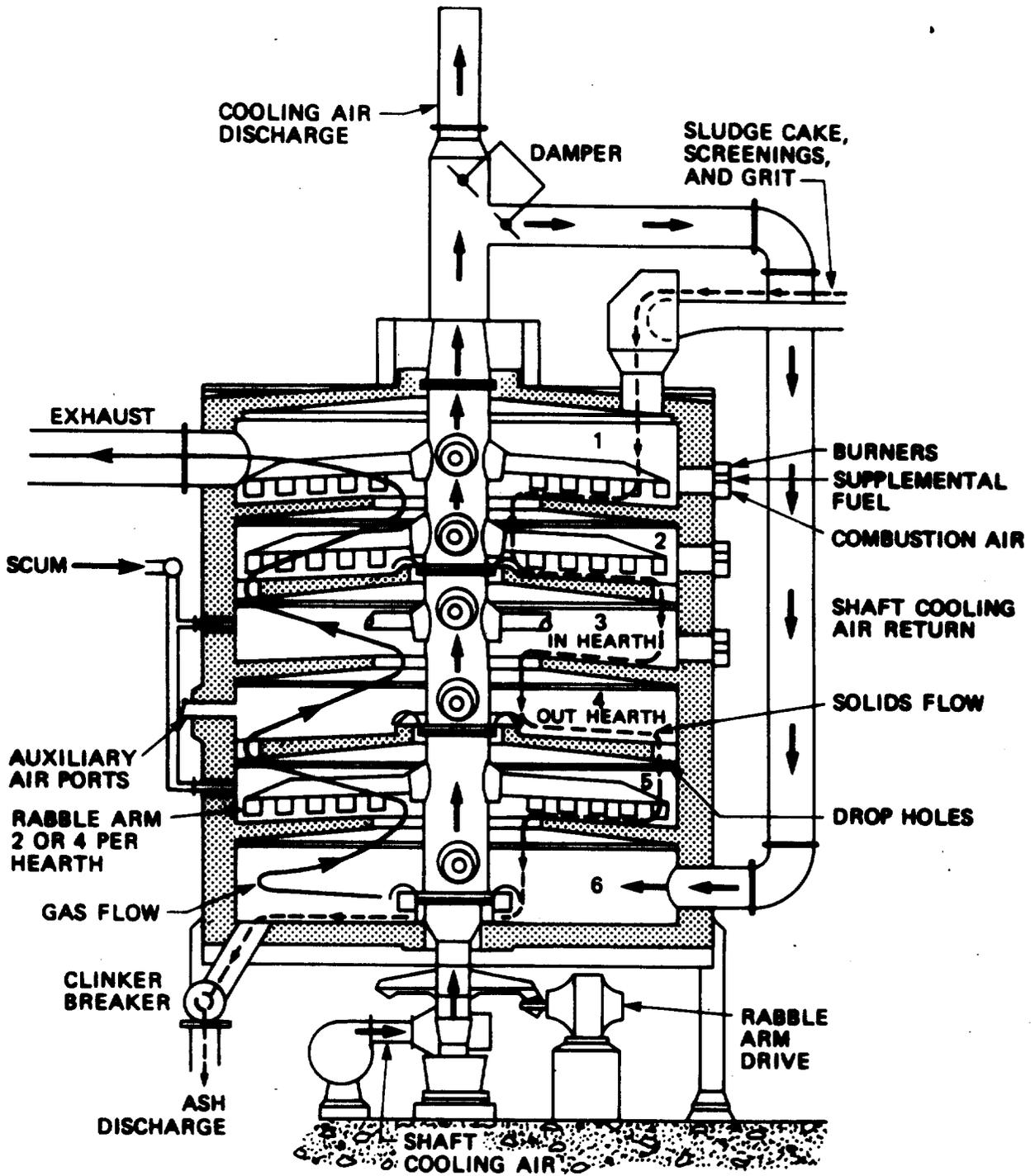


Figure 3. Cross section of a multiple-hearth furnace. <sup>11</sup>

and breaks up the sludge, thus promoting drying and combustion. Openings on the periphery of each hearth permit the sludge to drop by gravity onto the next level. Eight gas-fired burners are positioned, two to a hearth, in hearths 1, 3, 5 and 6 to aid in sludge combustion. Once the unit is at operating temperature sludge combustion is autogeneous. The incinerator temperature is measured and recorded at every hearth level.

The combustion reaction inside the multiple hearth incinerator can be considered to occur in four district zones as shown on Figure 4.

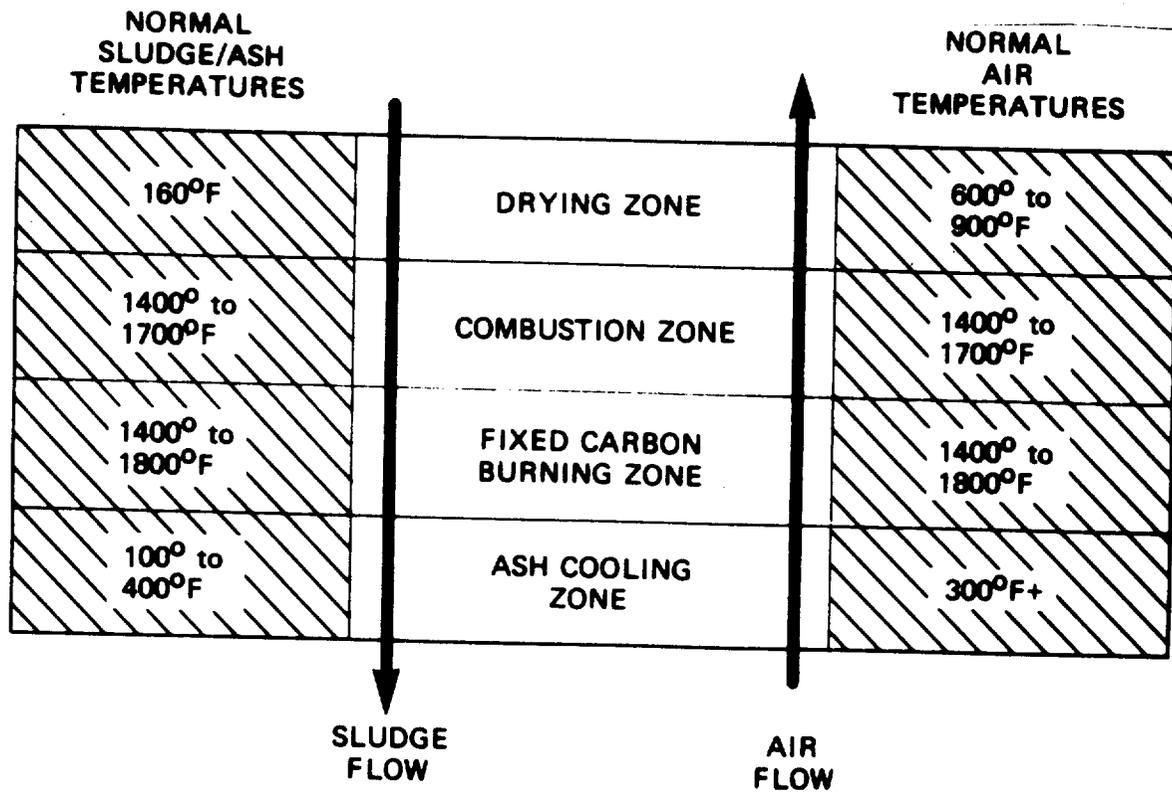


Figure 4. Process zones in a multiple-hearth furnace.<sup>11</sup>

The first zone, which consists of the upper hearths, is the drying zone. Most of the water is evaporated in the drying zone. The second zone, generally consisting of the central hearths, is the combustion zone. In this zone the majority of combustibles are burned and temperatures reach 1400°F to 1800°F (760°C to 980°C). The third zone is the fixed carbon burning zone, where the remaining carbon is oxidized to carbon dioxide. The fourth zone includes the lowest hearths and is the cooling zone. In this zone, ash is cooled by the incoming combustion air. The sequence of these zones is always the same, but the number of hearths in each zone is dependent on the quality of the feed, the design of the furnace, and the operational conditions.

Ash from sludge combustion is carried from Hearth No. 7 by screw conveyor and bucket elevator to an ash holding hopper. This ash is subsequently hauled by truck to an onsite landfill.

Flue gas generated by sludge combustion is ducted from the incinerator to a precooler where the temperature is lowered to approximately 150°F (66°C). The precooler consists of 12 nozzles arranged in two rows and utilizes city water. The use of city water represents a change in this system. Prior to 1979, chlorinated effluent from the clarifiers was used in both the precooler and the scrubber. Corrosion, due to condensation of acid gases generated by sludge combustion, has been a maintenance problem with the precooler. The entire unit has been replaced since the facility opened in 1973. Plant personnel are investigating the use of an alkali additive to combat this problem.

From the precooler, flue gases pass into the impinjet gas scrubber, manufactured by the W. W. Sly Manufacturing Company. The scrubber utilizes Impingement Baffle Plates to effect particulate control. Flue gases enter the bottom of the unit and flow upward through several impingement plate stages where impingement scrubbing action takes place. The gas finally passes through an entrainment separator where water carry over is eliminated. Water is introduced at the top of the baffle plate stages and flows downward by gravity, counter-current to the flue gases. Particulate laden water is drawn off the bottom of the scrubber and fed to the primary clarifiers in the plant. The scrubber and precooler combined use a total of 380 gallons per minute of city water. The differential pressure across the scrubber is continuously measured and recorded in the control room.

The flue gases leave the scrubber and pass through an induced draft fan before exiting to the atmosphere through a 3-foot diameter stack located directly above the incinerator. The stack is easily accessible from the roof of the Treatment Plant Building. An emergency bypass stack is also installed on the incinerator. It provides for direct atmospheric exhaust of incinerator gases should a power failure occur in the scrubber system.

Plant personnel attempt to run the incinerator as close to steady state as is possible. For scheduled and unscheduled incinerator/scrubber maintenance, sludge can be retained and allowed to build up for several days in the primary clarifiers and gravity thickener tanks. Once back on line, the incinerator can be operated continuously, 7 days per week until sludge quantities return to normal levels.

A new hearth was installed in the incinerator on level No. 2 in 1981. New rabble arm teeth were installed in hearths No. 3 and No. 4 in 1980 and 1981, respectively. An in-stack oxygen meter was originally installed in the exhaust stack, downstream of the induced draft fan. This meter is no longer operating.

The facility underwent significant equipment modification and upgrading in 1983. Under this program a new precooler, scrubber to stack breeching, rubber lined induced draft fan, stainless steel spray bars in the scrubber and a new stack were installed. The new equipment, including the fan, were of the same size or capacity as the items replaced. In addition, new burners were installed on hearths Nos. 3 and 5. In all, the air pollution control aspect of the incinerator was completely reconditioned prior to the February 1984 test.

#### PLANT OPERATION DURING TESTING

One unique aspect of the New Bedford test series was the goal of insuring "typical" incinerator operating conditions during each stack test run. Since one of the objectives of conducting the test series was to document historical emissions from the incinerator, no attempt was made by GCA or EPA personnel to optimize combustion conditions. Plant personnel were requested to operate the unit as they normally do, making adjustments to combustion controls, sludge feed rates and scrubber operation as they deemed necessary. The result of

this approach is PCB destruction efficiency results which describe actual, and not optimum, multiple hearth incinerator conditions. This distinction is important to note, since a wide variety of operating conditions were observed throughout the 3 Run test series. These variations will be subsequently discussed.

Another factor related to plant operation which potentially affected the accuracy of the reported values concerns measurement of inlet and outlet solid and liquid feed streams for the incinerator. The wastewater treatment plant is not equipped to continuously measure all streams of interest with regards to a PCB destruction efficiency test of the incinerator. For example, the sludge feed rate into the incinerator can only be measured before and/or after each test run. Attempts to use other less restrictive methods were either unsuccessful due to equipment related problems with the measurement device (e.g., the Doppler equipment) or due to potential inaccuracies in the measurement technique (e.g., back calculation of sludge feed using ash generation rates and sludge ash composition data). Similar measurement problems were encountered in estimating water flow rates into and out of the precooler and scrubber. These measurement problems are more fully described in Section 4 of this report.

#### FACILITY OPERATING DATA

Operating data taken during each of the three test runs are presented in Tables 12 through 14. These data are taken by plant personnel on an hourly basis. The data presented in each table, when possible, span the test period in order to provide an indication of any trend in incinerator hearth temperatures and the other operating parameters. In addition, an average of each parameter is given for the period during which the test run was conducted. These average values can then be compared with each other to summarize the operating parameter differences that were noted during the entire test series. This summary is presented in Table 15.

Run 1 of the test series was attempted on January 31, 1984. However a problem with the sampling equipment forced premature termination of that run before its completion. Consequently no incinerator operating data were obtained or reported for this run.

TABLE 12. INCINERATOR OPERATING DATA, RUN 2, FEBRUARY 1, 1984, 13:07-16:36

Time	O <sub>2</sub> <sup>a</sup> conc. (%)	Hearth temperature (°F)							H7	H6	H5	Scrubber temperature Inlet	Cool air exchanger temp. (°F)	Furnace draft (in. W.C.)	Pressure		Free flow gpm	Comments
		H1	H2	H3	H4	H5	H6	H7							Scrubber psi	Precooler psi		
0800	11	640	760	1280	1645	1100	790	210	110	55	260	0.1	3 in.	20	300			
0900	10	900	790	1350	1720	1100	790	210	145	55	295	0.1	2.5	20	300			
1000	9	1000	890	1450	1500	1100	795	205	145	55	310	0.08	3	20	300			
1100	8	1100	910	1400	1440	1100	790	205	150	55	325	0.08	3	20	295			
1200	8	1100	890	1540	1520	1100	790	200	150	55	330	0.1	3	20	295			
1300	8	1125	900	1590	1595	1130	800	200	150	55	330	0.1	3	20	295			
1400	7.5	1145	945	1550	1525	1125	795	200	155	55	345	0.11	3	20	295			
1500	8	1150	950	1540	1520	1120	790	200	155	55	345	0.11	3	20	295			
1600		Not recorded as plant personnel stopped sludge feed and commenced "burnout" of the unit.																
Average <sup>b</sup> during test	7.8	1140	932	1560	1547	1125	795	200	153	55	340	0.11	3	20	295			

<sup>a</sup>O<sub>2</sub> concentration recorded by plant personnel

<sup>b</sup>Included 1300-1500 readings, excluded 1600 reading

TABLE 13. INCINERATOR OPERATING DATA, RUN 3, FEBRUARY 6, 1984, 15:08-18:26

Time	Hearth temperature (°F)					Scrubber temperature			Cool air exchanger temp. (°F)	Furnace draft (in. W.C.)	Pressure		Free flow gpm	Comments	
	H1	H2	H3	H4	H5	H6	H7	Inlet			Outlet	Scrubber			Precooler
O <sub>2</sub> <sup>a</sup> conc. (%)											psi	psi			
0700-1200	Recalculated leak in precooler door; adjusted mazerators to even consistency of feed														
1300	660	750	1210	1055	1100	890	205	110	50	295	0.11	4	20	295	Burner 3 and 4 on awhile
1445	625	720	1160	1460	1110	890	205	115	55	300	0.12	4	20	295	
1600	525	700	1100	1425	1100	890	225	100	50	280	0.12				
1700															Burners 2 and 4 on
1800	625	760	1250	1350	1100	875	225	100	50	300	0.10				
1900															Burners 3 and 4 off
2000															Light, watery feed
2100															
2200	575	700	1150	1560	1100	880	225	110	50	290	0.10				
Average <sup>b</sup> during test	592	727	1170	1412	1103	885	218	105	52	293	0.11	4	20	295	

<sup>a</sup>No O<sub>2</sub> recordings due to equipment malfunction

<sup>b</sup>Includes 1445-1800 readings

TABLE 14. INCINERATOR OPERATING DATA, RUN 4, FEBRUARY 7, 1984, 10:19-13:42

Time	O <sub>2</sub> <sup>a</sup> conc. (%)	Hearth temperature (°F)							Scrubber temperature		Cool air exchanger temp. (°F)	Furnace draft (in. w.c.)	Pressure		Free flow gpm	Comments
		H1	H2	H3	H4	H5	H6	H7	Inlet	Outlet			Scrubber psi	Precooler psi		
0800	17	640	750	1208	1100	1100	890	220	100	50	290	0.10	3.5	20	290	
0900		Bypassed incinerator due to inconsistent feed (too watery)														
1000		Commenced feed at 10:20														
1100	15	600	740	1160	1170	1100	890	225	100	50	295	0.11	3.5	20	290	
1200		Bypassed incinerator due to poor (watery) sludge feed														
1300	16.5	620	775	1100	1110	1100	840	225	95	50	300	0.11				
1400	16	620	740	1120	1100	1100	850	225	95	50	290	0.12				"Lousy feed"
Average <sup>b</sup> during test	15.8	613	752	1127	1100	1100	860	225	97	50	295	0.11	3.5	20	290	

<sup>a</sup>O<sub>2</sub> concentration recorded by plant personnel

<sup>b</sup>Includes 1100-1400 readings

TABLE 15. AVERAGE INCINERATOR OPERATING DATA, RUN 2, 3 AND 4

Test run	Test date	O <sub>2</sub> conc. (%)	Hearth temperature (°F)							Scrubber temperature		Cool air exchanger temp. (°F)	Furnace draft (in. W.C.)	Pressure		Free flow gpm
			H1	H2	H3	H4	H5	H6	H7	Inlet	Outlet			Scrubber	Precooler	
2	2-1-84	7.8	1140	932	1560	1547	1125	795	200	153	55	340	0.11	3	20	295
3	2-6-84	NA <sup>b</sup>	592	727	1170	1412	1103	885	218	105	52	293	0.11	4	20	295
4	2-7-84	15.8	613	752	1127	1127	1100	860	225	97	50	295	0.11	3.5	20	290

<sup>a</sup>O<sub>2</sub> concentrations recorded by plant personnel

<sup>b</sup>Not available due to equivalent malfunction

Run 2 was conducted on February 1, 1984 from 1:07 p.m. to 4:36 p.m. Prior to the start of the run the incinerator was brought to steady state operation by the treatment plant operators. As indicated on Table 12, each hearth of the incinerator, with one exception, was maintained within a 50°F temperature range. Incinerator operators control these temperatures principally through control of the sludge feed rate. A faster rate of feed will typically move the principal burning zone of the sludge to a lower hearth (e.g., from hearth Nos. 3 to 4). This will cause a corresponding increase in the temperature of this hearth. Plant operators can visually examine the burning sludge through inspection ports located at each hearth level. At times they note the principal sludge burning zone on the operating log.

In addition to sludge feed rate, hearth temperatures can be regulated by use of the auxiliary fuel (natural gas) burners or by controlling the consistency (moisture content) of the sludge. The natural gas burners are operated automatically, and an increase from pilot firing to full firing, depending on the temperature of each hearth. Natural gas consumption is monitored on a daily basis by plant personnel. For Run 2, natural gas consumption totaled 10,000 cubic feet. For Runs 3 and 4, 5,000 cubic feet per run were used.

Control of the sludge consistency (solids content) is maintained by temporarily halting sludge feed to the incinerator and rerouting the sludge back to the clarifiers. This is accomplished by utilizing a bypass line that is installed between the macerators and the centrifuge. When a plant operator notes a high moisture content ("soupy") feed, or if he notes a decrease in the upper hearth temperatures, or upper hearth burning is attributed to excess moisture, he will temporarily bypass the incinerator to thicken the sludge. This condition may be maintained from 10 minutes to several hours, according to plant personnel. This bypass technique is employed frequently at the plant, and although not a problem during Run 2, did occur during one of the later test runs.

One other plant operating condition that did occur during Run 2 was the stoppage of sludge feed that occurs after a sludge burn cycle. This condition is referred to as "burnout" by plant personnel. This procedure was initiated roughly 30 minutes before the end of Run 2. During burnout, which lasts some 30 to 45 minutes, all sludge feed is halted and the hearths are allowed to

burn out the remaining sludge. This practice leads to a rapid buildup in temperatures in the upper hearths. Plant personnel have indicated that temperatures in the 1750-1800°F range are reached in hearths 2 and 3 during this condition. The effects of burnout are most noticeable on the concentrations of CO, CO<sub>2</sub> and O<sub>2</sub> which were measured during Run 2. Graphs of these data, which are presented in Section 4, indicate a rapid decrease in stack gas carbon monoxide and carbon dioxide concentrations, and a rapid increase in oxygen concentrations as a result of this burnout procedure.

The plant operating data for Run 3 are presented in Table 13. There was a greater variation in hearth temperatures during this run than during Run 2. This was attributed to normal fluctuations in sludge feed rate and consistency. In addition, the temperatures in all hearths were lower than during Run 2. This was especially noticeable for the uppermost hearth, No. 1. No reason was given in the operating log for this change in operating temperature.

Table 14 presents incinerator operating data that were taken during Run 4. These data, while demonstrating less variation in individual hearth temperatures than Run 3, have an overall temperature profile similar to that for Run 3. These temperatures are again significantly lower than those noted during Run 2. In addition, as noted on Table 14, plant personnel commented on the poor (watery) consistency of the sludge feed ("lousy feed"). In addition, they attempted to improve this condition by temporarily bypassing the feed back to the clarifiers. No note was made in the operating log for this run of the total time that elapsed between the start of this bypass and the resumption of sludge feed to the incinerator. However, based on the next (1300) recorded hourly operating data, the hearth temperatures did not increase substantially, and it is assumed that the bypass condition was temporary, lasting less than 15 minutes.

A comparison of the average operating data for each run can be made by examining the data presented in Table 15. Note the wide variation in recorded parameters between runs. The oxygen concentration varied significantly between Runs 2 and 4, those for which plant O<sub>2</sub> data are available. This condition was also noted by the continuous emission monitoring data obtained by GCA, and reported in Section 4. As was discussed earlier, the hearth temperatures were at a maximum during Run 2, and decreased in the subsequent

test runs. This decrease in temperatures was also noted for the scrubber inlet and cool air exchanger temperatures. The water flow rate into the scrubber did not change appreciably between any of the runs.

In summary, incinerator operating data obtained during the three test runs indicated a significant variation in hearth temperatures from one run to the next. Run 2 exhibited temperatures far above those recorded during subsequent tests. In addition, the end of Run 2 was conducted while the incinerator commenced a burnout mode of operation. These data collectively exhibit the wide variation in operating conditions that identify "normal" operation at this facility, and demonstrate, by this variation, that it is difficult to easily characterize one set of operating conditions as "typical" for sewage sludge incinerators.

## SECTION 4

### SAMPLING PROCEDURES

The New Bedford sewage sludge incinerator test series included the collection of three sets of flue gas samples from the incinerator's scrubber stack, and the grab sampling of six process streams. In addition, continuous emission monitors were used to measure carbon dioxide, oxygen and carbon monoxide concentrations in the stack gas. A complete detailed description of the sampling and analysis plan for this sampling effort is presented in Appendix A of this report. The Quality Assurance Project Plan associated with this effort is presented in Appendix B. A brief description of each sample collection method is given in the following subsections highlight the principal techniques employed.

#### MODIFIED METHOD 5 TRAIN

A Modified Method 5 train was configured for the simultaneous collection of particulates, PCBs, PCDD and PCDF. A schematic of this train is presented in Figure 5. Use of this combined train is a change from previous PCB destruction efficiency efforts which employed two separate stack sampling trains; one for PCBs and a second for PCDD and PCDF. This combined train was first used during an August 1982 test of the motor tanker Vulcanus<sup>12</sup> and was recently proposed for a test of the Point Woronzof (Alaska) sewage treatment plant incinerator.<sup>13</sup> Use of this train simplifies the collection of all stack gas samples and eliminates errors associated with attempting to simultaneously collect two sets of stack samples.

The sampling train was operated in accordance with the procedures outlined in EPA Reference Method 5. Accordingly, representative samples of the flue gas were obtained during each run from 48 points along two

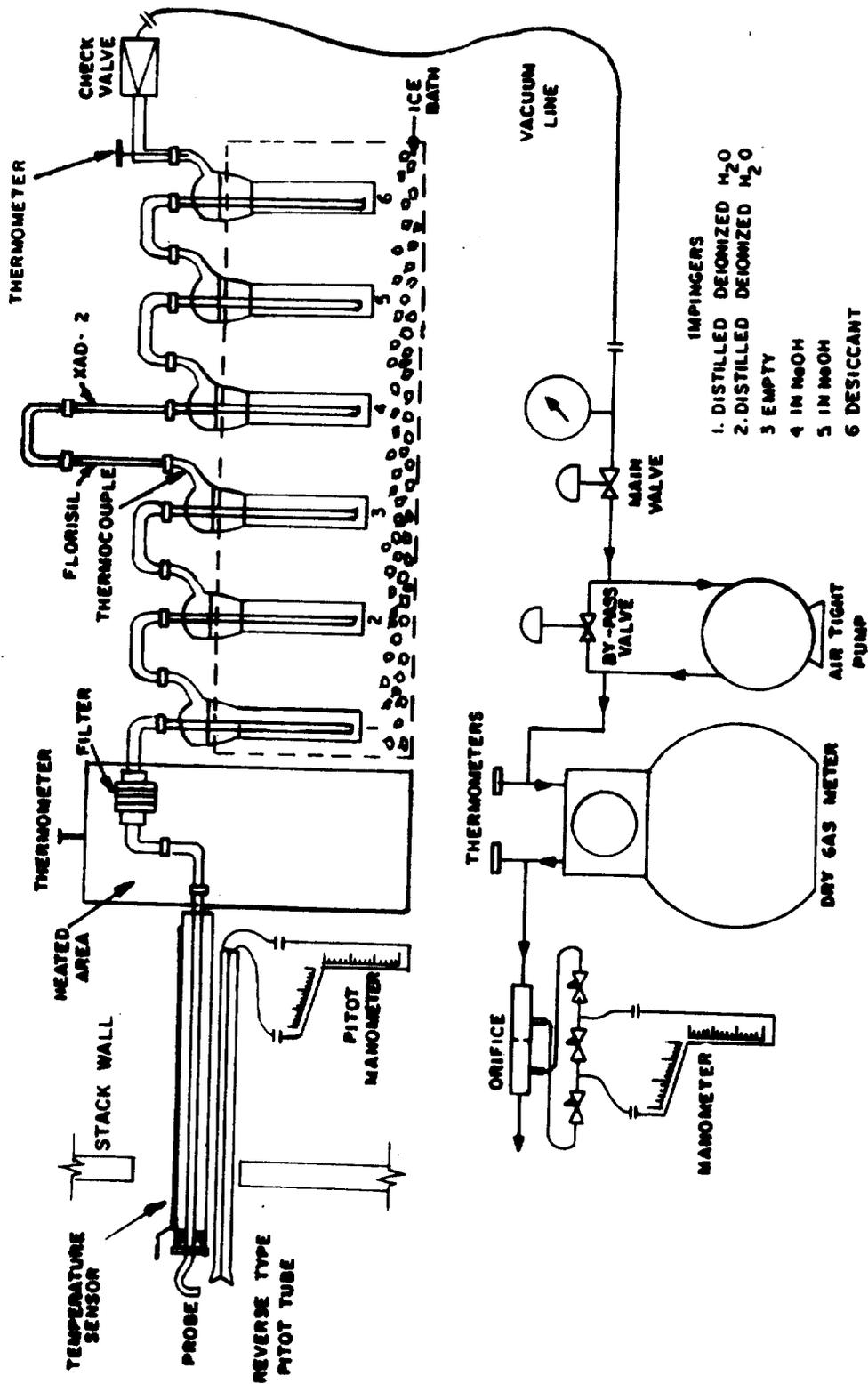


Figure 5. PCB sampling train.

perpendicular diameters of the stack. Specifications of the points were calculated by guidelines in EPA Reference Method 1. Sampling was conducted for 4 minutes per point for a total of 192 minutes.

The sample train consists of a glass lined heat traced probe with a stainless steel button hook nozzle. The probe is equipped with an attached pitot tube and thermocouple. The probe is followed by a heated 4-inch glass fiber filter (Reeve Angel 934 AH). Downstream of the filter are a series of impingers and organic sorbent traps. The impingers contained the following:

- First: 100 ml distilled deionized water
- Second: 100 ml distilled deionized water
- Third: empty
- Fourth: 1N sodium hydroxide
- Fifth: 1N sodium hydroxide
- Final: dessicant.

At the outlet to the third impinger, a sorbent trap containing 7.5 grams of Florisil is followed by an XAD-2 trap containing nine grams of sorbent. The temperature of the traps were maintained at less than 68°F throughout the sampling period.

The impingers were followed by a pump, dry gas meter and a calibrated orifice.

As required by EPA sampling protocol, a field bias blank modified Method 5 train was set up and recovered on each run day.

Recovery procedures of the sampling system were:

1. Remove the sampling train to the predetermined recovery area (the GCA truck).
2. Note the condition of the trains.
3. Remove and seal the sorbent tubes and particulate filter. Label the containers.
4. Measure and record the volume of the first two impingers in a precleaned glass graduated cylinder. Store the impinger contents in a precleaned amber glass container seal and label properly.

5. Rinse the probe, nozzle, filter housing and first two impingers with acetone followed by a hexane rinse. Store the contents in precleaned amber glass container. Seal and label.
6. Measure and record the volume of impingers 4 and 5. Rinse each impinger three times with DDI water and store the sample in a precleaned 1 liter LPE container.
7. Be sure all containers are labeled, sealed and the liquid level marked. Log all samples on the Chain of Custody sheet.

Results from the Modified Method 5 sampling are presented in Section 2. QC procedures implemented are further discussed in the Quality Assurance section (Section 6) of this report.

A complete listing of samples which taken in the flue gas stream, and from the various incinerator inlet and outlet streams is presented in Table 16.

#### CONTINUOUS MONITORING SYSTEM

A portable continuous emissions monitoring system (CEMS) was used to document combustion conditions. The monitoring system consisted of a gas conditioning system and sensors for carbon monoxide, oxygen and carbon dioxide.

A schematic diagram of the CEMS is shown in Figure 6. Briefly, the sample is extracted from the flue stream at a point of average concentration and passed through a glass fiber filter for particulate removal. The gas exiting the filter element is then dried passing it through a condenser for moisture removal. Gas analysis was performed using the instruments and operating ranges specified in Table 17.

Continuous monitoring of the flue gas was performed over the entire length of the proposed testing. On each day of flue gas testing the following sequence was implemented:

1. Arrive onsite, inspect condition of equipment.
2. Set up and leak check conditioning system through manifold.
3. Connect all three analyzers to manifold and individual strip chart recorders.

TABLE 16. SAMPLES COLLECTED AT NEW BEDFORD MUNICIPAL INCINERATOR

	Sample	Source	Species to be analyzed	Number	
Modified Method 5 train	Particulate filter	Train	PCDF, PCDD	3	
	Particulate rinses	Train	PCDF, PCDD	3	
	Florisil resin	Florisil tube	PCBs, PCDF, PCDD	3	
	XAD-2 resin	XAD-2 tube	PCBs, PCDF, PCDD	3	
	Impinger water	Impingers	PCBs, PCDF, PCDD	3	
	Impinger catch (1N NaOH)	Impingers	HCl	3	
	Solvent rinses of train	System rinse	PCBs, PCDF, PCDD	3	
	Method blanks	XAD-2 resin blank	XAD-2 lot	PCBs, PCDD, PCDF	3
		Florisil resin blank	Florisil lot	PCBs	3
		Filter lot blank	Filter lot	PCBs	3
Hexane blank		Hexane lot	PCBs	3	
Acetone blank		Acetone lot	PCBs	3	
Impinger water blank		Organic-free laboratory water	PCBs	3	
1N NaOH		NaOH lot	Cl	3	
Other		Sludge feed	Screw conveyor at incinerator inlet	PCBs	21
		Ash	Ash hopper	PCBs, PCDF, PCDD	3
		Precooler/scrubber water feed	Precooler inlet	PCBs	3
	Scrubber water effluent	Scrubber drain	PCBs	3	
	Centrifuge water	Centrifuge outlet	PCBs	3	
	Plant influent wastewater	Head of plant	PCBs	3	

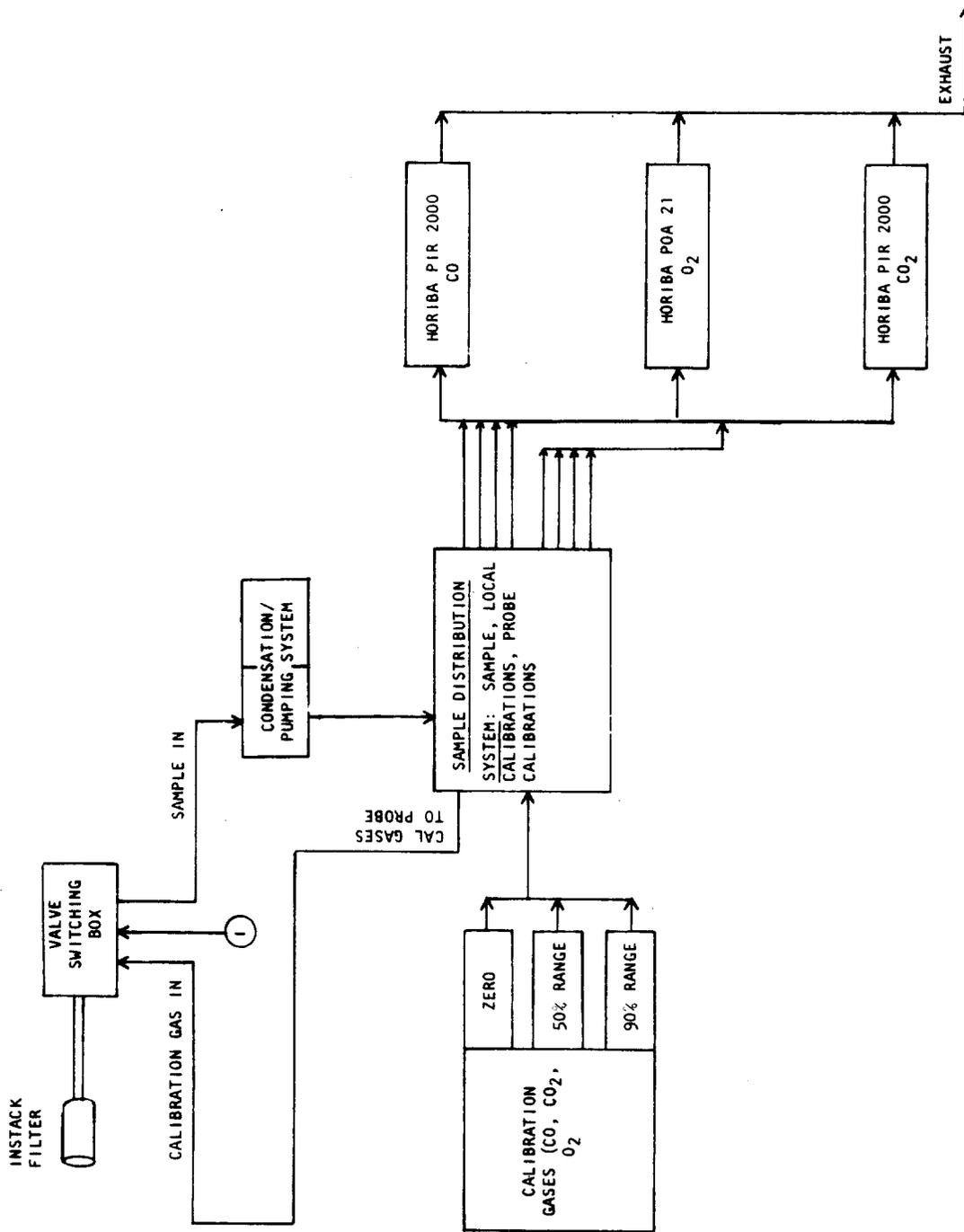


Figure 6. Continuous monitor flow schematic.

TABLE 17. ANALYZER SPECIFICATIONS OF CONTINUOUS MONITORS

	Horiba PIR 2000 CO <sub>2</sub> Analyzer	Horiba PIR 2000 CO Analyzer	Horiba POA 21 OPE 315 O <sub>2</sub> Analyzer
Operating Sensitivity Ranges	0-5%, CO <sub>2</sub> , FS 0-15% CO <sub>2</sub> , FS 0-25% CO <sub>2</sub> , FS	0-500 ppm CO, FS 0-1500 ppm CO, FS 0-2500 ppm CO, FS	0-5% O <sub>2</sub> , FS 0-20% O <sub>2</sub> , FS 0-50% O <sub>2</sub> , FS
Operating Temperature Ranges	24°F -122°F	24°F - 122°F	32°F - 122°F <95% Humidity
Analysis Method	Nondispersive Infrared	Nondispersive Infrared	Polarographic
Linearity	+1% FS	+1% FS	+0.5% of Full Scale
Accuracy	+1% of Full Scale	+1% of Full Scale	+1% of Full Scale
Drift	+1% of Full Scale in 24 hours in both zero and span	+1% of Full Scale in 24 hours in both zero and span	<5% Full Scale for 24 hours in both zero and span
Noise Level	<0.5% of Full Scale in most sensitive range	<0.5% of Full Scale in most sensitive range	<25% of Full Scale in most sensitive range

4. Perform initial calibration of all monitors with zero, mid and high span certified gases. Note and make any adjustments on the monitors. All calibration gases are introduced through the conditioning system sample probe.
5. Monitor O<sub>2</sub>, CO<sub>2</sub> and CO throughout the flue gas testing making sure to mark the strip charts noting the beginning and end of the test runs.
6. At the end of the run, recalibrate the monitors and note all values on the appropriate data sheet to determine monitor drift.
7. Monitoring data was reduced and presented as 3-minute averages.

The continuous monitoring system inspection, installation and operation was performed in accordance with the Horiba Instruments Incorporated Instruction Manual. Documentation of the quality control procedures implemented are further discussed in the QA section of this report.

#### CONTINUOUS EMISSION MONITORS RESULTS

Concentrations of carbon dioxide, oxygen and carbon monoxide were recorded at 3-minute intervals during the Modified Method 5 run. Results are graphically presented in Figures 7 through 12.

Average values for CO, CO<sub>2</sub> and O<sub>2</sub> for each run are reported below:

Run	2	3	4
O <sub>2</sub> , %	7.0	11.5	13.8
CO <sub>2</sub>	9.5	7.0	3.9
CO, ppm	449	1385	1029

#### PROCESS STREAM SAMPLING PROCEDURES

Grab samples of each of the incinerator inlet and outlet feed streams were manually collected at timed intervals during each run. For all but the sludge feed samples, the individual grab samples were composited in a 1250 ml wide mouth amber glass bottle with a teflon lined cap. The samples were labeled, sealed and cooled to 4°C during transport to GCA for laboratory analysis.

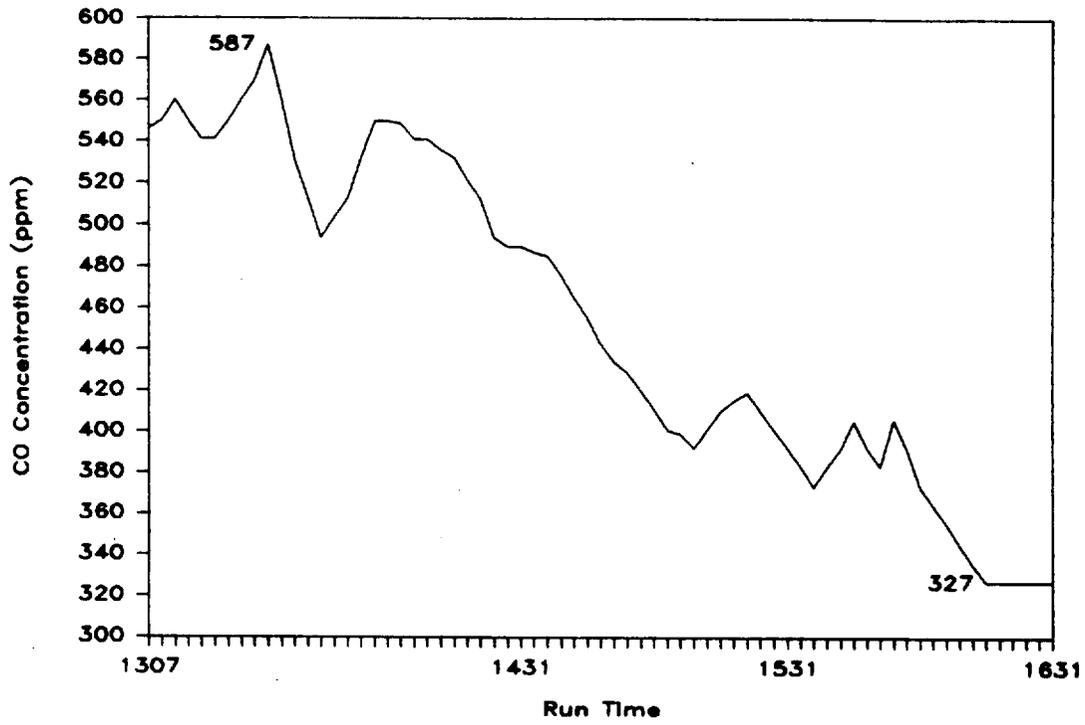


Figure 7. Run 2 carbon monoxide concentration.

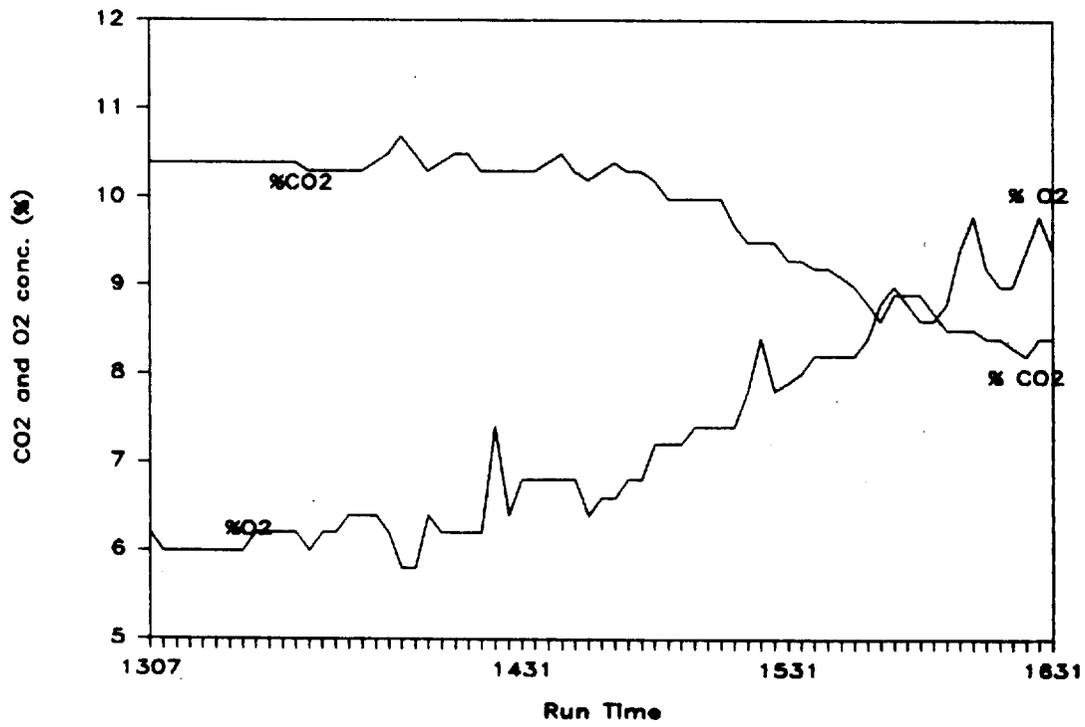


Figure 8. Run 2 carbon dioxide and oxygen concentration.

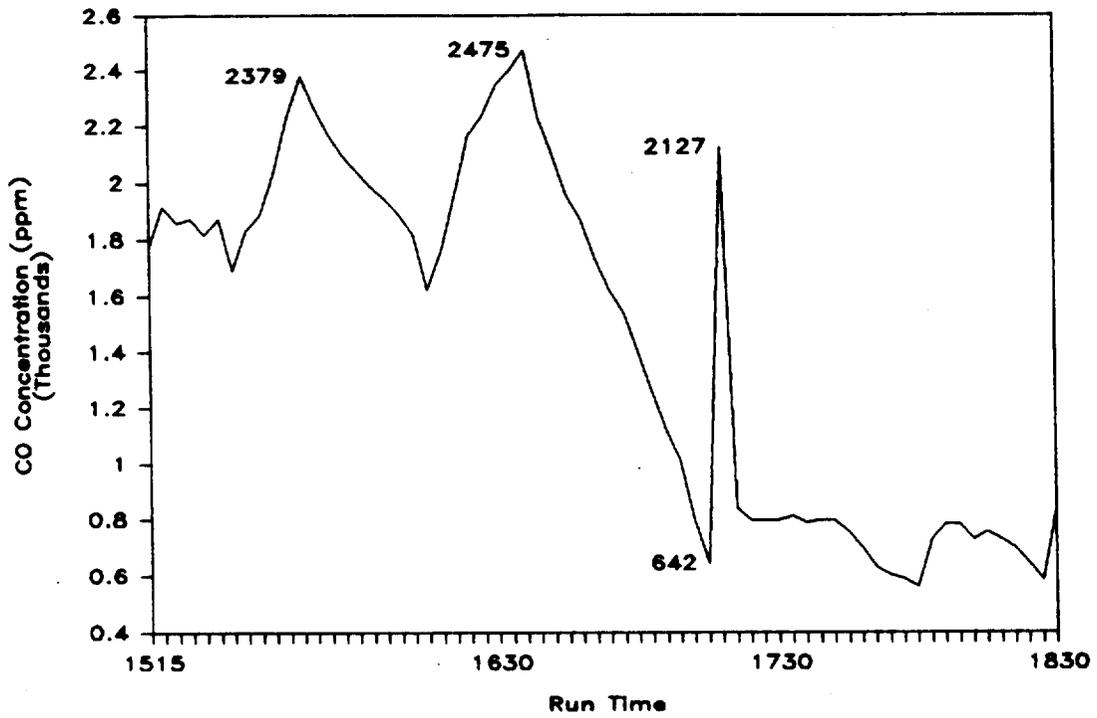


Figure 9. Run 3 carbon monoxide concentration.

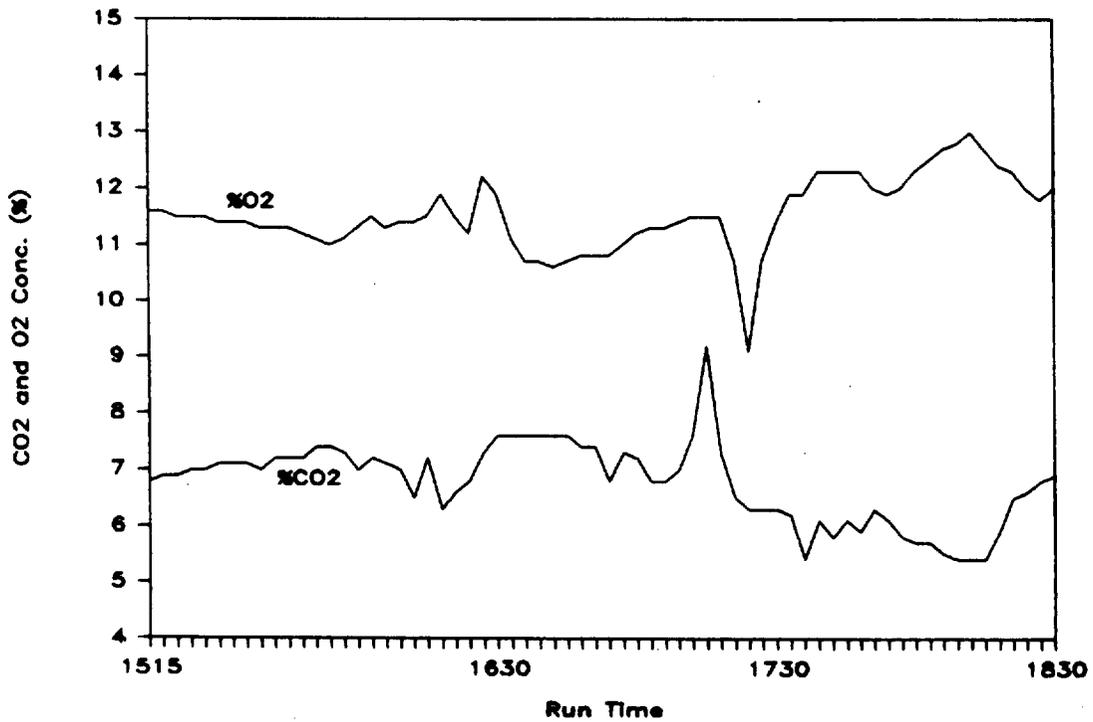


Figure 10. Run 3 carbon dioxide and oxygen concentration.

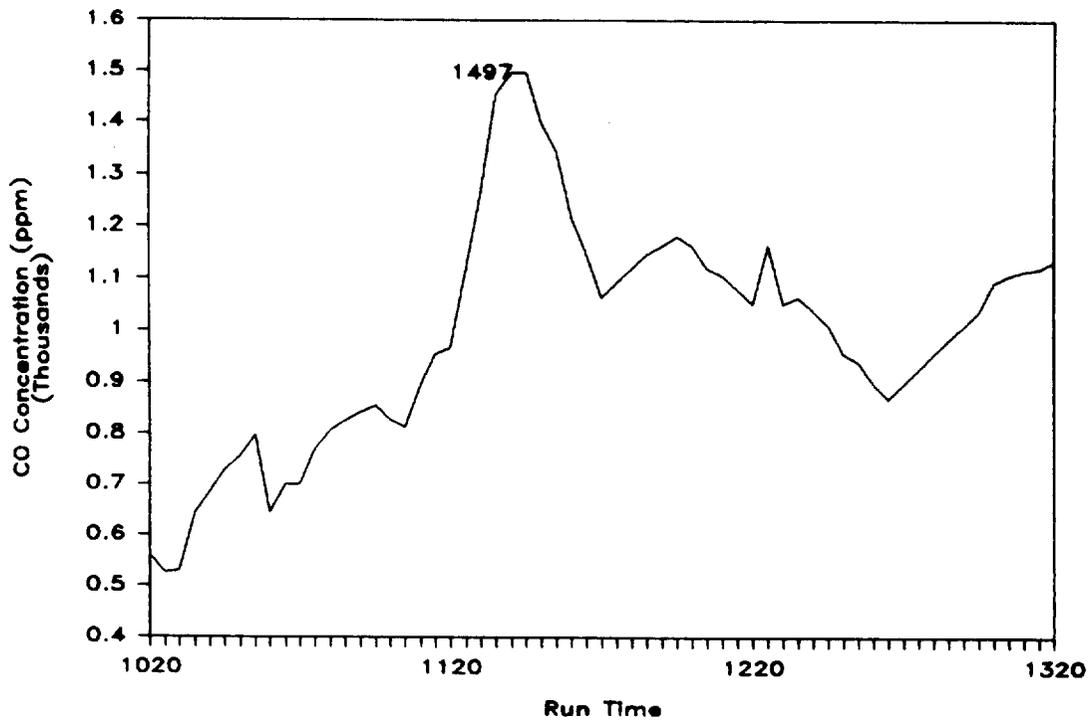


Figure 11. Run 4 carbon monoxide concentration.

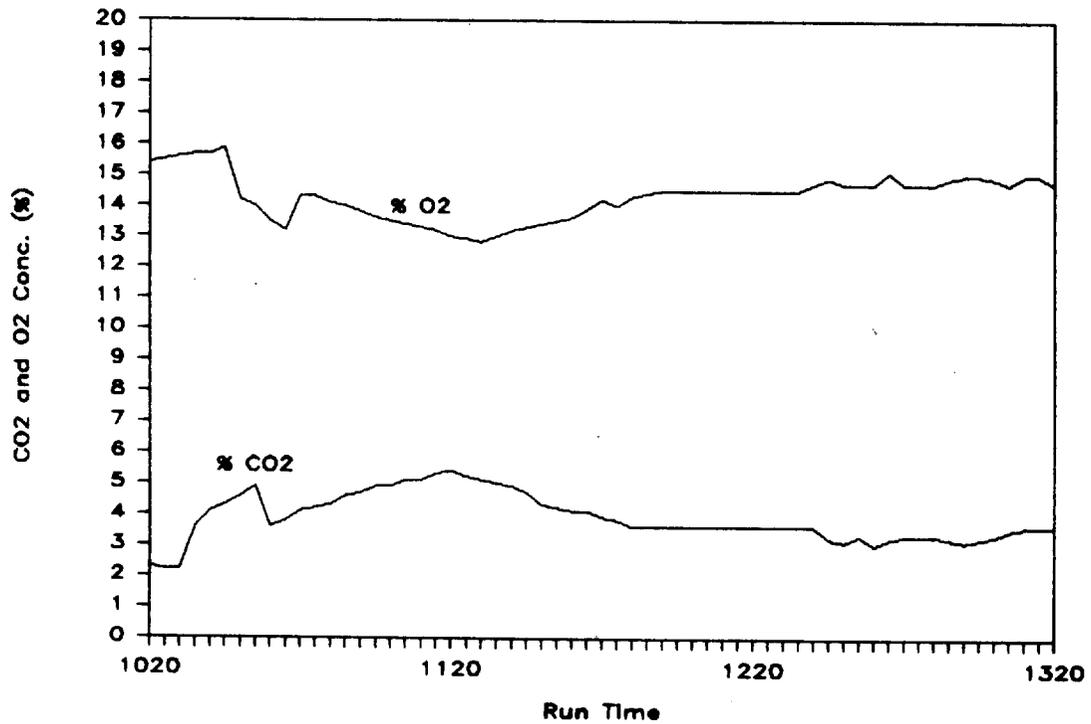


Figure 12. Run 4 carbon dioxide and oxygen concentration.

The following subsections briefly describe each process stream and the sample collected.

### Sludge Feed

Sludge feed samples were collected at the end of the screw converter, immediately prior to the entrance to the incinerator. Grab samples, each approximately 200 ml in volume were collected at the beginning of each run and at 30-minute intervals during each run. These samples were not composited in order to provide an indication of the change of sludge PCB concentration over time.

### Sludge Feed Rate

No direct, in-line sludge flow rate meter is installed at the New Bedford facility. Consequently, it was necessary to provide for one or more alternative measurement techniques. The approach preferred by GCA sampling personnel prior to the conduct of the test series was use of an ultrasonic doppler flowmeter. This instrument (Model 500, manufactured by Dynasonics, Inc., Naperville, Illinois) had been successfully used by GCA during a previous sludge incinerator test.<sup>14</sup> It offers the advantage of being lightweight, portable and it can be mounted on the exterior of any readily accessible pipe in the facility with a diameter in excess of 1 inch. The doppler instrument emits an ultrasonic signal which is altered by any solids contained in a liquid stream. The instrument measures the shifts in frequency that occur to this signal and converts this shift to a flow velocity reading. This reading, in combination with the cross sectional area of the pipe, can be used to calculate the volumetric flow rate within the pipe.

GCA field personnel electronically field calibrated the meter at the New Bedford facility, by a bucket and stop watch method. The Doppler was initially placed on the sludge feed line located between the macerator and the centrifuges. This was the only line of concern at the plant which could be completely bypassed to provide this weight per unit time calibration. One instrument reading was made each test run on this sludge line, as well as on the centrifuge liquid effluent line, the scrubber and pre-cooler feed lines and

the scrubber effluent stream. The sludge feed rate into the incinerator was to have been calculated by subtracting the centrifuge liquid effluent value from the centrifuge inlet reading.

At the conclusion of the first valid sampling run (Run 2) the doppler flow readings were analyzed, and a mass balance of sludge into the incinerator, and water inlet and effluents from the precooler/scrubber were calculated. These calculations indicated that the doppler readings were questionable, as the sum of the effluent streams was greater than the sum of the influent streams. GCA field personnel concluded that various factors may have led to erroneous doppler readings, including variable solids loading of the sludge, a rough surface lining on the interior of the pipe(s), and a variable flow rate in each measured pipe. This problem precluded use of doppler measurements for the determination of incinerator sludge feed and other stream measurements.

GCA field personnel, in Runs 3 and 4, relied upon the reverse screw feed method as the primary indicator of incinerator sludge feed rates. This technique involves reversing the screw feed to the incinerator and measuring the amount (weight) of sludge captured in a predetermined time. A sludge feed rate, in units of pounds of sludge per minute, can then be calculated. This bucket and stop watch technique was run three times immediately before and after Runs 3 and 4. The numerical average of the six sludge feed rates obtained for each run was then used to characterize the sludge feed rate during the entire run. This average value for Run 3 was 2533.5 lb/hr and for Run 4, 1945.0 lb/hr. It should be noted that there was a noticeable decline in the sludge feed rates during each run. At the start of Run 3, the calculated sludge feed rate was 4269.2 lb/hr while at the end of this run, the rate has dropped to 797.8 lb/hr. Similarly, for Run 4, the initial sludge feed rate was 3422 lb/hr, but this rate had dropped to 468 lb/hr by the end of the test. No reason was given for this drop off in sludge feed.

As no direct reverse screw measurement had been obtained during Run 2, rate was estimated by comparing the relationship between the reverse screw sludge rate values obtained during Runs 3 and 4 and the corresponding doppler reading that was taken on the centrifuge inlet line. A doppler reading had been taken during Run 2. Therefore a proportion was established between

reverse screw sludge feed rates and the doppler readings to provide an estimate of Run 2 sludge feed. This value, 2451.4 lb/hr, is used in all calculations concerning the sludge feed rate for Run 2.

Due to the importance associated with incinerator sludge field rates, for both particulate emission compliance determinations and PCB mass balance calculations, alternate methods of estimating sludge feed were investigated. Two additional feed rate methods were employed; the F-factor approach and the ash feed rate method.

The F-factor approach has been used in many fossil fuel stack test applications. This method utilizes an elemental analysis of the fuel being burned (or incinerated) to provide an indication of the amount of fuel consumed or combustible material burned. This method requires the following information to calculate the sludge feed rate:

- Elemental breakdown of sludge (C, H, N, O, S);
- Btu value of dry sludge, Btu/lb;
- Btu value of auxiliary fuel (natural gas), Btu/lb;
- F-factor of dry sludge, dry standard cubic feed (dscf)/10<sup>6</sup> Btu;
- F-factor of auxiliary fuel (natural gas), dscf/10<sup>6</sup> Btu;
- Volume of natural gas combusted, cubic feet; and
- Stack gas volumetric flow rate, dry standard cubic feed.

To perform these calculations, an ultimate analysis of a composite sludge sample was required. This analysis and a set of F-factor calculations are provided in Appendix C. The natural gas consumed by the incinerator during each run was obtained by GCA field personnel from a flow meter installed at the facility. These data indicated that 10,000 cubic feet were burned during Run 2, while 5000 cubic feet were consumed per run during Runs 3 and 4. Natural gas consumption is required in the F-factor calculation to account for the amount of flue gas generated by natural gas. This quantity is subtracted from the total flue gas rate to provide an estimate of sludge generated flue gas. This value is then used to estimate sludge consumption.

The F-factor calculation approach resulted in sludge feed rate estimated of 1255.9, 1427.0 and 1401.1 lb/hr for Test Runs 2, 3, and 4, respectively. These values are for dry sludge feed rates, as opposed to the wet (as fired) sludge rates reported previously for the reverse screw technique. The composited sludge analysis presented in Appendix C cites a sludge solids content of 22.3 percent (moisture content of 77.7 percent). When the F-factor sludge rate is adjusted to account for this sludge moisture content, we note estimated (wet) feed rates of 5631.8, 6399.1 and 6283.0 lb/hr for Test Runs 2, 3 and 4 respectively. These values are from 2.3 to 3.2 times the feed rates calculated using the reverse screw method. The reason for this discrepancy is unclear, however, it may be due to the failure of the F-factor calculation to take into account the excess air that is needed for both natural gas and sludge combustion. Nevertheless the F-factor approach does provide for a second set of sludge feed rate estimates.

The third technique employed to estimate sludge feed rates was a back calculation using the ash collection rate. Ash sampling procedures are documented in another subsection of the sampling procedures. Ash sampling methodology permitted calculation of an average ash generation rate for each test run. Three ash generation rates were calculated for each run and the numerical average of these three was used to characterize the ash rate for the entire test. These average ash rates were 56.7, 86.7 and 30 lb/hr for Test Runs 2, 3 and 4, respectively. If these ash generation rates are used in conjunction with the average ash content of the sludge, then an average sludge generation rate can be estimated. The average sludge ash content was measured as part of the ultimate analysis of the composite sludge sample. This analysis is presented in Appendix C, and indicates a sludge ash content of 4.9 percent. If this value (in decimal form) is divided into the ash rates, estimated sludge feed rates of 1156.5, 1768.7 and 612.2 lb/hr (wet basis) can be calculated for Runs 2, 3 and 4, respectively. These ash derived rates are from 31 to 70 percent of the sludge feed rates calculated using the reverse screw technique.

The reason for this discrepancy is unknown. A summary of all estimated sludge feed rates is presented in Table 18. In addition, an average of the three sets of calculated feed rates is presented. As is evident from the table, there is as much an order of magnitude difference between the maximum

TABLE 18. NEW BEDFORD SLUDGE FEED RATE DETERMINATIONS

Run <sup>a</sup>	Pounds of sludge per hour		
	2	3	4
Date	2-1-84	2-6-84	2-7-84
F-factor method <sup>b</sup>			
Dry <sup>a</sup>	1255.9	1427.0	1401.1
Wet	5631.8	6399.1	6283.0
Reverse screw method			
Dry <sup>c</sup>	546.7 <sup>d</sup>	564.9	433.7
Wet	2451.4 <sup>d</sup>	2533.5	1945.0
Ash feed method			
Dry <sup>c</sup>	257.9	394.4	136.5
Wet	1156.5	1768.7	612.2
Average			
Dry	686.8	795.4	657.1
Wet	3079.8	3567.0	2946.6

<sup>a</sup>Note Run 1 was halted due to an equipment leak.

<sup>b</sup>See Appendix C for calculations.

<sup>c</sup>All dry sludge feed rates were calculated based on the composite sludge sample solids content (22.3% solids)

<sup>d</sup>A reverse screw check of the sludge feed rate was not conducted for Run 2. The feed rate provided was based on the centrifuge inlet rate and the average centrifuge moisture removal efficiency (see text).

and minimum feed rate determinations. Rates estimated using the F-factor approach are always the greatest, while feed rates back-calculated from ash generation values are always the smallest of the three techniques. The reverse screw technique provides a median value in all cases and this approach is considered the most accurate as it is the only method that directly measures sludge feed. It is recommended that this sludge rate be considered the most representative.

#### Hopper Ash

Ash samples were collected from the feed line to the ash storage hopper. These samples were collected at the start of each run and at 60-minute intervals. The rate at which ash was generated was calculated during the collection of each sample by means of a bucket and a stop watch. All ash entering the storage hopper during a pre-set time period was captured and weighed onsite. As previously discussed this technique yielded average ash generation rates of 53.3, 86.7 and 30 lb/hr for Runs 2, 3 and 4, respectively. Each sample was collected in a 250 ml amber glass jar, sealed with a teflon lined cap, labeled and forwarded to GCA for analysis.

#### Precooler/Scrubber Water Feed

The precooler/scrubber water feed was sampled from a tap located on the precooler inlet. Four 250 ml samples were taken; one at the start of each run and at 45-minute intervals thereafter. These samples were composited in a 1250 ml amber glass bottle. One composite sample per run was submitted for analysis.

Precooler and scrubber inlet water feed rates for the New Bedford incinerator are measured continuously and recorded hourly by plant personnel.

A check of these values was attempted, through use of the doppler flowmeter. However, due to the metering problems previously described, this attempt was unsuccessful. Consequently, the plant readings were assumed to be correct. These flow rates included 295 gallons per minute (gpm) of "free flow" water entering the scrubber above the scrubber plates, 42 gpm of

precooler water feed and an additional 28 gpm of water fed to the scrubber at the spray bars. The total of 365 gpm did not vary significantly between stack test runs.

### Scrubber Effluent

Samples of the precooler/scrubber effluent were taken from a tap located on the scrubber line drain. These samples were taken at the beginning of each run and at 45-minute intervals thereafter. Each sample comprised approximately 500 ml. The four samples for each run were composited in a precleaned 3000 ml amber glass container. One composite sample per run was submitted for analysis.

The precooler/scrubber effluent is not directly measured at the facility. In addition, GCA's attempt to use the doppler flowmeter for this stream was unsuccessful. Consequently this effluent rate was calculated, by doing a moisture balance over the entire incineration system. This mass balance calculation is presented in Appendix C. The resulting estimated precooler/scrubber effluent rate was 371 gpm.

SECTION 5  
ANALYTICAL PROTOCOLS

INTRODUCTION

As noted earlier in Section 4 samples were collected during each of three test days at the New Bedford facility. Three complete sets of flue gas samples and associated field blanks were analyzed for polychlorinated biphenyls (PCBs), polychlorinated dibenzodioxin (PCDDs), polychlorinated dibenzofuran (PCDFs) and other organic components. A fourth sample set collected on the first test day was not suitable for analysis due to problems encountered during the sampling activities. This sample set was aborted as a consequence.

In addition, the following analyses were conducted on a variety of influent and effluent streams at the New Bedford Wastewater Treatment Plant: twenty sludge feed samples were analyzed for PCBs; hopper ash grab samples from each test run were analyzed for PCBs, PCDDs, PCDFs and other organic components; precooler/scrubber water feed samples from each test run were analyzed for PCBs and other organics; scrubber water effluent samples from each test series were analyzed for PCBs; centrifugate water samples and plant influent wastewater samples were also analyzed for PCBs and other organics.

The discussion to follow will provide further details on the analytical protocols used including sample preparatory procedures unique to each matrix as well as the subsequent gas chromatographic (GC/ECD) and mass spectrometric (GC/MS) procedures employed.

## SAMPLE PREPARATION

### Flue Gas Samples--Gas Phase

The gas phase is defined as all train samples except the particulate filter and probe rinse which are addressed in the next section. Each of the three sets of steady state runs and corresponding field and method blanks were analyzed for the following parameters:

- PCBs as positional isomer classes in both the particulate and gaseous phases.
- PCDDs/PCDFs--again as positional isomer classes in both the particulate and gaseous phases.
- Other Organics--Results for other major organic components in each of the particulate and gaseous phases and not members of the above organic classes.
- Chloride--in the NaOH impingers only.

Each gas phase sample set contains the following sample types:

- Impingers/Organic condensate
- Back half rinse (acetone/hexane)
- Florisil sorbent
- XAD-2 sorbent
- Aqueous impinger (NAOH)

There are also corresponding field blanks associated with each of the above sample types. There is one complete field biased blank set for each of the respective test days.

A summary of the analysis scheme appropriate for the gas phase samples is provided in Figure 13. Pertinent instruction on the preparation and analysis of each sample type are provided below.

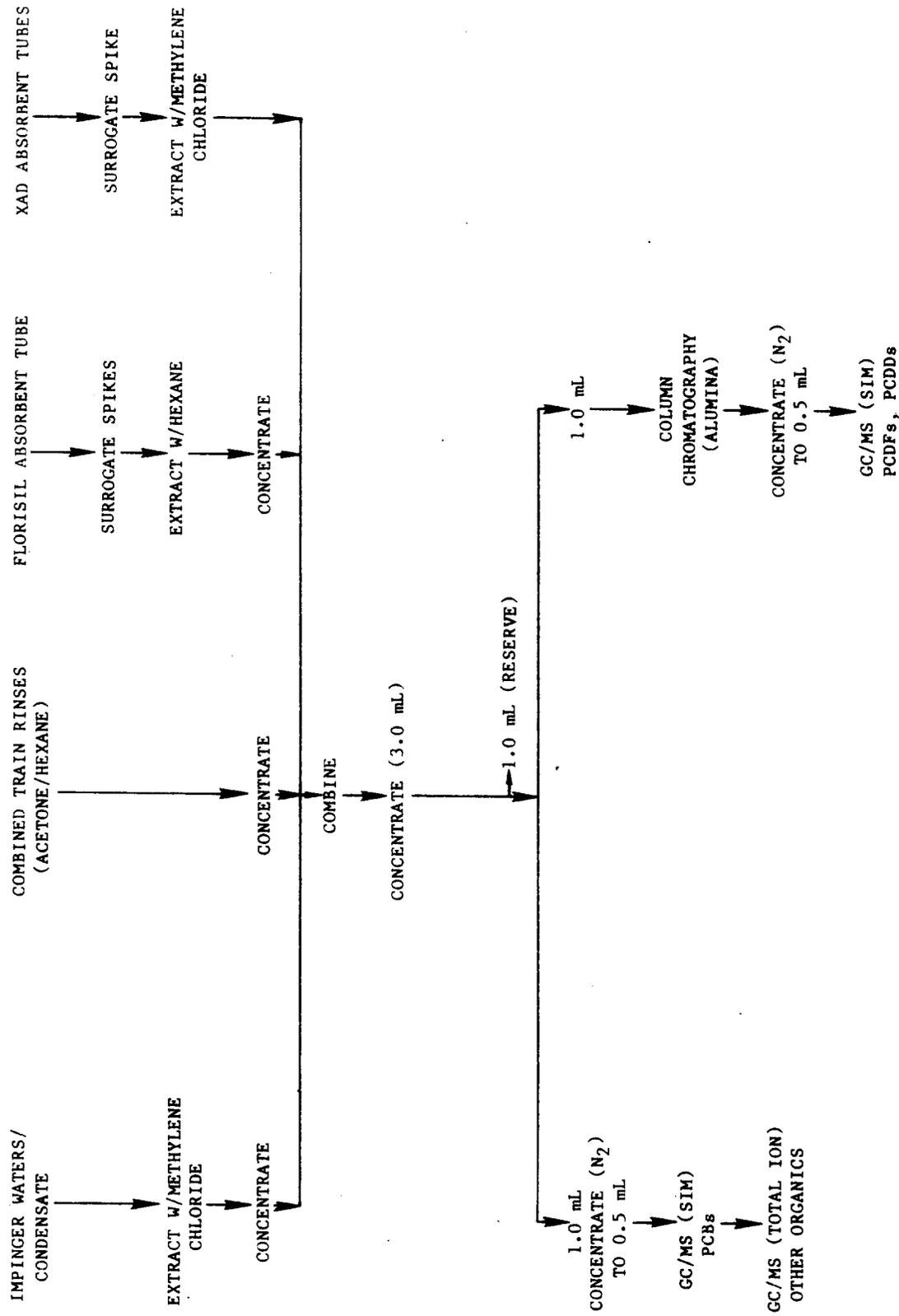


Figure 13. Analytical flow scheme--gas phase flue gas samples.

#### Impingers/Condensate--

The organic and inorganic condensate samples were combined representing the contents of the first three impingers. Each of these composite aqueous samples was extracted sequentially (3X) with methylene chloride. Extracts from each sample were combined and held for eventual combination with the corresponding train rinses and sorbent extracts.

#### Train Rinses--Back Half (Acetone/Hexane)--

Each set of back-half rinses were combined and held for combination with the corresponding impinger and sorbent extracts.

#### Florisil Sorbent--

The physical condition of each sorbent tube was noted and recorded prior to actual analysis (e.g., color, percent moisture, and other characteristics). Each sorbent tube and associated blanks (method and field) was fortified with a surrogate cocktail prior to removal from the trap. Spiking levels of 10-20 g were used for each of the following:  $d_3$ -trichlorobenzene,  $d_{10}$ -biphenyl,  $d_{12}$ -chrysene and decafluorobiphenyl (DFB).

The spiked sorbent was then transferred to an "E" size soxhlet extractor and subsequently extracted overnight with hexane. Each of the glass sorbent tubes was rinsed with a series of solvent washings as follows: 1 x 10 ml acetone followed by 3 x 10 ml of hexane. All rinses were transferred into the corresponding soxhlet extractor. Upon completion of the extraction cycle the solvent was held for combination with all other solvent extracts for eventual Kuderna-Danish concentration.

#### XAD-2 Sorbent--

The physical condition of each sorbent tube was noted and recorded prior to actual analysis (e.g., color, percent moisture, other characteristics). Each sorbent tube and associated blanks (method and field) was fortified with the following surrogate mixture prior to removal from the trap:  $d_3$ -trichlorobenzene,  $d_{10}$ -biphenyl,  $d_{12}$ -chrysene and decafluorobiphenyl (DFB). Spiking levels of 10-20 g were used for each component. The spiked sorbent was then transferred to an "E" size soxhlet thimble. Each tube was rinsed with acetone (1 x 10 ml) followed by (3 x 10 ml) rinses with hexane.

All rinses were transferred into the corresponding soxhlet extractor. Upon completion of the extraction cycle all of the existing solvent extracts were combined and concentrated via KD to 10.0 ml. A 5.0 ml aliquot was removed and held in reserve. The second aliquot was further reduced in volume via N<sub>2</sub> blowdown to 0.5 ml (500 µl) and submitted directly for GC/MS analysis. This combined extract represents the total gas phase portion of each flue gas sample.

#### Aqueous Impingers (NaOH)--

Impinger aliquot were initially analyzed using ion chromatography, however, matrix interferences precluded quantitation by this method. Subsequent quantitation was performed utilizing a Technicon Auto Analyzer II according to the protocol specified in Method 325.2.<sup>15</sup> This automated, colorimetric procedure involves the addition of mercuric, thiocyanate and ferric nitrate solutions to both standards and samples followed by an absorbance measurement, at a wavelength of 480 nm. Working standards in the range of 1 to 30 mg/l were prepared daily from a 1000 mg/l chloride stock solution. Sample concentrations were determined by comparison of sample percent scale to the established standard curve.

#### Flue Gas Samples--Particulate Phase

The combined probe rinse and particulate filter samples from each flue gas train constituted the stack particulate samples. Preparation of these samples proceeded in the following manner: particulate weights and probe rinse residue weights were recorded. The filter and probe rinse solids were then combined and extracted in an "F" soxhlet using methylene chloride. Prior to extraction each thimble was spiked with 50 µg of the following surrogates: d<sub>3</sub>-trichlorobenzene, d<sub>10</sub>-biphenyl, d<sub>12</sub>-chrysene and decafluorobiphenyl. Extracts were reduced to a final volume using a Kuderna-Danish evaporator. GC/MS analyses for PCBs, PCDDs, PCDFs and other organic components were conducted using the instrumental operating parameters previously cited for gas phase extracts. Extracts for PCDD/PCDF analysis were subjected to cleanup using acid/base partitioning and alumina column chromatography. This procedure is described in Method 613 of the EPA/EMSL Methods for Organic

Chemical Analysis of Municipal and Industrial Wastewater (EPA-600/4-82-057). A schematic of the analytical flow scheme pertinent to the flue gas particulate samples is provided in Figure 14.

### Sludge Feed

Grab samples of sludge feed were taken at 1/2 hour intervals during each of the test series resulting in a total of seven samples per 3 hour period. Analyses were conducted on each of these samples. A 10 g aliquot was removed from each sample and air-dried. Each of these samples, in turn, was extracted overnight in a soxhlet-extractor using a hexane/acetone (1/1) solvent system. Each resultant extract was reduced to 5.0 ml using a Kuderna-Danish apparatus. An aliquot of each extract was submitted for PCB analysis using a gas chromatograph fitted with an electron capture detector (GC/ECD). All samples were subjected to additional florisil column chromatography as described in Method 608 of Reference 16.

Identification of an Aroclor mixture by GC/ECD requires a chromatograph match of several peaks between sample and standard. The relative sizes of the individual peaks within each mixture forms a pattern which aids specific Aroclor identification. In environmental samples, however, alteration of the Aroclor pattern can occur due to degradation of individual PCB isomers or the presence of interfering peaks. When this pattern alteration occurs as with the present program samples, an alternative quantitation procedure, developed by Webb and McCall (Reference 17) is frequently employed.

All sludge feed extracts were quantitated against p,p'-DDE as a retention time reference using the instrumental analysis conditions listed on Table 19. Quantitation was performed by applying the computation rules described by Webb and McCall to determine the total amount of PCB present. Results are reported as ppm (mg/kg) of Aroclor 1242, 1254, or 1260 on a dry weight basis.

In addition to PCB analysis via GC/ECD on each grab sample, PCB analyses as positional isomer categories were conducted via GC/MS on composite samples from each test series. In this instance a 1.0 ml aliquot was removed from each of the uncleaned seven sample extracts representing each test run. The composite extract was fortified with the same surrogate cocktail used for the flue gas and hopper ash samples. Spiking levels of 5-10 µg for each of these

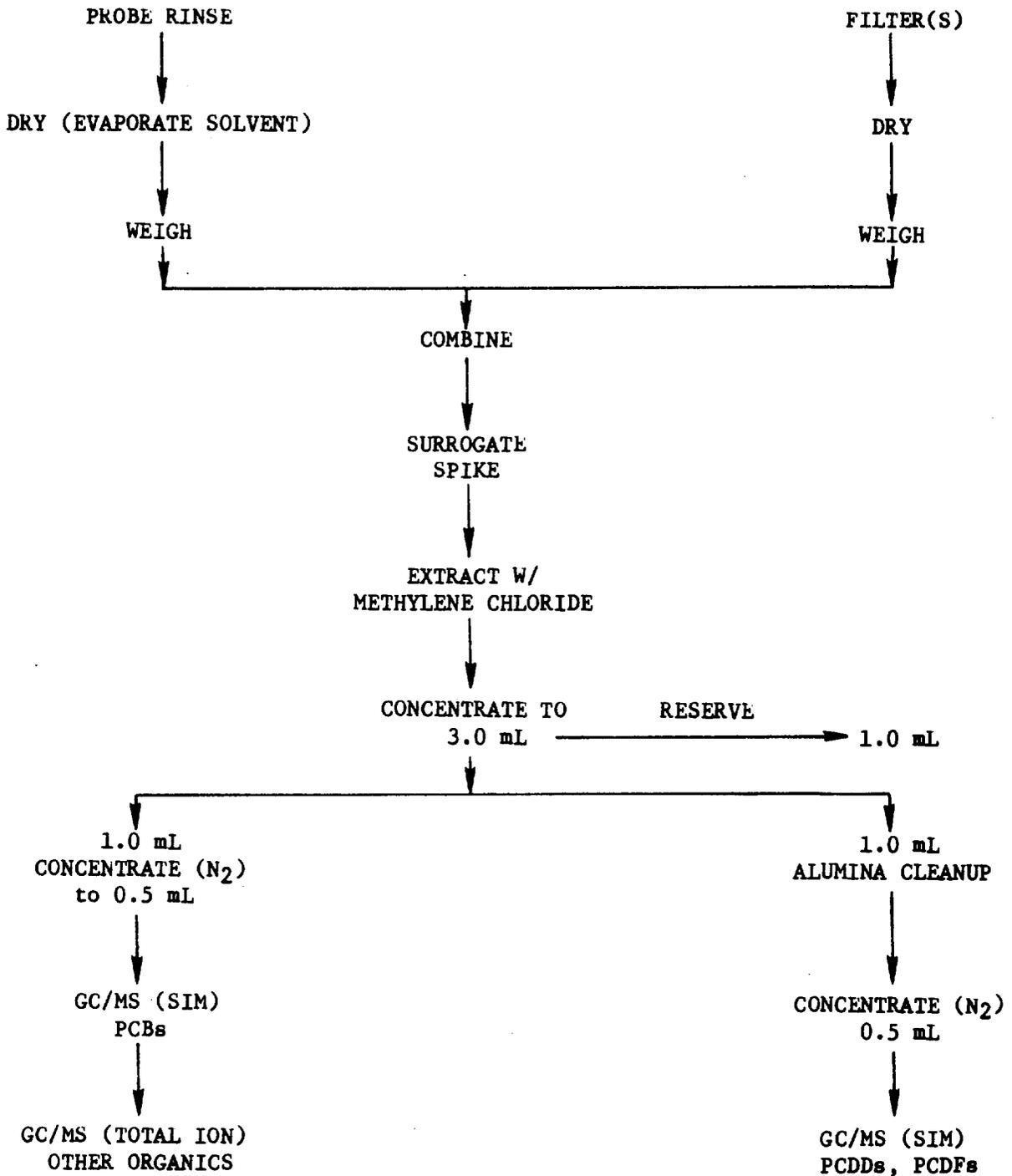


Figure 14. Organic analysis flow scheme--particulate phase flue gas samples.

TABLE 19. GC/ECD CONDITIONS FOR PCB ANALYSIS

Instrument	Hewlett-Packard 5840A with Ni <sup>63</sup> electron capture detector and HP 7671A automatic sampler
Column	3% SE-30 on 80/100 Chromosorb WHP, 6 ft x 2 mm
Temperatures	
Column	185°C (Aroclors 1254, 1260), 175°C (Aroclor 1242)
Injector	270°C
Detector	350°C
Injector volume	4.0 µl
Run time	30 min
Carrier flow	UHP argon/methane, 35 ml/min

components were used. Each combined extract was further reduced in volume under a gentle stream of prepurified  $N_2$  to 100  $\mu$ l. GC/MS analyses provided results on a ppm (mg/kg) basis for each of the positional isomer categories.

#### Hopper Ash Samples

Three grab samples were taken from the hopper during each test run. As noted earlier they were taken at approximately 1 hour intervals during each test run.

Twenty-five (25 g) gram aliquots were taken from each of the three grab samples and composited resulting in a 75 g composite sample for each test series. Each composite sample was transferred to an "F" size soxhlet thimble and fortified with a surrogate cocktail containing the following components:  $d_3$ -trichlorobenzene,  $d_{10}$ -biphenyl,  $d_{12}$ -chrysene and decafluorobiphenyl (DFB). Samples were extracted overnight with methylene chloride. Extracts were reduced in volume to 10.0 ml using a Kuderna-Danish evaporative concentrator. As was the case with the flue gas particulate samples (Figure 13), a 5.0 ml aliquot was reserved. The remaining 5.0 ml portion was further reduced to 0.5 ml employing a gentle stream of prepurified  $N_2$  for GC/MS analyses. GC/MS analyses were conducted in the selected ion (SIM) mode for isomers of PCBs, PCDFs and PCDDs. GC/MS analyses for additional organics, not members of the above chemical classes, were conducted in the total ion mode. Additional cleanup procedures were implemented as needed to permit unambiguous identification of PCDD, PCDF and PCB isomer classes. This included the use of acid partitioning ( $H_2SO_4$ ) and alumina column chromatography. All results from GC/MS analyses are reported in units of ppb or  $\mu$ g/kg of hopper ash.

#### Aqueous Samples--GC ECD

Analyses were conducted on the following types of water samples collected during each test series.

- Precooler/Scubber Water Feed--3 grab samples per run. These were composited so as to create a single representative sample per run (e.g., one liter total).

- Centrifuge Water--3 grab samples per run. These were composited into a single sample per run.
- Plant Influent Wastewater--3 grab samples per run. Each of these samples were prepared and analyzed separately.

One liter aliquots of each of the above sample types were extracted in a separatory funnel with methylene chloride as recommended in EPA Method 608. Each extract was dried using a sodium sulfate column and reduced in volume to 10.0 ml using a Kuderna-Danish apparatus. A 5.0 ml aliquot of each sample was removed for PCB analysis (pattern matching) employing a gas chromatograph in combination with an electron capture detector. Florisil column chromatography procedures were employed as needed to facilitate identification and quantitation of aroclor (PCB) mixtures. Results of all GC/ECD analyses are provided in units of  $\mu\text{g}/\text{l}$  (ppb) of Aroclor 1242, 1254 or 1260 using the Webb and McCall quantitation procedure cited previously.

#### Aqueous Samples--GC/MS

Each of the five aqueous sample streams from each test series were analyzed further using gas chromatography/mass spectrometry. Details on additional sample preparation procedures are provided below.

##### Scrubber Effluent--

A 5.0 ml aliquot from each 10.0 ml extract was reduced under a stream of prepurified  $\text{N}_2$  to 100  $\mu\text{l}$ , GC/MS analyses were conducted for each of ten PCB positional isomer categories. All results are provided in units of  $\mu\text{g}/\text{l}$  (ppb).

##### Plant Influent Wastewater--

A single composite sample was prepared from the three grab samples taken during each run. A one liter composite sample representing each test series was fortified with a surrogate mixture and extracted (3X) with methylene chloride per EPA Method 625. Each of the acid and base-neutral fractions was reduced in volume to 1.0 ml in a Kuderna-Danish apparatus. The concentrations were combined and a 1.0 ml aliquot was removed for GC/MS analyses as described later in this section.

#### Scrubber Water/Precooler Feed and Centrifuge Water--

Samples from the above categories that contained elevated levels of PCBs (GC/ECD) were further analyzed using GC/MS to include: qualitative analysis for PCBs (presence or absence confirmation). In this instance 1/2 (e.g., 5.0 ml) of the existing extract was reduced in volume using N<sub>2</sub> to 100 µl for PCB confirmation in the total ion mode. In addition each of these extracts simultaneously underwent GC/MS analyses for the identification and quantitation of major organic components with particular emphasis on chlorinated organics (e.g., chlorinated pesticides, chlorinated benzenes, chlorinated naphthalenes (PCNs)).

#### GC/ECD ANALYSIS--PCBs

PCB analyses of all previously designated sample extracts were conducted using a Hewlett-Packard 5840 gas chromatograph fitted with a Ni<sup>63</sup> electron capture detector. This includes each of the aqueous sample types discussed previously, as well as the sewage sludge feed grab samples collected during each test series. Instrument calibration was provided using Aroclor 1242 and Aroclor 1254 reference materials. (Initial qualitative GC/ECD analyses of samples had indicated that these Aroclors were the predominant species). EPA/EMSL check samples were analyzed to verify instrument calibration for each of these Aroclor mixtures. Webb-McCall quantitative procedures were used for all samples because chromatographic profiles did not coincide with those of the aroclor reference materials. Results for all aqueous samples are provided in units of µg/l while those of the sludge feed samples are provided on a mg/kg (ppm) dry weight basis.

#### GC/MS ANALYSIS

##### Polychlorinated Biphenyls (PCBS)

Analyses for PCBs as positional isomer categories were conducted using a Hewlett-Packard 5985 quadruple mass spectrometer (GC/MS). A summary of instrument operating conditions pertinent to these analyses is contained in

Table 20. Each of the extracts noted earlier was spiked with an internal standard solution containing  $d_8$ -naphthalene and  $d_{10}$ -anthracene. Spectra were acquired in the continuous scan mode over the range of 45 to 450 amu.

Instrument calibration was established using at least one isomer from each of ten PCB positional isomer categories. Standard reference materials, obtained from ultrascientific (Hope, R.I.), were used to establish pertinent chromatographic (RT, RRT) and mass spectral (RF) identification criteria for each of these positional isomer categories.

Mass spectral response factors relative to the closest eluting internal standard were determined for each of the compounds listed in Table 21. Serial dilutions of each stock calibration mixture ranging in concentration from 10 ng/ l to 400 ng/ l were used to establish the working calibration curve prior to sample analysis. Individual component response factors (RF) were derived from the following mathematical relationship:

$$RF = \frac{A_x W_{IS}}{A_{IS} W_x}$$

where  $A_x$  is the area of the quantitative ion of compound x,  $W_x$  is the quantity in ng eliciting the area response,  $A_{IS}$  is the area of the quantitative ion of the appropriate internal standard ( $d_8$ -naphthalene  $m/e = 136$ ,  $d_{10}$ -anthracene  $m/e = 188$ ) and  $W_{IS}$  is the quantity in ng eliciting the area response. Average component response factors are provided in Table 21.

The identification and quantitation of polychlorinated biphenyls (e.g., Aroclor mixtures) were based upon the analysis of the positional isomers listed in Table 21. These isomers, representing unit increments in chlorine substitution from mono- to decachlorobiphenyl were used to establish a retention time window for each isomer group. The characteristic ions for each isomer group were determined from the mass spectral data obtained during the analysis of standard reference materials. Data obtained for each sample was then reviewed for the presence of the chlorine isotope clusters typical of PCB compounds. The extracted ion profiles for the primary ions of all PCBs were obtained. All peaks noted on the extracted ion profile for a given isomer group falling within the assigned retention time window for that isomer group were then examined.

TABLE 20. GC/MS OPERATING CONDITIONS

---

Instrument	Hewlett Packard 5985, quadruple mass spectrometer
GC Conditions	
Column	DB-5 30M fused silica capillary (or equivalent)
Temperature program	70°C held for 2 min then 15°/min to 300°C and held
Injector type	Grob w/0.5 min sweep time
Injector temperature	265°C
Injection volume	1 $\mu$ l, splitless
Column flow	UHP helium, 0.3 ml/min
MS Conditions	
Emission	300 $\mu$ A
Electron energy	70 eV
Scan time	1.0 s/scan
Mass interval	45 to 450 amu
Source temperature	200°C

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TABLE 21. SUMMARY OF MASS SPECTRAL IDENTIFICATION CRITERIA--PCB POSITIONAL ISOMERS

Compound	Quantitative ion	Internal standard <sup>a</sup>	Typical RT (minutes)	Average RFB
2-chlorobiphenyl	186	d <sub>10</sub>	14.7	0.159
4-chlorobiphenyl	186	d <sub>10</sub>	15.9	0.260
2,5-dichlorobiphenyl	186	d <sub>10</sub>	18.1	0.505
3,3'-dichlorobiphenyl	186	d <sub>10</sub>	19.5	0.495
2,3',5-trichlorobiphenyl	256	d <sub>10</sub>	19.8	0.121
2,4,5-trichlorobiphenyl	256	d <sub>10</sub>	19.8	0.120
2,3'4',5-tetrachlorobiphenyl	292	d <sub>10</sub>	22.4	0.051
2,2'4,5,5-pentachlorobiphenyl	326	d <sub>10</sub>	23.1	0.029
2,2'4',4',6,6'-hexachlorobiphenyl	326	d <sub>10</sub>	22.8	0.023
2,2'3,4,5,5',6-heptachlorobiphenyl	394	d <sub>10</sub>	26.6	0.007
2,2'3,3',4,4',5,5'-octachlorobiphenyl	179	d <sub>10</sub>	29.0	0.005
2,2'3,3',4,5,5',6,6'-nonchlorobiphenyl	197	d <sub>10</sub>	30.0	0.017
2,2'3,3',4,4',5,5',6,6'-decachlorobiphenyl	214	d <sub>10</sub>	31.0	0.013

<sup>a</sup>Response factors calculated relative to most closely eluting internal standard, either d<sub>8</sub>-naphthalene or d<sub>10</sub>-anthracene.

<sup>b</sup>Response factor of component calculated versus the appropriate internal standard using auto integration program.

The values shown here represent average component response factors.

Assignments to a specific isomer group were made upon satisfying the following criteria: (a) the primary ions were present as part of a chlorine isotope cluster, (b) additional ions characteristic of the isomer group were present in the component spectra at the expected relative intensities, and (c) the balance of the spectra was consistent with chlorine substitution on a biphenyl molecule. Peaks satisfying these criteria were assigned to the appropriate positional isomer group and quantitated using the response factor (RF) generated for the reference material with the same molecular mass as the component peak. Additionally, a GC/MS analysis in the total ion (TI) mode was performed to confirm the presence of PCB detected in selected ion monitoring (SIM) analyses. This confirmation was necessitated by the complexity of the sample matrices encountered.

#### Polychlorinated Dibenzodioxins/Polychlorinated Dibenzofurans (PCDDs/PCDFs)

Analyses for PCDDs/PCDFs as positional isomer categories were conducted using a Hewlett-Packard 5985 quadrupole mass spectrometer. Instrument operating conditions were identical to those followed for PCB analysis with the exceptions that spectra were acquired in the selected ion mode (SIM). Instrument calibration was established using a series of PCDD and PCDF positional isomer as listed in Table 22. These isomers were used to establish pertinent chromatographs and mass spectral identification criteria for each of eight PCDD and PCDF positional isomer categories. As was the case with the PCB analyses the PCDD/PCDF isomer listed in Table 22 were used to establish retention time windows for each of the positional isomer categories. A summary of these retention time windows and the characteristic ions (SIM) related for each category is provided in Table 23.

Criteria for assignment to a specific isomer group followed the protocol provided earlier for PCB isomers. Component concentrations were provided using the response factor (RF) generated for the reference material with the same molecular mass as the component peak. Additional confirmatory analyses were performed as stated previously for PCB isomers.

TABLE 22. SUMMARY LISTING OF POLYCHLORINATED DIBENZOFURAN (PCDF) AND POLYCHLORINATED DIBENZODIOXIN (PCDD) REFERENCE MATERIALS

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Dibenzofuran

Dibenzo-p-dioxin

1-Chlorodibenzo-p-dioxin

2-Chlorodibenzo-p-dioxin

2,3,-Dichlorodibenzo-p-dioxin

2,7-Dichlorodibenzo-p-dioxin

1,2,4-Trichlorodibenzo-p-dioxin

1,2,3,7,8-Pentachlorodibenzo-p-dioxin

1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin

Heptachlorodibenzo-p-dioxin

Octachlorodibenzo-p-dioxin

2,7-Dichlorodibenzofuran

Octachlorodibenzofuran

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TABLE 23. GC/MS SIM ANALYSIS FOR DIBENZOFURANS AND DIBENZO-P-DIOXINS---  
COMPONENT IDENTIFICATION CRITERIA

Retention time window	Isomer class	Ions monitored	Mass dwell time (milliseconds)
10.0 - 12.5 minutes	Dibenzofuran	168 139	100
	Dibenzo-p-dioxin	184 128	100
	Chlorodibenzofurans	202 204	100
	Chlorodibenzo-p-dioxins	218 220 155	100
12.5 - 15.2 minutes	Dichlorodibenzofurans	236 238	100
	Dichlorodibenzo-p-dioxins	252 254 189	100
	Trichlorodibenzofurans	270 272	100
	Trichlorodibenzo-p-dioxins	288 290 218	100
15.2 - 23.0	Tetrachlorodibenzofurans	304 306 308	80
	Tetrachlorodibenzo-p-dioxins	320 322 257	80
	Pentachlorodibenzofurans	338 340 342	80
	Pentachlorodibenzo-p-dioxins	356 358 293	80
23.0 - 25.0	Hexachlorodibenzofurans	372 374	150
	Hexachlorodibenzo-p-dioxins	388 390	150
	Heptachlorodibenzofurans	406 408 410	150
	Heptachlorodibenzo-p-dioxins	424 426	150
25.0 - 27.5	Octachlorodibenzofuran	440 442 444	150
	Octachlorodibenzo-p-dioxin	458 460 462	150

## Other Organics

The identification and quantitation of additional chlorinated organic components not classified as PCBs, PCDDs or PCDFs followed the criteria noted below.

A background-corrected spectrum of the component was first obtained by computer subtraction. A probability-based library search (PBS) was then conducted by computer routine, comparing the unknown spectra with those of the EPA/NIH libraries. The computer search provided up to 10 possible matches. The spectra of library matches were compared directly to the unknown spectrum by the operator. For positive identification, the following conditions were required:

- The intensity, relative to the base peak, of all major peaks (greater than 50 percent of base peak) agreed within 20 percent.
- All peaks present in the library spectrum at more than 20 percent of the base peak were present in the unknown spectrum.
- The unknown spectrum must not have any peaks present more than 30 percent of the base peak that are not seen in the library spectrum or are not clearly attributable to coeluting compounds.

If the library search did not provide a positive match, the unknown spectra were reviewed for major peaks and fragmentation patterns. Tentative identifications were made by the operator and verified by comparison of available reference spectra (EPA/NIH libraries) to the background corrected component spectra. Component concentrations were calculated relative to the closest eluting internal standard. All values represent approximations due to inherent variabilities in component response factors in the absence of reference materials.

## SECTION 6

### QUALITY ASSURANCE

#### INTRODUCTION

This section briefly summarizes QA/QC activity on this project since January 1984. Subsections deal with Sampling and Field Measurements QC, Laboratory Analysis QC, and Performance and System Audits. To curtail the size of this section, most raw QC data (instrument calibration data sheets, custody forms, analysts notebook pages, etc.) have been omitted. However, the Analytical QC subsection contains data reports for most, but not all, of the QC samples specific to this project.

A Sampling and Analysis Test Plan and a Quality Assurance Project Plan were initially prepared for this test series in 1982. These plans were subsequently revised in December 1983 and January 1984, respectively. The revised Sampling and Analysis Plan appears in Appendix A of this report and the QA project plan appears in Appendix B. A system audit was conducted in February 1984 to check on adherence to this QA Plan; the summary audit report is included in this section.

#### SAMPLING AND FIELD MEASUREMENTS QUALITY CONTROL

Quality control procedures implemented during the field sampling deal mainly with equipment calibration, sampling procedures, field data reduction, and sample chain-of-custody. Specific activities are summarized below.

##### Equipment Calibration

Equipment and instrumentation used during the project participated in GCA's routine preventive maintenance and calibration program. Calibration of

the equipment used for reference method testing was performed in accordance with the procedures outlined in the EPA publication "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III: Stationary Source Specific Methods." Calibration work sheets for the dry gas meters, orifices, pitot tubes, thermocouples and sample nozzles are presented in Appendix D of this report. Calibration of the carbon monoxide and oxygen continuous monitors was performed at the beginning and end of each one hour test run. All calibrations were within tolerance.

Gravimetric determinations were performed in GCA's environmentally controlled balance room. Room conditions are maintained at less than 50 percent relative humidity and at 15° to 25°C; actual conditions are recorded daily by the balance room technician. The Mettler H54 balance was used to weigh particulate filters and the Mettler H-15 balance was used to weigh the front-half rinses that were dried down in beakers. Each balance was checked with Class S weights in the approximate weight range of the sample before and after each weighing session. Blanks were weighed with each set of samples and were included in all final calculations for weight determinations.

Additional presampling activities included preparation of all sample containers to be used in the test series. Sample glassware and containers were specifically precleaned using the following wash and rinse sequence to minimize the possibility of inadvertent sample contamination.

1. Soap and water wash;
2. Tap water rinse;
3. No-chromic acid soak;
4. DI water rinse;
5. Acetone rinse;
6. Hexane rinse; and
7. Air dry and seal with precleaned foil.

A Field/Laboratory Procedure Coordination Form was completed to ensure: proper sample container selection, use of approved QC'd reagents and clear sample identification. A copy of this form is provided in Appendix D.

### Sampling Procedures and Field Data Reduction

The personnel involved in the field activities of this project implemented sampling procedures in accordance with GCA's Sampling and Analysis Test Plan, QA Project Plan and EPA protocols. Leak checks of the sampling system and its components were performed prior to and at the end of each run. This information and all other pertinent sampling data were recorded on designated sampling forms.

The percent isokinetic flow was calculated manually in the field using standardized calculation forms to ensure valid sampling conditions. The majority of the source sampling data was reduced using a Texas Instruments TI-59 Desk Top Calculator with printer at GCA. These data were checked by two members of the field crew before being submitted to the field team leader for use in the individual summary reports.

Field-biased blanks of all reagents and sorbent resins were collected. These samples were prepared and handled in manners identical to those used when handling the actual samples. This procedure aids in identifying areas of contamination from the field. Method blanks were also collected. An in-depth discussion of these procedures is presented in the QA Project Plan.

### Chain-of-Custody Procedures

Chain-of-custody of all samples collected was initiated in the field. All samples were clearly labeled, sealed and their level marked prior to storage for shipment to GCA. Each sample was listed on the packing sheet/custody form to document its collection and the responsible field sample custodian. Sample codes, as designated on the Field/Laboratory Coordination Form and in the Test/QA Plan were followed throughout the program. At GCA, custody of the samples was transferred to the Sample Bank Manager who logged the samples in the Master Log and assigned a GCA Control Number to each.

sample. This control number identified each sample throughout all further handling and analysis. For the New Bedford project, these control numbers were 36233 to 36335.

#### LABORATORY ANALYSIS QUALITY CONTROL

Quality Control (QC) protocols implemented for this program, as specified in the Program Test and Quality Assurance Plan, included the use of method blanks, field-biased blanks, laboratory control spikes, and a series of deuterated spikes contained in each sample designated for GC/MS analysis. A brief synopsis of each of these control elements is provided below. A more detailed discussion of each element, including results for each analytical category, is provided in subsequent portions of this section.

#### Blank Samples

##### Method Blanks--

Appropriate method blanks containing all reagents used in the preparation and analysis of samples were processed through the entire analytical scheme to assess spurious contamination arising from reagents, glassware and other materials used in the analysis. No PCBs, PCDDs, or PCDFs were detected in laboratory method blanks.

##### Field-Biased Blanks--

Blank filters and reagents used in the collection of samples accompanied actual samples during transfer in the field and transfer to the sample bank for storage to account for contamination which might be introduced during these operations. No PCBs, PCDDs, or PCDFs were detected in field-biased blanks.

##### Calibration Blanks--

These are blanks which are used in instrument calibration and contain all reagents used in the preparation of calibration standards except the standard itself.

### Laboratory Control Samples

Laboratory Control Samples (LCS) include both replicate samples and spiked samples (i.e., those fortified with predetermined quantities of the components of interest) which accompany program samples throughout the analytical scheme. Results from the analysis of control samples can provide information on analytical precision and spike recovery.

### Instrument Check Samples

These samples are usually prepared from an EPA/EMSL concentrate or other suitable reference materials and are used on a daily basis to verify existing instrument calibration. Results from the analysis of these check samples are entered in the instrument logbook.

### Surrogate Spikes

Samples requiring analysis by GC/MS are routinely surrogate-spiked with a series of deuterated analogues of the components of interest. During this program, each sample requiring analysis for PCBs, PCDDs and PCDFs was spiked with a mixture containing  $d_3$ -trichlorobenzene,  $d_{10}$ -biphenyl, and  $d_{12}$ -chrysene and decafluorobiphenyl. It was anticipated that these compounds would assess the behavior of actual components in individual program samples during the entire preparative and analysis scheme. However, it should be noted that recent data from an interlaboratory study of semivolatile compounds do not indicate strong relationships between surrogate recoveries and recoveries of the components of interest (EPA Method Study 30; EPA 600/4-84-053).<sup>18</sup>

### Sludge Feed

GC/ECD--

Total PCB-concentrations in the sludge feed were determined by GC/ECD as designated in the revised Test Plan (Appendix A). One sludge sample with a measurable concentration of PCBs was run in triplicate. The results of these

analyses are presented in Table 24. It can be seen from this table that the relative standard deviation (RSD) is less than 10 percent for this analysis.

#### GC/MS--

Sludge samples to be analyzed by GC/MS were spiked with a surrogate mixture prior to concentration of the composite extract. As noted earlier this surrogate mixture contained  $d_3$ -trichlorobenzene,  $d_{10}$ -biphenyl,  $d_{12}$ -chrysene and decafluorobiphenyl. Surrogate recoveries for each of the samples are presented in Table 25. In addition, duplicate solvent spikes of chlorobiphenyl isomers were prepared and analyzed with the sludge samples concentrates. Results of GC/MS analysis of solvent spikes are shown in Table 26.

Surrogate recoveries in the sludge extracts seem to have been biased positively by the presence of background interference in the GC/MS total ion mode. Recoveries of chlorobiphenyl isomers average 88 percent for the mono- through heptachlorobiphenyls and 36 percent for the higher substituted isomers. This decrease in recovery for the latter may be attributed to the poor chromatographic behavior of the higher molecular weight compounds. Recoveries for chlorobiphenyl isomers were not effected by interferences as were the earlier eluting surrogate compounds.

#### Water Samples--

PCB concentrations in the precooler/scrubber water feed, the scrubber water effluent, the centrifuge water and the plant influent wastewater were analyzed for total Aroclor PCBs using GC/ECD. The GC/ECD served as a screening tool for GC/MS analysis. Only the scrubber effluent, however, was analyzed by GC/MS for each of ten positional isomer categories. Quality control techniques employed on these samples included two method blanks, spiking of two aqueous samples with EPA/EMSL WP 679 concentrates containing Aroclor 1016 or Aroclor 1254, and spiking of one sample with the same chlorobiphenyl isomer mixture used for the sludge samples. Recovery efficiencies of the EPA/EMSL mixture are reported in Table 27, were well within the 95 percent confidence interval established by EPA. The chlorobiphenyl data presented in Table 28, indicate average recoveries of 72 to 120 percent.

TABLE 24. QUALITY CONTROL RESULTS. TRIPLICATE ANALYSIS  
OF SLUDGE SAMPLE (GCA 36299)--GC/ECD

Relative standard deviation (%)		
Aroclor 1242	Aroclor 1254	Aroclor 1260
8.1	7.1	ND

ND = None detected (<1 mg/kg)

TABLE 25. QUALITY CONTROL RESULTS--SLUDGE SAMPLE SURROGATE RECOVERIES

GCA control no.	Run no.	Surrogate recovery (%) <sup>a</sup>			Decafluoro- biphenyl
		d <sub>3</sub> -trichloro- benzene	d <sub>10</sub> -biphenyl	d <sub>12</sub> -chrysene	
36292	2	110	140	240	160
36298	3	130	150	400	160
36305	4	130	200	220	160
Average percent recovery (%)		120	160	290	160
Standard deviation (s <sub>x</sub> )		12	32	99	0
Relative standard deviation, %		10	20	34	0

<sup>a</sup>Approximately 10 µg of each component was spiked onto the sludge extract.

TABLE 26. QUALITY CONTROL RESULTS--SLUDGE SAMPLE  
CHLOROBIPHENYL RECOVERIES

Component	Quantity added (µg)	Quantity recovery (g)				Average recovery (%)
		A	B	C	X	
Chlorobiphenyl	50	47	42	96 <sup>a</sup>	44	88
Trichlorobiphenyl	28	28	21	100 <sup>a</sup>	24	86
Tetrachlorobiphenyl	26	32	24	99 <sup>a</sup>	28	108
Pentachlorobiphenyl	21	20	14	67 <sup>a</sup>	17	81
Hexachlorobiphenyl	26	26	14	77 <sup>a</sup>	20	77
Heptachlorobiphenyl	8.7	2.4	1.4	5.1 <sup>a</sup>	1.9	22
Nonachlorobiphenyl	23	5.8	5.1	13 <sup>a</sup>	5.4	23
Decachlorobiphenyl	21	6.4	2.4	2.8	13	62

<sup>a</sup>Data rejected as outlier based on Dixon's test for extreme observations.<sup>17</sup>

TABLE 27. QUALITY CONTROL RESULTS--GC/ECD ANALYSIS OF EPA/EMSL  
WP 679 CONCENTRATES, AQUEOUS SAMPLES

Component	Percent recovery	
	Concentrate No. 1	Concentrate No. 11
Aroclor 1016	70	a
Aroclor 1254	a	89

<sup>a</sup>Not a component of EPA/EMSL concentrate.

TABLE 28. QUALITY CONTROL RESULTS--GC/MS ANALYSIS  
OF AQUEOUS SAMPLES CHLOROBIPHENYL ISOMER SPIKES

Component	Total $\mu$ g				Average recovery (%)
	Sample 1		Sample 2		
	Expected	Recovered	Expected	Recovered	
Chlorobiphenyl	170	180	340	230	87
Trichlorobiphenyl	92	110	180	120	93
Tetrachlorobiphenyl	87	100	170	140	98
Pentachlorobiphenyl	69	81	140	100	94
Hexachlorobiphenyl	85	100	170	130	97
Heptachlorobiphenyl	29	42	60	56	120
Nonachlorobiphenyl	78	96	160	150	110
Decachlorobiphenyl	71	47	140	110	72

#### Ash--

Sludge ash samples were analyzed for each of ten positional PCB isomer categories, each of eight positional PCDD/PCDF isomer categories and other organics not members of these organic classes (i.e., other chlorinated organics, polynuclear aromatics, etc). All analyses were conducted employing electron impact GC/MS. Quality control procedures included one method blank, one replicate sample, surrogate spikes of each sample using the four components noted earlier and two lab control spikes containing a series of PCB, PCDD and PCDF positional isomers. The lab spikes contained a representative mixture of each of these organic classes (i.e., complete range of volatility and chlorine substitution). Unfortunately, none of the components of interest were detected. The surrogate spike recovery efficiencies are presented in Table 29. Lab control spike data for one sample are presented in Table 30. The duplicate spike was inadvertently lost during the preparatory stage of the analysis.

#### Flue Gas Samples--

Particulate Samples--Analyses of the particulate filter and the probe rinse (front half rinse) was conducted after particulate weights had been taken by field personnel for particulate emission rate calculations. Quality control samples associated with this analysis included one field-biased blank per run, one method blank, surrogate spikes of each sample with the four components previously described and two lab control spikes, which were prepared with a series of PCB, PCDD and PCDF positional isomers. Surrogate spike recovery efficiencies for these samples are presented in Table 31, while lab control spike data are presented in Table 32. As footnoted in Table 31, two of the sample extracts went dry after PCB analysis was complete, but prior to surrogate analysis. The recoveries obtained for the third sample are consistent with those reported in a previous study.<sup>5</sup>

Sorbent Samples--Each flue gas sampling train contained the following samples; back half rinse (acetone/hexane), organic condensate, florisil sorbent, XAD-2 sorbent, aqueous impinger (NaOH), and aqueous impingers.

TABLE 29. QUALITY CONTROL RESULTS--ASH SAMPLE SURROGATE RECOVERIES

GCA control no.	Run no.	Surrogate recovery (%) <sup>a</sup>			
		d <sub>3</sub> -trichloro- benzene	d <sub>10</sub> -biphenyl	d <sub>12</sub> -chrysene	Decafluoro- biphenyl
36312	2	59	87	47	88
36315	3	63	82	102	62
36318	4	50	97	83	74
Average percent recovery (%)		57	89	77	75
Standard deviation (s <sub>x</sub> )		6.6	7.6	28	13
Relative standard deviation, %		12	8.5	36	17

<sup>a</sup>20 to 50 µg of each component was spiked onto the ash.

TABLE 30. QUALITY CONTROL RESULTS--GC/MS ANALYSIS OF LABORATORY CONTROL SPIKE PREPARED WITH ASH SAMPLES

Component	Quantity spiked (µg)	Quantity recovered (µg)	Percent recovery
Chlorobiphenyl	86	83	96
Trichlorobiphenyl	84	51	61
Tetrachlorobiphenyl	44	45	100
Pentachlorobiphenyl	34	28	82
Hexachlorobiphenyl	42	43	100
Heptachlorobiphenyl	14	39	280
Nonachlorobiphenyl	39	100	260
Decachlorobiphenyl	36	83	230
Chlorodibenzodioxin	140	200	140
Dichlorodibenzodioxin	94	140	150
Trichlorodibenzodioxin	82	61	74
Tetrachlorodibenzodioxin	72	42	58
Dichlorodibenzofuran	24	21	88

TABLE 31. QUALITY CONTROL RESULTS--FLUE GAS PARTICULATE  
SAMPLE SURROGATE RECOVERIES

GCA control no.	Run no.	Surrogate recovery (%) <sup>a</sup>			
		d <sub>3</sub> -trichloro- benzene	d <sub>10</sub> -biphenyl	d <sub>12</sub> -chrysene	Decafluoro- biphenyl
36239	2 <sup>b</sup>	42	8	39	60
36240	3 <sup>b</sup>	14	74	54	15
36241	4	68	130	120	84
Average percent recovery (%)		41	71	71	53
Standard deviation (s <sub>x</sub> )		27	34	43	35
Relative standard deviation, %		66	37	60	66

<sup>a</sup>45 to 60 µg of each component was spiked onto the particulate.

<sup>b</sup>Sample extract went dry prior to analysis for surrogate recoveries, but subsequent to PCB S/M analysis.

TABLE 32. QUALITY CONTROL RESULTS--GC/MS ANALYSIS OF  
FLUE GAS PARTICULATE SPIKES

Component	Quantity spiked ( $\mu\text{g}$ )	Quantity recovered ( $\mu\text{g}$ )		Percent recovery	
		Sample 1	Sample 2	Sample 1	Sample 2
Chlorobiphenyl	86	170	72	200	84
Trichlorobiphenyl	84	140	120	170	140
Tetrachlorobiphenyl	44	120	87	270	200
Pentachlorobiphenyl	34	100	61	290	180
Hexachlorobiphenyl	42	120	90	280	210
Heptachlorobiphenyl	14	23	26	160	180
Nonachlorobiphenyl	39	30	51	77	130
Decachlorobiphenyl	36	82	100	270	280
Chlorodibenzodioxin	140	69	61	49	44
Dichlorodibenzodioxin	94	170	91	180	97
Trichlorodibenzodioxin	82	180	190	220	230
Tetrachlorodibenzodioxin	72	100	120	170	170
Dichlorodibenzofuran	24	41	44	170	180

Quality control measures associated with these samples included one complete method blank comprised of each sample type (i.e., sorbent, water, etc.), two sets of field-biased blanks, surrogate spikes of each of the sorbent tubes (florisil, XAD-2) using the four surrogate components previously discussed and five laboratory control spikes containing a series of PCB, PCDD and PCDF positional isomers. Two sets of these lab control spikes were added to the florisil and XAD-2 sorbent samples; while the remaining 3 sets of spiked tubes are being held for a sampling stability study, which is subsequently discussed. Surrogate spike recovery data are presented in Table 33, while the two sets of lab control spike recovery efficiencies are documented in Table 34.

### Sample Stability Spikes

A small scale storage stability study was conducted to assess potential effects of long-term sample storage conditions on the flue gas samples. Three sets of spiked tubes containing components of interest; PCBs, PCDDs and PCDFs (analytes from each of ten PCB positional isomer categories) were placed in refrigerated storage for a period of 150 days. Storage conditions were identical to those for samples themselves during the February to July time period of this year. These samples were subsequently analyzed by GC/MS. Actual train sorbent tubes were used for this study which provides an evaluation of component depreciation due to long-term storage. In addition, aliquots of the same spiking solution (both surrogate and component mixtures) were stored under the same conditions. Results of this study are presented in Table 35.

The persistent nature of the components of interest makes it unlikely that any significant loss will have occurred; however, the spiking solution includes ten PCB positional isomers so that the stability of positional isomer categories can be verified. The analytical protocol for the sorbent tube samples includes a solvent rinse of the glass tubes which has been demonstrated to effectively recover PCBs adsorbed onto glass sample containers.<sup>19</sup> The analysis of the stored spiking solution will provide data relevant to other stored sample extracts, as well as to the sorbent tube samples.

TABLE 33. QUALITY CONTROL RESULTS--FLUE GAS SORBENT SAMPLE RECOVERIES

GCA control no.	Run no.	Surrogate recovery (%) <sup>a</sup>							
		d <sub>3</sub> -trichloro- benzene		d <sub>10</sub> -biphenyl		d <sub>12</sub> -chrysene		Decafluoro- biphenyl	
		Florisol	XAD-2	Florisol	XAD-2	Florisol	XAD-2	Florisol	XAD-2
36268	2	90		130		91		160	
36269	3	95		74		100		90	
36270	4	120		52		120		130	
Average percent recovery (%)		102		85		104		130	
Standard deviation (s <sub>x</sub> )		16		40		15		35	
Relative standard deviation, %		16		47		14		27	

<sup>a</sup>10 µg of each component was spiked onto each sorbent tube.

TABLE 34. QUALITY CONTROL RESULTS--GC/MS ANALYSIS  
OF FLUE GAS SORBENT SAMPLES

	Quantity	Quantity recovered		Percent recovery	
		Sample 1	Sample 2	Sample 1	Sample 2
Chlorobiphenyl	86	110	130	130	150
Trichlorobiphenyl	84	77	70	92	83
Tetrachlorobiphenyl	44	40	62	91	140
Pentachlorobiphenyl	34	32	50	94	150
Hexachlorobiphenyl	42	48	63	110	150
Heptachlorobiphenyl	14	24	19	170	140
Nonachlorobiphenyl	39	48	42	120	110
Decachlorobiphenyl	36	22	34	61	94
Chlorodibenzodioxin	140	93	150	70	110
Dichlorodibenzodioxin	94	230	140	240	150
Trichlorodibenzodioxin	82	150	120	180	150
Tetrachlorodibenzodioxin	72	150	76	210	100
Dichlorodibenzofuran	24	51	51	210	210

TABLE 35. QUALITY CONTROL RESULTS--PCB, PCDD, PCDF STORAGE STABILITY STUDY--GC/MS

Component	Quantity spiked (µg)	Quantity recovered--florisil			Average percent recovery--florisil			Quantity recorded XAD-2			Average percent recovery XAD-2		
		Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
Chlorobiphenyl	98												
Trichlorobiphenyl	84												
Tetrachlorophenyl	44												
Pentachlorobiphenyl	34												
Hexachlorobiphenyl	42												
Heptachlorobiphenyl	14												
Nonachlorobiphenyl	39												
Decachlorobiphenyl	36												
Chlorodibenzodioxin	140												
Dichlorodibenzodioxin	94												
Trichlorodibenzodioxin	82												
Tetrachlorobenzodioxin	72												
Dichlorodibenzofuran	24												

## PERFORMANCE AND SYSTEM AUDITS

### Sampling and Measurements Performance Audits

No GCA performance audits of sampling and field measurement were conducted. However, the EPA 0982 Method 5 audit was completed in June 1983. A mean percent different of 2.4 percent between GCA's reported values and EPA's expected values was achieved for the five dry gas meters checked during this audit.

### Laboratory Analysis Performance Audits

Several sets of QC samples prepared at GCA by spiking compounds of interest into the flue gas, sludge and ash matrices served to audit the accuracy of analytical work. Although identified as QC samples, their concentrations were not known to the analyst. In addition, EPA Quality Control Concentrations or NBS Standard Reference Materials were used to assess the analytical work. The results achieved on these samples are presented in this section.

### System Audit

A system audit is an overall evaluation of a project to check on the use of appropriate QC measures and ensure that the quality system planned for the project has been implemented. Such an audit of sampling and field measurements was conducted by GCA's QA Staff in February 1984. The summary audit report is presented in this subsection.

GCA TECHNOLOGY DIVISION

PARTICULATE / NONPARTICULATE FIELD DATA  
CODING FORM  
PG 1 of 2

CLIENT EPA SHEET 1 OF 3  
 PROJECT No. AW PREPARED BY  
 PLANT N.B. FINECHESTER PITOT NUMBER and SIDE  
 RUN No. 4 PITOT TUBE CP  
 LOCATION MAN STREET FILTER No. / THIMBLE No. 1  
 DATE 7 Feb 84 AMBIENT TEMP OF 30  
 OPERATOR AW R.V. BAR PRESS., in. Hg 29.86  
 SAMPLE BOX NO. 1726 ASSUMED MOISTURE, % 3  
 METER BOX NO.          HEATER BOX SETTING, °F 250  
 METER ΔH<sub>2</sub>O 1.913 NOZZLE #/DIA., in. 622 1  
 Y FACTOR .997 PROBE LENGTH 5 ft  
 PROBE HEATER SETTING 250

\* FIELDS FOR NONPARTICULATE RUN

POINT	DISTANCE IN INCHES	CLOCK TIME	DRY GAS METER, CF	PITOT in. H <sub>2</sub> O ΔP	OR-FICE ΔH in H <sub>2</sub> O		DRY GAS TEMP. °F		PUMP VACUUM IN Hg GAUGE	BOX TEMP °F	IMPINGER TEMP °F	STACK PRESS in Hg	STACK TEMP °F	NULL ANGLE (DEGREES)
					DESIRED	ACTUAL	INLET	OUTLET						
1	START	10:19	708.599	.005	.82	.82	72	73	5	237	33		52	
2		40	710.5	.005	.82	.82	74	75	5	259	35		51	
3		80	712.5	.005	.82	.82	74	77	5	270	35		52	
4		120	714.5	.005	.82	.82	75	80	5	268	36		51	
5		160	716.5	.005	.82	.82	76	83	5	270	35		51	
6		200	718.5	.005	.82	.82	76	85	5	268	35		51	
7		240	720.6	.005	.82	.82	77	87	5	268	34		51	
8		280	722.5	.005	.91	.91	78	89	5	267	35		51	
9		320	724.6	.005	.91	.91	79	91	5	265	35		51	
10		360	726.7	.005	.91	.91	81	93	5	259	36		51	
11		400	728.8	.005	.91	.91	82	94	5	262	36		52	
			TOTAL											

COMMENT

10.14  
11.14  
36  
45

METER LEAK CHECK:  
 BEFORE TEST: .02 CF SEC 15 in. Hg  
 AFTER TEST: \_\_\_\_\_ CF SEC \_\_\_\_\_ in. Hg  
 PITOT LEAK CHECK   
 ORSAT LEAK CHECK \_\_\_\_\_  
 STATIC PRESSURE  
 PORT 0026  
 in. H<sub>2</sub>O \_\_\_\_\_  
 in. Hg \_\_\_\_\_

7 March 1984

The liquid and solids sampling was conducted according to the procedures in the QA Plan and two additional samples were collected. One was the plant influent (raw sewage), however, only the liquid portions could be sampled because the solids settled out and could not be reached by the rope and bucket sampling method. The other additional sample was the centrifuge water and there were no problems obtaining this. Chain-of-custody procedures were followed and all collected samples were placed in ice chests and stored in the locked GCA van until transfer to the GCA Sample Bank.

None of the influent/effluent lines of the incineration system were fitted with any type of measuring device. A portable Doppler meter was used to measure the liquid flow and a stopwatch and collection bucket was used to measure the flow of the solids. The Doppler meter was calibrated in duplicate at the start of the test run; it showed stable readings at the scrubber inlet and the centrifuge during the test.

After field testing was concluded, the auditor spot-checked calculations on instrument calibration sheets and Method 5 field data sheets for run four; no errors were found. The project notebooks of active staff members were also checked. The Project Manager's notebook had clear, dated entries of phone calls noting persons involved, agreements or questions, etc. Important reference materials and calculations of assumed Incinerator Feed Rates were also included. The field notebook was issued just before the testing was begun. It contained a daily log of the field testing, noting any problems and recommendations, and calculations of the sampling volume needed to find detectable levels of PCBs. The Doppler meter readings and the volume of Natural Gas used during each run were also included in this notebook.

In summary, with the exceptions noted in this report, the QC procedures stated in the QA Plan were followed.

  
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QA Scientist

JF/cf

cc: M. Atkinson  
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APPENDIX A  
SAMPLING AND ANALYSIS TEST PLAN

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY  
Research Triangle Park, NC 27711

Contract No. 68-02-3168  
Technical Service Area 3  
Assignment Nos. 58, 79 and 99

SAMPLING AND ANALYSIS  
PROTOCOLS FOR THE MULTIPLE  
HEARTH SEWAGE SLUDGE INCINERATOR  
AT THE NEW BEDFORD MUNICIPAL  
WASTEWATER TREATMENT PLANT

Amended Test Plan

August 1984

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## SECTION 1

### INTRODUCTION

The Environmental Protection Agency is currently conducting a comprehensive evaluation on the occurrences, transport mechanisms and fate of polychlorinated biphenyls (PCBs) within the New Bedford Harbor Area. Among those processes presently under investigation as a source of PCBs is the Multiple Hearth Incinerator at the New Bedford Wastewater Treatment Plant.

This treatment plant is of considerable interest since it presently processes both municipal and industrial wastewater from the city of New Bedford. It is estimated that industries contribute 60 percent of the total plant influent while municipal sources account for the remaining 40 percent. Two of the industrial facilities that feed the treatment plant are known to have used significant amounts of PCBs. While these plants no longer use PCB, quantities of this environmental contaminant still remaining in their sewer lines can potentially be flushed out of the plants and into their wastewater discharge. At the Municipal Treatment Plant, the PCBs can adhere to the solid sludge and undergo subsequent processing with the sludge. In the plant's multiple hearth incinerator this sludge is combusted and PCBs potentially released as a gaseous, or solid waste emission.

Given the relatively low operating temperature of the Multiple Hearth Incinerator and low PCB destruction efficiencies (<97%) reported for these units, serious consideration should be given to emissions of polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF), and other chlorinated combustion by-products potentially formed during the incineration process. Tests conducted in 1977 at this incinerator reported measurable concentrations of PCB in the incinerator flue gas emissions and process water.<sup>1</sup> However, no data presently exists on PCDD and PCDF emissions from the unit. Based on limited results available on the New Bedford Municipal Incinerator, a complete sampling and analysis program has been designed to establish the fate of PCBs within the treatment plant facility.

The subsequent sections of this test plan will address the sampling and analysis protocols chosen for the New Bedford facility. This program is designed to provide a complete mass balance of all incinerator process streams for PCBs including gaseous and particulate emissions, sludge feed, hopper ash, pre-cooler and scrubber water feeds, scrubber water effluent and centrifuge effluent.

Analysis for PCDFs and PCDDs will be conducted on gaseous and particulate emissions as well as hopper ash. Complete organic chemical analysis will be conducted on composite samples of the sludge feed. This analysis will address major components including PCBs, PCDFs, and PCDDs. In addition, grab samples of sludge feed will be collected and analyzed for total PCBs. It is anticipated that these measurements will provide necessary data on the variability of PCB content in the influent sludge.

## SECTION 2

### SAMPLING AND ANALYSIS

#### SAMPLING PROTOCOL

##### Flue Gas Sampling Train

The proposed approach for sampling PCBs in the incinerator flue gas will include three complete tests of the incinerator at normal operation. These tests will commence only after normal incinerator hearth temperatures have been established (i.e., temperature in hearths 3, 4 and 5 greater than 650°C [1100°F]).

A modified Method 5 train will be used for the simultaneous collection of particulates, HCl, PCBs, PCDDs and PCDFs. A schematic of the train is presented in Figure 1.

The sampling train will be operated in accordance with the procedures outlined in EPA Method 5. Accordingly, representative samples of the flue gas will be obtained from predetermined sampling points in the stack. The specifications of these points will be calculated in accordance with the guidelines set forth in EPA Method 1. The sample train will consist of a glass-lined, heat-traced probe with a stainless steel button hook nozzle. The probe is equipped with a thermocouple and pitot tubes. The flue gas will pass through the probe and then through a heated glass fiber filter (Reeve Angel 934 AH filter paper). Downstream of the heated filter, the sample gas will pass through two impingers containing distilled deionized water, an empty knockout impinger, and two organic sorbent traps containing florisol and XAD-2 resins, respectively. The temperature of the sample gas entering the sorbent traps will be maintained at less than 68°F throughout the sampling period. The sorbent traps are followed by three impingers, the first two containing 100 ml of 1N NaOH for HCl collection and the third containing dessicant. The impingers will be followed by a pump, dry gas meter and calibrated orifice.

The sampling and velocity traverse will be conducted along two perpendicular diameters of the stack. A total of 24 points will be sampled on each diameter resulting in a final total 48 sampling points. The sampling time will be 4 minutes per point for a total sampling time of 192 minutes. Figures 2 and 3 present the stack schematic and the layout of stack sampling points, respectively.

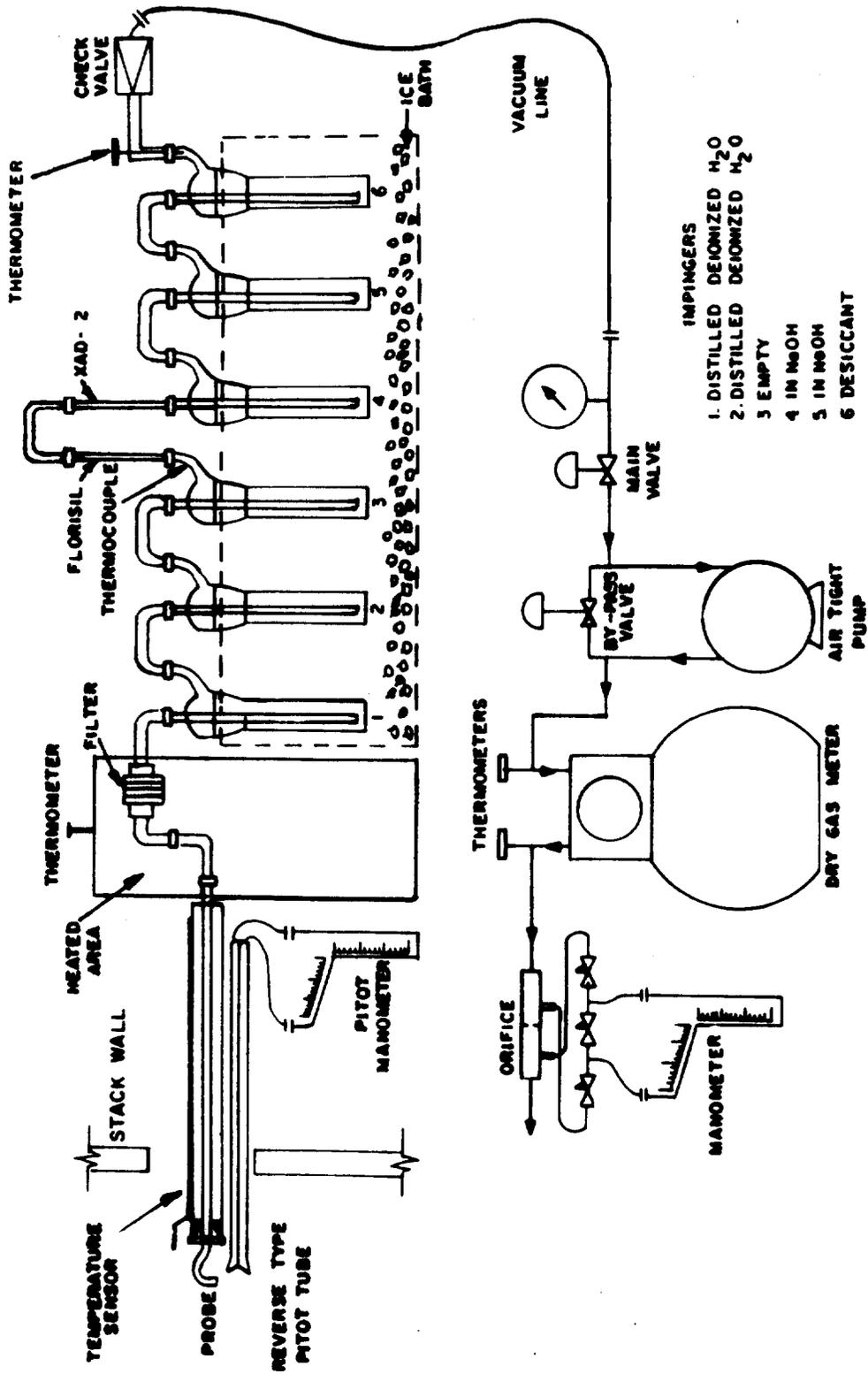
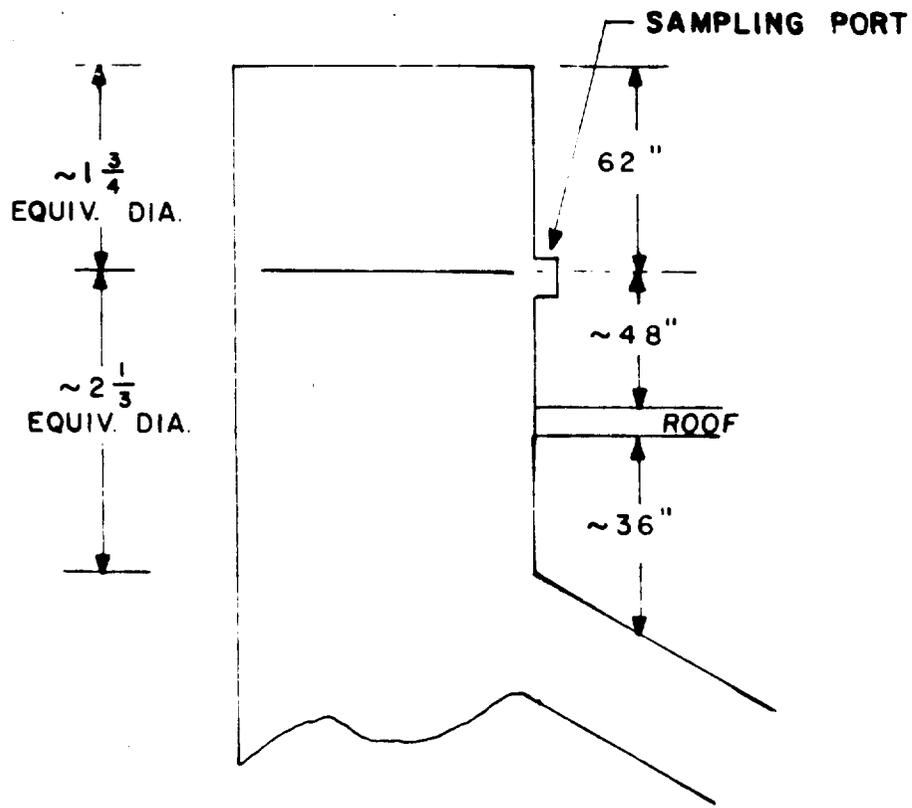


Figure 1. Flue gas sampling train.



OUTER CIRCUMFERENCE OF STACK = 113.5" =  $\sim 3'$  I.D.

Figure 2. Schematic of stack.

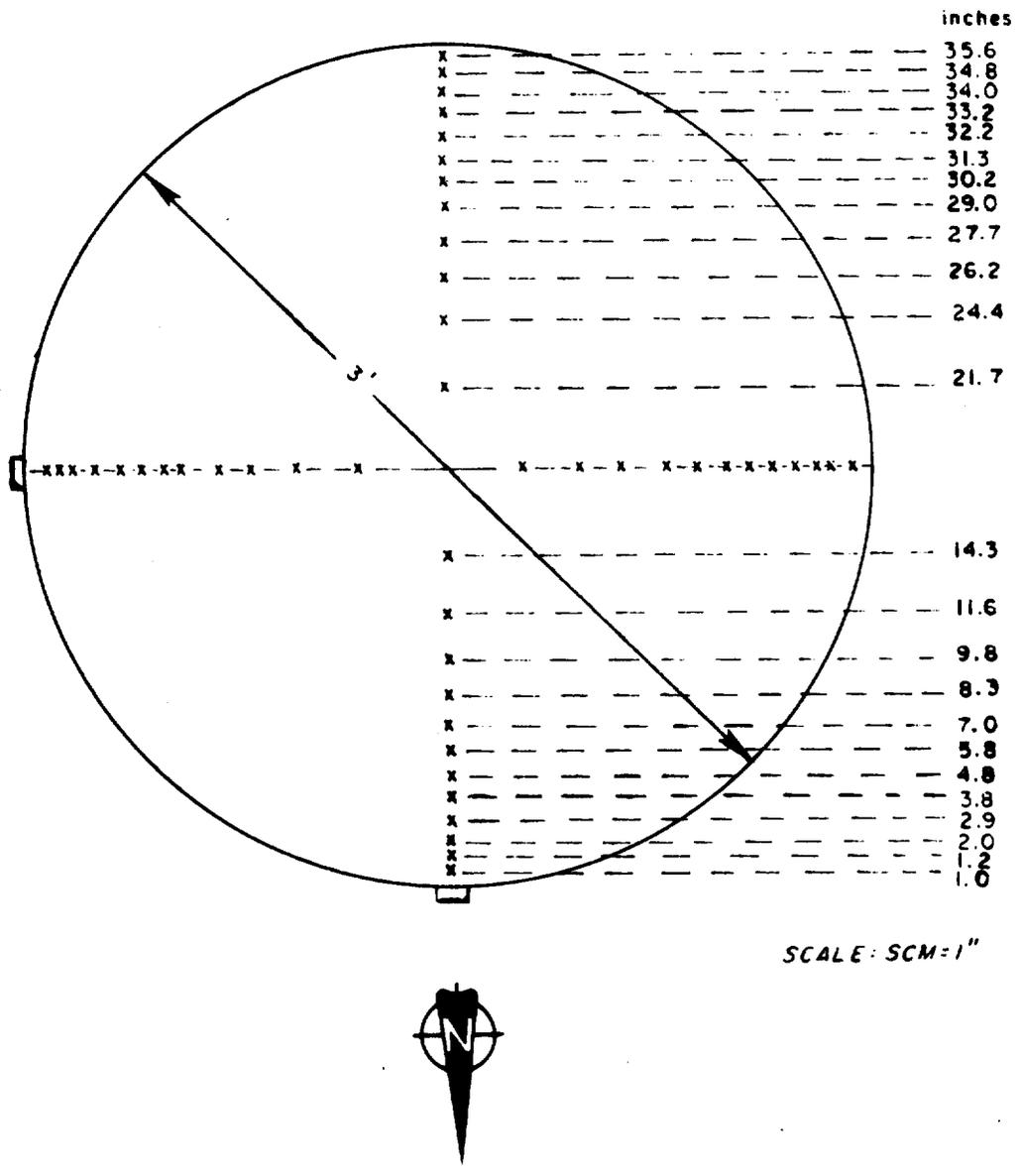


Figure 3. Stack sampling points.

The stack inner diameter will be checked prior to sampling, although it is presumed that Figures 2 and 3 adequately represent this sampling location. An additional, integrated sample of flue gas will be collected during each of the three tests for fixed gas analysis ( $\text{CO}_2$ ,  $\text{O}_2$ ,  $\text{CO}$ ). All of the sampling and analysis procedures will be carried out in accordance with EPA Method 3.

Sampling will be isokinetic (+10 percent) with readings of flue gas parameters recorded at every sampling point during the traverse. In the event that isokinetic sampling cannot be maintained, the train will be shut down and the problem remedied. In the event that steady operation is not maintained, or there are atypical fluctuations in monitored gas parameters ( $\text{CO}$ ,  $\text{O}_2$ ), the testing will be stopped until these conditions are stabilized. Steady operation of the incinerator will be the responsibility of Treatment Plant personnel, but the flue gas parameters and composition will be monitored by GCA. Any changes will be noted and relayed to Treatment Plant personnel so that appropriate action can be taken. Suggested parameters for monitoring by Treatment Plant personnel are listed in Table 1.

The recovery procedures for the flue gas sampling system will be:

1. Remove the sampling train to the predetermined recovery area.
2. Note the condition of the train (e.g., color of the desiccant, resin condition, etc.).
3. Remove, seal and label the florisil and XAD-2 tubes with the codes X-FL and X-XR, respectively. The run number (X) should precede all sample codes (e.g., 1-FL).
4. Brush and rinse the sampling probe liner and front half of the filter assembly with equal amounts of acetone and methylene chloride. Label the sample with the code X-FH.
5. Recover the particulate filter into its original glass petri dish and label with the code X-PF.
6. Measure the volume of the impingers 1, 2 and 3 in a precleaned graduated cylinder and transfer to an amber glass sample bottle labeled X-Cond-O.
7. Rinse the impingers 1, 2 and 3 with acetone followed by hexane into an amber glass container and measure and label X-BH.
8. Measure and recover impingers 4 and 5 into a Nalgene container and label X-IMP.
9. Record the weight gain of the desiccant contained in the final impinger.
10. Ensure that all sample containers are properly sealed, labeled and the liquid levels marked. Log all samples in sample packing list.

TABLE 1. INCINERATOR OPERATIONAL PARAMETERS TO BE MONITORED  
 BY PLANT PERSONNEL AT THE NEW BEDFORD MUNICIPAL  
 WASTEWATER TREATMENT PLANT

Incinerator temperatures	Normal range (°F)
Hearth 1	1000-1900
2	1000-1900
3	1000-1900
4	1000-1900
5	1000-1900
6	700-1000
7	200-500
Scrubber inlet temperature	120-150
Scrubber outlet temperature	40-60
Cool air exchanger temperature	250-325
Incinerator draft	0.0-0.45 in. W.G.
Scrubber Differential Pressure	2.0-5.0 in. W.G.
Natural Gas Fuel Rate	ft <sup>3</sup> /min <sup>a</sup>

<sup>a</sup>Rate will be measured daily.

EPA sampling protocol requires the use of a blank flue gas train for each day of sampling in order to provide blank corrections. This train must be set up and recovered each day of sampling.

Figure 4 is a flow diagram for the sample recovery of the flue gas train. Table 2 presents a complete listing of the samples to be taken during the entire testing program.

### Continuous Monitoring

A continuous monitoring system will be used to monitor CO, CO<sub>2</sub> and O<sub>2</sub>. The continuous monitoring system will be equipped with a gas conditioning system and continuous chart recorders. The flue gas will be extracted from the stack and drawn through a flue gas conditioning system to remove moisture (by condensation) and particulates (by filtration through glass fiber filter media).

Oxygen concentrations will be determined using a Horiba Model POA 21 Polarographic O<sub>2</sub> Analyzer with a measuring range of 0 to 20 percent O<sub>2</sub> full scale. The analyzer will be calibrated at 0 percent O<sub>2</sub> with ultrapure nitrogen and with two other appropriate span gases before and after each test.

Carbon dioxide concentrations will be determined using a Horiba Model PIR 2000 NDIR Carbon Dioxide Analyzer with a measuring range of 0 to 25 percent CO<sub>2</sub>. This monitor will be calibrated with a zero and two span gases in an analogous fashion to the previously described O<sub>2</sub> monitor.

Carbon monoxide concentrations will be determined using a Horiba Model PIR 2000 NDIR CO Analyzer with a measuring range of 0-500 ppm CO full scale. This will be calibrated with a zero and two span gases in an analogous fashion to the O<sub>2</sub> and CO<sub>2</sub> analyzers.

Table 3 lists the analyzer specifications for all the above determinations.

Four strip chart recorders will be used to record all monitoring data. The data will be corrected for calibration drift, if any, and reduced to 15-minute averages. Maximum and minimum values for each test period will also be determined.

The continuous emission monitors will be calibrated twice daily from Airco cylinders containing certified ( $\pm 1$  percent) calibration gases. This gas will be prepared according to EPA protocol 1.

### Testing Schedule

The proposed sampling program will include three complete tests of the incinerator at normal load or steady state operation. It is anticipated that at least two complete tests can be collected during a given 24 hour period of continuous incinerator operation. The sludge will be fed to the incinerator after normal hearth temperatures have been established. The sampling will require approximately 3 hours for each test.

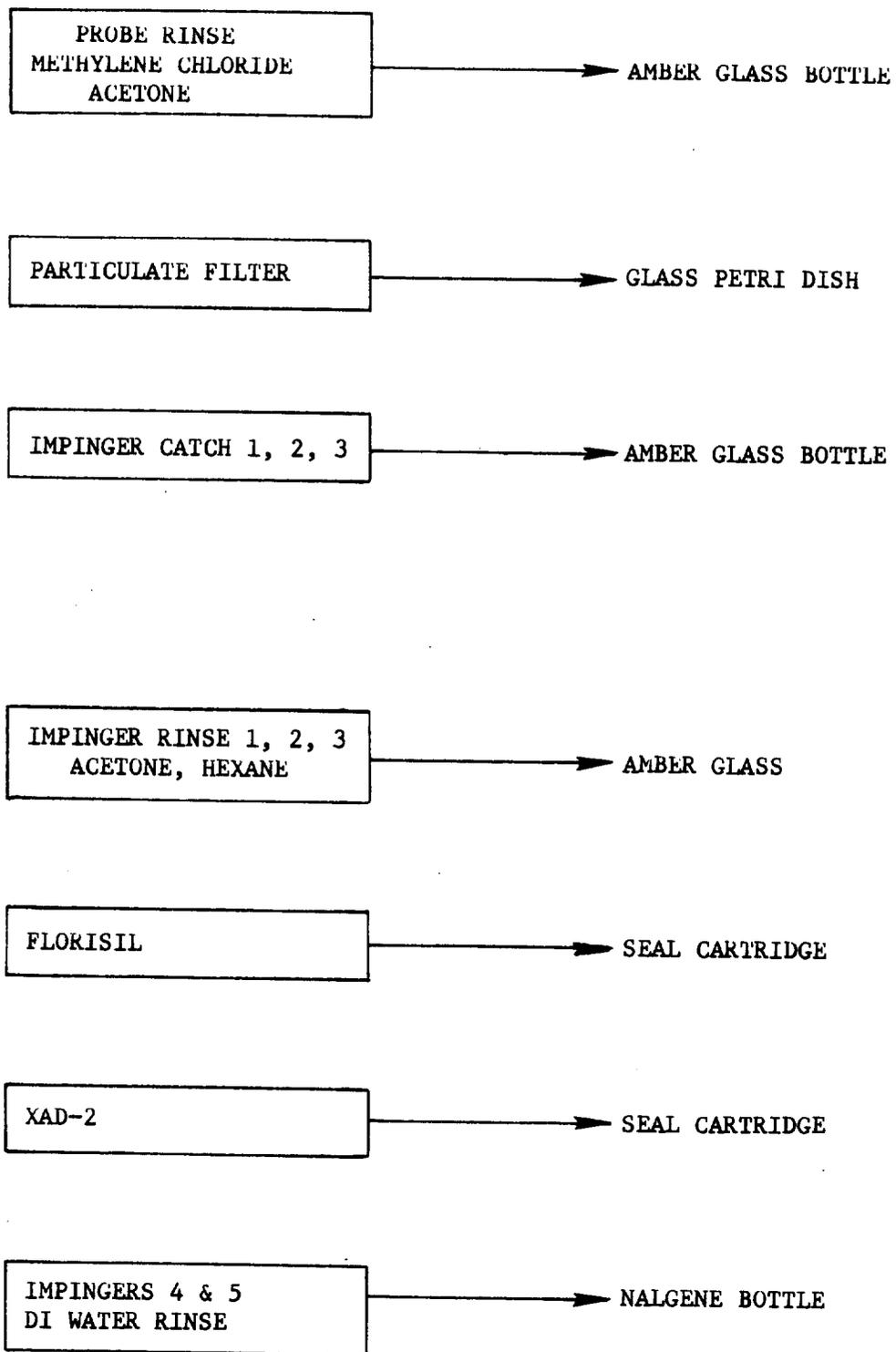


Figure 4. Flue gas train sample recovery.

TABLE 2. SAMPLES TO BE COLLECTED AT NEW BEDFORD MUNICIPAL INCINERATOR

Matrix	Sample	Source	Species to be analyzed	Number	
Flue Gas Modified Method 5 train	Particulate filter	Train	PCBs, PCDDs, PCDFs, Other Organics	3	
	Particulate rinses	Train	PCBs, PCDDs, PCDFs, Other Organics	3	
	Florisil resin	Florisil tube	PCBs, PCDDs, PCDFs, Other Organics	3	
	XAD-2 resin	XAD-2 tube	PCBs, PCDDs, PCDFs, Other Organics	3	
	Impinger water	Impingers	PCBs, PCDDs, PCDFs, Other Organics	3	
	Impinger catch (1N NaOH)	Impingers	Chloride	3	
	Solvent rinses of train	System rinse	PCBs, PCDDs, PCDFs, Other Organics	3	
	Method Blanks (One Complete Train Per Run)	XAD-2 resin blank	XAD-2 lot	PCBs, PCDDs, PCDFs, Other Organics	3
		Florisil resin blank	Florisil lot	PCBs, PCDDs, PCDFs, Other Organics	3
		Filter lot blank	Filter lot	PCBs, PCDDs, PCDFs, Other Organics	3
Hexane blank		Hexane lot	PCBs, PCDDs, PCDFs, Other Organics	3	
Acetone blank		Acetone lot	PCBs, PCDDs, PCDFs, Other Organics	3	
Impinger water blank		Organic-free laboratory water	PCBs, PCDDs, PCDFs, Other Organics	3	
1N NaOH		NaOH lot	Chloride	3	
Solids		Sludge feed	Screw conveyor at incinerator inlet	PCBs	21
		Ash	Ash hopper	PCBs, PCDDs, PCDFs, Other Organics	9
Waters		Precooler/scrubber water feed	Precooler inlet	PCBs, Other Organics	9
	Scrubber water effluent	Scrubber drain	PCBs	9	
	Centrifuge water	Centrifuge outlet	PCBs, Other Organics	9	
	Plant influent wastewater	Head of plant	PCBs, Other Organics	9	

TABLE 3. ANALYZER SPECIFICATIONS OF CONTINUOUS MONITORS

	Horiba PIR 2000 CO <sub>2</sub> analyzer	Horiba PIR 2000 CO analyzer	Horiba POA 21 OPE 315 O <sub>2</sub> analyzer
Operating Sensitivity Ranges	0-5% CO <sub>2</sub> , FS 0-15% CO <sub>2</sub> , FS 0-25% CO <sub>2</sub> , FS	0-500 ppm CO, FS 0-1500 ppm CO, FS 0-2500 ppm CO, FS	0-5% O <sub>2</sub> , FS 0-20% O <sub>2</sub> , FS 0-50% O <sub>2</sub> , FS
Operating Temperature Ranges	24°F - 122°F	24°F - 122°F	32°F - 122°F 95% Humidity
Analysis Method	Nondispersive Infrared	Nondispersive Infrared	Polarographic
Linearity	+1% FS	+1% FS	+0.5% of Full Scale
Accuracy	+1% of Full Scale	+1% of Full Scale	+1% of Full Scale
Drift	+1% of Full Scale in 24 hours in both zero and span	+1% of Full Scale in 24 hours in both zero and span	5% Full Scale for 24 hours in both zero and span
Noise level	0.5% of Full Scale in most sensitive range	0.5% of Full Scale in most sensitive range	0.25% of Full Scale in most sensitive range

### Sludge

A grab sampling procedure will be used to obtain a composite sludge sample. The sludge sampling location will be at the end of the screw conveyor, as the sludge enters the incinerator. Grab samples, each 50 ml in volume, will be obtained at 30 minute intervals throughout each test run. These samples will be collected in 100 ml wide mouth amber glass jars with teflon lined caps. The 21 jars, 7 for each test run, will be clearly labeled in sequential order and then transported to the laboratory for analysis.

### Ash

Ash samples will also be obtained by grab sampling. These samples will be taken from the feed line to the ash storage hopper. Sample size will be approximately 75 ml and will be collected at 60 minute intervals during each 3-hour test run. All ash samples will be transferred into 250 ml wide mouth glass bottles lined with a teflon lined cap. Each of nine samples, three for each test run, will be clearly labeled and then transported to the laboratory for analysis.

### Precooler/Scrubber Water Feed

The precooler/scrubber water feed will be sampled from a tap located on the precooler inlet. Three samples will be collected during the course of each 3-hour run. It is anticipated that a total of nine such samples will be collected. All sample bottles will be clearly labeled and then transported to the laboratory for analysis.

### Precooler/Scrubber Water Effluent

Samples of the precooler/scrubber water effluent will be taken at hourly intervals during each test run. Again, assuming sampling runs of three hours in duration three separate scrubber water effluent samples will be collected per test. A total of nine samples will result from the three test programs. All samples will be taken from a tap located on the scrubber drain line. All samples will be placed in wide mouth amber glass jars and transported to the laboratory for analysis.

### Centrifugate Water

Aqueous samples will be collected at hourly intervals from a tap located at the base of the centrifuge. Three samples will be taken during each three hour test period. A total of nine samples will result from the three scheduled runs. All samples will be placed in wide mouth amber glass jars and transported to the laboratory for subsequent analysis.

### Plant Influent Wastewater

Influent samples will be collected at hourly intervals from the head of the plant in the vicinity of the grid screen. Three grab samples will be taken during each test run. A total of nine samples will result from the test program. All samples will be placed in wide mouth amber glass jars and transported to the laboratory for subsequent analysis.

A summary of the sampling procedures for the flue gas, sludge, ash and aqueous samples is provided in Table 4.

#### Field Parameter Measurements

During each of the three tests, various physical parameters will be measured and monitored by the sampling contractor, including continuous monitoring of the flue gas for CO and O<sub>2</sub>. While there are no minimum or maximum values for CO and O<sub>2</sub> set forth in the Federal Register for sewage sludge incinerators, as there are for Annex I incinerators and high efficiency boilers, monitoring and recording of the flue gas composition will aid the post-test review of PCB destruction efficiency. All monitors will be equipped with a gas conditioning system and each will be calibrated prior to and during use as required.

Other physical parameters will also be measured during this testing program. These parameters include the flue gas velocity, static pressure and temperature. These measurements shall be made prior to each run for the determination of proper nozzle size and sampling rate.

Since there are presently no meters in place to monitor sludge and pre-cooler/scrubber inlet and outlet water stream flows a portable noninvasive flow monitor will be used. As there is no available location for direct on-line monitoring of sludge flow as it enters the incinerator, flow monitors will be placed at both the inlet and outlet of the centrifuge. The inlet monitor will record sludge flow and the outlet monitor will record exit meter flow. These measurements in conjunction with the density of sludge "as fired" will provide sludge flow data.

The solids and PCB content of the sludge "as fired" will be determined, and these measurements will later be used to calculate the dry sludge feed rate and PCB input rate to the incinerator for each test.

Sludge flow rate measurements will also be provided by a supplementary technique involving reversing the screw conveyor and weighing the sludge caught in a plastic bag during a specific time interval. This will be done immediately before and after each test run. In addition, if the flue gas sampling is halted during a sampling run for any reason, the sludge feed may be measured during this interruption. The results of these measurements will be used to verify on an instantaneous basis the calibration of the portable flow meter.

Immediately before the start of each test run the ash collection hopper will be emptied. This action can be coordinated with treatment plant personnel. At the conclusion of each run, the volume of ash in the hopper will be measured. Laboratory analysis of the ash samples will yield the bulk density of the ash (in pounds per cubic foot) and organic analysis as noted earlier. These factors will allow the calculation of the ash production rate (in pounds per minute, hour, etc.) and emission rates for PCBs and combustion by-products such as PCDFs and PCDDs potentially formed during the incineration process.

TABLE 4. TEST RUN SAMPLING PROCEDURES AT NEW BEDFORD SEWAGE SLUDGE INCINERATOR

---

Stream A - Input sludge

Sampling location: Immediately after the end of the screw conveyor as the sludge enters the incinerator.

Number of samples per run: Seven grab samples of approximately 50 ml each, collected in individual 100 ml wide mouth jars with Teflon lined caps, will be collected at 30-minute intervals, beginning with the start of each test.

Stream B - Ash

Sampling location: Feed to the ash storage hopper.

Number of samples per run: Three grab samples of approximately 75 ml each, transferred and composited in a 250 ml wide mouth glass bottle with Teflon lined cap, will be collected at 60 minute intervals, beginning with the start of each test.

Stream C - Precooler and scrubber water feeds

Sampling location: Tap located on precooler feed line.

Number of samples per run: Three samples of approximately 250 ml each, transferred and composited in a 1250 ml wide mouth amber glass bottle with Teflon lined cap, will be collected at 60 minute intervals in duplicate, beginning with the start of each test.

Stream D - Scrubber water effluent

Sampling location: Tap located on scrubber drain line.

Number of samples per run: Three samples at approximately 500 ml each, transferred and composited in a 3000 ml wide mouth amber glass bottle with Teflon lined cap, will be collected at 60 minute intervals in duplicate, beginning with the start of each test.

---

(continued)

TABLE 4 (continued)

---

Stream E - Stack gas

Sampling location: Existing ports on 3' diameter stack.

Number of sample  
points: A total of 48 points, 24 along each diameter  
of the stack.

---

## Sampling Requirements

The stack sampling program will require 4 days of testing. A total of three tests of at least 3 hours each will be conducted over a two day period assuming 24 hour periods of continuous unit operation. The incinerator operation will be at steady state for the tests.

Incinerator operation including sludge feed and incinerator conditions such as hearth temperatures and precooler/scrubber water feed will be the responsibility of New Bedford wastewater treatment plant personnel. All data gathered will be made available to the testing contractor for assessment purposes.

Other requirements to be provided by plant personnel include the following:

- Adequate electrical power for both stack sampling trains.
- Laboratory area in an accessible location for sample recovery.
- Parking for truck nearby stack location.

All samples collected will be inventoried in the field. Chain of custody will be maintained through the use of log books. Upon receipt at the GCA laboratory, the samples will be checked against the inventory sheets and assigned laboratory log numbers. All sample handling will be controlled to prevent loss or alteration of samples. All data sheets will be bound and filed.

## ANALYTICAL PROTOCOLS

### Introduction

As noted earlier in Table 2, analyses will be conducted on sample sets collected during each of three test runs at the New Bedford facility. Each of three complete sets of flue gas samples and associated field blanks will be analyzed for polychlorinated biphenyls (PCBs), polychlorinated dibenzodioxins (PCDDs) polychlorinated dibenzofurans (PCDFs) and other organic components. In addition, approximately 21 sludge feed samples will be analyzed for PCBs; hopper ash grab samples from each test run will be analyzed for PCBs, PCDDs, PCDFs and other organic components; precooler/scrubber water feed samples from each test run will be analyzed for PCBs and other organics; scrubber water effluent samples from each test series will be analyzed for PCBs; centrifugate water and plant influent wastewater samples collected during each test run will also be analyzed for PCBs and other organics. The description that follows will provide further details on the analyses of each of these matrices.

### Flue Gas Samples--Gas Phase

The Gas Phase is defined as all train samples except the particulate filter and probe rinse which are addressed in the next section. Each of the three sets of steady state runs and corresponding field and method blanks will be analyzed for the following parameters:

- PCBs as positional isomer classes in both the particulate and gaseous phases.
- PCDDs/PCDFs - again as positional isomer classes in both the particulate and gaseous phases.
- Other Organics - Results for other major organic components in each of the particulate and gaseous phases and not members of the above organic classes.
- Chloride - in the NaOH impingers only.

Each flue gas sample set contains the following sample types:

- Back half rinse (acetone/hexane)
- Organic condensate and Aqueous impingers (DI H<sub>2</sub>O)
- Florisil sorbent
- XAD-2 sorbent
- Aqueous impingers (NaOH)

There are also corresponding field blanks associated with each of the above sample types. There is one complete field-biased blank set for each of the respective test days.

A summary of the organic analysis scheme appropriate for the flue gas samples (gas phase) is provided in Figure 5. Pertinent instructions on the preparation and analysis of each sample type is provided below.

Back-Half Rinses (Acetone/Hexane)--

Hold for combination with aqueous extracts. Combined extracts will be concentrated to a final volume using Kuderna-Danish evaporative concentrator.

Organic Condensate/Impingers (DI H<sub>2</sub>O)--

Should be combined prior to extraction. Sequential extractions should be performed using methylene chloride (3x). Extracts should be held for eventual combination with the train rinses and sorbent extracts.

Impingers (NaOH)--

Analyze for chloride content.

GC/MS Analyses (PCBs, PCDFs, PCDDs, Other Organics)--

Analyses for PCBs as positional isomer categories will be conducted using a Hewlett-Packard 5985 GC/MS operating in the total ion mode. A summary of GC/MS operating conditions suggested for these analyses is provided in Table 5. Instrument calibration should be established using at least one isomer from each of ten PCB positional isomer categories. Use of the following representative isomers as well as the parent biphenyl is suggested:

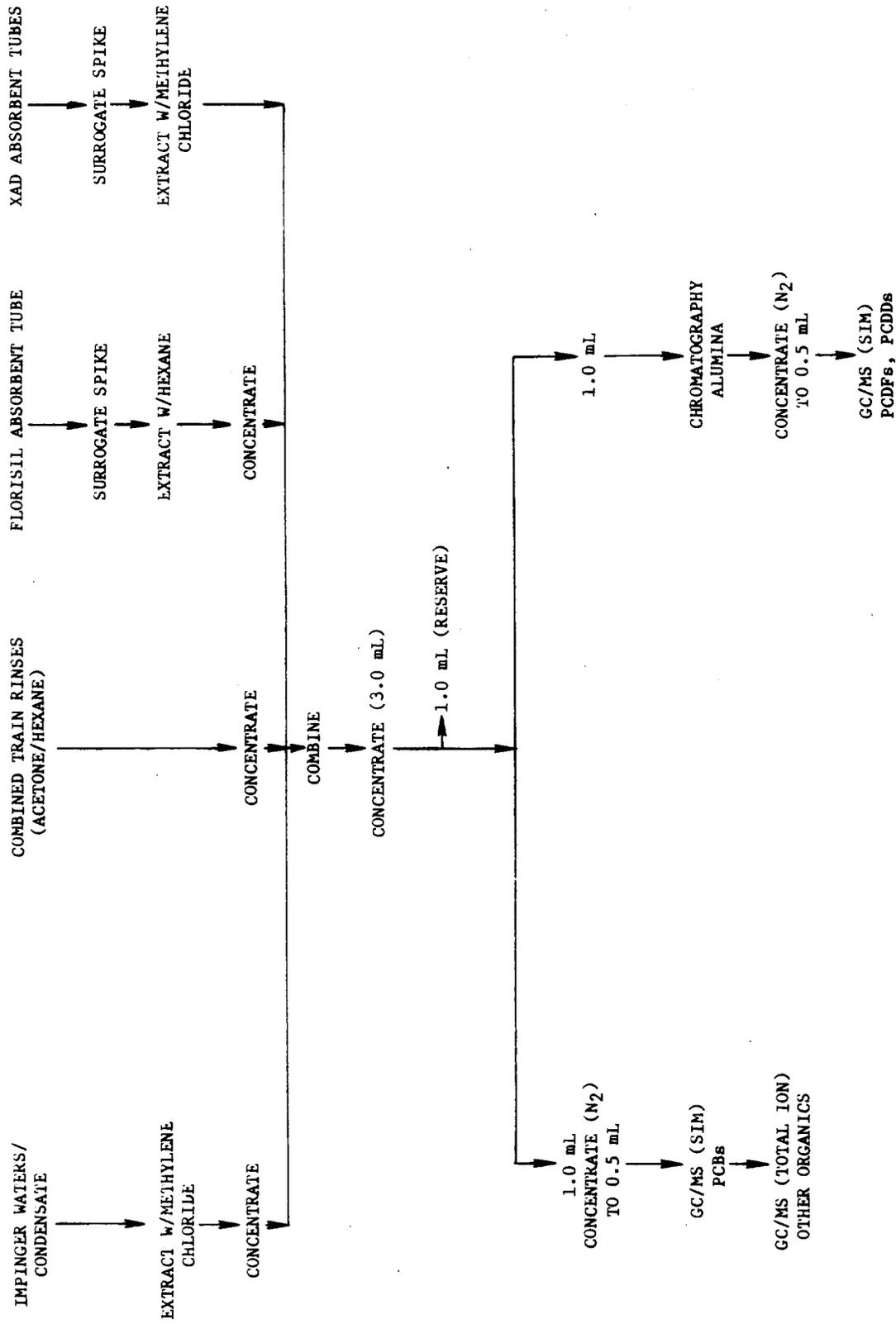


Figure 5. Organic analysis flow scheme--gas phase flue gas samples.

TABLE 5. GC/MS OPERATING CONDITIONS

---

Instrument	Hewlett Packard 5985, quadrupole mass spectrometer
GC Conditions	
Column	DB-5 30M fused silica capillary (or equivalent)
Temperature program	50°C held for 2 min then 10°/min to 300°C and held
Injector type	Grob w/0.5 min sweep time
Injector temperature	275°C
Injection volume	1 $\mu$ l, splitless
Column flow	UHP helium, 0.5 ml/min
MS Conditions	
Emission	300 $\mu$ A
Electron energy	70 eV
Scan time	1.0 s/scan
Mass interval	45 to 450 amu
Source temperature	200°C

---

- 2-Chlorobiphenyl
- 4-Chlorobiphenyl
- 3,3'-Dichlorobiphenyl
- 2,5-Dichlorobiphenyl
- 2,3,5-Trichlorobiphenyl
- 2,4,5-Trichlorobiphenyl
- 2,3,4,5-Tetrachlorobiphenyl
- 2,2',4,4',5-Pentachlorobiphenyl
- 2,2',4,4',6,6'-Hexachlorobiphenyl
- 2,2',3,4,5,6,6'-Heptachlorobiphenyl
- 2,2',3,3',4,4',5,5'-Octachlorobiphenyl
- 2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl
- 2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl

Analyses for PCDDs/PCDFs as positional isomer categories will also be conducted using the Hewlett-Packard 5985 GC/MS. Instrument conditions will be identical to those cited in Table 5, with the exceptions that spectra will be acquired in the selected ion mode (SIM). Instrument calibration will be provided for a variety of chlorinated dioxins and chlorinated dibenzofurans. A suggested listing of representative isomers for the GC/MS screening protocols are shown below.

- Dibenzofuran
- 2,8-Dichlorodibenzofuran
- Dibenzo-p-dioxin
- 1-Chlorodibenzo-p-dioxin
- 2-Chlorodibenzo-p-dioxin
- 2,6-Dichlorodibenzo-p-dioxin
- 1,2,4-Trichlorodibenzo-p-dioxin
- 1,2,3,4-Tetrachlorodibenzo-p-dioxin

The identification and quantitation of additional organic components not classified as PCBs, PCDDs or PCDFs will follow the criteria noted below.

A background-corrected spectrum of the component will first be obtained by computer subtraction. A probability-based library search (PBS) will then be conducted by computer routine, comparing the unknown spectra with those of the EPA/NIH libraries. The computer search will provide up to 10 possible matches. The spectra of library matches will then be compared directly to the unknown spectrum by the operator. For positive identification, the following conditions are required:

- The intensity, relative to the base peak, of all major peaks (greater than 50 percent of base peak) must agree within 20 percent.
- All peaks present in the library spectrum at more than 20 percent of the base peak must be present in the unknown spectrum.
- The unknown spectrum must not have any peaks present at more than 30 percent of the base peak that are not seen in the library spectrum or are not clearly attributable to coeluting compounds.

If the library search does not provide a positive match, the unknown spectra will be reviewed for major peaks and fragmentation patterns. Component concentrations will be calculated relative to the closest eluting internal standard.

#### Flue Gas Samples--Particulate Phase

The combined probe rinse and particulate filter samples from each flue gas train will constitute the stack particulate samples. Analyses of these samples will proceed in the following manner: particulate weights and probe rinse residue weights will be recorded. The filter and probe rinse solids will be combined prior to extraction. Extracts will be performed in an "F" soxhlet using methylene chloride. Prior to extraction each thimble will be spiked with the following surrogate mixture: d<sub>3</sub>-trichlorobenzene, d<sub>10</sub>-biphenyl, d<sub>12</sub>-chrysene and decafluorobiphenyl. Spiking levels of 20-50 µg are recommended. Extracts will be reduced to a final volume using a Kuderna-Danish evaporator. GC/MS analyses for PCBs, PCDDs, PCDFs and other organic components will be conducted using the instrumental operating parameters previously cited for gas phase extracts. A schematic of the analytical flow scheme pertinent to the flue gas particulate samples is provided in Figure 6.

#### Hopper Ash Samples

Three grab samples will be taken from the hopper during each test run. As noted earlier they will be taken at approximately 1 hour intervals during each test run.

Thirty-five gram (35 g) aliquots will be taken from each of the three grab samples and composited resulting in a 105 g composite sample for each test series. Each composite sample will be transferred to an "F" size soxhlet thimble and fortified with a surrogate cocktail containing the following components: d<sub>3</sub>-trichlorobenzene, d<sub>10</sub>-biphenyl, d<sub>12</sub>-chrysene and decafluorobiphenyl (DFB). Samples will be extracted overnight with methylene

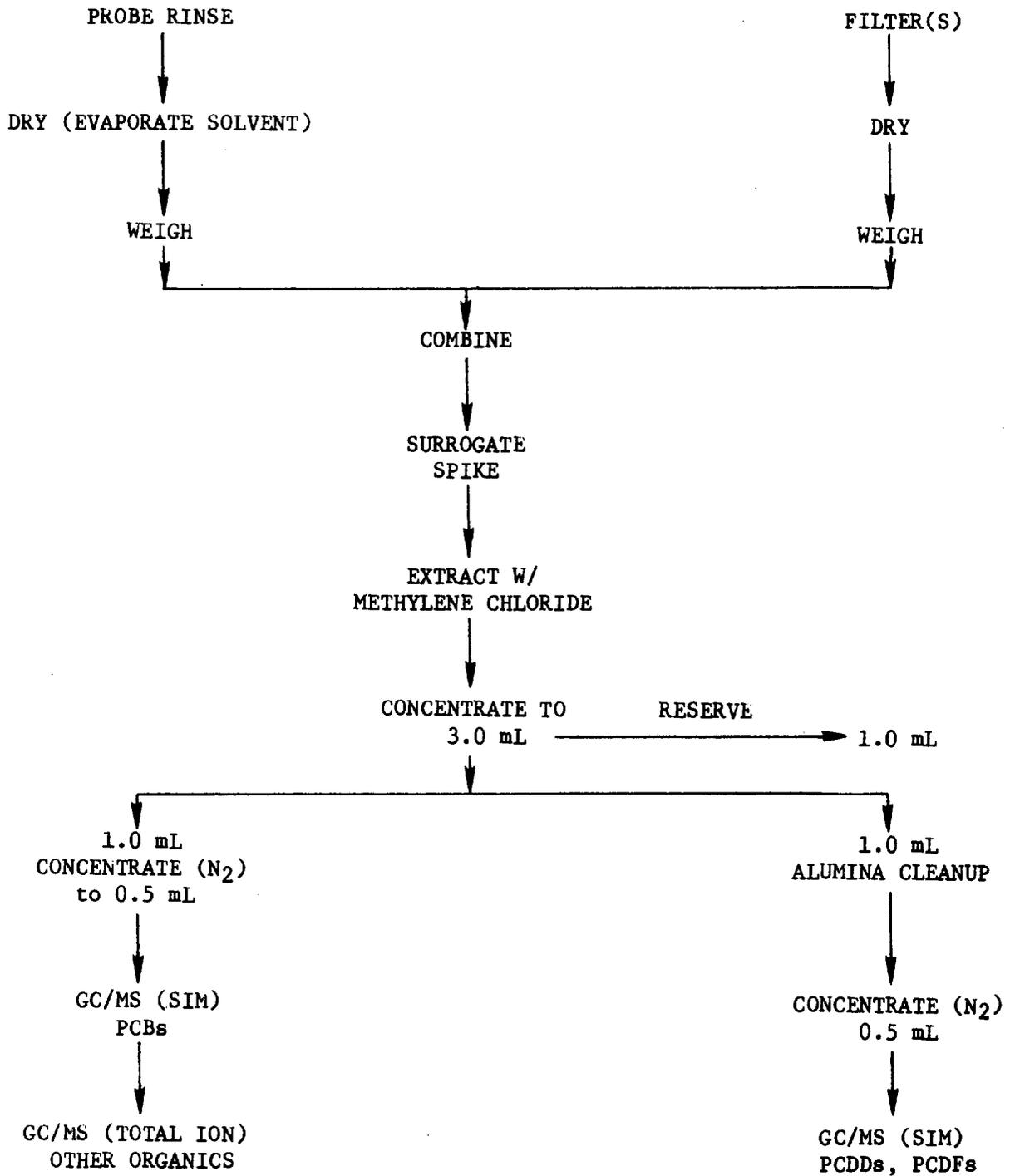


Figure 6. Organic analysis flow scheme--particulate phase flue gas samples.

chloride. Extracts will be reduced in volume to 10.0 ml using a Kuderna-Danish evaporative concentrator. A 3.0 ml aliquot will be held in reserve. A 3.0 ml portion will be further reduced to 0.5 ml, or the minimum attainable volume, for GC/MS analysis in the selected ion mode (SIM) for isomers of PCBs. GC/MS analyses for additional organics, not members of the above chemical classes, will be conducted in the total ion mode. Alumina cleanup procedures will be implemented on an additional 3.0 ml aliquot to permit unambiguous identification of PCDD and PCDF isomer classes.

#### Sludge Feed Samples

Grab samples of sludge feed will be taken at 1/2 hour intervals during each of the test series resulting in a total of seven samples per 3 hour period. Analyses will be conducted on each of these samples. A 10 g aliquot will be removed from each sample and air-dried. Each of these samples, in turn, will be extracted overnight in a soxhlet-extractor using a hexane/acetone (1/1) solvent system. Each resultant extract will be reduced to 5.0 ml using a Kuderna-Danish apparatus. An aliquot of each extract will be submitted for PCB analysis using a gas chromatograph fitted with an electron capture detector (GC/ECD). Samples displaying a complex or indistinguishable pattern will be subjected to additional cleanup procedures including acid ( $H_2SO_4$ ) partitioning or florisil column chromatography. Webb-McCall procedures will further be used on samples not displaying a complete Aroclor pattern after these cleanup procedures.

In addition to PCB analysis via GC/ECD on each grab sample, PCB analyses as positional isomer categories will be conducted via GC/MS on composite samples from each test series. In this instance a 1.0 ml aliquot will be removed from each of the seven uncleaned sample extracts representing each test run. The composite extract will be fortified with the same surrogate cocktail used for the flue gas and hopper ash samples. Spiking levels of 5-10  $\mu g$  for each of these components are recommended. Each combined extract will be further reduced in volume under a gentle stream of prepurified  $N_2$  to the minimum volume attainable, ideally 100  $\mu l$ .

#### Aqueous Samples

##### GC ECD--

Analyses will be conducted on the following types of water samples collected during each test series.

- Precooler/Scubber Water Feed--3 grab samples per run. These will be composited so as to create a single representative sample per run (e.g., one liter total).
- Scrubber Water Effluent--3 grab samples per run. These will be composited into a single representative sample per run (e.g., one liter).
- Centrifuge Water--3 grab samples per run. These will be composited into a single sample per run.

- Plant Influent Wastewater--3 grab samples per run. Each of these samples will be prepared and analyzed separately.

One liter aliquots of each of the above sample types will be extracted in a separator funnel with methylene chloride as recommended in EPA Method 608. Each extract will be dried using a sodium sulfate column and reduced in volume to 10.0 ml using a Kuderna-Danish apparatus. A 5.0 ml aliquot of each sample will be removed for PCB analysis (pattern matching) employing a gas chromatograph in combination with an electron capture detector. Florisil column chromatography and acid partitioning ( $H_2SO_4$ ) procedures will be employed as needed to facilitate identification and quantitation of aroclor (PCB) mixtures.

#### GC/MS--

Each of the five aqueous sample streams from each test series will be analyzed further using gas chromatography/mass spectrometry. Details on additional sample preparation procedures are provided below.

Scrubber Effluent--A 5.0 ml aliquot from each 10.0 ml extract will be reduced under a stream of prepurified  $N_2$  to 100  $\mu$ l GC/MS analyses will be conducted for each of ten PCB positional isomer categories.

Plant Influent Wastewater--A single composite sample will be prepared from the three grab samples taken during each run. A one liter composite sample representing each test series will be fortified with a surrogate mixture and extracted (3X) with methylene chloride per EPA Method 625. Each of the acid and base-neutral fractions will be reduced in volume to 1.0 ml in a Kuderna-Danish apparatus. Analysis by GC/MS will be performed for each of the EPA Method 625 analytes. Additional major organic components in the extract will be identified and quantitated as described previously using EPA/NIH libraries.

Scrubber Water/Precooler Feed and Centrifuge Water--Samples from the above categories that contained elevated levels of PCBs (GC/ECD) will be further analyzed using GC/MS to include qualitative analysis for PCBs (presence or absence confirmation). For this analysis 1/2 (e.g., 5.0 ml) of the existing extract will be reduced in volume using  $N_2$  to 100  $\mu$ l for PCB confirmation in the total ion mode. In addition each of these extracts will simultaneously undergo GC/MS analyses for the identification and quantitation of major organic components with particular emphasis on chlorinated organics (e.g., chlorinated pesticides, chlorinated benzenes, chlorinated naphthalenes (PCNs)).

#### GC/ECD Analysis--PCBs--

PCB analyses of all previously designated sample extracts will be conducted using a Hewlett-Packard 5840 gas chromatograph fitted with a  $Ni^{63}$  electron capture detector. This includes each of the aqueous sample types discussed previously, as well as the sewage sludge feed grab samples collected during each test series. A summary of pertinent GC/ECD operating conditions is provided in Table 6. Instrument calibration will be provided using Aroclor 1242 and Aroclor 1254 reference materials, since these were the

TABLE 6. GC/ECD CONDITIONS FOR PCB ANALYSIS

---

Instrument	Hewlett-Packard 5840A with Ni <sup>63</sup> electron capture detector and HP 7671A automatic sampler
Column	1.5% OV-17/1.95% QF-1 on 100/120 Chromosorb WHP, 6 ft x 2 mm
Temperatures	
Column	185°C
Injector	270°C
Detector	350°C
Injector volume	4.0 µl
Run time	30 min
Carrier flow	UHP argon/methane, 35 ml/min

---

predominant Aroclors noted in the 1977 test series. EPA/EMSL check samples will be analyzed to verify instrument calibration for each of these Aroclor mixtures. Pattern recognition procedures will be used for quantitation of each of the two Aroclor mixtures (peak summations). Webb-McCall quantitative procedures will be used in instances where chromatographic profiles do not coincide with those of the Aroclor reference materials.

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APPENDIX B

QUALITY ASSURANCE PROJECT PLAN

EPA CONTRACT NO. 68-02-3168  
TECHNICAL SERVICE AREA 3  
WORK ASSIGNMENT NOS. 58, 79 AND 99  
(GCA 1-619-058, 1-619-079 and 1-619-099)

REVISED  
QUALITY ASSURANCE PROJECT PLAN FOR  
SAMPLING AND ANALYSIS ACTIVITIES  
FOR THE MULTIPLE HEARTH SEWAGE SLUDGE  
INCINERATOR AT THE NEW BEDFORD  
MUNICIPAL WASTEWATER TREATMENT PLANT

GCA CORPORATION  
GCA/TECHNOLOGY DIVISION  
Bedford, Massachusetts 01730

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## 1.0 PROJECT DESCRIPTION

The objective of this program is to quantify the atmospheric emissions of polychlorinated biphenyls (PCBs) from the New Bedford Wastewater Treatment Plant Multiple Hearth Incinerator. GCA will also attempt to establish the fate of PCBs within the treatment plant facility by the collection and analysis of samples from applicable process streams. Included in this evaluation is the determination of levels of PCBs, PCDD, PCDF and other chlorinated combustion byproducts. The work to be performed has been broken down into the four tasks listed below.

- Task 1 - Develop Test Plan and QA Plan--These plans will describe the test site, streams to be sampled, sampling and analysis procedures, project organization and QA/QC measures to be implemented, thus providing a concise overview of the entire program. These plans will be implemented after approval by the EPA Project Officer.
- Task 2 - Conduct Field Sampling Program--Samples will be collected as described in the Test Plan and Section 4.0 of this Plan. The primary objective of this portion of the program is to provide the representative samples necessary for determination of the fate of PCBs in this incinerator.
- Task 3 - Conduct Laboratory Analyses--A variety of analyses will be conducted on program samples. The analytical techniques are discussed in detail in the Test Plan and addressed in Section 7.0 of this QA Plan.
- Task 4 - Prepare Final Report--The results of this program will be presented in a comprehensive, final report.

## 2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

Figure 2-1 presents GCA's organization chart for this project showing the individuals responsible for each element of the overall task. The key individual responsible for QA is the Division QA Manager who reports directly to the Division General Manager. The department QC Coordinators report directly to their Department Manager and the QA Manager, enabling them to implement QC measures on all projects independent of the project manager. The responsibilities of these individuals on this project are briefly described below.

### 2.1 QA MANAGER'S RESPONSIBILITIES

The Division QA Manager is the responsible Quality Assurance Officer for this project. She has aided in the development of the QA Project Plan and reviewed and approved the plan before its submittal to the Project Officer. She will ensure that any necessary revisions are made and she will check on implementation of the QA Plan during the life of the project, scheduling performance or system audits as necessary.

She will initiate or follow-up on corrective actions and aid in preparation of a section of the Final Report summarizing QA/QC activities and including estimates of the precision, accuracy and completeness of data achieved. Quality problems found and corrective actions taken will be described.

### 2.2 FIELD AND LABORATORY QC COORDINATOR'S RESPONSIBILITIES

The Environmental Measurements Department (Field) and the Laboratory Analysis Department QC Coordinators oversee and implement the ongoing QC program within their departments. They have aided in the preparation of this QA Plan and will ensure that the required QC procedures are followed. They will initiate corrective actions as necessary, and maintain and report the QC records and results for this project.

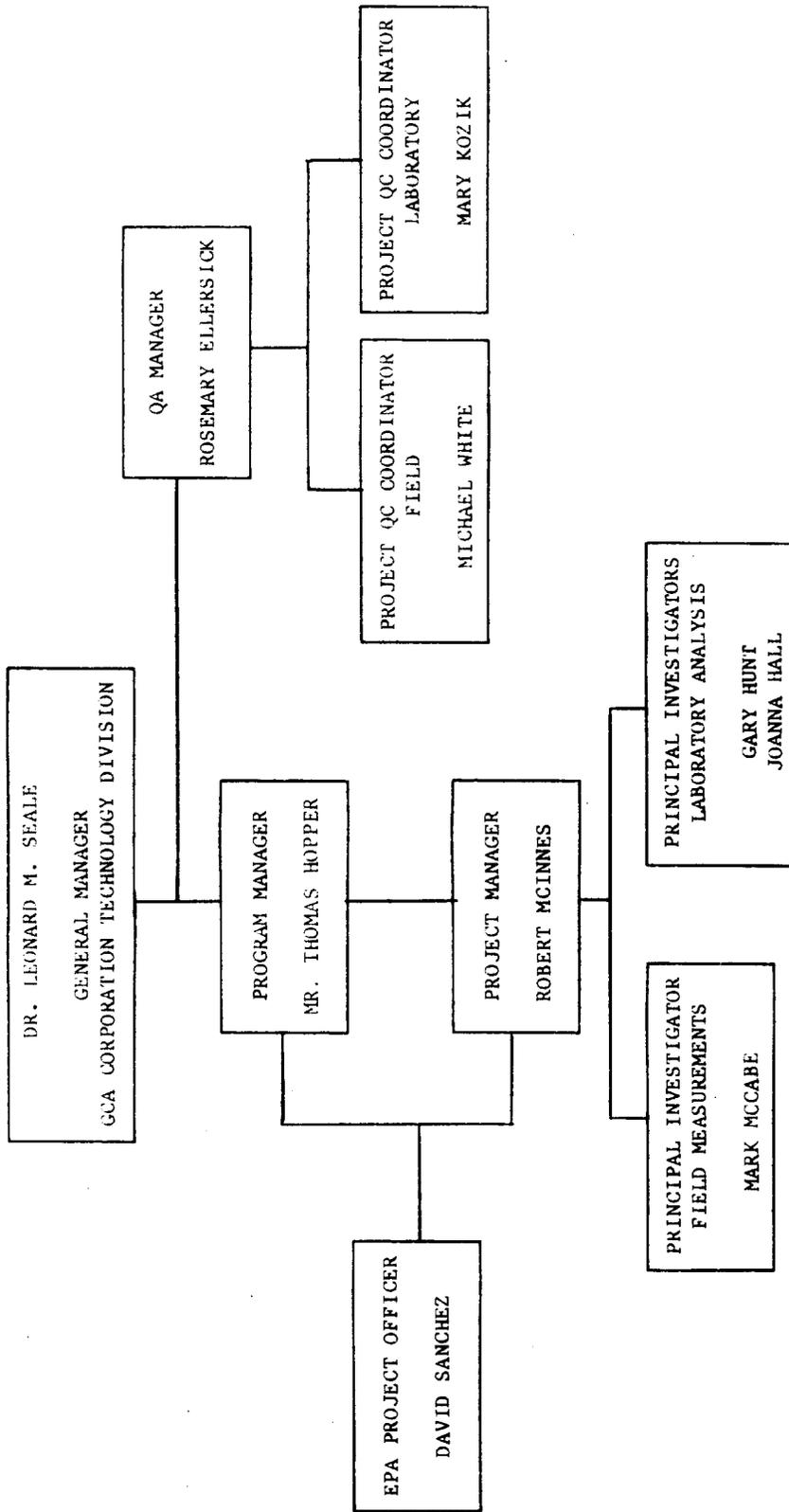


Figure 2-1. Project organization and responsibility.

### 3.0 QA OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY

#### 3.1 PRECISION, ACCURACY AND COMPLETENESS

The collection of data which can be used in mass balance calculations to determine the fate of PCBs during the incineration process requires that sampling and analysis procedures be conducted with properly operated and calibrated equipment by trained personnel. Precision and accuracy goals for sampling procedures are shown in Table 3-1; Table 3-2 presents the precision and accuracy goals for the analytical procedures.

Every attempt will be made to have all data generated be valid data. However, realistically, some samples may be lost in laboratory accidents and some results may be deemed questionable based on internal QC procedures. The objective will be to have 95 percent of the data valid.

#### 3.2 REPRESENTATIVENESS AND COMPARABILITY

It is recognized that the usefulness of the data is also contingent upon meeting the criteria for representativeness and comparability. Wherever possible, reference methods and standard sampling procedures will be used. The QA objective is that all measurements be representative of the media and operation being evaluated. The detailed requirements for gaseous and particulate sampling given in Reference Methods 1-5, and the continuous monitoring requirements given in Performance Specification Tests 3 and 4 will be followed to ensure representative sampling of flue gases. The frequent grab sampling of sludge, ash and process water during each flue gas test run should provide representative samples of these media, comparable to the flue gas samples.

The corresponding QA objective is that all data resulting from sampling and analysis be comparable with other representative measurements made by GCA or another organization on this or a similar incinerator operating under similar conditions. The use of published sampling and analytical methods and standard reporting units will aid in ensuring the comparability of the data.

TABLE 3-1. QA OBJECTIVES FOR PRECISION, ACCURACY AND COMPLETENESS--  
FIELD SAMPLING

Measurement (parameter)	Precision (standard deviation)	Accuracy (relative accuracy)	Completeness
Particulate Matter (EPA Reference Method 5)	12% RSD	Not determined	95%
Sorbent Trap-Florisil, XAD (Flue Gas - Train)	<u>+50%</u>	<u>+50%</u>	95%
Fixed Gases (CO, O <sub>2</sub> , CO <sub>2</sub> ) (Continuous Monitor)	<u>&lt;2.5%</u>	<u>+10%</u>	95%

RSD = Relative standard deviation.

TABLE 3-2. QA OBJECTIVES FOR PRECISION, ACCURACY AND COMPLETENESS--  
LABORATORY ANALYSIS

Measurement method	Matrix	Precision (relative standard deviation)	Accuracy	Completeness
GC-ECD	Flue gas	≤20%	+20%	95%
	Sludge	≤20%	+20%	95%
	Ash	≤20%	+20%	95%
	Water	≤20%	+20%	95%
GC/MS	Flue gas	≤30%	+30%	95%
	Sludge	≤30%	+30%	95%
	Ash	≤30%	+30%	95%

## 4.0 SAMPLING PROCEDURES

### 4.1 FLUE GAS SAMPLING PROCEDURES

The sampling procedures for semivolatile organics (PCBs, PCDDs, PCDFs) in the incinerator flue gas will include three complete tests of the incinerator in normal operation. Each of these tests will be conducted under normal operating conditions.

#### 4.1.1 Flue Gas Sampling Train

A modified Method 5 train will be used for the simultaneous collection of particulates, HCl, PCBs, PCDDs, and PCDFs. A schematic of the train is presented in Figure 4-1.

The sampling train will be operated in accordance with the procedure outlined in EPA Method 5. Accordingly, representative samples of the flue gas will be obtained from predetermined sampling points in the stack. The specifications of these points will be calculated in accordance with the guidelines set forth in EPA Method 1. The sample train will consist of a glass-lined, heat-traced probe with a stainless steel button hook nozzle. The probe is equipped with a thermocouple and pitot tubes. The flue gas will pass through the probe and then through a heated glass fiber filter (Reeve Angel 934 AH filter paper). Downstream of the heated filter, the sample gas will pass through two impingers containing distilled deionized water, an empty knockout impinger, and two organic sorbent traps containing florisisil and XAD-2 resins, respectively. The temperature of the sample gas entering the sorbent traps will be maintained at less than 68°F throughout the sampling period. The sorbent traps are followed by a series of three impingers. The first and second impingers will contain 100 ml of 1N NaOH for HCl collection. The third impinger will contain a known amount of dessicant for moisture removal. The impingers will be followed by a pump, dry gas meter and calibrated orifice.

The sampling and velocity traverses will be conducted along two perpendicular diameters of the stack. A total of 24 points will be sampled on

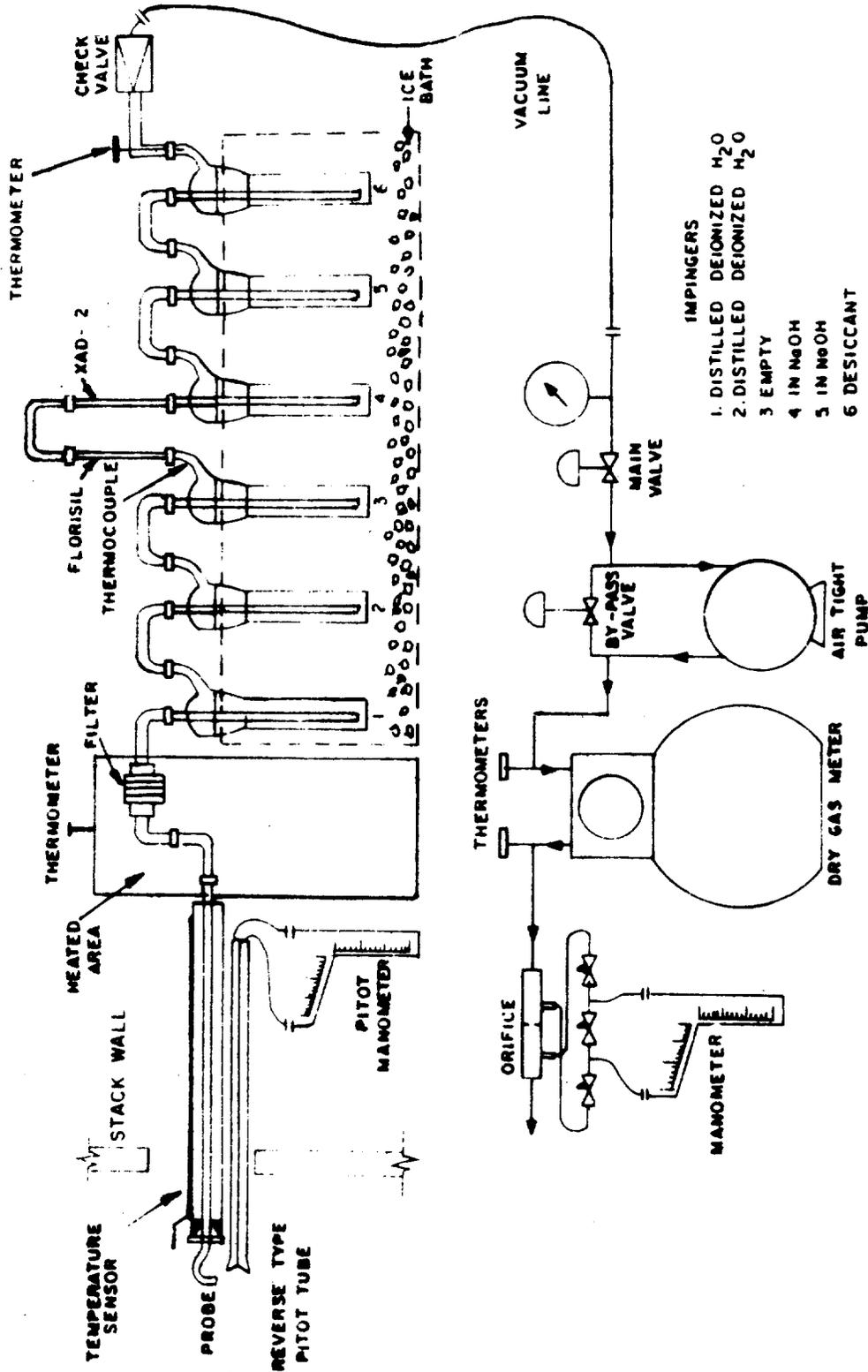


Figure 4-1. Modified Method 5 train.

each diameter, resulting in a final total of 48 sampling points. The sampling time will be 4 minutes per point for a total sampling time of 192 minutes per run.

As required by EPA sampling protocol, a blank train will be set up and recovered with the flue gas sampling train each day in order to provide appropriate blank corrections.

The recovery procedures for the sampling system will be:

1. Remove the sampling train to the predetermined recovery area.
2. Note the condition of the trains (e.g., improper color, florisil/XAD-2 condition, etc.).
3. Remove and seal the florisil and XAD-2 tubes. These tubes should be properly marked as X-FL and X-XR, respectively. The run number (X) should precede all sample codes (e.g., 1-FL).
4. Measure the volume of the impinger catches (impingers 1, 2 and 3) in a precleaned glass graduated cylinder. Pour the impinger contents into a precleaned amber glass container with a Teflon cap. Label the sample X-COND-0.
5. Rinse the impingers, probe and nozzle thoroughly with acetone followed by hexane. This rinse volume should be put into a precleaned amber glass container with a Teflon cap. Label the sample X-PR.
6. Measure the volume of impingers 4 and 5 and pour contents into a nalgene container. Label the sample as X/MP.
7. Record the weight gained by the silica gel impinger.
8. Be sure that all containers are properly sealed, labeled, and the liquid level marked. Log all samples on sample packing list.

#### 4.1.2 Continuous Monitoring System

A portable continuous emissions monitoring system (CEMS) will be used to document combustion conditions. The monitoring system consists of a gas conditioning system and sensors for CO, CO<sub>2</sub> and O<sub>2</sub>.

A schematic diagram of the CEMS is shown in Figure 4-2. Briefly, the sample is extracted from the flue stream at a point of average concentration and passed through a glass fiber filter for particulate removal. The gas exiting the filter element is then dried by passing it through a condenser for primary moisture removal. Gas analysis is performed using the instruments and operating ranges specified in Table 4-1.

Continuous monitoring of the flue gas will be performed over the entire length of the proposed testing. On each day of flue gas testing the following sequence will be implemented:

1. Arrive onsite, inspect condition of equipment.
2. Set up and leak check conditioning system through manifold.
3. Connect all three analyzers to manifold and individual strip chart recorders.
4. Perform initial calibration of all monitors with zero, mid and high span certified gases. Note and make any adjustments on the monitors. All calibration gases are introduced through the conditioning system sample probe.
5. Monitor O<sub>2</sub>, CO<sub>2</sub> and CO throughout the flue gas testing making sure to mark the strip charts noting the beginning and end of the test runs.
6. At the end of the run, recalibrate the monitors and note all values on the appropriate data sheet to determine monitor drift.
7. Monitoring data will be reduced and presented as 15-minute averages.

The continuous monitoring system inspection, installation and operation will be performed in accordance with the Horiba Instruments Incorporated Instruction Manual.

#### 4.2 SAMPLING PROCEDURES FOR SOLIDS

Sludge and ash samples will not be accurately weighed or measured in the field. The amount of sample collected will be kept approximately the same for each type of sample by using the specified size container and filling it to the indicated fraction of its capacity. The volumes cited below are based on that fraction and are included to give an estimate of sample size.

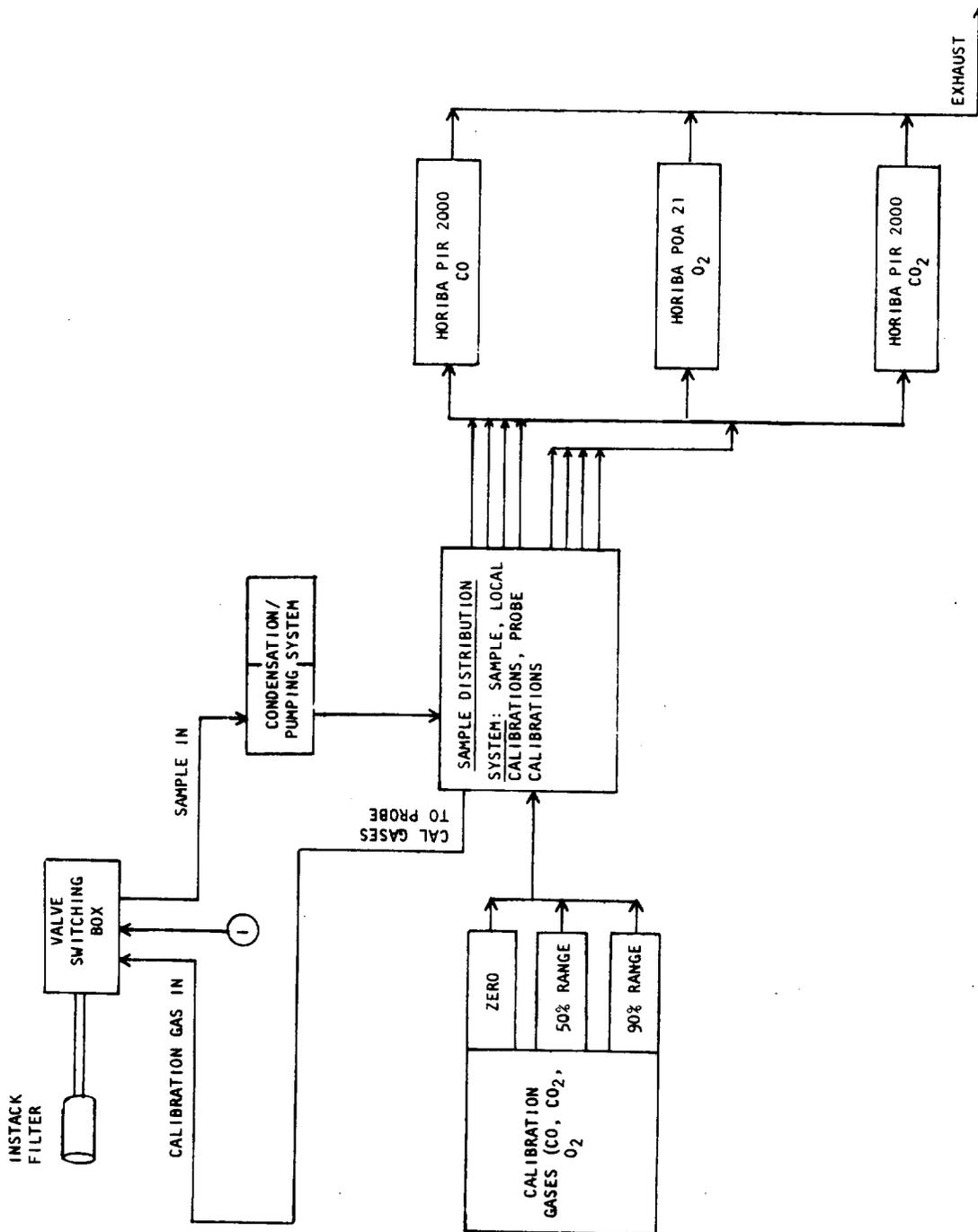


Figure 4-2. Continuous monitor flow schematic.

TABLE 4-1. ANALYZER SPECIFICATIONS OF CONTINUOUS MONITORS

	Horiba PIR 2000 CO <sub>2</sub> Analyzer	Horiba PIR 2000 CO Analyzer	Horiba FOA 21 OPE 315 O <sub>2</sub> Analyzer
Operating Sensitivity Ranges	0-5% CO <sub>2</sub> , FS 0-15% CO <sub>2</sub> , FS 0-25% CO <sub>2</sub> , FS	0-500 ppm CO, FS 0-1500 ppm CO, FS 0-2500 ppm CO, FS	0-5% O <sub>2</sub> , FS 0-20% O <sub>2</sub> , FS 0-50% O <sub>2</sub> , FS
Operating Temperature Ranges	24° F - 122° F	24° F - 122° F	32° F - 122° F <95% Humidity
Analysis Method	Nondispersive Infrared	Nondispersive Infrared	Polarographic
Linearity	+ - 1% FS	+ - 1% FS	+ - 0.5% of Full Scale
Accuracy	+ - 1% of Full Scale	+ - 1% of Full Scale	+ - 1% of Full Scale
Drift	+ - 1% of Full Scale in 24 hours in both zero and span	+ - 1% of Full Scale in 24 hours in both zero and span	< 5% Full Scale for 24 hours in both zero and span
Noise Level	<0.5% of Full Scale in most sensitive range	<0.5% of Full Scale in most sensitive range	< .25% of Full Scale in most sensitive range

A grab sampling procedure will be used to obtain a series of seven sludge samples during each test run. The sludge sampling location will be at the end of the screw conveyor, as the sludge enters the incinerator. Grab samples, each approximately 50 ml in volume, will be collected at the beginning and at 30 minute intervals throughout each test run. These samples will be collected in 250 ml wide mouth amber glass jars with teflon-lined caps. The 28 jars, seven for each test run, will be clearly labeled in sequential order, maintained at 4°C and then transported to the laboratory for analysis.

Ash samples will also be obtained by grab sampling. These samples will be taken from the ash storage hopper at the beginning, and at 60-minute intervals during each test run. Sample size will be approximately 75 ml or one third of a 250 ml wide-mouth amber glass jar. All ash samples from one test will be transferred and composited in a 500 ml wide-mouth amber glass bottle with a teflon-lined cap. The three composite bottles, one for each test run, will be clearly labeled, maintained at 4°C and transported to the laboratory for analysis.

#### 4.3 SAMPLING PROCEDURES FOR WATER

The precooler/scrubber water feed will be sampled from a tap located on the precooler inlet. Four 250 ml samples will be taken at 45-minute intervals. These samples will be transferred and composited in a 1250 ml wide mouth amber glass bottle with a teflon-lined cap. The three composite bottles shall be clearly labeled and then transported to the laboratory for analysis.

Samples of the precooler/scrubber water effluent will be taken from a tap located on the scrubber drain line at 45-minute intervals during each test run. The volume of each sample will be 500 ml. The samples will be transferred and composited in a 3000 ml wide mouth amber glass bottle with a teflon-lined cap. The three composite bottles will be clearly labeled, maintained at 4°C and then transported to the laboratory for analysis.

#### 4.4 ADDITIONAL SAMPLING QC CONSIDERATIONS

A primary factor in the successful accomplishment of this program will be the pre-test coordination of field and laboratory staff members, concerning sample containers, adsorbents, solvents, impinger solutions and field reagent

blanks. Such planning will minimize the possibility of inadvertent sample contamination in the field. Additional information on field/laboratory coordination is contained in Section 5.0, Sample Custody.

The preparation of all glassware will involve the following sequence: acid soak, alcoholic KOH soak (performed to eliminate visible greases, if present), deionized water rinse, acetone rinse and hexane rinse. Glass sample bottles with Teflon-lined caps and glass petri dishes for the storage of Method 5 particulate filters will be prepared in the same manner.

A blank will be generated in the field for each sorbent, solvent and reagent used in sampling. These field-biased reagent blanks will be later used to correct for any analytical interferences introduced during sample recovery. In addition to reagent blanks, there will be a blank train set up and recovered with the sample trains. These field-biased blank trains will provide a check on the cleanliness of sampling equipment and set-up procedures.

#### 4.5 PREPARATION OF REAGENTS AND SOLVENTS

Organic solvents and laboratory water will be checked for purity before use. All organic solvents will be Burdick and Jackson "Distilled in Glass" grade or J. T. Baker "Resi-Analyzed" grade; the laboratory water is provided by a Continental Water Systems Corporation Cartridge System and meets ASTM criteria for Type I water.

The florisol adsorbent and glass wool packing will be soxhlet-extracted for 20 to 24 hours using a mixture of 85 percent hexane and 15 percent methylene chloride. The florisol will be subsequently dried and activated at 130°C for at least 16 hours before packaging and sealing in adsorbent tubes.

The XAD-2 resin will be soxhlet-extracted using the sequence described in the IERL-RTP Procedures Manual: Level 1 Environmental Assessment (EPA-600/7-89-201). As a quality control check on the cleaned XAD-2, two 50-100 g samples of the cleaned resin will be soxhlet extracted in methylene chloride for 16 to 24 hours. After drying and concentration to 2 ml, the extract will be subjected to Total Chromatographable Organics (TCO) and Gravimetric (GRAV) analysis. The acceptance limits are, for TCO, 10 µg/g and for GRAV, 20 µg/g.

All samples will be maintained at 4°C until the time of transfer to the GCA/Technology Division Sample Bank.

## 5.0 SAMPLE CUSTODY

The purpose of chain-of-custody procedures is to document the identity of the sample and its handling from its first existence as a sample until analysis and data reduction are completed. Custody records trace a sample from its collection through all transfers of custody until it is transferred to the analytical laboratory. Internal laboratory records then document the custody of the sample through its final disposition.

### 5.1 FIELD SAMPLING OPERATIONS

The importance of uncontaminated reagents, collection media and sample containers in collecting valid samples is well recognized by GCA. The collection medium actually becomes part of the sample itself.

Figure 5-1, the Field Reagent Prep Data Sheet, is used to document the preparation of absorbing solutions and reagents brought to the field collection site. The Field/Laboratory Procedure Coordination Form shown in Figure 5-2 is initiated by the Environmental Measurements Department (Field) for all sample collection projects involving analysis of the collected samples at GCA or elsewhere. Each type of sample to be collected is listed individually and assigned a unique identification number. Based on the type of sample and the analysis to be performed, the appropriate sample container and field preservative are specified. Approved lots of solvents and reagents are listed by the Laboratory Analysis Department QC Coordinator who must give final approval to the form. One or both of these forms are used as appropriate to the sample collection task.

Preprinted sample identification tags are used by GCA to ensure that the required information is entered in the field. Each collected sample including duplicates and field blanks shall have a completely filled-in sample tag securely attached. In addition, the sample identification number is marked on the container with a permanent marker so that the sample can be properly identified even if the tag is separated from the sample.

Figure 5-3 shows the general use GCA sample label and chain-of-custody seal that will be used to identify and seal samples in the field. Figure 5-4 shows a general use chain-of-custody record. This three-part carbonless copy





GCA TECHNOLOGY DIVISION ●●▲

DATE: \_\_\_\_\_ SAMPLE NO.: \_\_\_\_\_

SAMPLE DESCRIPTION: \_\_\_\_\_

SAMPLE METHOD: \_\_\_\_\_

ADJUSTMENTS: \_\_\_\_\_

COLLECTED BY: \_\_\_\_\_

COMMENTS: \_\_\_\_\_

\_\_\_\_\_

SHIPPED: \_\_\_\_\_ REC'D: \_\_\_\_\_

 GCA/Technology Division Chain of Custody Sample Seal	FIELD	DATE	SEALERS INITIAL
	SAMPLE NO		
	RUN #	SAMPLE DESCRIPTION	
	SEALERS NAME (PRINT)	SEAL BROKEN BY & DATE	

Figure 5-3. Sample Label and Chain of Custody Seal.



form is based on NEIC format and will be used to document sample transfer in the field and from sampling personnel to the laboratory.

## 5.2 LABORATORY OPERATIONS

All samples submitted to the GCA/Technology Division Analytical Laboratory will be brought to the Sample Bank Manager, Jackie Ferragut, who will continue the chain of custody by assigning a GCA Control Number to each sample on receipt; this number identifies the sample through all further handling. The sample will be recorded in the bound Master Sample Log under its GCA Control Number. A Master Log page is not depicted here because the hand-written records do not reproduce well; however, each page of the Master Log has the following format:

- GCA Control Number
- Sample description
- Sample condition
- Signature of person completing sample record
- Date of sample receipt

GCA/Technology Division maintains large, locked, refrigerated and nonrefrigerated storage areas with provision for hazardous material storage. After necessary preservation or subdivision, the Sample Bank Manager will store each sample in the appropriate area under its GCA Control Number.

The Sample Bank Manager will initiate a page (Figure 5-5) for each sample in the Custody Book and ensure that each handling of the sample is appropriately documented. Each analyst working with the sample will first go to the Sample Bank Manager and record in the Custody Book actions taken on the sample thereby maintaining the chain of custody of the original sample.

When sample preparation and analysis procedures necessitate the transfer of samples between two analysts within the laboratory, a Sample Custody Transfer form (Figure 5-6) is required. This document serves as a supplement to the Custody Notebook record of sample handling and becomes part of the permanent project file.



SAMPLE CUSTODY TRANSFER

PURPOSE

Procedure/Analysis required \_\_\_\_\_

(General information only - analyst MUST refer to Project File for specific details.)

Instrumentation required \_\_\_\_\_

BACKGROUND

Client \_\_\_\_\_

Contract (Charge) No. \_\_\_\_\_ Work Order No. \_\_\_\_\_

SAMPLES

General description of sample type(s) \_\_\_\_\_

List of samples (by GCA Control No.):

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Additional samples (QC-blanks, QC-spikes, etc.):

\_\_\_\_\_  
\_\_\_\_\_

Total number of samples \_\_\_\_\_

Comments \_\_\_\_\_

TRANSFER

From \_\_\_\_\_ Date \_\_\_\_\_

Received by \_\_\_\_\_ Date \_\_\_\_\_

(Location of samples \_\_\_\_\_)

When completed, make 3 copies - one each for originator, recipient, and Task Manager.

RETURN ORIGINAL TO PROJECT FILE.

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Figure 5-6. Sample Custody Transfer form.

All materials such as field and laboratory notebooks and logbooks, field and laboratory data records, correspondence, reports, sample tags, chain-of-custody records and instrument printouts will be clearly labeled with the project number and become a permanent part of the project file.

## 6.0 CALIBRATION PROCEDURES AND FREQUENCY

Calibration procedures for field and laboratory instrumentation are described in the following sections.

### 6.1 SOURCE SAMPLING EQUIPMENT

Calibration of the field sampling equipment will be performed, prior to and at the conclusion of, the field sampling effort. Copies of the initial calibration sheet will be submitted to the field team leader to take onsite for reference, and to the project file. Calibrations will be performed as described in the EPA publication "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods;" acceptance limits are listed below.

- Sample meter system--leak checked, then compared against a wet test meter to a ratio average of  $Y \pm 0.01Y$ .
- Thermocouples--compared to mercury in glass thermometer to accuracy of  $\pm 1.5$  percent, or the use of a constant correction factor.
- Field Barometer--calibrated initially versus mercury in glass barometer to  $\pm 0.01$  in. Hg. The calibration will be checked before and after each field test.
- Nozzles--calibrated with micrometer to the nearest 0.001 inch.
- Triple beam balance--checked with class S weights to  $\pm 1$  mg.
- Type S pitot tube and probe assembly--All dimension specifications are met or calibrated against Type P pitot. Mounted in an interference free manner.
- Calibration of continuous monitors. All calibration gases will be purchased as prepared EPA protocol 1 calibration gases. The instruments will be calibrated prior to the burn by injecting through the probe standards corresponding to 0, 50 and 90 percent of the analytical range. Responses resulting from these injections will be subsequently used to construct a calculation equation in the form:

$$\text{ppm (\%)} = M (\% \text{ chart}) + b$$

where ppm (%) is the concentration of the gas being measured,  
% chart is the response noted on the strip chart recorder,  
M and b are the slope and intercept of the equation.

The calibration equation will be constructed by plotting the span gas and resulting responses as shown in Figure 6-1. A line of best fit will be drawn through the span gas coordinate pairs. If all points do not fall on the best fit line, the line will be passed through the zero pair and averaged between the upper two span responses. The calibration equation will be calculated from the plot as follows:

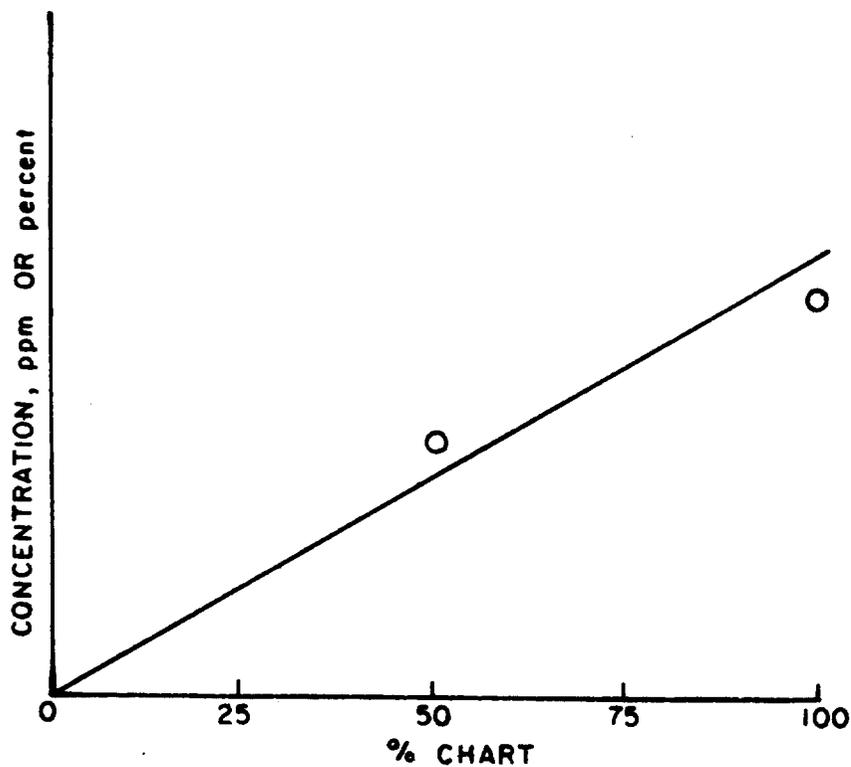


Figure 6-1. Determination of a calibration equation.

Determine the slope (m) as follows:

$$M = \frac{Y_1 - Y_2}{X_1 - X_2}$$

where  $X_1$ ,  $Y_1$  and  $X_2$ ,  $Y_2$  are coordinate pairs determined by the best fit line.

The intercept (b) is determined as follows:

$$b = \left( \frac{\% \text{ chart}}{100} \right) \text{conc. at full scale}$$

The acceptance procedure for an initial calibration involves inserting the responses obtained from the gas injections into the calibration equation and solving for the concentration. The result calculated from the equation is compared for difference to the accepted value of the gas as follows:

$$\% \text{ error} = \frac{\text{Obs} - \text{Acc}}{\text{Acc}} \times 100$$

where Obs is the calculated value  
Acc is the accepted value of the calibration gas.

If both upscale spans are within +2.5 percent error, the calibration is acceptable. For cases where this criteria are not met the instrument linearity (for NDIRS) and/or span gas values must be checked.

## 6.2 ANALYTICAL INSTRUMENTATION

### 6.2.1 Hewlett-Packard 5840A Gas Chromatograph with Ni<sup>63</sup> Electron Capture Detector

#### Calibration Standards--

1. Prepare stock solutions for each Aroclor at concentrations of 1  $\mu\text{g}/\mu\text{l}$  using materials available from the EPA/RTP Reference Standards Repository. Chlorinated biphenyl standards will be

prepared using materials obtained from Ultra Scientific, Inc., Hope, Rhode Island. Use the specified purity of each lot of the compound in calculating the standard concentration. Prepare stock solutions every 6 months or as needed.

2. Prepare the working standards by dilution of the stock solution. The working standards will be prepared as needed.
3. Verify working standard solutions by analysis of Aroclor quality control check samples (EPA/EMSL, Cincinnati, Ohio) before use as calibration standards.

#### Calibration Procedures--

1. Calibrate the instrument daily using 4 to 5 calibration (working) standards.
2. Analyze a laboratory control sample. If the reported values are within 10 percent of the expected values, analysis may proceed.
3. Enter all instrument operating conditions and quality control results in the instrument logbook. The analyst's notebook must contain all information regarding standard preparation. Sign and date all entries.

#### 6.2.2 Hewlett-Packard 5985 GC/MS

#### Calibration Standards--

1. Prepare stock solutions of the chlorinated biphenyls at a concentration of 1  $\mu\text{g}/\mu\text{l}$  using materials obtained from Ultra Scientific, Inc., Hope, Rhode Island. Use the specified purity of each compound lot in calculating the standard concentration. Prepare stock solutions every 6 months or as needed.
2. Prepare working standards by dilution of the stock solutions. The working standards are prepared as needed.
3. Verify standard solutions by analysis of an independent standard. If possible, standards for the various isomers will be obtained from Dr. David Longfellow, Bethesda, Maryland; these standards will be used for verification of the commercial standards before their use as calibration standards.

Calibration Procedures--

1. Calibrate the instrument daily using a minimum of three calibration (working) standards.
2. The following instrumental conditions are normally used:

GC Conditions

- column 1.5 percent OV-17/1.95 percent QF-1 on 100/120 Chromosorb WHP, 6 ft x 2 mm
- injection
  - mode splitless
  - temperature 225°C
- temperature program 160°C for 2 minutes, then 5°C/min to 225°C and hold.

Mass Spectrometer Conditions

- mode mass scan 50 to 450 amu
  - electron energy 70 eV
  - scan time 0.8 sec/scan
3. Analyze a laboratory control sample. If the reported values are within established acceptance limits (generally  $\pm 20$  percent), analysis may proceed.
  4. Enter all instrument operating conditions and quality control results in the instrument logbook. The analyst's notebook must contain all information regarding standard preparation. Sign and date all entries.

## 7.0 ANALYTICAL PROCEDURES

As discussed in Section 4.0, Sampling Procedures, a single sampling train will be utilized during each of the three test runs. This single train will permit collection of PCBs as well as chlorinated dibenzofurans and chlorinated dioxins, combustion by-products potentially formed during the incineration process.

### 7.1 FLUE GAS SAMPLES - PARTICULATE

A total of three runs will be conducted for stack gas emissions. Each of these trains will generate five types of samples: (1) impingers/condensates; (2) combined train (solvent) rinses; (3) a florisil cartridge; (4) an XAD cartridge; and (5) particulates collected on a filter. The combined probe rinse and particulate filter sample will constitute the particulate sample. Analysis of these samples will proceed in the following manner.

1. Record particulate weights for each of the recovered filter catches and probe rinses.
2. Aliquot the filters, and probe rinse solids and combine the aliquots of each for extraction.
3. Surrogate spike the combined sample from each run with an appropriate deuterated analogue and soxhlet extract for 24 hours in methylene chloride.
4. Reduce each extract to 3.0 ml using a Kuderna-Danish evaporative concentrator.
5. Remove 1.0 ml of the 3.0 ml extract and maintain in reserve. Subject 1.0 ml to alumina column cleanup and then separately reduce it and remaining 1.0 ml aliquot to 0.5 ml via the N<sub>2</sub> blowdown prior to GC/MS analyses.
6. Analyze the extracts by selected ion monitoring GC/MS for polychlorinated dioxins and polychlorinated dibenzofurans. Instrument calibration will be provided for a variety of chlorinated dioxins and chlorinated dibenzofurans; a list of representative isomers for the GC/MS screening protocols is shown below:
  - Dibenzofuran
  - 2,8-Dichlorodibenzofuran

- Dibenzo-p-dioxin
  - 1-Chlorodibenzo-p-dioxin
  - 2-Chlorodibenzo-p-dioxin
  - 2,6-Dichlorodibenzo-p-dioxin
  - 1,2,4-Trichlorodibenzo-p-dioxin
  - 1,2,3,4-Tetrachlorodibenzo-p-dioxin
7. Analyze each of the same extracts for each of ten PCB positional isomer categories again employing GC/MS procedures.

Use of the following representative PCB isomers is suggested:

- Biphenyl
  - 2-Chlorobiphenyl
  - 4-Chlorobiphenyl
  - 3,3'-Dichlorobiphenyl
  - 2,5'-Dichlorobiphenyl
  - 2,3',5-Trichlorobiphenyl
  - 2,4,5-Trichlorobiphenyl
  - 2,3',4',5-Tetrachlorobiphenyl
  - 2,2',4,4',5-Pentachlorobiphenyl
  - 2,2',4,4',6,6'-Hexachlorobiphenyl
  - 2,2',3,4,5,6,6'-Heptachlorobiphenyl
  - 2,2',3,3',4,4',5,5'-Octachlorobiphenyl
  - 2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl
  - 2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl
8. Analyze each of these same extracts for other organic components not members at the PCB, PCDD or PCDF chemical categories.

## 7.2 FLUE GAS SAMPLES - GAS PHASE (SORBENTS/IMPINGERS, ETC.)

The remaining sample types from each train will constitute the gas phase portion of each flue gas sample set. This will include the following types of samples: solvent rinses, condensate, aqueous impinger, florisol sorbent and XAD-2 sorbent. The analysis of these samples will proceed as follows:

1. Transfer the impinger condensate waters from the sampling train to a separatory funnel and extract with three 100 ml portions of methylene chloride. Combine these extracts with that of the florisol tube, XAD-2 extract and the corresponding hexane/acetone train rinses.
2. Surrogate spike each XAD-2 cartridge and soxhlet extract with methylene chloride overnight. Retain the solvent extract for combination with the impinger extracts and train rinses.
3. Surrogate spike the contents of each florisol tube using appropriate deuterated analogues and soxhlet extract with hexane overnight. After cooling, the solvent extract will be combined with the impinger/condensate extract, XAD-2 extract and corresponding train rinse.
4. Dry the combined extract using a sodium sulfate column and concentrate to 3.0 ml using a Kuderna-Danish apparatus.
5. Remove 1.0 ml for reserve. Subject 1.0 ml to alumina column cleanup and separately reduce it and the remaining 1.0 ml to 500  $\mu$ l using  $N_2$  blowdown.
6. Proceed with GC/MS analyses for PCBs, PCDDs, PCDFs, and other organics as noted earlier for flue gas particulate samples.

## 7.3 SLUDGE FEED SAMPLES

### 7.3.1 GC/ECD Analysis

1. Remove a 10 g aliquot from each of the 21 grab samples. Air-dry the sample and soxhlet extract with 1:1 hexane: acetone according to the procedure detailed in Reference 7.
2. Concentrate the extract to 5.0 ml by rotary evaporation or using a Kuderna-Danish apparatus.
3. Submit an aliquot of each extract for PCB analysis using a gas chromatograph fitted with an electron capture detector (GC/ECD).

4. All GC/ECD measurements will be conducted on a gas chromatograph fitted with a Ni<sup>63</sup> electron capture detector (e.g., HP5840A). All gas chromatographic peaks will be recorded and integrated using an appropriate data system. Qualitative pattern matching, if needed, will be provided after comparison of the sample elution pattern with Aroclor mixtures.
5. Pattern recognition procedures should be used for quantitation (peak summation). Samples displaying a complex or indistinguishable pattern may warrant Alumina<sup>8</sup> column or acid-partitioning.<sup>10</sup> Webb-McCall procedures should be substituted for samples not displaying a complete Aroclor pattern.
6. All data should be reported on both a dry and wet weight basis (mg/kg). The detection limit for these analyses should be 1 ppm (mg/kg) on a dry weight basis.

#### 7.3.2 GC/MS Analysis

1. Remove a 1.0 ml aliquot from each of seven sludge extracts for each test run. Combine these aliquots to create a composite sample for each of the three runs.
2. Fortify each of these composite samples with a surrogate cocktail.
3. The combined extract should be reduced to 100  $\mu$ l using a stream of nitrogen.
4. Proceed with PCB analyses (positional isomers) as noted before for flue gas samples.

#### 7.4 HOPPER ASH SAMPLES

1. Composite 35 g from each of three grab samples taken during each test series.
2. Spike each 105 g sample with a surrogate mixture and extract overnight with methylene chloride in a soxhlet apparatus.
3. Reduce each extract to 10.0 ml using a Kuderna-Danish apparatus. Remove 3.0 ml for reserve.
4. Reduce a 3.0 ml aliquot to the minimum attainable volume.
5. Subject 3.0 ml to alumina column cleanup and concentration to the minimum attainable volume.
6. Proceed with GC/MS analyses for PCBs, PCDDs, PCDFs and other organics as before for flue gas samples.

## 7.5 AQUEOUS SAMPLES

Analyses will be conducted on the following types of water samples:

- Precooler/Scrubber Water Feed - 3 grab samples per run. These should be composited so as to create a single representative sample per run (e.g. one liter total).
- Scrubber Water Effluent - 3 grab samples per run. Composite into a single representative sample per run (e.g. one liter).
- Centrifuge Water - 3 grab samples per run. Composite into a single sample per run. May require filtration prior to extraction due to large quantities of solids. If this is the case then aqueous samples should be analyzed as described below and solid filtered material should be analyzed separately per sludge analysis procedure.
- Plant Influent Wastewater - 3 grab samples per run. Each at these should be analyzed separately.
  1. Extract 1 liter samples of each of the above with methylene chloride per procedures outlined in EPA Method 608.10
  2. Dry extracts using a sodium sulfate column and concentrate to 10.0 ml using a Kuderna-Danish apparatus.
  3. Proceed with gas chromatographic (GC/ECD) analyses discussed earlier for sludge feed samples. Detection limits of 1  $\mu\text{g}/\text{l}$  are needed. GC/MS analyses will also be required for these samples, since the GC/ECD will serve only as a screening tool.

### 7.5.1 GC/MS - Scrubber Effluent

1. Reduce 5.0 ml aliquot of each extract to 100  $\mu\text{l}$  using a stream at prepurified  $\text{N}_2$ .
2. Proceed with GC/MS analysis for each of ten PCB positional isomer categories as stated earlier for flue gas samples.

### 7.5.2 GC/MS - Plant Influent Wastewater

1. A single composite sample should be prepared from the three grab samples taken during each run.
2. Extract one liter composite sample with methylene chloride and proceed with analysis as in EPA Method 625.

3. Provide results in  $\mu\text{g/l}$  for major components in each extract (acids, base-neutrals, other major compounds).

#### 7.5.3 GC/MS - Scrubber Water Feed/Precooler Centrifuge Water

Samples containing elevated levels of PCBs will require GC/MS analysis to include: qualitative analysis for PCBs (presence or absence confirmation). In this instance, 1/2 (e.g. 5.0 ml) of the existing extract should be reduced in volume using  $\text{N}_2$  to 100  $\mu\text{l}$  (PCB confirmation using total ion mode should be used). In addition, each of these extracts should simultaneously undergo GC/MS analyses for the identification and quantitation of major components with particular emphasis on chlorinated organics (e.g., chlorinated pesticides, chlorinated benzenes, chlorinated naphthalenes (PCNs)). Again, these analyses should be conducted in accordance with procedures provided earlier for flue gas samples.

## 8.0 DATA REDUCTION, VALIDATION AND REPORTING

Extensive QC measures will be used to ensure the generation of reliable data from sampling and analysis activities. Proper collection and organization of accurate information followed by clear and concise reporting of the data is a primary goal in all projects.

### 8.1 FIELD DATA REDUCTION

Appendix A of this QA Plan presents the standardized forms that will be used to record sampling data. The data collected will be reviewed in the field by at least two field crew members. Errors or discrepancies will be noted in the field log book.

Figure 8-1 shows the data flow scheme; Appendix B gives the calculations used to determine concentrations. In practice, these forms are used as worksheets in the field to check on isokinetic sampling conditions. The bulk of the data is computer-processed at GCA to yield the following information:

- Particulate emissions
  - Grains per standard cubic foot
  - Grains per standard cubic foot at 12 percent CO<sub>2</sub>
  - Pounds per hour
- HCl emissions
  - ppm, actual
  - pounds per hour
- Organic emissions
  - ppm, actual
- Volumetric flow rate
  - Cubic feet per minute, dry basis, standard conditions
  - Cubic feet per minute, actual

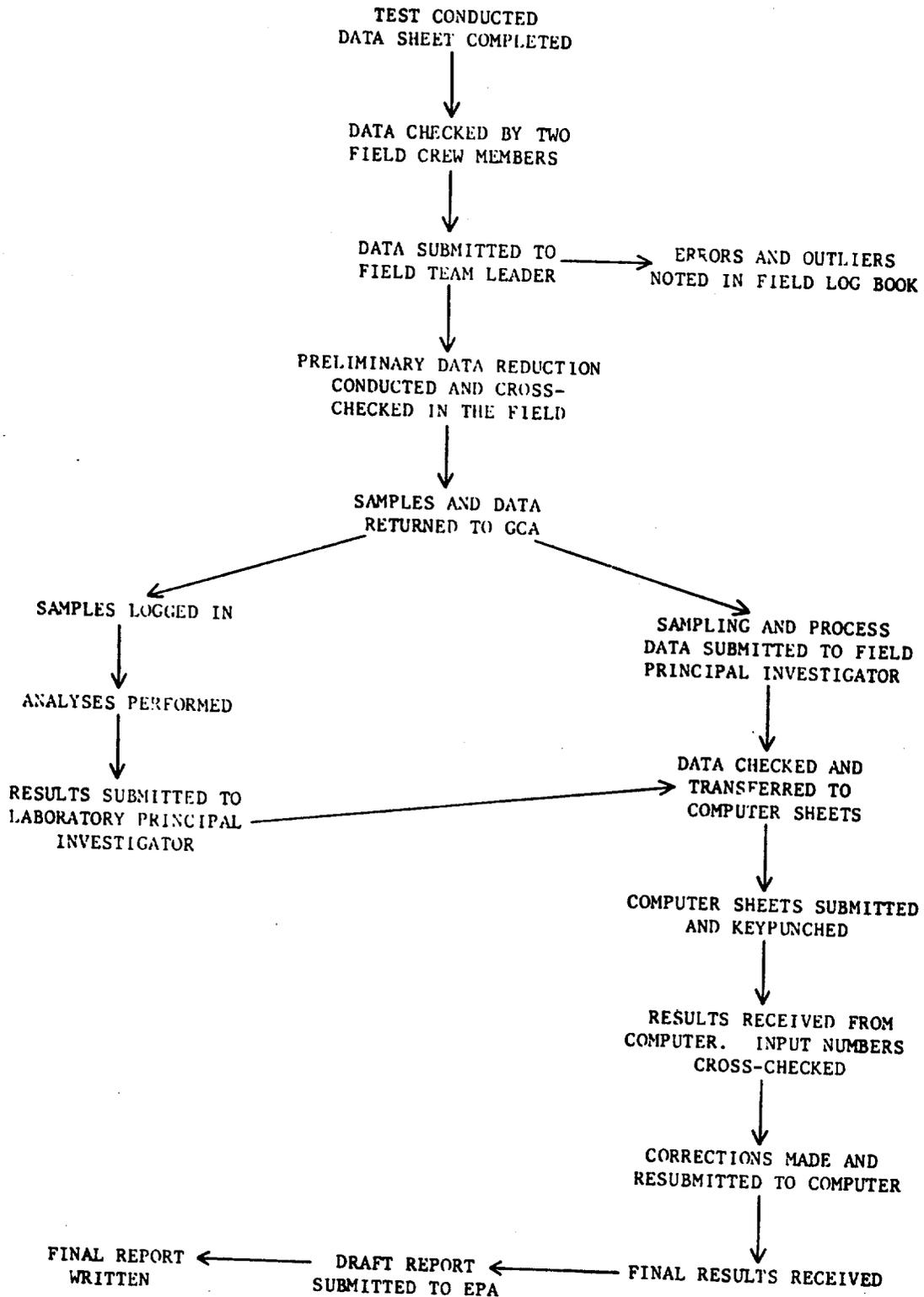


Figure 8-1. Data flow scheme.

- Continuous Monitoring Data--the monitoring data will be reduced and presented in terms of 15 minute averages for the parameters listed below:
  - Carbon monoxide--ppm
  - Carbon dioxide--percent
  - Oxygen--percent
- Dry molecular weight--to nearest tenth gram
- Liquid grab samples
  - Weight per unit volume
- Solid grab samples
  - Weight per unit volume

## 8.2 LABORATORY ANALYSIS DATA REDUCTION

Analysis results will be reduced to concentration units specified in EPA's instructions or the analytical procedure, using the equations given in the analytical procedures. If units are not specified, data from the analysis of water samples will be reported in units of mg/l. Data from the analysis of solid samples will be converted to units of mg/kg using the following equation:

$$X_w = X_v \times V \times \text{d.f.} \div w$$

where  $X_w$  = reported value, mg/kg

$X_v$  = reported sample value, mg/l

$V$  = sample volume, l

d.f. = dilution factor

$w$  = sample weight, kg

Data from the analysis of air samples will be reported as  $\mu\text{g}/\text{m}^3$ . This will be calculated by dividing the total weight of the substance detected by the volume of air sampled. Appropriate blank corrections will be applied in all cases.

### 8.3 DATA VALIDATION

Data validation is the process of filtering data and accepting or rejecting it on the basis of sound criteria. GCA/Technology Division supervisory and QC personnel will use validation methods and criteria appropriate to the type of data and the purpose of the measurement. Records of all data will be maintained, even that judged to be an "outlying" or spurious value. The persons validating the data will have sufficient knowledge of the technical work to identify questionable values.

The following criteria will be used to evaluate the field sampling data:

- Use of approved test procedure
- Steady state operation of the process
- Use of properly operating and calibrated equipment
- Leak checks conducted before and after tests
- Use of reagents that have passed QC checks

The criteria listed below will be used to evaluate the analytical data:

- Use of approved analytical procedure
- Use of properly operating and calibrated instrumentation
- Acceptable results from analyses of EPA QC samples for PCBs in water and sediment (i.e., the reported values should fall within the EPA 95 percent confidence interval for these samples)
- Precision and accuracy achieved should be comparable to that achieved in previous PCB analytical programs

### 8.4 IDENTIFICATION AND TREATMENT OF OUTLIERS

Any data point which deviates markedly from others in its set of measurements will be investigated; however, the suspected outlier will be recorded and retained in the data set while it is investigated. One or both of the following tests will be used to identify outliers.

Dixon's test for extreme observations<sup>11,15</sup> is an easily computed procedure for determining whether a single very large or very small value is consistent with the remaining data. The one-tailed t test for difference<sup>11</sup> may also be used in this case. Reference 10 contains calculation formats and tables of critical values for these tests. It should be noted that these tests are designed for testing a single value. If more than one outlier is suspected in the same set of data, the statistical sources listed will be consulted and the most appropriate test of hypothesis will be used.

Since an outlier may result from unique circumstances at the time of sample analysis or data collection, those persons involved in the analysis and data reduction will be consulted. This may provide an experimental reason for the outlier. Further statistical analyses will be performed with and without the outlier to determine its effect on the conclusions. In many cases, two data sets will be reported, one including and one excluding the outlier.

In summary, every effort will be made to include the outlying value in the reported data. If the value is rejected, it will be identified as an outlier, reported with its data set and its omission noted.

## 8.5 DATA REPORTING

Figure 8-1 shows the field data reduction, validation and reporting process; Figure 8-2 depicts the analytical data reduction, validation and reporting process. Key personnel who will handle data gathering and evaluation are shown in Figure 2-1, the Project Organization Chart.

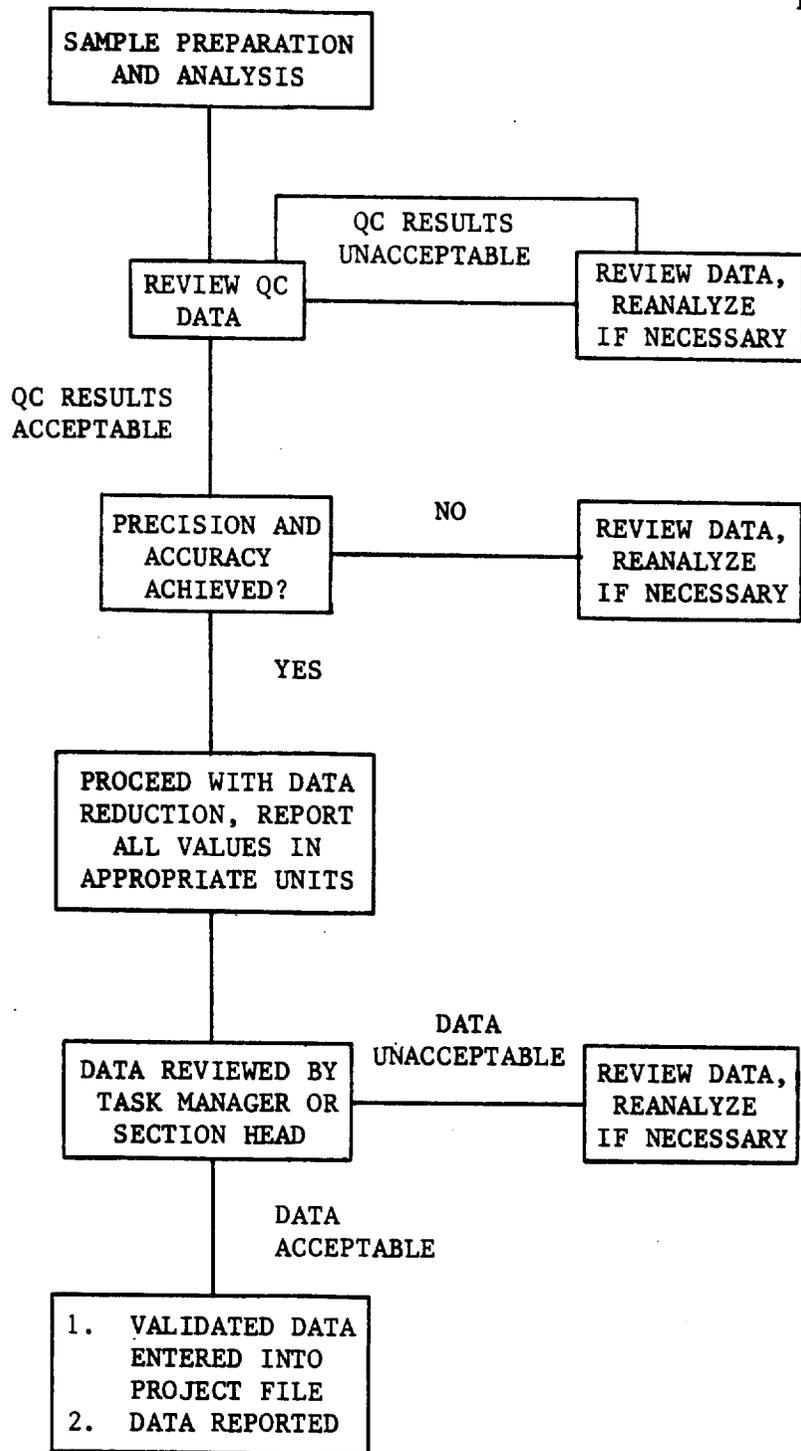


Figure 8-2. GCA analytical data reporting scheme.

## 9.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

Quality control checks will be performed to ensure the collection of representative samples and the generation of valid analytical results on these samples. These checks will be performed by project participants throughout the program under the guidance of the QA Manager and the Field and Laboratory Department QC Coordinators.

### 9.1 SAMPLING QC PROCEDURES

#### 9.1.1 Sampling Equipment QC Checks and Frequency

Calibration of the field sampling equipment will be performed, prior to and at the conclusion of, the field sampling effort. Copies of the calibration sheet will be submitted to the field team leader to take on site for reference, and the project file. Calibrations will be performed as described in the EPA publication "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods;" Section 6.1 presents acceptance limits.

#### 9.1.2 Span Drift Check of Continuous Monitors

The continuous monitoring equipment will be checked during the course of each test by inputting a combination span gas through the probe for analysis. This gas will correspond to the CO and O<sub>2</sub> limits (80 ppm and 3 percent, respectively). The CO<sub>2</sub> span gas will contain approximately 12 to 13 percent CO<sub>2</sub> (mid-span level). Since these gases are all contained in the same cylinder, the use of this technique does not take the monitors out of service for any appreciable time period.

The responses obtained from these span checks will be reduced into units of concentration using the appropriate calibration equation. These data will be plotted on a control chart to determine if a significant drift has occurred. Limits on the control charts are as follows:

- CO--100  $\pm$  12 ppm
- O<sub>2</sub>--6  $\pm$  0.5 percent
- CO<sub>2</sub>--13  $\pm$  0.3 percent

These limits were determined by using the 24 hour drift criteria (contained in PST No. 2: 2.5 percent of span). Data falling outside the chart limits will necessitate corrective actions.

#### 9.1.3 Sample Collection QC Checks

Field-biased blanks of reagents and collection media (deionized water, filters, florisil, XAD-2, solvent rinses, etc.) will be placed in appropriately cleaned and sized sample containers in the field and handled in the same way as actual field samples, to provide a QC check on sample handling. Samples will be collected in sequence or in duplicate to monitor the collection, handling and analysis procedures.

#### 9.1.4 Sample Collection QC Checks and Frequency

- One blank modified Method 5 train of each sampling day.
- One field-biased blank on each reagent and collection medium used on each sampling day and not contained in the sampling trains.
- Seven sequential sludge samples during each test run.

### 9.2 ANALYTICAL QC PROCEDURES

GCA's Quality Control program for laboratory analysis makes use of a number of different types of QC samples to document the validity of the generated data. The following types of QC samples are used routinely:

#### 1. Blank Samples

- a. Field-Biased Blanks--Blank samples which have been exposed to field and sampling conditions in order to assess possible contamination from the field.

- b. Method Blanks--Blanks which are processed through the sample preparation procedures to account for contamination introduced in the laboratory.
- c. Calibration Blanks--Blanks used in instrument calibration; these blanks contain the reagents used in preparing instrument calibration standards except the parameters of interest.
2. Duplicate Samples--A second aliquot of a sample carried through all sample preparation and analysis procedures to verify the precision of the analytical method.
3. Spiked Samples--Samples will be spiked with the parameters of interest at a level two to three times the method detection limit. Samples requiring organic analyses are routinely surrogate spiked with an appropriate deuterated analogue.
4. EPA QC Materials--EPA Quality Control check samples for PCBs in water (WP679 concentrates 1-16) or PCBs in Sediment (WP978, groups 1-3) will be used as appropriate.

Specific quality control protocols for this project will include the following:

- GC/ECD analysis of replicate sludge feed samples.
- GC/MS analysis of aqueous samples containing Aroclor spikes.
- GC/MS analysis of duplicate aqueous spikes containing series of PCB isomers.
- GC/MS analysis of duplicate spiked (PCB, PCDD, PCDF) sorbent samples (florisil, XAD-2).
- GC/MS analysis of duplicate lab spikes containing a series of PCB, PCDD and PCDF isomers.

The duplicate and spiked samples may be submitted as known QC samples, termed laboratory control samples (LCS), or "blind" QC samples, those which are not recognizable to the analyst. LCS are routinely used to ensure that the analytical process is in control. The type and frequency of use of each of these QC measures is summarized below.

- Instrument QC Checks and Frequency
  - daily calibration

- analyze LCS daily before sample analysis; reported values must be within established control limits
- analyze a calibration check sample after every 10 samples; reported value must be within 5 percent of original value.
- Preparation and Analysis Procedure QC Checks and Frequency
  - method blank with each group of 20 or fewer samples
  - laboratory control sample and duplicate with each group of 20 or fewer samples
  - "blind" quality control sample with each group of samples received.

Reagents used in the laboratory are normally of analytical reagent grade or higher purity; each lot of acid or solvent used is checked for acceptability prior to lab use. All reagents are labeled with the date received and date opened. The quality of the laboratory deionized water is routinely checked.

## 10.0 QUALITY ASSURANCE PERFORMANCE AUDITS, SYSTEM AUDITS AND FREQUENCY

GCA/Technology Division's quality assurance program includes both performance and system audits as independent checks of the quality of data obtained from sampling, analysis, and data gathering activities. Every effort is made to have the audit assess the measurement process in normal operation. Either type of audit may show the need for corrective action.

### 10.1 PERFORMANCE AUDITS

The sampling, analysis, and data handling segments of a project are checked in performance audits. A different operator/analyst performs these audit operations to ensure the independence of the quantitative results.

The EPA Project Officer will be requested to obtain audit gases for the continuous monitors. It is anticipated that the monitors will be audited once during the program. The results of the most recent Method 5 Interlaboratory Study will be reported as an audit of the Dry Gas Meters.

EPA Quality Control concentrates and NBS Standard Reference Materials will be used to assess the analytical work. The Laboratory QC Coordinator will direct the inclusion in the sample load of QC samples appropriate to the analyses performed in each batch of 20 or fewer samples so that they are not recognizable to the analyst. In addition, any appropriate interlaboratory study samples which are available during this program will be analyzed to further audit the analytical work.

### 10.2 SYSTEM AUDITS

A system audit will be conducted by the Division QA Manager at least once during the program to ensure that the elements outlined in the Project QA Plan are functioning.

Run 2

Aug.  
cc.

Time	CO	PPM
1307	540	
10	552	
13	560	
16	552	
19	541	
22	541	
25	552	
28	560	
31	569	
34	587	
37	560	
40	531	
43	513	
46	494	
49	524	
52	513	
55	533	
58	552	
1401	550	
04	549	
07	541	
10	541	
13	536	
16	532	
19	522	
22	513	
25	494	

Time	CO	PPM
28	490	
31	490	
34	487	
37	485	
40	476	
43	465	
46	455	
49	443	
52	434	
55	427	
58	420	
1501	411	
04	401	
07	399	
10	392	
13	401	
16	410	
19	415	
22	419	
25	410	
28	401	
31	392	
34	383	
37	373	
40	383	
43	392	
46	405	

Time	CO	PPM
49	392	
52	383	
55	406	
58	392	
1601	373	
04	364	
07	355	
10	345	
13	335	
16	327	
19	327	
22	327	
25	327	
28	327	
1631	327	

## 11.0 PREVENTIVE MAINTENANCE PROCEDURES AND SCHEDULES

GCA/Technology Division follows an orderly program of positive actions to prevent the failure of equipment or instruments during use. This preventive maintenance and careful calibration helps to assure accurate measurements from field and laboratory instruments.

All equipment that is scheduled for field use is cleaned and checked prior to calibration. Once the equipment has been calibrated, sample trains are assembled and leak checked in order to reduce problems in the field. An adequate supply of spare parts is always taken in the field to minimize downtime from equipment failure. Upon return to GCA, an Equipment Evaluation is written and submitted to the department QC Coordinator to point out problems with critical equipment (sample trains, DTRs, pumps, pH meters, etc.) and ensure required maintenance is completed before use in the next project. Table 11-1 summarizes maintenance procedures and their frequency for field sampling equipment.

The CEM systems are operated and maintained in accordance to the Horiba operation manual. Maintenance is performed on a regular scheduled basis prior to use in the field. If extended down time of the monitors is incurred during the sample run an Orsat analyzer will be used. Hourly grab samples will be collected and analyzed for CO<sub>2</sub>, O<sub>2</sub> and CO.

In the analytical laboratories routine maintenance procedures are followed for glassware, water supply, reagents, and analytical balances. These procedures are contained in GCA's Analytical QC Manual. Table 11-2 summarizes maintenance procedures and their frequency for the major laboratory instrumentation to be used in this program.

TABLE 11-1. MAINTENANCE PROCEDURES AND FREQUENCY FOR  
FIELD SAMPLING EQUIPMENT

Equipment	Maintenance procedure/frequency	Spare parts
Vacuum system	<ul style="list-style-type: none"> <li>● Before and after each sample trip; <ol style="list-style-type: none"> <li>1. Check oil and oiler jar</li> <li>2. Leak check</li> <li>3. Vacuum gauge functional</li> </ol> </li> <li>● Yearly or as needed; <ol style="list-style-type: none"> <li>1. Replace valves in pump</li> </ol> </li> </ul>	Additional meter box
Manometer	<ul style="list-style-type: none"> <li>● Before and after each sample trip; <ol style="list-style-type: none"> <li>1. Leak check</li> <li>2. Check fluid for discoloration or visible matter</li> </ol> </li> <li>● Yearly or as needed; <ol style="list-style-type: none"> <li>1. Disassemble and clean</li> <li>2. Replace fluid</li> </ol> </li> </ul>	Spare fluid
Dry gas meter	<ul style="list-style-type: none"> <li>● Before and after each sample trip; <ol style="list-style-type: none"> <li>1. Check meter dial for erratic rotation</li> </ol> </li> <li>● Every 3 months <ol style="list-style-type: none"> <li>1. Remove top plate and check for excessive oil or corrosion</li> <li>2. Disassemble and clean</li> </ol> </li> </ul>	
Nozzles	<ul style="list-style-type: none"> <li>● Before and after each test <ol style="list-style-type: none"> <li>1. No dents, corrosion or other damage</li> </ol> </li> </ul>	
Miscellaneous		Fuses, fittings. Variable transformers.
Diaphragm pump	<ul style="list-style-type: none"> <li>● Before and after each test; <ol style="list-style-type: none"> <li>1. Leak check. Change diaphragm if needed.</li> </ol> </li> <li>● Yearly or as needed <ol style="list-style-type: none"> <li>1. Disassemble and clean</li> </ol> </li> </ul>	
Orsat Analyzer	<ul style="list-style-type: none"> <li>● Before each test; <ol style="list-style-type: none"> <li>1. Leak check</li> <li>2. Inspect for damage</li> </ol> </li> <li>● After each trip <ol style="list-style-type: none"> <li>1. Replace reagents after 10 runs</li> </ol> </li> </ul>	Reagents
Tedlar bags	<ul style="list-style-type: none"> <li>● Before each test <ol style="list-style-type: none"> <li>1. Leak check</li> <li>2. Inspect for damage</li> </ol> </li> </ul>	

TABLE 11-2. MAINTENANCE PROCEDURES AND SCHEDULE FOR MAJOR INSTRUMENTATION

Instrument	Maintenance procedure/schedule	Spare parts
Hewlett-Packard 5840A Gas Chromatograph with Ni <sup>63</sup> Detector <sup>a</sup>	<ol style="list-style-type: none"> <li>1. Change septa daily.</li> <li>2. Check syringe for burrs daily.</li> <li>3. Change gas line dryers quarterly.</li> <li>4. Leak check when installing new analytical column.</li> <li>5. Periodically check inlet system for residue buildup.</li> </ol>	<ol style="list-style-type: none"> <li>1. 10 1 syringes</li> <li>2. Inlet septa</li> </ol>
Hewlett-Packard 5993 GC/MS <sup>b</sup>	<ol style="list-style-type: none"> <li>1. Replace pump oils annually.</li> <li>2. Change septa daily.</li> <li>3. Change gas line dryers quarterly.</li> <li>4. Replace Electron Multiplier as needed.</li> </ol>	<ol style="list-style-type: none"> <li>1. Syringes</li> <li>2. Septa</li> </ol>

<sup>a</sup>Hewlett-Packard Gas Chromatography Instrument Manual, 5840A, Hewlett-Packard, Palo Alto, CA.

<sup>b</sup>HP 5993 GC/MS System Users Manual, Hewlett-Packard, Palo Alto, CA.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA  
PRECISION, ACCURACY, REPRESENTATIVENESS,  
COMPARABILITY AND COMPLETENESS

12.1 PRECISION

12.1.1 Reference Method Testing and Analytical Measurements

Precision will be determined by the analysis of replicate samples and will be expressed as the standard deviation, s, which is determined according to the following equation:

$$S = \sqrt{\frac{\sum_{i=1}^N X_i^2 - \frac{1}{N} \left( \sum_{i=1}^N X_i \right)^2}{N-1}}$$

where S = standard deviation

$X_i$  = individual measurement result

N = number of measurements

Relative standard deviation may also be reported. If so, it will be calculated as follows:

$$RSD = 100 \left( \frac{S}{\bar{X}} \right)$$

where RSD = relative standard deviation, expressed in percent

S = standard deviation

$\bar{X}$  = arithmetic mean of replicate measurements

12.1.2 Continuous Monitoring System

Precision will be estimated from the periodic span check data for each monitor using mid-span Manufacturer's Certified Reference Material (CRM) gases

as noted in Section 9.1. The following equations will be used to estimate precision from at least five data points:

Percent difference ( $d_i$ )

$$d_i = \frac{Y_i - X_i}{X_i} (100)$$

where:  $Y_i$  = Monitor indicated concentration from the i-th span check.

$X_i$  = The span check reference concentration for the i-th precision check.

Mean percent difference ( $\bar{d}_j$ )

$$\bar{d}_j = \frac{1}{n} \sum_{i=1}^n d_i$$

where:  $n$  = number of valid precision checks made during the test period  $j$ .

Standard deviation of the percent difference ( $S_j$ )

$$S_j = \sqrt{\frac{1}{n-1} \sum_{i=1}^n d_i^2 - \frac{1}{n} \left( \sum_{i=1}^n d_i \right)^2}$$

Upper and lower 95 percent probability limits (UPL and LPL) will be computed as follows:

$$UPL = \bar{d}_j + 1.96 S_j$$

$$LPL = \bar{d}_j - 1.96 S_j$$

## 12.2 ACCURACY

### 12.2.1 Reference Method Tests and Analytical Measurements

Accuracy will be estimated from the analysis of "blind" QC samples whose true values are known to the Laboratory QC Coordinator. Accuracy will be expressed as percent recovery or as relative error. The formulas to calculate these values are:

$$\text{Percent Recovery} = 100 \left( \frac{\text{Measured Value}}{\text{True Value}} \right)$$
$$\text{Relative Error} = 100 \left( \frac{\text{Measured Value} - \text{True Value}}{\text{True Value}} \right)$$

Post sampling field calibration checks on the dry gas meter will be performed at the conclusion of each modified Method 5 run.

### 12.2.2 Continuous Monitoring System

Relative accuracy will be estimated from the period span check data for each monitor using CRM gases. The cylinder value will be used as the reference value. The following equations will be used:

The arithmetic differences will be computed as follows:

$$X_i = C_m - C_r$$

where  $X_i$  is the difference between the current concentration and previous calibration value

$C_m$  is the monitor indicated concentration

$C_r$  is the reference value concentration

Next, the arithmetic mean of the individual differences will be calculated:

$$\bar{X} = \frac{\sum X_i}{n}$$

where  $\bar{X}$  is the mean of the differences

$X_i$  are the individual differences

$n$  is the number of data points.

The confidence interval at the 95 percent confidence level will be calculated as follows:

$$CI_{95} = \frac{t_{.975}}{n(n-1)} \sqrt{n \sum (X_i^2) - \sum (X_i)^2}$$

where  $CI_{95}$  is the 95 percent confidence interval

$t_{.975}$  is a statistical "t factor"

$n$  is the number of data points

$X_i$  are the individual differences

The Relative Accuracy (R.A.) will be calculated from the preceding values:

$$R.A. = \frac{\bar{X} + [CI95]}{C_r}$$

### 12.3 COMPLETENESS

Completeness will be reported as the percentage of all measurements made whose results are judged to be valid. The procedures to be used for validating data and determination of outliers are contained in Section 8.0 of this QA Plan. The following formula will be used to estimate completeness:

$$C = 100 \left( \frac{V}{T} \right)$$

where C = percent completeness

V = number of measurements judged valid

T = total number of measurements

### 13.0 CORRECTIVE ACTION

The acceptance limits for the sampling and analyses to be conducted in this program will be those stated in the method or defined by EPA's Project Officer. The corrective actions are likely to be immediate in nature and most often will be implemented by the analyst or Project Manager; the corrective action will usually involve recalculation, reanalysis, or repeating a sample run. GCA's ongoing corrective action policy is described here.

#### 13.1 IMMEDIATE CORRECTIVE ACTION

Specific QC procedures and checklists are designed to help analysts detect the need for corrective action. Often the person's experience will be more valuable in alerting the operator to suspicious data or malfunctioning equipment.

If a corrective action can be taken at this point, as part of normal operating procedures, the collection of poor quality data can be avoided. Instrument and equipment malfunctions are amenable to this type of action and GCA's QC procedures include troubleshooting guides and corrective action suggestions. The actions taken should be noted in field or laboratory notebooks but no other formal documentation is required, unless further corrective action is necessary. These on-the-spot corrective actions are an everyday part of the QA/QC system.

Corrective action during the field sampling portion of a program is most often a result of equipment failure or an operator oversight and may require repeating a run. When equipment is discovered to be defective (i.e., pre- and post-sampling leak check) it is repaired or replaced and a correction factor is established as per the EPA method. If a correction factor is unacceptable the run is repeated. Operator oversight is best avoided by having field crew members audit each others work before and after a test. Every effort is made by the field team leader to ensure that all QC procedures are followed. Economically, it is preferred to repeat a run during a particular field trip rather than return at a later date.

Corrective action for the continuous monitors will involve constructing a new 3-point calibration equation. In order to minimize the time that the analyzer is offline, the span gases will be sampled intermittently (e.g., zero, flue gas, mid span, flue gas, high span).

Corrective action for analytical work would include recalibration of instruments, reanalysis of known QC samples and, if necessary, of actual field samples.

If the problem is not solved in this way, more formalized long-term corrective action may be necessary.

### 13.2 LONG-TERM CORRECTIVE ACTION

The need for this action may be identified by standard QC procedures, control charts, performance or system audits. Any quality problem which cannot be solved by immediate corrective action falls into the long-term category. GCA uses a system to ensure that the condition is reported to a person responsible for correcting it who is part of the closed-loop action and follow-up plan. It is patterned after the system described in Reference 10.

The essential steps in the closed-loop corrective action system are:

- Identify and define the problem.
- Assign responsibility for investigating the problem.
- Investigate and determine the cause of the problem.
- Determine a corrective action to eliminate the problem.
- Assign and accept responsibility for implementing the corrective action.
- Establish effectiveness of the corrective action and implement it.
- Verify that the corrective action has eliminated the problem.

Documentation of the problem is important to the system. A Corrective Action Request Form (shown in Figure 13-1) is filled out by the person finding the quality problem. This form identifies the problem, possible causes and

CORRECTIVE ACTION REQUEST FORM NO. \_\_\_\_\_

Originator \_\_\_\_\_ Date \_\_\_\_\_

Person Responsible for Replying \_\_\_\_\_ Contract Involved \_\_\_\_\_

Description of problem and when identified: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

State cause of problem, if known or suspected: \_\_\_\_\_

\_\_\_\_\_

Sequence of Corrective Action: (If no responsible person is identified, bring this form directly to QA Manager.)

State Date, Person, and Action Planned:

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

CA Initially Approved By: \_\_\_\_\_ Date: \_\_\_\_\_

Follow-up Dates \_\_\_\_\_

Final CA Approval By: \_\_\_\_\_ Date: \_\_\_\_\_

Information copies to:

RESPONSIBLE PERSON: \_\_\_\_\_

QA MANAGER: \_\_\_\_\_

DEPARTMENT MANAGER: \_\_\_\_\_

Figure 13-1. Corrective Action Request Form.

the person responsible for action on the problem. The responsible person may be an analyst, field team leader, department QC coordinator or the QA Manager. If no person is identified as responsible for action, the QA Manager investigates the situation and determines who is responsible in each case.

The Corrective Action Request Form includes a description of the corrective action planned and the date it was taken, and space for follow-up. The QA Manager checks to be sure that initial action has been taken and appears effective and, at an appropriate later date, checks again to see if the problem has been fully solved. The QA Manager receives a copy of all Corrective Action Forms and then enters them in the Corrective Action Log. This permanent record aids the QA Manager in follow-up and makes any quality problems visible to management; the log may also prove valuable in listing a similar problem and its solution.

## 14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

### 14.1 INTERNAL REPORTS

The Analytical Laboratory QC Coordinator and the Environmental Measurements Department QC Coordinator prepare written monthly reports on QC activities for their Department Manager and the Division QA Manager. These reports detail the results of quality control procedures, problems encountered and any corrective action which may have been required.

All Corrective Action Forms are submitted to the QA Manager for initial approval of the corrective action planned and a copy is provided to the department manager. All system audit reports are provided to the project manager, department manager and the Technology Division General Manager.

### 14.2 REPORTS TO EPA

Each data transmittal will contain a summary of QC activities; this summary will include:

- Estimates of precision, accuracy and completeness of reported data
- Quality problems found
- Corrective actions taken

The final report will include a section summarizing QA/QC activities during the program. The Laboratory and Environmental Measurements QC Coordinators and the Division QA Manager will participate in preparing this section.

15.0 REFERENCES

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APPENDIX A  
FIELD DATA SHEETS









GCA/TECHNOLOGY DIVISION  
 SOURCE PARTICULATE SAMPLING  
 TRAIN ASSEMBLY, RECOVERY AND ANALYTICAL REPORT SHEET

Appendix A  
 Revision 0  
 August 3, 1982  
 Page 6 of 8

Run Date: \_\_\_\_\_ Client: \_\_\_\_\_  
 Run No.: \_\_\_\_\_ W. O. No.: \_\_\_\_\_  
 Sample Box No.: \_\_\_\_\_ Plant: \_\_\_\_\_  
 Operator: \_\_\_\_\_ Sampling Location: \_\_\_\_\_

**FRONT HALF**

Laboratory Results

Nozzle and Probe (Cyclone Bypass)-Acetone Wash, Lab No.: \_\_\_\_\_ Residue \_\_\_\_\_ mg  
 Cyclone and Flask-Acetone Wash Lab No.: \_\_\_\_\_ Residue \_\_\_\_\_ mg

<u>Thimble No.</u>	<u>Lab No.</u>	<u>Weight Results</u>
_____	_____	_____ mg

Thimble particulate weight \_\_\_\_\_ mg

<u>Filter No.</u>	<u>Lab No.</u>	<u>Weight Results</u>
_____	_____	_____ mg

Filter particulate weight \_\_\_\_\_ mg

FRONT HALF Sub Total \_\_\_\_\_ mg

**BACK HALF**

Impinger WATER and Water Wash of Impingers  
 Connectors and Back Half of Filter Holder  
 Lab No.: \_\_\_\_\_

Collected on 0.22µ Filter \_\_\_\_\_ mg  
 Chloroform-ether Extract \_\_\_\_\_ mg  
 Aqueous Residue \_\_\_\_\_ mg

ACETONE WASH of  
 Impinger, Connectors and Back Half of  
 Filter Holder  
 Lab No.: \_\_\_\_\_

Residue \_\_\_\_\_ mg  
 BACK HALF Sub Total \_\_\_\_\_ mg

**TOTAL TRAIN**

TOTAL WEIGHT (Front & Back) \_\_\_\_\_ mg

**MOISTURE**

Impingers: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 Final Volume Total \_\_\_\_\_  
 Initial Volume Total \_\_\_\_\_  
 Net Volume \_\_\_\_\_

Silica Gel  
 Weight after test: \_\_\_\_\_  
 Weight before test: \_\_\_\_\_  
 Net Weight: \_\_\_\_\_  
 Container No.: 1. \_\_\_\_\_ 2. \_\_\_\_\_ 3. \_\_\_\_\_ 4. \_\_\_\_\_  
 TOTAL NET WEIGHT-Silica Gel \_\_\_\_\_ gm  
 NET VOLUME-Impingers \_\_\_\_\_ ml  
 TOTAL MOISTURE \_\_\_\_\_ gm

Comments: \_\_\_\_\_

LAB: DATE RECEIVED \_\_\_\_\_  
 DATE REPORTED \_\_\_\_\_

Train Assembled by: \_\_\_\_\_  
 Sample Recovered by: \_\_\_\_\_  
 Sample Analysed by: \_\_\_\_\_

PARTICULATE ANALYSIS

Lab No. \_\_\_\_\_ WO No. \_\_\_\_\_

Client \_\_\_\_\_ Date Received \_\_\_\_\_

Description \_\_\_\_\_

<p>A. <u>SAMPLE VOLUME</u></p> <p>_____ mL</p>	<p>B. <u>WASH VOLUME</u></p> <p>_____ mL</p> <p>_____ mL</p> <p>_____ mL</p> <p>_____ mL</p> <p>Total _____ mL</p>	<p>C. <u>BLANK CORRECTION</u></p> <p>Sample: _____ mL × _____ g/mL = _____ gm</p> <p style="text-align: right;">+</p> <p>Wash: _____ mL × _____ g/mL = _____ gm</p> <p style="text-align: right;">=</p> <p>TOTAL _____ gm</p>
------------------------------------------------	--------------------------------------------------------------------------------------------------------------------	-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------

D. TARE WEIGHTS

	No.	Wt.
Container		_____ g
Filter		_____ g
Thimble		_____ g
Total		_____ g

CONTAINER NO.

\_\_\_\_\_

0.22/0.45  
μ  
FILTER NO.

\_\_\_\_\_

E. GROSS WEIGHTS

<u>RH/°F</u>	<u>Date/Time</u>		<u>RH/°F</u>	<u>Date/Time</u>	
____/____	____/____	(1) _____ g	____/____	____/____	(4) _____ g
____/____	____/____	(2) _____ g	____/____	____/____	(5) _____ g
____/____	____/____	(3) _____ g	____/____	____/____	(6) _____ g

Final Gross Weight \_\_\_\_\_ g

Total Tare Weight - \_\_\_\_\_ g

Residue Weight \_\_\_\_\_ g

Blank Weight - \_\_\_\_\_ g

+

g

E. NET WEIGHT

Remarks: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_



APPENDIX B  
FIELD DATA REDUCTION

Facility \_\_\_\_\_  
 Source \_\_\_\_\_  
 Run \_\_\_\_\_

Job No. \_\_\_\_\_  
 Date \_\_\_\_\_  
 Calc/Review \_\_\_\_\_

SAMPLE CALCULATIONS

Particulate Isokinetic Sampling

I. Calculations for stack volume and Isokinetic Ratio

Time	Dry Gas Meter ft <sup>3</sup>	Pitot ΔP, in. H <sub>2</sub> O	Orifice ΔH, in. H <sub>2</sub> O	Dry Gas Temp °F In Out	Stack Static Pressure in. H <sub>2</sub> O	Stack Temp °F
T	VM	ΔP	PM	TMI TMO	PST	TS

1. DN = Nozzle Diameter, inches \_\_\_\_\_ in.
2. PB = Barometric Pressure, inches Hg \_\_\_\_\_ in. Hg
3. TT = Net Sampling Time, minutes \_\_\_\_\_ min.
4. VM = VM final - VM initial = Sample Gas Volume, ft<sup>3</sup> \_\_\_\_\_ ft<sup>3</sup>
- 4A. VML = Use only if any final or intermediate leak check rate is over 0.02 cfm

LI = Leak rate after any given sampling period, cfm

TLI = Total time of sampling period in which leak occurred, min.

$$VML = VM - [(L1 - 0.02) TL1 + (L2 - 0.02) TL2 + (L3 - 0.02) TL3 + L4 + 0.02) TL4]$$

$$= ( \quad ) - [ ( \quad - 0.02 ) ( \quad ) + ( \quad - 0.02 ) ( \quad ) + ( \quad - 0.02 ) ( \quad ) + ( \quad - 0.02 ) ( \quad ) ]$$

$$= ( \quad ) - [ ( \quad ) + ( \quad ) + ( \quad ) + ( \quad ) ]$$

$$= ( \quad ) - ( \quad ) = \underline{\hspace{2cm}} \text{ ft}^3$$

5. TM = Average Dry Gas Temperature at Meter, °F

$$TM = \frac{\text{Avg. TMI} + \text{Avg. TMO}}{2} = \underline{\hspace{2cm}} \text{ °F}$$

6. PM = Average Orifice Pressure Drop, inches H<sub>2</sub>O

$$PM = \text{Avg. } \Delta H = \underline{\hspace{2cm}} \div 13.6 = \underline{\hspace{2cm}} \text{ in. Hg}$$

7. Volume of dry gas sampled at standard conditions,<sup>a</sup> dscf

$$VMSTD = \frac{528 (Y)(VM) \left( PB + \frac{PM}{13.6} \right)}{29.92 (TM + 460)} \quad Y = \text{dry gas meter calibration factor } \underline{\hspace{2cm}}$$

$$= \frac{528 ( \quad ) ( \quad ) ( \quad )}{29.92 ( \quad )} = \underline{\hspace{2cm}} \text{ ft}^3$$

Job No. \_\_\_\_\_  
 Run No. \_\_\_\_\_

8.  $VW = \text{Total Water Collected} = \text{gm H}_2\text{O Silica gel} + \text{ml Imp. H}_2\text{O} = \text{ml}$   
 $( \quad ) + ( \quad ) = \underline{\hspace{2cm}}$

9. Volume of water vapor at standard conditions, scf  
 $VW \text{ gas} = 0.04715 \times VW = \text{scf} = 0.04715 ( \quad ) = \underline{\hspace{2cm}} \text{ft}^3$

10. Percent moisture in stack gas

$$\% M = \frac{100 \times VW \text{ gas}}{VMSTD + VW \text{ gas}} = \frac{100 ( \quad )}{( \quad ) + ( \quad )} = \frac{( \quad )}{( \quad )} = \underline{\hspace{2cm}} \%$$

11. Mole fraction of dry gas

$$MD = \frac{100 - \%M}{100} = \frac{100 - ( \quad )}{100} = \underline{\hspace{2cm}}$$

12. Molecular weight of dry stack gas

$$\begin{aligned} MWD &= (\%CO_2 \times \frac{44}{100}) + (\%O_2 \times \frac{32}{100}) + [(\%CO + \%N_2) \times \frac{28}{100}] \\ &= ( \quad \times 0.44) + ( \quad \times 0.32) + ( \quad \times 0.28) \\ &= ( \quad ) + ( \quad ) + ( \quad ) = \underline{\hspace{2cm}} \text{lb/lb mole dry} \end{aligned}$$

12A.  $\%EA = \% \text{ Excess Air} = \frac{[(\% O_2 - 0.5 (\%CO))] \times 100}{[(0.264(\%N_2)) - (\% O_2) + 0.5 (\% CO)]}$   
 $= \frac{( \quad - \quad ) \times 100}{[(0.264 ( \quad )) - ( \quad ) + 0.5 ( \quad )]}$   
 $= \frac{( \quad ) - 100}{( \quad ) - ( \quad ) + ( \quad )} = \underline{\hspace{2cm}} \%$

13. Molecular weight of wet stack gas

$$\begin{aligned} MW &= MWD \times MD + 18 (1 - MD) \\ &= ( \quad ) ( \quad ) + 18(1 - \quad ) \\ &= ( \quad ) + ( \quad ) = \underline{\hspace{2cm}} \text{lb/lb mole wet} \end{aligned}$$

14. AS = Stack Area, square inches

Circular,  $= \left( \frac{\text{Stack diameter}}{2} \right)^2 \pi = \left( \frac{\quad}{2} \right)^2 \pi = \underline{\hspace{2cm}} \text{sq. in.}$

Rectangular, = Length  $\times$  width = (  $\quad$  ) (  $\quad$  ) =  $\underline{\hspace{2cm}}$  sq. in.

15. PS = Stack Pressure, absolute, inches Hg = PB  $\pm$  AV PST

PST = Stack static pressure

Job No. \_\_\_\_\_  
 Run No. \_\_\_\_\_

$$\text{PST in. Hg} = \frac{\text{PST in. H}_2\text{O}}{13.6} = \frac{\quad}{13.6} = \quad \text{in. Hg}$$

$$\text{PS} = \text{PB} + \text{Avg. PST} = (\quad) (\quad) = \quad \text{in. Hg}$$

16. TS = Average Stack Temperature, \_\_\_\_\_ °F + 460 = \_\_\_\_\_ °R

$$\text{TS} = \text{Average TS} \quad \sqrt{\text{TS}_{\text{AV}} + 460} = \quad$$

17.  $\text{SDE}_{\text{AV}} = (\sqrt{\Delta P})_{\text{AV}} \times \sqrt{\text{TS}_{\text{AV}} + 460} = (\quad) (\quad) = \quad$

18. Stack gas velocity at stack conditions, afpm

$$\text{VS} = 5130^{(e)} \times C_p \times \text{Avg. (SDE)} \times \left[ \frac{1}{\text{PS} \times \text{MW}} \right]^{\frac{1}{2}} = \text{afpm} \quad C_p = \text{pitot tube coefficient}$$

$$= 5130 \times (\quad) \times (\quad) \times \left[ \frac{1}{(\quad)(\quad)} \right]^{\frac{1}{2}} = \quad \text{afpm}$$

19. Stack gas volumetric flow rate at standard conditions, <sup>c</sup> dscfm

$$Q_s = \frac{528 \times \text{VS} \times \text{AS} \times \text{MD} \times \text{PS}}{(29.92)(144)(\text{TS} + 460)} = \text{dscfm}$$

$$= \frac{528 (\quad) \times (\quad) \times (\quad) \times (\quad)}{29.92 \times 144 (\quad)} = \quad \text{dscfm}$$

20. Stack gas volumetric flow rate at stack conditions, acfm<sup>d</sup>

$$Q_a = \frac{29.92 \times Q_s (\text{TS} + 460)}{(528) (\text{PS}) (\text{MD})} = \text{acfm}$$

$$= \frac{29.92 (\quad) (\quad)}{528 (\quad) (\quad)} = \quad \text{acfm}$$

21. Percent isokinetics

$$\% I = \frac{1,039^{(f)} \times (\text{TS} + 460) \times \text{VMSTD}}{\text{VS} \times \text{TT} \times \text{PS} \times \text{MD} \times (\text{DN})^2}$$

<sup>a</sup> Dry standard cubic feet at 68°F (528R) and 29.92 in. Hg.

<sup>b</sup> Standard conditions at 68°F (528R) and 29.92 in. Hg.

<sup>c</sup> Dry standard cubic feet per minute at 68°F (528) and 29.92 in. Hg.

<sup>d</sup> Actual cubic feet per minute

<sup>e</sup>  $5130 = 85.5 \frac{\text{ft}}{\text{sec}} \left[ \frac{(\text{lb/lb mole})(\text{in. Hg})}{(^{\circ}\text{R})(\text{in. H}_2\text{O})} \right]^{\frac{1}{2}} \times 60 \text{ sec/min}$

<sup>f</sup>  $1039 = \frac{29.92 \text{ in. Hg}}{528 \text{ DegR}} \times \frac{144 \text{ in.}^2}{\text{ft}^2} \times \frac{4}{\pi} \times 100$

Job No. \_\_\_\_\_  
 Run No. \_\_\_\_\_

$$= \frac{1,039 \times ( ) \times ( )}{( ) \times ( ) \times ( ) \times ( ) \times ( )^2}$$

$$= \underline{\hspace{2cm}} \%$$

II. Calculations for grain loading and emission rates

22. Particulate, gr/dscf

$$\text{gr/dscf} = 0.0154 \times \frac{\text{mg}}{\text{VMSTD}} = \frac{0.0154 ( )}{( )} = \underline{\hspace{2cm}} \text{ gr/dscf}$$

23. Particulate at stack conditions, gr/acf

$$\text{gr/acf} = \frac{528 \times \text{gr/dscf} \times \text{PS} \times \text{MD}}{29.92 (\text{TS} + 460)}$$

$$= \frac{528 ( ) \times ( ) \times ( )}{29.92 ( )} = \underline{\hspace{2cm}} \text{ gr/acf}$$

24. Particulate, lb/hr conc. method

$$\text{lb/hr} = \frac{60 \text{ min/hr} \times \text{gr/dscf} \times \text{QS}}{7000 \text{ gr/lb}} = \frac{60 \text{ min/hr}}{7000 \text{ gr/lb}} \times ( ) \times ( )$$

$$= \underline{\hspace{2cm}} \text{ lb/hr}$$

25. Particulate lb/hr area method =  $0.132 \times \frac{\text{gms particulate} \times \text{AS}}{\pi \left(\frac{\text{DN}}{2}\right)^2 \times \text{TT}}$

$$= \frac{0.132 \times ( ) \times ( )}{\pi \left(\frac{( )}{2}\right)^2 \times ( )} = \underline{\hspace{2cm}} \text{ lb/hr}$$

26.  $\frac{\text{lb/hr area} \times 100}{\text{lb/hr conc.}} = \frac{( )}{( )} \times 100 = \underline{\hspace{2cm}} \% \text{ I}$

27. Particulate combustion lb/10<sup>6</sup> Btu heat input method

lb/hr = avg. of area and conc. method = \_\_\_\_\_

10<sup>6</sup> Btu from fuel flow, steam generation or heat rate = \_\_\_\_\_

$$\frac{\text{lb/hr}}{10^6 \text{ Btu hr}} = \frac{( )}{( )} = \underline{\hspace{2cm}} \text{ lb/10}^6 \text{ Btu}$$

28. lb/10<sup>6</sup> Btu F Factor method =

$$\frac{\text{gr/dscf}}{7000} \times F \times \frac{20.9}{(20.9 - \% \text{ O}_2)} = \frac{\hspace{1cm}}{7000} \times ( ) \times \frac{20.9}{[20.9 - ( )]}$$

$$= \frac{\hspace{1cm}}{7000} \times ( ) \times \frac{(20.9)}{( )} =$$

$$= \underline{\hspace{2cm}} \text{ lb/10}^6 \text{ Btu}$$

Job No. \_\_\_\_\_  
 Run No. \_\_\_\_\_

29. Density of stack gas

$$\begin{aligned} \text{a. Wet at stack condition} &= \text{MW lb/lb mol} \left/ \left[ 21.85 \times \left( \frac{\text{TS} + 460}{\text{PS}} \right) \right] \right. \\ &= ( \quad ) \left/ \left[ 21.85 \times \left( \frac{\quad}{\quad} \right) \right] = ( \quad ) \left/ ( \quad ) = \underline{\quad} \frac{\text{lb}}{\text{lb mole wet}} \right. \end{aligned}$$

$$\begin{aligned} \text{b. Dry at } 68^{\circ}\text{F (528R) and 29.92 in. Hg} &= \text{MWD}/385.6 \\ &= ( \quad )/385.6 = \underline{\quad} \frac{\text{lb}}{\text{lb mole dry}} \end{aligned}$$

30. Exhaust gas flow rate

$$\begin{aligned} \text{a. lb/hr dry} &= \text{QS} \times 60 \times \text{density dry} \\ \text{FRS} &= ( \quad ) \times 60 \times ( \quad ) = \underline{\quad} \text{lb/hr} \end{aligned}$$

$$\begin{aligned} \text{b. lb/hr wet} &= \text{QA} \times 60 \times \text{density wet} \\ \text{FRA} &= ( \quad ) \times 60 \times ( \quad ) = \underline{\quad} \text{lb/hr} \end{aligned}$$

$$\begin{aligned} \text{31. gr/dscf at 12\% CO}_2 &= \text{gr/dscf} \times \frac{12}{\% \text{ CO}_2} \\ &= ( \quad ) \times \left( \frac{12}{\quad} \right) = \underline{\quad} \text{gr/dscf} \end{aligned}$$

$$\begin{aligned} \text{32. gr/dscf at 50\% excess air} &= \frac{100 + \text{EA}}{150} \times \text{gr/dscf} \\ &= \left( \frac{\quad}{150} + 100 \right) \times ( \quad ) = \underline{\quad} \text{gr/dscf} \end{aligned}$$

$$\begin{aligned} \text{33. lb pollutant/1000 lb flue gas at 12\% CO}_2 \\ \text{wet or dry} &= \frac{\text{lb pollutant/hr}}{\text{FRA or FRS}} \times \frac{12}{\% \text{ CO}_2} \times 1000 \\ &= \left( \frac{\quad}{\quad} \right) \left( \frac{12}{\quad} \right) \times 1000 \\ &= \underline{\quad} \text{lb/1000 lb} \end{aligned}$$

APPENDIX C  
SAMPLE CALCULATIONS

## DETERMINATION OF SCRUBBER EFFLUENT FLOW RATE

The flow rate of the scrubber effluent was calculated by doing a moisture balance over the system in accordance with the equations presented below. The equation input parameters and results are presented in Table C-1.

$$E_R = M_s + M_f + M_c + F_r - M_{stk}$$

- where
- $E_R$  = scrubber effluent flow rate, gal/min
  - $M_s$  = moisture flow rate into incinerator due to sludge, gal/min
  - $M_f$  = moisture flow rate into incinerator due to fuel and sludge conversion, gal/min
  - $M_c$  = moisture flow rate into incinerator due to combustion air, gal/min
  - $F_R$  = scrubber and precooler feed flow rate, gal/min
  - $M_{stk}$  = moisture flow rate out of incinerator in flue gas, gal/min

$$M_s = S_{RW} \times f_w \times 0.1198 \text{ gal H}_2\text{O/lb H}_2\text{O}$$

- where
- $S_{RW}$  = sludge feed rate (wet basis), lb/min
  - $f_w$  = water fraction = 1 - solids fraction

$$M_f = (G_R C_{wg} + S_{Rd} F_v C_{ws}) (0.1198 \text{ gal H}_2\text{O/lb H}_2\text{O})$$

$$M_f = (G_R \times 0.099 + S_{Rd} \times 0.59) (0.1198)$$

- where
- $G_R$  = natural gas firing rate, ft<sup>3</sup>/min
  - $C_{wg}$  = water wt/volume of natural gas burned (0.099 lb H<sub>2</sub>O/ft<sup>3</sup> gas)
  - $S_{Rd}$  = sludge feed rate (dry basis), lb/min
  - $f_v$  = volatile fraction (0.80)\*
  - $C_{ws}$  = water weight/weight of volatile (0.74 lb H<sub>2</sub>O/lb volatile)\*

---

\*Design parameter

TABLE C-1. DETERMINATION OF SCRUBBER EFFLUENT FLOW RATE

Kun No.	Date	Time	$S_{rw}$ lb/min	$f_w$	$M_g$ gal/min	$G_r$ ft <sup>2</sup> /min	$S_{rd}$ lb/min	$M_f$ gal/min	$M_c$ gal/min	$F_r$ gal/min	$Q_{dstp}$ ft <sup>3</sup> /min	$t_{dg}$	$M_{stk}$ gal/min	Scrubber effluent flow rate, gal/min
2	2/1/84	1307 to 1636	40.9	0.78	3.82	47.84	9.00	1.20	1.36	365	1883	0.99	0.11	371
3	2/6/84	1508 to 1826	42.2	0.78	3.94	25.25	9.28	0.96	1.19	365	1665	0.99	0.09	371
4	2/7/84	1019 to 1342	32.4	0.78	3.03	24.63	7.13	0.80	0.96	365	1665	0.99	0.09	370

$$M_c = C_A (\rho_a G_R A_g + S_{Rd} f_v A_v) (0.1198 \text{ gal H}_2\text{O/lb H}_2\text{O})$$

$$M_c = 0.0096 (0.920 \times G_R + S_{Rd} \times 10.9)$$

where  $C_A$  = water weight/weight of combustion air (0.08 lb H<sub>2</sub>O/lb dry air)\*

$A_g$  = air required for gas combustion at 20 percent excess air  
(12,432 ft<sup>3</sup> air/ft<sup>3</sup> gas)\*\*

$A_v$  = air required for volatiles combustion at 75 percent excess air  
(13.60 lb air/lb volatile)

$\rho_a$  = air density at 70°F (0.074 lb/ft<sup>3</sup>)

$$F_R = F_p + F_s$$

where  $F_p$  = precooler feed flow = 42 gpm

$F_s$  = scrubber feed design flow = 340 gpm

$M_{stk} = (Q_{dstp}/f_{dg} - Q_{dstp}) (0.005567 \text{ gal H}_2\text{O/ft}^3 \text{ H}_2\text{O vapor})$

where  $Q_{dstp}$  = dry volumetric flue gas flow rate at STP (70°F + 29.92 in. Hg),

$f_{dg}$  = dry gas fraction

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\*From psychrometric chart assuming 70°F and 50 percent RH

\*\*Design parameter

Facility \_\_\_\_\_  
 Source \_\_\_\_\_  
 Run \_\_\_\_\_

Job No. \_\_\_\_\_  
 Date \_\_\_\_\_  
 Calc/Review \_\_\_\_\_

SAMPLE CALCULATIONS

Particulate Isokinetic Sampling

I. Calculations for stack volume and Isokinetic Ratio

Time	Dry Gas Meter ft <sup>3</sup>	Pitot ΔP, in. H <sub>2</sub> O	Orifice ΔH, in. H <sub>2</sub> O	Dry Gas Temp °F In Out	Stack Static Pressure in. H <sub>2</sub> O	Stack Temp °F
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T	VM	ΔP	PM	TMI	TMO	PST	TS
1. DN - Nozzle Diameter, inches							_____ in.
2. PB = Barometric Pressure, inches Hg							_____ in. Hg
3. TT = Net Sampling Time, minutes							_____ min.
4. VM = VM final - VM initial = Sample Gas Volume, ft <sup>3</sup>							_____ ft <sup>3</sup>

4A. VML = Use only if any final or intermediate leak check rate is over 0.02 cfm

LI = Leak rate after any given sampling period, cfm

TLI = Total time of sampling period in which leak occurred, min.

VML = VM - [(L1 - 0.02) TL1 + (L2 - 0.02) TL2 + (L3 - 0.02) TL3 + L4 + 0.02) TL4]

= ( ) - [( - 0.02)( ) + ( - 0.02)( ) + ( - 0.02)( ) + ( - 0.02)( )]

= ( ) - [( ) + ( ) + ( ) + ( )]

= ( ) - ( ) = \_\_\_\_\_ ft<sup>3</sup>

5. TM = Average Dry Gas Temperature at Meter, °F

TM =  $\frac{\text{Avg. TMI} + \text{Avg. TMO}}{2}$  = \_\_\_\_\_ °F

6. PM = Average Orifice Pressure Drop, inches H<sub>2</sub>O

PM = Avg. Δ.H = \_\_\_\_\_ ÷ 13.6 = \_\_\_\_\_ in. Hg

7. Volume of dry gas sampled at standard conditions, <sup>a</sup> dscf

VMSTD =  $\frac{528 (Y)(VM)(PB + \frac{PM}{13.6})}{29.92 (TM + 460)}$  Y = dry gas meter calibration factor \_\_\_\_\_

=  $\frac{528 ( ) ( ) ( )}{29.92 ( )}$  = \_\_\_\_\_ ft<sup>3</sup>

Job No. \_\_\_\_\_

Run No. \_\_\_\_\_

8.  $VW = \text{Total Water Collected} = \text{gm H}_2\text{O Silica gel} + \text{ml Imp. H}_2\text{O} = \text{ml}$   
 $( \quad ) + ( \quad ) = \underline{\hspace{2cm}}$

9. Volume of water vapor at standard conditions, scf

$VW_{\text{gas}} = 0.04715 \times VW = \text{scf} = 0.04715 ( \quad ) = \underline{\hspace{2cm}} \text{ft}^3$

10. Percent moisture in stack gas

$\% M = \frac{100 \times VW_{\text{gas}}}{VMSTD + VW_{\text{gas}}} = \frac{100 ( \quad )}{( \quad ) + ( \quad )} = \frac{( \quad )}{( \quad )} =$   
 $= \underline{\hspace{2cm}} \%$

11. Mole fraction of dry gas

$MD = \frac{100 - \%M}{100} = \frac{100 - ( \quad )}{100} = \underline{\hspace{2cm}}$

12. Molecular weight of dry stack gas

$MWD = (\%CO_2 \times \frac{44}{100}) + (\%O_2 \times \frac{32}{100}) + [(\%CO + \%N_2) \times \frac{28}{100}]$   
 $= ( \quad \times 0.44 ) + ( \quad \times 0.32 ) + ( \quad \times 0.28 )$   
 $= ( \quad ) + ( \quad ) + ( \quad ) = \underline{\hspace{2cm}} \text{lb/lb mole dry}$

12A.  $\%EA = \% \text{ Excess Air} = \frac{[(\% O_2 - 0.5 (\%CO))] \times 100}{[(0.264(\%N_2)) - (\% O_2) + 0.5 (\% CO)]}$   
 $= \frac{( \quad - \quad ) \times 100}{[(0.264 ( \quad )) - ( \quad ) + 0.5 ( \quad )]}$   
 $= \frac{( \quad ) \times 100}{( \quad ) - ( \quad ) + ( \quad )} = \underline{\hspace{2cm}} \%$

13. Molecular weight of wet stack gas

$MW = MWD \times MD + 18 (1 - MD)$   
 $= ( \quad ) ( \quad ) + 18(1 - \quad )$   
 $= ( \quad ) + ( \quad ) = \underline{\hspace{2cm}} \text{lb/lb mole wet}$

14. AS = Stack Area, square inches

Circular,  $= \left( \frac{\text{Stack diameter}}{2} \right)^2 \pi = \left( \frac{\quad}{2} \right)^2 \pi = \underline{\hspace{2cm}} \text{sq. in.}$

Rectangular, = Length  $\times$  width =  $( \quad ) ( \quad ) = \underline{\hspace{2cm}} \text{sq. in.}$

15. PS = Stack Pressure, absolute, inches Hg = PB  $\pm$  AV PST

PST = Stack static pressure

Job No. \_\_\_\_\_

Run No. \_\_\_\_\_

$$\text{PST in. Hg} = \frac{\text{PST in. H}_2\text{O}}{13.6} = \frac{\quad}{13.6} = \quad \text{in. Hg}$$

$$\text{PS} = \text{PB} \pm \text{Avg. PST} = (\quad) (\quad) = \quad \text{in. Hg}$$

16. TS = Average Stack Temperature, \_\_\_\_\_ °F + 460 = \_\_\_\_\_ °R

$$\text{TS} = \text{Average TS} \quad \sqrt{\text{TS}_{\text{AV}} + 460} = \quad$$

17.  $\text{SDE}_{\text{AV}} = (\sqrt{\Delta P})_{\text{AV}} \times \sqrt{\text{TS}_{\text{AV}} + 460} = (\quad) (\quad) = \quad$

18. Stack gas velocity at stack conditions, afpm

$$\text{VS} = 5130^{(e)} \times C_p \times \text{Avg. (SDE)} \times \left[ \frac{1}{\text{PS} \times \text{MW}} \right]^{\frac{1}{2}} = \text{afpm} \quad C_p = \text{pitot tube coefficient}$$
$$= 5130 \times (\quad) \times (\quad) \times \left[ \frac{1}{(\quad)(\quad)} \right]^{\frac{1}{2}} = \quad \text{afpm}$$

19. Stack gas volumetric flow rate at standard conditions, <sup>c</sup> dscfm

$$Q_s = \frac{528 \times \text{VS} \times \text{AS} \times \text{MD} \times \text{PS}}{(29.92)(144)(\text{TS} + 460)} = \text{dscfm}$$
$$= \frac{528 (\quad) \times (\quad) \times (\quad) \times (\quad)}{29.92 \times 144 (\quad)} = \quad \text{dscfm}$$

20. Stack gas volumetric flow rate at stack conditions, <sup>d</sup> acfm

$$Q_a = \frac{29.92 \times Q_s (\text{TS} + 460)}{(528) (\text{PS}) (\text{MD})} = \text{acfm}$$
$$= \frac{29.92 (\quad) (\quad)}{528 (\quad) (\quad)} = \quad \text{acfm}$$

21. Percent isokinetics

$$\% I = \frac{1,039^{(f)} \times (\text{TS} + 460) \times \text{VMSTD}}{\text{VS} \times \text{TT} \times \text{PS} \times \text{MD} \times (\text{DN})^2}$$

<sup>a</sup> Dry standard cubic feet at 68°F (528R) and 29.92 in. Hg.

<sup>b</sup> Standard conditions at 68°F (528R) and 29.92 in. Hg.

<sup>c</sup> Dry standard cubic feet per minute at 68°F (528) and 29.92 in. Hg.

<sup>d</sup> Actual cubic feet per minute

<sup>e</sup>  $5130 = 85.5 \frac{\text{ft}}{\text{sec}} \left[ \frac{(1\text{b}/1\text{b mole})(\text{in. Hg})}{(\text{R})(\text{in. H}_2\text{O})} \right]^{\frac{1}{2}} \times 60 \text{ sec/min}$

<sup>f</sup>  $1039 = \frac{29.92 \text{ in. Hg}}{528 \text{ DegR}} \times \frac{144 \text{ in.}^2}{\text{ft}^2} \times \frac{4}{\pi} \times 100$

Job No. \_\_\_\_\_  
Run No. \_\_\_\_\_

$$= \frac{1,039 \times ( ) \times ( )}{( ) \times ( ) \times ( ) \times ( ) \times ( )^2}$$

$$= \underline{\hspace{2cm}} \%$$

II. Calculations for grain loading and emission rates

22. Particulate, gr/dscf

$$\text{gr/dscf} = 0.0154 \times \frac{\text{mg}}{\text{VMSTD}} = \frac{0.0154 ( )}{( )} = \underline{\hspace{2cm}} \text{gr/dscf}$$

23. Particulate at stack conditions, gr/acf

$$\text{gr/acf} = \frac{528 \times \text{gr/dscf} \times \text{PS} \times \text{MD}}{29.92 (\text{TS} + 460)}$$

$$= \frac{528 ( ) \times ( ) \times ( )}{29.92 ( )} = \underline{\hspace{2cm}} \text{gr/acf}$$

24. Particulate, lb/hr conc. method

$$\text{lb/hr} = \frac{60 \text{ min/hr} \times \text{gr/dscf} \times \text{QS}}{7000 \text{ gr/lb}} = \frac{60 \text{ min/hr}}{7000 \text{ gr/lb}} \times ( ) \times ( )$$

$$= \underline{\hspace{2cm}} \text{lb/hr}$$

25. Particulate lb/hr area method =  $0.132 \times \frac{\text{gms particulate} \times \text{AS}}{\pi \left(\frac{\text{DN}}{2}\right)^2 \times \text{TT}}$

$$= \frac{0.132 \times ( ) \times ( )}{\pi \left(\frac{\hspace{1cm}}{2}\right)^2 \times ( )} = \underline{\hspace{2cm}} \text{lb/hr}$$

26.  $\frac{\text{lb/hr area} \times 100}{\text{lb/hr conc.}} = \frac{( )}{( )} \times 100 = \underline{\hspace{2cm}} \% \text{ I}$

27. Particulate combustion lb/10<sup>6</sup> Btu heat input method

lb/hr = avg. of area and conc. method = \_\_\_\_\_

10<sup>6</sup> Btu from fuel flow, steam generation or heat rate = \_\_\_\_\_

$$\frac{\text{lb/hr}}{10^6 \text{ Btu hr}} = \frac{( )}{( )} = \underline{\hspace{2cm}} \text{lb}/10^6 \text{ Btu}$$

28. lb/10<sup>6</sup> Btu F Factor method =

$$\frac{\text{gr/dscf}}{7000} \times F \times \frac{20.9}{(20.9 - \% \text{ O}_2)} = \frac{\hspace{1cm}}{7000} \times ( ) \times \frac{20.9}{[20.9 - ( )]}$$

$$= \frac{\hspace{1cm}}{7000} \times ( ) \times \frac{(20.9)}{( )} =$$

$$= \underline{\hspace{2cm}} \text{lb}/10^6 \text{ Btu}$$



SOURCE \_\_\_\_\_ CALCULATED BY \_\_\_\_\_  
 RUN \_\_\_\_\_ DATE \_\_\_\_\_

F FACTOR CALCULATION (530R)

% H = \_\_\_\_\_ % C = \_\_\_\_\_ % S = \_\_\_\_\_ % N = \_\_\_\_\_  
 % O = \_\_\_\_\_ GCV = \_\_\_\_\_ Btu/lb \_\_\_\_\_

$$F = \frac{10^6}{GCV} [ 3.64 (\%H) + 1.53 (\%C) + 0.57 (\%S) + 0.14 (\%N) - 0.46 (\%O) ]$$

$$F = \frac{10^6}{GCV} [ 3.64 ( ) + 1.53 ( ) + 0.57 ( ) + 0.14 ( ) - 0.46 ( ) ]$$

$$F = \frac{10^6}{GCV} [ ( ) + ( ) + ( ) + ( ) - ( ) ]$$

$$F = 10^6 [ \quad ]$$

F = \_\_\_\_\_ dscf/10<sup>6</sup> Btu

SOURCE New Bedford Sludge Incinerator

CALCULATED BY \_\_\_\_\_

RUN \_\_\_\_\_

DATE \_\_\_\_\_

F FACTOR CALCULATION (530R)

% H = 5.42      % C = 37.2      % S = 0.44      % N = 1.58

% O = 31.4      GCV = 6929      Btu/lb

F =  $10^6 \left[ \frac{3.64}{\text{GCV}} (\%H) + 1.53 (\%C) + 0.57 (\%S) + 0.14 (\%N) - 0.46 (\%O) \right]$

F =  $10^6 \left[ \frac{3.64(5.42)}{\text{GCV}} + \frac{1.53(39.2)}{\text{GCV}} + \frac{0.57(0.44)}{\text{GCV}} + 0.14(1.58) - 0.46(31.4) \right]$

F =  $10^6 \left[ \frac{19.73}{\text{GCV}} + \frac{59.98}{\text{GCV}} + \frac{0.2508}{\text{GCV}} + (0.2212) - (14.44) \right]$

F =  $10^6 \left[ \frac{65.742}{6929} \right]$

F = 9487.9      dscf/10<sup>6</sup> Btu



JOB NO. \_\_\_\_\_

PROJECT WSS Seaford Sludge Incinerator

SUBJECT incinerator feed rate

Run 2

$$\frac{10000 \text{ ft}^3 \text{ natural gas}}{132 \text{ minutes}} * \frac{1060 \text{ BTU}}{\text{ft}^3 \text{ nat gas}} * \frac{3710 \text{ dsf}}{10^6 \text{ BTU}} = \frac{507.3 \text{ dsf}}{\text{min}}$$

generated by nat gas

$$\frac{10^6 \text{ BTU}}{4488 \text{ dsf}} * \frac{\text{Lb dry sludge}}{6729 \text{ BTU}} * \frac{60 \text{ min}}{\text{hr}} * \left[ \frac{(1833.4 - 507.3) \text{ dsf}}{\text{min}} \right] = \frac{1255.9 \text{ Lb dry sludge}}{\text{hr}}$$

Run 3

$$\frac{2000 \text{ ft}^3 \text{ nat gas}}{182 \text{ min}} * \frac{1060 \text{ BTU}}{\text{ft}^3 \text{ nat gas}} * \frac{3710 \text{ dsf}}{10^6 \text{ BTU}} = \frac{101.5 \text{ dsf}}{\text{min}}$$

generated by nat. gas

$$\frac{10^6 \text{ BTU}}{4488 \text{ dsf}} * \frac{\text{Lb dry sludge}}{6729 \text{ BTU}} * \frac{60 \text{ min}}{\text{hr}} * \left[ \frac{(1665.1 - 101.5) \text{ dsf}}{\text{min}} \right] = \frac{1427.0 \text{ Lb dry sludge}}{\text{hr}}$$

Run 4

$$\frac{2000 \text{ ft}^3 \text{ nat gas}}{172 \text{ min}} * \frac{1060 \text{ BTU}}{\text{ft}^3 \text{ nat gas}} * \frac{3710 \text{ dsf}}{10^6 \text{ BTU}} = \frac{130.0 \text{ dsf}}{\text{min}}$$

generated by nat. gas

$$\frac{10^6 \text{ BTU}}{4488 \text{ dsf}} * \frac{\text{Lb dry sludge}}{6729 \text{ BTU}} * \frac{60 \text{ min}}{\text{hr}} * \left[ \frac{(1665.1 - 130.0) \text{ dsf}}{\text{min}} \right] = \frac{1401.1 \text{ Lb dry sludge}}{\text{hr}}$$

1401.1



GCA CORPORATION  
Technology Division

213 Burlington Road  
Bedford, Massachusetts 01730  
Telephone: 617-275-5444  
Telex: 92-3339

SHEET 1 OF       

BY M. White

DATE 7/6/87

JOB NO. \_\_\_\_\_

PROJECT New Bedford Sewage Sludge Incinerator

SUBJECT Incinerator Feedrate Calculations

CH'K. BY \_\_\_\_\_

DATE CH'K. \_\_\_\_\_

B'K. CH'K. BY \_\_\_\_\_

Run 2

$$\frac{10^6 \text{ BTU}}{1488 \text{ dscf}} * \frac{\text{Dry Lb of Sludge}}{6729 \text{ BTU}} * \frac{60 \text{ MIN}}{\text{hr}} * \frac{1225.4 \text{ dscf}}{\text{MIN}} = \frac{1719.9 \text{ LB dry}}{\text{hr}}$$

Run 3

$$0.9126 * \frac{1265.1 \text{ dscf}}{\text{MIN}} = \frac{1519.7 \text{ LB dry}}{\text{hr}}$$

Run 4

$$0.9126 * \frac{1665.2 \text{ dscf}}{\text{MIN}} = \frac{1519.7 \text{ LB dry}}{\text{hr}}$$

Volume of Natural Gas Fired (NL)

	2	3	4
CF) 10000	2000	2000	2000
(MIN) 142	177	142	



GILBERT ASSOCIATES, INC., P. O. Box 1498, Reading, PA 19603/Tel. 215 775-2600

**CERTIFICATE OF ANALYSIS**

LABORATORY NO: 28967

RECEIVED: 5/14/84

REPORTED: 5/31/84

CLIENT: GCA Corp, 213 Burlington Rd.  
Bedford, MA 01730

SAMPLE DESCRIPTION: Sludge Feed  
36298-304 Composite  
Grab  
Sampled 5/9/84 by Mark McCabe/Joe Vitale

-----

		AS RECEIVED	DRY BASIS
Total Moisture	%	77.7	
Ash	%	4.90	22.0
Heating Value	Btu/lb	1545	6929 ✓
Sulfur	% S	0.10	0.44
Carbon	% C	8.75	39.2
Hydrogen (excluding H in moisture)	% H	1.21	5.42
Hydrogen (including H in moisture)	% H	9.90	
Nitrogen	% N	0.35	1.58
Oxygen (excluding O in moisture)	% O	7.00	31.4
Oxygen (including O in moisture)	% O	76.0	

Respectfully submitted,

R. M. Large, Program Supervisor  
Laboratory Services

MAH

cc: Mark McCabe (2)

Project 1-619-099

DATA REPORT SHEET

Chloride

Sample Matrix Impinger Solution Analysis Date 9/10/84

Analytical Method 325.2 Instrument Technicon AutoAnalyzer II

GCA Control No.	Sample Identification	Concentration (mg/l)
36274	Run 2	31.0
36275	Run 3	21.7
36276	Run 4	20.0
36277	Run 2 & 3 FBB	0.5
36278	Run 4 FBB	1.0
36279	Component Blank	< 0.5

- QC Data:
- 1. Lab Control Sample 104% Recovery
  - 2. Sample # 36276 & Spike 108% Recovery
  - 3. Duplicates 36274 A 31.3 mg/l ) 31.00  
36274 B 30.7 mg/l )

\*Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, March 1979.



# Run 2

5 min  
Average  
CO<sub>2</sub> + O<sub>2</sub>

Time	CO <sub>2</sub> %	O <sub>2</sub> %	Time	CO <sub>2</sub> %	O <sub>2</sub> %	Time	CO <sub>2</sub> %	O <sub>2</sub> %
1307	10.4	6.2	1428	10.3	6.4	1549	8.8	8.4
1310	10.4	6.0	1431	10.3	6.2	1553	8.6	8.8
1313	10.4	6.0	1434	10.3	6.2	1555	8.9	9.0
1316	10.4	6.0	1437	10.4	6.8	1558	8.9	8.8
1319	10.4	6.0	1440	10.5	6.8	1601	8.9	8.6
1322	10.4	6.0	1443	10.3	6.8	1604	8.7	8.6
1325	10.4	6.0	1446	10.2	6.4	1607	8.5	8.8
1328	10.4	6.0	1449	10.3	6.6	1610	8.5	9.4
1331	10.4	6.2	1452	10.4	6.6	1613	8.5	9.8
1334	10.4	6.2	1455	10.3	6.8	1616	8.4	9.2
1337	10.4	6.2	1458	10.3	6.8	1619	8.4	9.0
1340	10.4	6.2	1501	10.2	7.2	1622	8.2	9.0
1343	10.3	6.0	1504	10.0	7.2	1625	8.2	9.4
1346	10.3	6.2	1507	10.0	7.2	1628	8.4	9.8
1349	10.3	6.2	1510	10.0	7.4	1631	8.4	9.4
1352	10.3	6.4	1513	10.3	7.4			
1355	10.3	6.4	1516	10.0	7.4			
1358	10.4	6.4	1519	9.7	7.4			
1401	10.5	6.2	1522	9.5	7.8			
1404	10.7	5.8	1525	9.5	8.4			
1407	10.5	5.8	1528	9.5	7.8			
1410	10.3	6.4	1531	9.3	7.8			
1413	10.4	6.2	1534	9.3	8.0			
1416	10.5	6.2	1537	9.2	8.2			
1419	10.5	6.2	1540	9.2	8.2			
1422	10.3	6.2	1543	9.1	8.2			
1425	10.3	7.4	1546	9.0	8.2			

Run 2

Aug.  
cc.

Time	CO	PPM
1307	540	
10	552	
13	560	
16	552	
19	541	
22	541	
25	552	
28	560	
31	569	
34	587	
37	560	
40	531	
43	513	
46	494	
49	524	
52	513	
55	533	
58	552	
1401	550	
04	549	
07	541	
10	541	
13	536	
16	532	
19	522	
22	513	
25	494	

Time	CO	PPM
28	490	
31	490	
34	487	
37	485	
40	476	
43	465	
46	455	
49	443	
52	434	
55	427	
58	420	
1501	411	
04	401	
07	399	
10	392	
13	401	
16	410	
19	415	
22	419	
25	410	
28	401	
31	392	
34	383	
37	373	
40	383	
43	392	
46	405	

Time	CO	PPM
49	392	
52	383	
55	406	
58	392	
1601	373	
04	364	
07	355	
10	345	
13	335	
16	327	
19	327	
22	327	
25	327	
28	327	
1631	327	

# Run 2

15 min +  
Total Averages

Time	CO <sub>2</sub>	CO	O <sub>2</sub>	% CO <sub>2</sub> = $\frac{\text{CO}_2}{\text{CO}_2 + \text{CO} + \text{O}_2} \times 100$ ↓ (91)
1307 → 1322 =	10.4	548	6.0	99.5
1322 → 1337 =	10.4	565	6.1	99.5
1337 → 1352 =	10.3	511	6.2	99.5
1352 → 1407 =	10.5	544	6.1	99.5
1407 → 1422 =	10.4	529	6.2	99.5
1422 → 1437 =	10.3	489	6.8	99.5
1437 → 1452 =	10.3	455	6.6	99.6
1452 → 1507 =	10.2	412	7.0	99.6
1507 → 1522 =	10.1	407	7.5	99.6
1522 → 1537 =	9.4	392	8.0	99.6
1537 → 1552 =	8.9	391	8.4	99.6
1552 → 1607 =	8.8	378	8.8	99.6
1607 → 1622 =	8.4	332	9.3	99.6
1622 → 1631 =	8.3	327	9.5	99.6
$\Sigma X$	126.8	6280	102.5	
$\bar{X}$	9.75	449	7.32	99.5

Jun 3

5 minute  
Averages

Time	%	%CO <sub>2</sub>	PPMCO	Time	%	CO <sub>2</sub>	PPM
1515	11.6	6.8	1114	1703	11.3	6.7	1260
1518	11.6	6.9	1117	1706	11.3	6.8	1119
1521	11.5	6.9	1120	1709	11.4	6.8	1222
1524	11.5	7.0	1123	1712	11.5	7.0	7918
1527	11.5	7.0	1126	1715	11.5	7.0	642
1530	11.4	7.1	1129	1718	11.5	7.0	1171
1533	11.4	7.1	1132	1721	10.7	6.7	840
1536	11.4	7.1	1135	1724	9.1	6.2	748
1539	11.3	7.0	1138	1727	10.7	6.5	772
1542	11.3	7.0	1141	1730	11.4	6.8	799
1545	11.3	7.0	1144	1733	11.9	7.2	813
1548	11.2	7.0	1147	1736	11.9	6.9	775
1551	11.1	7.0	1150	1739	12.3	7.1	712
1554	11.0	7.0	1153	1742	12.3	6.9	718
1557	11.1	7.0	1156	1745	12.3	6.1	158
1600	11.3	7.0	1159	1748	12.3	5.9	699
1603	11.5	7.0	1162	1751	12.0	6.7	630
1606	11.3	7.0	1165	1754	11.9	6.1	603
1609	11.4	7.1	1168	1757	12.0	6.0	587
1612	11.4	7.1	1171	1800	12.3	6.7	821
1615	11.5	7.1	1174	1803	12.5	6.7	1111
1618	11.9	7.1	1177	1806	12.7	6.8	112
1621	11.5	7.1	1180	1809	12.8	6.9	623
1624	11.2	7.1	1183	1812	13.0	6.8	719
1627	12.2	7.1	1186	1815	12.7	6.8	711
1630	11.9	7.0	1189	1818	12.4	6.9	711
1633	11.1	7.6	1192	1821	12.3	6.7	591
1636	10.7	7.5	1195	1824	12.0	6.7	545
1639	10.7	7.5	1198	1827	11.8	6.7	585
1642	10.0	7.5	1201	1830	12.0	6.7	248
1645	10.7	7.5	1204				
1648	10.8	7.5	1207				
651	10.8	7.5	1210				
1654	10.8	6.8	1213				
1657	11.0	7.5	1216				
1700	11.2	7.0	1219				

Run 3

15min + Total  
Averages

Time	CO <sub>2</sub>	CO	O <sub>2</sub>	Comb Est %
1515 - 1530	<del>6.95</del> 7.0	1853	11.52	97.4
1530 - 1545	7.12	1937	11.34	97.4
1545 - 1600	7.26	2191	11.14	97.1
1600 - 1615	7.00	1852	11.42	97.4
1615 - 1630	6.84	2095	11.74	97.0
1630 - 1645	7.60	2236	10.76	97.1
1645 - 1700	7.22	1634	10.92	97.3
1700 - 1715	7.48	965	11.40	95.7
1715 - 1730	6.54	1072	10.68	94.4
1730 - 1745	5.92	793	12.14	95.7
1745 - 1800	5.96	616	12.10	99.0
1800 - 1815	5.48	756	12.74	93.6
1815 - 1830	6.54	700	12.10	93.9
<del>1830</del>				
	$\Sigma X = 87.91$ 6.76 = 6.8%	$\Sigma X = 18,007$ $\bar{X} = 1385$ = 1385 ppm	$\Sigma X = 150$ $\bar{X} = 11.54$ = 11.5%	95.0

# Run 4

J. J. ...  
Ave.

Time	%	% CO <sub>2</sub>	PPM CO	Time	% CO <sub>2</sub>	% CO <sub>2</sub>	PPM CO
1020		2.1	750	1208	14.7		1160
1023		2.2	750	1211			1170
1026		2.2	750	1214			1180
1029		2.6	750	1217			1190
1032		4.1	750	1220			1200
1035		4.3	750	1223			1210
1038		4.6	750	1226			1220
1041	11.2	4.9	750	1229	14.7		1230
1044	11.5	5.2	750	1232	14.7		1240
1047	11.8	5.5	750	1235	14.9	3.2	1004
1050	11.9	4.1	750	1238	14.9	3.1	750
1053	11.9	4.2	750	1241	14.7	3.1	750
1056	11.9	4.3	750	1244	14.7	3.1	896
1059	11.9	4.4	750	1247	14.7	3.2	807
1102	11.9	4.4	750	1250	14.7	3.2	750
1105	11.9	4.4	750	1253	14.7	3.2	750
1108	11.9	4.9	750	1256	14.7	3.2	750
1111	11.9	5.1	750	1259	14.7	3.2	750
1114	11.9	5.1	896	1302	14.7	3.1	750
1117	11.9	5.1	750	1305	14.7	3.1	750
1120	11.9	5.1	750	1308	14.7	3.1	1091
1123	11.9	5.1	750	1311	14.7	3.1	1100
1126	11.9	5.1	750	1314	14.7	3.1	1114
1129	11.9	5.0	750	1317	14.7	3.1	1120
1132	11.9	4.1	750	1320	14.7	3.1	1130
1135	11.9	4.2	750				
1138	11.9	4.3	750				
1141	11.9	4.2	1344				
1144	11.9	4.1	1344				
1147	11.9	4.1	1344				
1150	11.9	4.1	1000				
1153	11.9	4.1	1011				
1156	11.9	4.1	1011				
1159	11.9	4.1	1011				
1202	11.9	4.1	1011				
1205	11.9	4.1	1011				

# Run 4

15min + Total  
Averages

Time	CO <sub>2</sub>	CO	O <sub>2</sub>	Temp
1020 1034	3.1	612	15.6	95.2
1034 1049	4.2	719	13.8	95.3
1049 1104	4.5	819	14.0	95.2
1104 1119	5.2	890	13.3	95.2
1119 1134	5.0	1366	13.0 13.7	97.3
1134 1149	4.1	1234	14.3	97.1
1149 1204	3.6	1140	14.5	96.9
1204 1219	3.6	1103	14.6	97.0
1219 1234	3.5	1464	14.8	96.0
1234 1249	3.2	910	14.9	97.2
1249 1304	3.2	980	14.9	97.2
1304 1319	3.5	1112		95.9
	ΣX = 50.2	ΣX = 12349	ΣX = 171.4	
	$\bar{x}$ = 3.86	$\bar{x}$ = 1029	$\bar{x}$ = 14.28	
	3.9%		14.3	97.4

APPENDIX D  
FIELD DATA SHEETS AND  
QUALITY ASSURANCE DATA

PARTICULATE / NONPARTICULATE FIELD DATA  
CODING FORM  
PG 1 of 2

CLIENT 274 SHEET 1 OF 2  
 PROJECT No. 1720 PREPARED BY 274  
 PLANT 274 PITOT NUMBER and SIDE 1  
 RUN No. 2 PITOT TUBE CP 1  
 LOCATION AMALGAM FILTER No. / THIMBLE No. 1  
 DATE 1/10/79 AMBIENT TEMP °F 57  
 OPERATOR PLS BAR PRESS. in. Hg 30.65  
 SAMPLE BOX NO. 1720 ASSUMED MOISTURE, % 10  
 METER BOX NO. 1720 HEATER BOX SETTING, °F 250  
 METER ΔH@ 1.715 NOZZLE #/DIA., in. 22  
 Y FACTOR 1.197 PROBE LENGTH 5  
 PROBE HEATER SETTING 250

\* FIELDS FOR NONPARTICULATE RUN

POINT	DISTANCE IN INCHES	CLOCK TIME	DRY GAS METER, CF	PITOT in. H <sub>2</sub> O ΔP	ORIFICE ΔH in. H <sub>2</sub> O		DRY GAS TEMP. °F		PUMP VACUUM in. Hg GAUGE	BOX TEMP °F	IMPINGER TEMP °F	STACK PRESS in. Hg	STACK TEMP °F	NULL ANGLE (DEGREES)
					DESIRED	ACTUAL	INLET	OUTLET						
1		10.7	500.245	.005	.76	.76	87	88	4	228	55	55		
2		4.0	500.2	.005	.76	.76	86	87	4	235	55	55		
3		8.0	500.1	.005	.76	.76	86	92	4	245	55	55		
4		12.0	500.0	.005	.76	.76	86	94	4	247	55	55		
5		16.0	507.0	.01	1.5	1.5	86	95	6	250	55	55		
6		20.0	510.4	.005	.76	.76	86	98	4	262	54	54		
7		24.0	512.5	.005	.76	.76	86	98	4	267	54	54		
8		28.0	514.4	.01	1.6	1.6	87	99	6	271	54	54		
9		32.0	517.5	.01	1.6	1.6	88	103	6	275	55	55		
10		36.0	520.5	.01	1.6	1.6	88	104	6	277	54	54		
11		40.0	522.6	.01	1.6	1.6	88	104	6	281	54	54		
TOTAL														

COMMENT

METER LEAK CHECK: BEFORE TEST: 01 CF SEC 15 in. Hg AFTER TEST: 01 CF SEC 15 in. Hg  
 PITOT LEAK CHECK:      PORT      STATIC PRESSURE       
 ORSAT LEAK CHECK:      in. H<sub>2</sub>O       
     in. Hg

PARTICULATE/NONPARTICULATE FIELD DATA  
CODING FORM  
PG. 2 of 2

SHEET 2 OF 3  
PREPARED BY AW

CLIENT CPA  
PROJECT NO. 100  
PLANT Rockwell  
RUN NO. 2  
LOCATION 1 Feb 87

POINT	DISTANCE IN INCHES	CLOCK TIME		DRY GAS METER, CF	PITOT in. H <sub>2</sub> O ΔP	ORIFICE ΔH in. H <sub>2</sub> O		DRY GAS TEMP. °F		PUMP VACUUM IN. Hg GAUGE	BOX TEMP. °F	IMPINGER TEMP. °F	STACK PRESS in. H <sub>2</sub> O in. Hg	STACK TEMP. °F °C	NULL ANGLE (DEGREES)
		ACTUAL	RUN			DESIRED	ACTUAL	INLET	OUTLET						
12			41.0	525.7	.87	1.6	1.6	87	125	6	256	39		53	
13			48.0	520.2	.01	1.6	1.6	89	105	6	267	37		54	
14			52.0	531.3	.01	1.6	1.6	89	106	6	271	42		54	
15			53.0	535.7	.01	1.6	1.6	90	106	6	222	31		54	
16			53.0	536.5	.01	1.6	1.6	89	106	6	251	40		53	
17			61.0	537.3	.01	1.6	1.6	90	106	6	267	38		53	
18			62	542.2	.01	1.6	1.6	90	106	6	275	38		53	
19			72	547.9	.01	1.6	1.6	90	106	6	232	37		53	
20			76	547.2	.005	1.6	1.6	90	106	4	252	38		53	
21			80	547.8	.005	1.6	1.6	90	105	4	264	37		53	
22			84	551.2	.005	1.6	1.6	91	104	4	262	34		52	
23			88	555.6	.005	1.6	1.6	91	105	4	272	32		52	
24			92	555.7	.005	1.6	1.6	91	105	4	225	39		52	
			96	557.5	.005	1.6	1.6	91	105	4					
			STOP												
1	START		3:00	552.502	.005	1.6	1.6	91	75	4	246	30		52	
2			7.0	560.7	.005	1.6	1.6	90	96	4	255	30		52	
3			8.0	562.3	.005	1.6	1.6	91	98	4	262	3		52	
4			12.0	567.2	.005	1.6	1.6	91	99	4	269	30		52	
5			16.0	566.2	.005	1.6	1.6	94	100	4	275	30		52	
6			20.0	565.1	.005	1.6	1.6	91	100	4	273	30		52	
7			24.0	570.1	.005	1.6	1.6	91	101	4	220	30		52	
TOTAL															

COMMENTS:  
Final

METER LEAK CHECK DURING TEST:  
METER READING START  
STOP

.02 CF 60 SEC 10 in. Hg  
.02 CF 60 SEC 10 in. Hg  
PORT in. H<sub>2</sub>O  
in. Hg

STATIC PRESSURE PORT in. H<sub>2</sub>O  
in. Hg

\* FIELDS FOR NONPARTICULATE RUN

96  
96/2  
15



GCA/TECHNOLOGY DIVISION  
 SOURCE PARTICULATE SAMPLING  
 TRAIN ASSEMBLY, RECOVERY AND ANALYTICAL REPORT SHEET

Run Date: 1 Feb 84 Client: EPA  
 Run No.: 2 W. O. No.: \_\_\_\_\_  
 Sample Box No.: 1726 Plant: New Bedford  
 Operator: AW Sampling Location: MAIN STACK

**FRONT HALF**

Laboratory Results

Nozzle and Probe (Cyclone Bypass)-Acetone Wash,  
 Cyclone and Flask-Acetone Wash

Lab No.: 36225 Residue 5.3 mg  
 Lab No.: \_\_\_\_\_ Residue \_\_\_\_\_ mg

Thimble No.	Lab No.	Weight Results
<u>NA</u>	_____	_____ mg
_____	_____	_____ mg
_____	_____	_____ mg
_____	_____	_____ mg

Thimble particulate weight NA mg

Filter No.	Lab No.	Weight Results
<u>GC-29</u>	_____	<u>12.06</u> mg
_____	_____	_____ mg
_____	_____	_____ mg
_____	_____	_____ mg

Filter particulate weight 12.06 mg

FRONT HALF Sub Total 17.36 mg

**BACK HALF**

Impinger WATER and Water Wash of Impingers  
 Connectors and Back Half of Filter Holder

Collected on 0.22µ Filter NA mg

Lab No.: \_\_\_\_\_

Chloroform-ether Extract NA mg

Aqueous Residue NA mg

ACETONE WASH of  
 Impinger, Connectors and Back Half of  
 Filter Holder

Residue 95.0 mg

Lab No.: \_\_\_\_\_

BACK HALF Sub Total 95.0 mg

**TOTAL TRAIN**

TOTAL WEIGHT (Front & Back) 112.36 mg

**MOISTURE**

Impingers: 1 1/2 200 ml  
415 200 ml  
100 ml 20

Silica Gel

Weight after test: 44.9

Weight before test: 329.4

Net Weight: 15.5

Container No.: 1. \_\_\_\_\_ 2. \_\_\_\_\_ 3. \_\_\_\_\_ 4. \_\_\_\_\_

Final Volume Total \_\_\_\_\_  
 Initial Volume Total 400 ml  
 Net Volume 415

TOTAL NET WEIGHT-Silica Gel \_\_\_\_\_ mg

NET VOLUME-Impingers \_\_\_\_\_ ml

TOTAL MOISTURE 30.5 mg

Comments: \_\_\_\_\_

LAB: DATE RECEIVED \_\_\_\_\_  
 DATE REPORTED 3/30/84 AW

Train Assembled by: AW + RV + M.M.  
 Sample Recovered by: " " "  
 Sample Analyzed by: \_\_\_\_\_

PARTICULATE ANALYSIS

Lab No. 56233  
 Client EPC

WO No. 1-619 091  
 Date Received 3/2/84

Description Front-Trail Runway  
Location of TR 40111 NB-MS-FH-2

A. SAMPLE VOLUME

250 mL

B. WASH VOLUME

\_\_\_\_\_ mL  
 \_\_\_\_\_ mL  
 \_\_\_\_\_ mL  
 \_\_\_\_\_ mL  
 Total \_\_\_\_\_ mL

C. BLANK CORRECTION

Sample: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm  
 +  
 Wash: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm  
 =  
 TOTAL \_\_\_\_\_ gm

D. TARE WEIGHTS

	No.	Wt.
Container	<u>4-1</u>	<u>105.0613</u> g
Filter	<u>02-21</u>	<u>0.62462</u> g
Thimble		_____ g
Total		_____ g

CONTAINER NO.  
4-1

0.22/0.45  
 FILTER NO.  
 \_\_\_\_\_

E. GROSS WEIGHTS

<u>RH/°F</u>	<u>Date/Time</u>	(1) _____ g	<u>RH/°F</u>	<u>Date/Time</u>	(4) _____ g
____/____	____/____	<u>105.0687</u>	____/____	____/____	_____
____/____	____/____	(2) _____ g	____/____	____/____	(5) _____ g
____/____	____/____	(3) _____ g	____/____	____/____	(6) _____ g

Final Gross Weight 105.0687 g  
 Total Tare Weight 105.0613 g  
 Residue Weight .0068 g  
 Blank Weight .0015 g

Filter  
0.63663  
0.62462  
.01205

↓  
.0053 g

E. NET WEIGHT

Remarks: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Analyst Rene Arango

GCA/TECHNOLOGY DIVISION

PARTICULATE ANALYSIS

Lab No. 36245 WO No. 1-12-1989  
 Client EPC Date Received 5/15/89

Description BACK-HALF BLIND  
GLACIOL & THERMOC MB-MB-BH-2

A. SAMPLE VOLUME 250 mL  
 B. WASH VOLUME \_\_\_\_\_ mL  
 C. BLANK CORRECTION  
 Sample: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm  
 +  
 Wash: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm  
 = \_\_\_\_\_ gm  
 Total \_\_\_\_\_ mL TOTAL \_\_\_\_\_ gm

D. TARE WEIGHTS

	No.	Wt.
Container	<u>4-7</u>	<u>110.5579</u> g
Filter		_____ g
Thimble		_____ g
Total		_____ g

CONTAINER NO.  
4-7

0.22/0.45  
 μ  
 FILTER NO.  
 \_\_\_\_\_

E. GROSS WEIGHTS

RH/OF	Date/Time	(1)	RH/OF	Date/Time	(4)
/	/	<u>110.5579</u> g	/	/	_____ g
/	/	_____ g	/	/	(5) _____ g
/	/	_____ g	/	/	(6) _____ g

Final Gross Weight 110.5579 g  
 Total Tare Weight 110.5579 g  
 Residue Weight 0.000 g  
 Blank Weight 0.0050 g  
 +  
0.0150 g

E. NET WEIGHT

Remarks: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Analyst Rene Arango

GCA/TECHNOLOGY DIVISION

PARTICULATE ANALYSIS

Lab No. 36237

WO No. 1-129-091

Client EPRI

Date Received 2/2/84

Description Front Door Room  
Acetone & Nonyl NB-MS-FH-F73B-2

A. SAMPLE VOLUME

20 mL

B. WASH VOLUME

\_\_\_\_\_ mL

\_\_\_\_\_ mL

\_\_\_\_\_ mL

\_\_\_\_\_ mL

Total \_\_\_\_\_ mL

C. BLANK CORRECTION

Sample: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm

+

Wash: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm

=

TOTAL \_\_\_\_\_ gm

D. TARE WEIGHTS

	No.	Wt.
Container	<u>4-5</u>	<u>0.0000</u> g
Filter		_____ g
Thimble		_____ g
Total		_____ g

CONTAINER NO.  
4-5

0.22/0.45  
μ  
FILTER NO.  
\_\_\_\_\_

E. GROSS WEIGHTS

RH/°F	Date/Time	(1)	g	RH/°F	Date/Time	(4)	g
/	/			/	/		
/	/	(2)	_____ g	/	/	(5)	_____ g
/	/	(3)	_____ g	/	/	(6)	_____ g

Final Gross Weight 109.6209 g

Total Tare Weight - 109.6200 g

Residue Weight .0009 g

Blank Weight - \_\_\_\_\_ g

+

.0009 g

E. NET WEIGHT

Remarks:

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

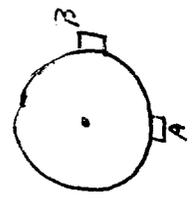


GCA TECHNOLOGY DIVISION

PARTICULATE / NONPARTICULATE FIELD DATA  
CODING FORM  
PG 1 of 2

SHEET L OF 3  
PREPARED BY AK  
PITOT NUMBER and SIDE \_\_\_\_\_  
PITOT TUBE CP \_\_\_\_\_  
FILTER No / THIMBLE No 1  
AMBIENT TEMP °F 49  
BAR PRESS, in. Hg 30.10 29.85  
ASSUMED MOISTURE, % 10.2  
HEATER BOX SETTING, °F 250  
NOZZLE #/DIA., in. 622 1  
PROBE LENGTH 3  
PROBE HEATER SETTING 250

VERY IMPORTANT - FILL IN ALL BLANKS  
READ AND RECORD AT THE START OF EACH TEST POINT  
SKETCH



CLIENT EPA  
PROJECT No \_\_\_\_\_  
PLANT A. B. Incinerator  
RUN No 3  
LOCATION Main Stack  
DATE 6 Feb 84  
OPERATOR AW  
SAMPLE BOX NO. 1726  
METER BOX NO. 1913  
METER ΔH@ 997

\* FIELDS FOR NONPARTICULATE RUN

POINT	DISTANCE IN INCHES	CLOCK TIME	DRY GAS METER, %	PITOT in. H <sub>2</sub> O ΔP	ORIFICE ΔH in. H <sub>2</sub> O		DRY GAS TEMP. °F		PUMP VACUUM IN Hg GAUGE	BOX TEMP °F	IMPINGER TEMP °F	STACK PRESS in. H <sub>2</sub> O in. Hg	STACK TEMP °F	NOZZLE ANGLE (DEGREES)
					DESIRED	ACTUAL	INLET	OUTLET						
1	SMRT	3:08	607.5%	.005	.72	.92	73	71	5	235	36		50	
2		4:0	606.9%	.005	.84	.84	73	72	4	244	37		51	
3		8:0	611.8	.005	.84	.84	73	75	4	263	38		51	
4		12:0	613.6	.005	.84	.84	73	79	4	274	38		51	
5		16:0	615.6	.005	.84	.84	74	82	4	262	38		51	
6		20:0	617.7	.005	.84	.84	75	85	4	224	38		51	
7		24:0	619.7	.005	.84	.84	76	87	4	242	38		51	
8		28:0	621.7	.005	.84	.84	77	90	4	264	39		51	
9		32:0	623.7	.005	.91	.91	78	92	4	272	39		51	
10		36:0	625.8	.005	.91	.91	79	93	4	261	38		51	
11		40:0	627.8	.005	.91	.91	81	95	4	245	39		50	
TOTAL														

METER LEAK CHECK: \_\_\_\_\_  
BEFORE TEST: 02 CF 60 SEC 15 in. Hg  
AFTER TEST: \_\_\_\_\_ CF \_\_\_\_\_ SEC \_\_\_\_\_ in. Hg  
PITOT LEAK CHECK  \_\_\_\_\_  
ORSAT LEAK CHECK \_\_\_\_\_  
STATIC PRESSURE  
PORT \_\_\_\_\_  
in. H<sub>2</sub>O \_\_\_\_\_  
in. Hg \_\_\_\_\_

CLIENT EPA

PARTICULATE/NONPARTICULATE FIELD DATA

SHEET 2 OF 3

PROJECT NO

CODING FORM  
PG. 2 of 2

PREPARED BY AW T JV

PLANT N-B

LOCATION MAIN STACK

RUN NO 3

DATE 6 Feb 81

POINT	DISTANCE IN INCHES	CLOCK TIME	DRY GAS METER, CF	PITOT IN. H <sub>2</sub> O ΔP	ORIFICE ΔH IN H <sub>2</sub> O		DRY GAS TEMP. OF		PUMP VACUUM IN. Hg GAUGE	BOX TEMP. OF	IMPINGER TEMP. OF	STACK PRESS. IN. H <sub>2</sub> O IN. Hg	STACK TEMP. OF °C	NULL ANGLE (DEGREES)
					DESIRED	ACTUAL	INLET	OUTLET						
12		47	629.1	.005	.91	.91	82	96	4	250	39		50	
13		48	632.1	.005	.91	.91	83	97	4	253	38		50	
14		52	634.2	.005	.91	.91	84	98	4	248	38		50	
15		56	626.3	.005	.91	.91	85	98	4	250	38		50	
16		60	635.4	.005	.91	.91	86	99	4	247	37		50	
17		64	640.6	.005	.91	.91	87	99	4	237	37		50	
18		68	642.6	.005	.91	.91	88	100	4	247	37		50	
19		72	644.7	.005	.91	.91	88	100	4	261	38		50	
20		76	646.7	.005	.91	.91	89	101	4	265	37		50	
21		80	648.7	.005	.91	.91	89	102	4	261	38		50	
22		84	651.0	.005	.91	.91	90	102	4	274	37		50	
23		88	653.0	.005	.91	.91	90	102	4	266	39		50	
24		92	655.1	.005	.91	.91	91	102	4	229	42		50	
	STOP	4.44	657.213	CHANGE										
1	START	4.50	657.411	.005	.91	.91	91	98	4	272	46		50	
2		4.0	659.4	.005	.91	.91	92	100	4	270	46		50	
3		8.0	661.6	.005	.91	.91	92	102	4	275	45		50	
4		12.0	663.7	.005	.91	.91	92	103	5	246	44		50	
5		16.0	665.8	.005	.91	.91	93	104	5	207	42		50	
6		20.0	668	.005	.91	.91	93	104	5	231	41		60	
TOTAL														

COMMENTS:

METER LEAK CHECK DURING TEST:

STOP

START

0.01 CF 60 SEC 8

CF SEC

in. Hg port change

STATIC PRESSURE PORT

in. H<sub>2</sub>O

in. Hg

\* FIELDS FOR NONPARTICULATE RUN

CLIENT EPA PROJECT NO. 101 PARTICULATE/NONPARTICULATE FIELD DATA SHEET 3 OF 3  
 CODING FORM PG 2 of 2 PREPARED BY AW

PLANT N.B. LOCATION MAIN STACK DATE 6 Feb 81  
 RUN NO. 3

POINT	DISTANCE IN INCHES	CLOCK TIME	DRY GAS METER, CF	PITOT IN. H <sub>2</sub> O ΔP	ORIFICE ΔH IN. H <sub>2</sub> O		DRY GAS TEMP. °F		PUMP VACUUM IN. Hg GAUGE	BOX TEMP. °F	IMPINGER TEMP. °F	STACK PRESS. in. H <sub>2</sub> O	STACK TEMP. °F	NULL ANGLE (DEGREES)
					DESIRED	ACTUAL	INLET	OUTLET						
7			670.1	.005	.91	.91	95	105	5	254	44		50	
8			672.2	.005	.91	.91	93	105	5	262	45		50	
9			674.3	.005	.91	.91	94	105	5	267	45		51	
10			676.4	.005	.91	.91	94	106	5	275	45		51	
11			678.6	.005	.91	.91	94	106	5	262	47		50	
12			680.6	.005	.91	.91	94	106	5	227	44		51	
13			682.8	.005	.91	.91	94	106	5	247	45		51	
14			684.8	.005	.91	.91	95	106	5	261	44		51	
15			686.9	.005	.91	.91	95	106	5	268	47		51	
16			689.0	.005	.91	.91	95	106	5	274	44		51	
17			691.1	.005	.91	.91	95	106	4	243	43		51	
18			693.2	.005	.91	.91	95	106	5	235	41		51	
19			695.3	.005	.91	.91	95	106	5	256	41		51	
20			697.4	.005	.91	.91	95	106	5	269	44		51	
21			699.6	.005	.91	.91	95	106	5	275	44		51	
22			701.7	.005	.91	.91	95	106	5	244	41		51	
23			703.8	.005	.91	.91	95	106	5	225	41		48	
24			705.9	.005	.91	.91	95	106	5	242	40		49	
			6.26											
			STOP											
			END											
TOTAL														

COMMENTS:

METER LEAK CHECK DURING TEST: METER READING START \_\_\_\_\_ STOP \_\_\_\_\_

CF 60 SEC 7 in. Hg FINAL in. Hg

STATIC PRESSURE PORT in. Hg

\* FIELDS FOR NONPARTICULATE RUN

GCA/TECHNOLOGY DIVISION  
SOURCE PARTICULATE SAMPLING  
TRAIN ASSEMBLY, RECOVERY AND ANALYTICAL REPORT SHEET

Run Date: 6 Feb 84 Client: EPA  
 Run No.: 3 W. O. No.: \_\_\_\_\_  
 Sample Box No.: 1726 Plant: N. B. Tinsley  
 Operator: AW Sampling Location: MAIN ST. LOC

**FRONT HALF**

Nozzle and Probe (Cyclone Bypass)-Acetone Wash,  
 Cyclone and Flask-Acetone Wash

Laboratory Results

Lab No.: 36076 Residue 11.5 mg  
 Lab No.: \_\_\_\_\_ Residue \_\_\_\_\_ mg

Thimble No.	Lab No.	Weight Results
<u>NA</u>	_____	_____ mg
_____	_____	_____ mg
_____	_____	_____ mg
_____	_____	_____ mg

Thimble particulate weight NA mg

Filter No.	Lab No.	Weight Results
<u>45-31</u>	_____	<u>59.13</u> mg
_____	_____	_____ mg
_____	_____	_____ mg
_____	_____	_____ mg

Filter particulate weight 59.13 mg

FRONT HALF Sub Total 70.63 mg

**BACK HALF**

Impinger WATER and Water Wash of Impingers  
 Connectors and Back Half of Filter Holder

Collected on 0.22µ Filter NA mg

Chloroform-ether Extract NA mg

Aqueous Residue NA mg

Lab No.: \_\_\_\_\_

ACETONE WASH of  
 Impinger, Connectors and Back Half of  
 Filter Holder

Residue 16.3 mg

BACK HALF Sub Total 16.3 mg

Lab No.: 36076

**TOTAL TRAIN**

TOTAL WEIGHT (Front & Back) 87.43 mg

**MOISTURE**

Impingers: 200 ml  
3.4

**Silica Gel**

Weight after test: 351

Weight before test: 339.1

1423.205 ml  
43 19.5 ml

Net Weight: 11.9

Container No.: 1. \_\_\_\_\_ 2. \_\_\_\_\_ 3. \_\_\_\_\_ 4. \_\_\_\_\_

Final Volume Total \_\_\_\_\_

TOTAL NET WEIGHT-Silica Gel \_\_\_\_\_ gm

Initial Volume Total \_\_\_\_\_

NET VOLUME-Impingers \_\_\_\_\_ ml

Net Volume 0.0

TOTAL MOISTURE 11.9 gm

Comments: \_\_\_\_\_

LAB: DATE RECEIVED \_\_\_\_\_  
 DATE REPORTED 3/20/84 MW

Train Assembled by: \_\_\_\_\_  
 Sample Recovered by: \_\_\_\_\_  
 Sample Analyzed by: \_\_\_\_\_



GCA/TECHNOLOGY DIVISION

PARTICULATE ANALYSIS

Lab No. 36,046 WO No. 1-619-094

Client EPa Date Received 5/5/87

Description Backflow/Bank  
Acetone & Toluene NB-115-BH-3

A. SAMPLE VOLUME B. WASH VOLUME C. BLANK CORRECTION

500 mL \_\_\_\_\_ mL Sample: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm  
 \_\_\_\_\_ mL \_\_\_\_\_ mL +  
 \_\_\_\_\_ mL \_\_\_\_\_ mL Wash: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm  
 \_\_\_\_\_ mL \_\_\_\_\_ mL =  
 Total \_\_\_\_\_ mL TOTAL \_\_\_\_\_ gm

D. TARE WEIGHTS

	No.	Wt.
Container	<u>45701005</u>	<u>0.22705</u> g
Filter	<u>6230</u>	<u>0.02705</u> g
Thimble		_____ g
Total		_____ g

CONTAINER NO.  
4-8

0.22/0.45  
μ  
FILTER NO.  
\_\_\_\_\_

E. GROSS WEIGHTS

RH/OF	Date/Time	(1)	RH/OF	Date/Time	(4)
___/___	___/___	<u>109.2418</u>	___/___	___/___	_____ g
___/___	___/___	(2) _____ g	___/___	___/___	(5) _____ g
___/___	___/___	(3) _____ g	___/___	___/___	(6) _____ g

Final Gross Weight 109.2418 g  
 Total Tare Weight - 109.2205 g  
 Residue Weight 0.0213 g  
 Blank Weight - 0.0045 g

Filter  
0.68616  
0.02705  
-0.59115

↓  
0.0103 g

E. NET WEIGHT

Remarks: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

GCA TECHNOLOGY DIVISION

PARTICULATE / NONPARTICULATE FIELD DATA  
CODING FORM  
PG 1 of 2

CLIENT EPA

PROJECT No. \_\_\_\_\_

PLANT A. B. FRIEDLANDER

RUN No. 4

LOCATION MAN STREET

DATE 7 Feb 84

OPERATOR AW T. R.V.

SAMPLE BOX NO. 1726

METER BOX NO. \_\_\_\_\_

METER ΔH<sub>2</sub>O 1.913

Y FACTOR .997

SHEET 1 OF 3

PREPARED BY AW

PITOT NUMBER and SIDE \_\_\_\_\_

PITOT TUBE CP \_\_\_\_\_

FILTER No. / THIMBLE No. 1

AMBIENT TEMP °F 30

BAR PRESS., in. Hg 29.86

ASSUMED MOISTURE, % 3

HEATER BOX SETTING, °F 250

NOZZLE #/DIA., in. 622 1

PROBE LENGTH 5 ft

PROBE HEATER SETTING 250

\* FIELDS FOR NONPARTICULATE RUN

VERY IMPORTANT - FILL IN ALL BLANKS

READ AND RECORD AT THE START OF EACH TEST POINT

SKETCH

POINT	DISTANCE IN INCHES	CLOCK TIME	DRY GAS METER, CF	PITOT in. H <sub>2</sub> O ΔP	OR-FICE ΔH in H <sub>2</sub> O		DRY GAS TEMP. °F		PUMP VACUUM IN Hg GAUGE	BOX TEMP °F	IMPINGER TEMP °F	STACK PRESS in Hg	STACK TEMP °F	NULL ANGLE (DEGREES)
					DESIRED	ACTUAL	INLET	OUTLET						
1	START	10:19	708.599	.005	.82	.82	72	73	5	237	33		52	
2		40	710.5	.005	.82	.82	74	75	5	259	35		51	
3		80	712.5	.005	.82	.82	74	77	5	270	35		52	
4		120	714.5	.005	.82	.82	75	80	5	268	36		51	
5		160	716.5	.005	.82	.82	76	83	5	270	35		51	
6		200	718.5	.005	.82	.82	76	85	5	268	35		51	
7		240	720.6	.005	.82	.82	77	87	5	268	34		51	
8		280	722.5	.005	.91	.91	78	89	5	267	35		51	
9		320	724.6	.005	.91	.91	79	91	5	265	35		51	
10		360	726.7	.005	.91	.91	81	93	5	259	36		51	
11		400	728.8	.005	.91	.91	82	94	5	262	36		52	
TOTAL														

COMMENT

10.14  
11.14  
36  
45

METER LEAK CHECK:

BEFORE TEST: .02 CF SEC 60 SEC 15 in. Hg

AFTER TEST: \_\_\_\_\_ CF SEC \_\_\_\_\_ SEC \_\_\_\_\_ in. Hg

PITOT LEAK CHECK

ORSAT LEAK CHECK \_\_\_\_\_

STATIC PRESSURE

PORT 0026 in. H<sub>2</sub>O

\_\_\_\_\_ in. Hg

COA TEST BY WYD/AM/NO

CLIENT EPA PROJECT NO. 103 PARTICULATE/NONPARTICULATE FIELD DATA SHEET 2 OF 3  
 PREPARED BY Am + JV CODING FORM PG. 2 of 2

PLANT NB LOCATION MAIN STACK DATE 7 Feb 84  
 RUN NO. 4

POINT	DISTANCE IN INCHES	CLOCK TIME		DRY GAS METER, CF	PICTG in. H <sub>2</sub> O ΔP	ORIFICE ΔH in. H <sub>2</sub> O		DRY GAS TEMP. °F		PUMP VACUUM IN. HG GAUGE	BOX TEMP. °F	IMPINGER TEMP. °F	STACK PRESS. in. H <sub>2</sub> O	STACK TEMP. °F	NULL ANGLE (DEGREES)
		ACTUAL	RUN			DESIRED	ACTUAL	INLET	OUTLET						
12			74	730.9	.005	.91	.91	83	95	5	269	35		53	
13			78	739.9	.005	.91	.91	83	95	5	270	37		51	
14			53	735.2	.005	.91	.91	84	96	5	262	37		51	
15			56	737.2	.005	.91	.91	84	95	5	264	35		50	
16			60	739.3	.005	.91	.91	85	96	5	272	36		50	
17			64	741.7	.005	.91	.91	86	97	5	275	37		50	
18			68	743.5	.005	.91	.91	87	98	5	239	38		50	
19			72	745.7	.0075	1.35	1.35	87	98	5	246	37		50	
20			76	748.2	.005	.91	.91	87	99	6	260	37		50	
21			80	750.4	.005	.91	.91	88	99	5	272	37		49	
22			84	752.5	.005	.91	.91	88	99	5	241	36		46	
23			88	754.6	.005	.91	.91	88	98	5	227	35		45	
24			92	756.7	.005	.91	.91	88	98	5	246	36		45	
			96	758.85											
			STOP	PORT	CHANGE										
1		11:45	START	759.191	.005	.91	.91	87	90	5	239	33		49	
2		12:00		761.5	.005	.91	.91	87	93	5	222	33		49	
3		12:00		763.7	.005	.91	.91	89	95	5	240	33		49	
4		12:00		765.7	.006	1.1	1.1	87	96	5	239	33		50	
5		12:00		767.8	.005	.91	.91	87	94	5	242	33		49	
6		12:00		770.0	.005	.91	.91	87	97	5	248	33		50	
7		12:00		772.1	.005	.91	.91	87	97	5	260	32		50	
				TOTAL											

COMMENTS:

METER LEAK CHECK DURING TEST: METER READING STOP \_\_\_\_\_ START \_\_\_\_\_

101 CF SEC 7 in. Hg  
 101 CF SEC 7 in. Hg → Port Change

STATIC PRESSURE PORT \_\_\_\_\_  
 in. H<sub>2</sub>O \_\_\_\_\_  
 in. Hg \_\_\_\_\_

\* FIELDS FOR NONPARTICULATE RUN



GCA/TECHNOLOGY DIVISION  
 SOURCE PARTICULATE SAMPLING  
 TRAIN ASSEMBLY, RECOVERY AND ANALYTICAL REPORT SHEET

Run Date: 7 Feb 84 Client: EPA  
 Run No.: 1 W. O. No.: \_\_\_\_\_  
 Sample Box No.: 1726 Plant: N.B. Turner  
 Operator: AW, RV Sampling Location: main street

**FRONT HALF**

Laboratory Results

Nozzle and Probe (Cyclone Bypass)-Acetone Wash, Lab No.: 44255 Residue 7.7 mg  
 Cyclone and Flask-Acetone Wash Lab No.: \_\_\_\_\_ Residue \_\_\_\_\_ mg

Thimble No.	Lab No.	Weight Results
<u>AH</u>	_____	_____ mg
_____	_____	_____ mg
_____	_____	_____ mg
_____	_____	_____ mg

Thimble particulate weight 0.4 mg

Filter No.	Lab No.	Weight Results
<u>22</u>	_____	<u>117.04</u> mg
_____	_____	_____ mg
_____	_____	_____ mg
_____	_____	_____ mg

Filter particulate weight 117.04 mg

FRONT HALF Sub Total 124.74 mg

**BACK HALF**

Impinger WATER and Water Wash of Impingers  
 Connectors and Back Half of Filter Holder  
 Lab No.: \_\_\_\_\_

Collected on 0.22µ Filter NA mg  
 Chloroform-ether Extract NA mg  
 Aqueous Residue NA mg

ACETONE WASH of  
 Impinger, Connectors and Back Half of  
 Filter Holder  
 Lab No.: \_\_\_\_\_

Residue 51.8 mg  
 BACK HALF Sub Total 51.8 mg

**TOTAL TRAIN**

TOTAL WEIGHT (Front & Back) 176.54 mg

**MOISTURE**

Impingers: 1+2 200  
4+5 200

Silica Gel

Weight after test: 354.1  
 Weight before test: 24.1

1+2, 205.2

4+5 195.76

Net Weight: 30.0

Container No.: 1. \_\_\_\_\_ 2. \_\_\_\_\_ 3. \_\_\_\_\_ 4. \_\_\_\_\_

Final Volume Total \_\_\_\_\_

TOTAL NET WEIGHT-Silica Gel \_\_\_\_\_ mg

Initial Volume Total \_\_\_\_\_

NET VOLUME-Impingers 0 ml

Net Volume 0.0

TOTAL MOISTURE 30.0 mg

Comments: \_\_\_\_\_

LAB: DATE RECEIVED \_\_\_\_\_  
 DATE REPORTED AW 3/20/84

Train Assembled by: \_\_\_\_\_  
 Sample Recovered by: \_\_\_\_\_  
 Sample Analyzed by: \_\_\_\_\_

GCA/TECHNOLOGY DIVISION

PARTICULATE ANALYSIS

Lab No. 36235 WO No. 1-619-099  
 Client EPA Date Received 3/3/84

Description front half Benzene  
Acetone & Naphthalene NB-45-EH-4

A. SAMPLE VOLUME 250 mL  
 B. WASH VOLUME \_\_\_\_\_ mL  
 C. BLANK CORRECTION  
 Sample: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm  
 +  
 Wash: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm  
 =  
 Total \_\_\_\_\_ mL TOTAL \_\_\_\_\_ gm

D. TARE WEIGHTS

	No.	Wt.
Container	<u>2-3</u>	<u>108.1340</u> g
Filter	<u>SE-32</u>	<u>0.00372</u> g
Thimble		_____ g
Total		_____ g

CONTAINER NO.  
2-3

0.22/0.45  
μ  
FILTER NO.  
\_\_\_\_\_

E. GROSS WEIGHTS

RH/°F	Date/Time	(1)	RH/°F	Date/Time	(4)
/	/	<u>108.1328</u>	/	/	_____ g
/	/	_____ g	/	/	(5) _____ g
/	/	_____ g	/	/	(6) _____ g

Final Gross Weight 108.1328 g  
 Total Tare Weight 108.1340 g  
 Residue Weight .0058 g  
 Blank Weight .0009 g  
+.0011  
.0079 g

7.14  
0.74076  
0.66372  
.11704

E. NET WEIGHT

Remarks: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Analyst Ronnie Arrigo



CCA/TECHNOLOGY DIVISION

PARTICULATE ANALYSIS

Lab No. 36238  
 Client EPL

WO No. 161104  
 Date Received 3/5/84

Description Acetone & Hexane Blank  
NB-145-FH-B

A. SAMPLE VOLUME

150 mL

B. WASH VOLUME

\_\_\_\_\_ mL  
 \_\_\_\_\_ mL  
 \_\_\_\_\_ mL  
 \_\_\_\_\_ mL  
 Total \_\_\_\_\_ mL

C. BLANK CORRECTION

Sample: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm  
 +  
 Wash: \_\_\_\_\_ mL × \_\_\_\_\_ g/mL = \_\_\_\_\_ gm  
 =  
 TOTAL \_\_\_\_\_ gm

D. TARE WEIGHTS

Container  
 Filter  
 Thimble  
 Total

No.	Wt.
<u>4-6</u>	<u>108.6318</u> g
_____	_____ g
_____	_____ g
_____	_____ g

CONTAINER NO.  
4-6

0.22/0.45  
 μ  
 FILTER NO.  
 \_\_\_\_\_

E. GROSS WEIGHTS

RH/°F	Date/Time	(1)	RH/°F	Date/Time	(4)
/	/	<u>108.6318</u> g	/	/	_____ g
/	/	_____ g	/	/	(5) _____ g
/	/	(3) _____ g	/	/	(6) _____ g

Final Gross Weight 108.6318 g  
 Total Tare Weight 108.6299 g  
 Residue Weight .0019 g  
 Blank Weight \_\_\_\_\_ g

.0019 g

E. NET WEIGHT

Remarks: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

The purpose of this form is to document prior approval from the Analytical Laboratory representative that the reagents, materials and procedures used in sample collection tasks are compatible with subsequent laboratory analysis requirements.

Field Team Leader: M. McCABE Anticipated Sampling Date 1/23/84  
 Sampling Task: POB TEST Site Identification NEW BEDFORD MINE  
 Contract Number: 1617-097

Field Sample Code	Sample Description	Container	Preservative	Reagent/Grade (Manufacturer and Lot Number)	Laboratory Comment
MS-PI	PLASTIC BOTTLE	GLASS	-	ACETONE 316106 KODAK MODEL 934AH	
EH	AMBER	AMBER	-	HEXANE 341097 ACETONE, FRODOX	RINSE OFF 1/17/83
BH	AMBER	AMBER	-	HEXANE 341097	IMP 1.23.83
CD-C	ORGANIC COND	AMBER	-	HEXANE 30/60 MESH	Cleaned up by R. Rebillard
FL	FLUORIDE	GLASS	-	HEXANE 701	
XR	XR-2	GLASS	-	HEXANE 701	
CD-I	LABORE COND	PEE	-	IN NaOH	10% ALICOUNT of - CD-C FISHER 673885-DI IMP 4.5.83
IMP	IMPINGEN SAITH	PEE	-	IN NaOH	
PEB	PART FULL-BLANK	GLASS	-	ACETONE 316106 HEXANE 341097	
F4B	PART FULL-BLANK	AMBER	-	WATER PROVIDED BY LAB	
CD-OB	CONDENSATE PLANK	AMBER	-	HEXANE 30/60 MESH	Cleaned up by R. Rebillard
FL B	FLUORIDE BLANK	GLASS	-	HEXANE 701	
XR B	XR-2 BLANK	GLASS	-	HEXANE 701	

Submitted [Signature] Follow-up Dates \_\_\_\_\_  
 Date 1/12/84 Final Laboratory Approval \_\_\_\_\_  
 Page 5 of 5 Date \_\_\_\_\_

Field/Laboratory Procedure Coordination

The purpose of this form is to document prior approval from the Analytical Laboratory representative that the reagents, materials and procedures used in sample collection tasks are compatible with subsequent laboratory analysis requirements.

Field Team Leader: M. McCABE Anticipated Sampling Date: 1/21/84  
 Sampling Task: PCB TEST Site Identification: NEW BEDFORD MCR  
 Contract Number: 1-619-019

Field Sample Code	Sample Description	Container	Preservative	Reagent/Grade (Manufacturer and Lot Number)	Laboratory Comment
-MS-CD-IB	IMPREGNATED BLANK	LPE		WATER PROVIDED BY LAP	
-MHB	IMPREGNATED PART-FILTER	LPE		IN ACID	FISH LOT 733025-24 exp. 7/85
-MS-2-FB	FIELD BASED PART-CATCH	Glass		REAGENT 316106	LOT 334134 ACID
-F4-FBB	FIELD BASED BLANK ORGANIC CARB	Glass		REAGENT LOT 341097	BEING QC'd
-MS-0-FBB	FIELD BASED BLANK POLYBUTYL	Glass		WATER PROVIDED BY MAB	
-FL-FBB	FIELD BASED BLANK XAD-2	Glass		30/60 MESH	Cleaned up by Richard
-Y3-FBB	FIELD BASED BLANK ORGANIC CARB	Glass		LOT QC 701	
-CD-7-FBB	FIELD BASED PART-CATCH	Glass		WATER PROVIDED BY LAP	FISH LOT 733025-24
-MS-P-FBB	FIELD BASED PART-CATCH	LPE		IN. NO. OF REAGENT 316106	
-BH-FBB	FIELD BASED PART-CATCH	Glass		REAGENT 341097	as above

Submitted by: [Signature] CC: M. McCABE  
 Date: 1/18  
 Page: 1 of 3  
 Follow-up Dates: 1/19/84  
 Final Laboratory Approval: [Signature]  
 Date: 1/19/84  
 GCA/Tech Div. 2/80



PRE-TEST NOZZLE CALIBRATION

Date 1/26/84 Set No. 72 SASS Calibrated by J. Lucheret

Nozzle identification number	D <sub>1</sub> mm, (in.)	D <sub>2</sub> mm, (in.)	D <sub>3</sub> mm, (in.)	ΔD, mm, (in.)	D <sub>avg</sub>
1-3	600	600	600	.003	.600
2-5	600	600	600	.001	.600
3-5	623	622	622	.001	.622
5-1	.757	.737	.737	.000	.737
5-2	.868	.870	.871	.003	.870

POST-TEST NOZZLE CALIBRATION

Date \_\_\_\_\_ Set No. \_\_\_\_\_ Calibrated by \_\_\_\_\_

where:

D<sub>1,2,3</sub> = nozzle diameter measured on a different diameter, mm (in.).  
 Tolerance = measure within 0.25 mm (0.001 in.).

ΔD = maximum difference in any two measurements, mm (in.).  
 Tolerance = 0.1 mm (0.004 in.).

D<sub>avg</sub> = average of D<sub>1</sub>, D<sub>2</sub>, D<sub>3</sub>.

Calibrated By J. B. Borne / R. Flaherty

Barometric Pressure, Pb = 30.17 in. Hg

Date 8 Nov 1983

Dry Gas Meter No. \_\_\_\_\_

Control Box No. 1726 - 9

Orifice manometer setting, in. H <sub>2</sub> O	Gas volume wet test meter V <sub>w</sub> , ft <sup>3</sup>	Gas volume dry gas meter V <sub>d</sub> , ft <sup>3</sup>	Temperature				Time 0. min	γ	ΔHg	Deviation	
			Wet test Meter t <sub>w</sub> , °F	Dry gas meter						Y	ΔH <sub>0</sub>
				Inlet t <sub>di</sub> , °F	Outlet t <sub>do</sub> , °F	Average t <sub>d</sub> , °F					
0.5	5	4.2	70	70	70	0.19	0.999	1.905			
1.0	5	4.2	70	70	70	0.19	0.992	1.946			
1.5	10	10.2	70	70	70	0.19	0.999	1.892			
Average								0.997	1.913		

Calculations:

$$V_d = V_w \left( \frac{P_b}{P_b - \Delta H_0} \right) \left( \frac{t_w + 460}{t_d + 460} \right)^2$$

$\frac{5 \text{ ft}^3 \times 30.17 \text{ in. Hg}}{30.17 \text{ in. Hg} - 1.913 \text{ in. Hg}} \left( \frac{70 + 460}{70 + 460} \right)^2 = 10.2 \text{ ft}^3$

Accuracy of wet test meter to dry test meter. Tolerance = ± 0.01  
 Orifice pressure differential that flows 0.75 cfm of air at 70° F and  
 30.2 inches of mercury, in. H<sub>2</sub>O. Tolerance = ± 0.15

Maintenance Checklist

- Oil Reservoir Level ; Knockout Jar
- Vacuum Gage ; Leak Check (No Leak) 15" Hg
- Leakers: Clean  Lubricate
- Check for Leaks ; Fluid Level ; Clean Surface
- Valve: Check for Click
- 2.5 amp. Probe Heater ; 7 amp. Pump ; 10 amp. Heater
- Connector  (Check with umbilical cord connected to hot box)
- ~~Probe Heater~~ (Check operation with Probe or light)

(All Items Must Be Checked and Initialed)



Technology Division

QC Okayed

Calibrated By F. Farley / R. Vachner

Barometric Pressure, Pb = 29.73 in. Hg

Date 21 March 1984

Dry Gas Meter No. \_\_\_\_\_

Control Box No. 1726

Orifice manometer setting, $\Delta H$ , in. H <sub>2</sub> O	Gas volume wet test meter $V_w$ , ft <sup>3</sup>	Gas volume dry gas meter $V_d$ , ft <sup>3</sup>	Temperature				Time $t_o$ , min	$\gamma$	$\Delta H_0$	Deviation	
			Wet test		Dry gas meter					$\gamma$	$\Delta H_0$
			Meter $t_w$ , °F	Inlet $t_{di}$ , °F	Outlet $t_{do}$ , °F	Average $t_d$ , °F					
0.5	5	5.678	62.5	68.5	71.5	70	13.11	.977	1.888		
1.0	5	5.1	62.5	71.5	73.5	72.5	9.26	.977	1.815		
2.0	10	10.232	62.5	72	78	75	13.18	.996	1.896		
Average								.977	1.884		

Calculations

$\gamma$	$\Delta H_0$
$\frac{V_w P_b (t_d + 460)}{V_d \left( P_b + \frac{\Delta H}{13.6} \right) (t_w + 460)}$	$\frac{0.0317 \Delta H}{P_b (t_d + 460)} \left[ \frac{(t_w + 460) \theta}{V_w} \right]^2$

$\gamma$  = Ratio of accuracy of wet test meter to dry test meter. Tolerance =  $\pm 0.01$   
 $\Delta H_0$  = Orifice pressure differential that gives 0.75 cfm of air at 70° F and 29.92 inches of mercury, in. H<sub>2</sub>O. Tolerance -  $\pm 0.15$

Maintenance Checklist

- Vacuum System: Oil Reservoir Level ; Knockout Jar
- Vacuum Gage ; Leak Chk (No Leak) 15" Hg
- Quick Connects: Clean  Lubricate
- Manometer: Check for Leaks ; Fluid Level ; Clean Surface
- Solenoid Valve: Check for Click
- Fuses: 2.5 amp. Probe Heater ; 7 amp. Pump ; 10 amp. Heater
- Amphenol Connector  (Check with umbilical cord connected to hot box)
- ~~Variable Transformer~~  (~~Check Operation with Probe or Light~~)

(All Items Must Be Checked and Initialed)



GCA CORPORATION  
Technology Division

QC Okayed \_\_\_\_\_



CHAIN OF CUSTODY RECORD  
WATER & WASTEWATER

CONTRACT NO: 1-619-099		ANALYSES			
SAMPLERS (Signatures)					
Sample Code	Seq. No.	Date	Source Description	Container	
				Size	G/P
12-MS-BH-2			DASH HALL BLDG		
-3			ASSIGNING AGENCY		
-4					
-FBB-1					
-FBB-2					
12-MS-CD-0-2			CREAMERY BUILDING		
-3					
-4					
-FBB-1					
-FBB-2					
-B					
12-MS-CD-0-1			INDOOR AIR COND. UNIT		
-3			5101 AUGUST ST. C.D.-0		
-4					
-FBB-1					
Relinquished by: M, H			Date/Time: 2/8/30	Received by:	
Relinquished by:			Date/Time:	Received by:	
Relinquished by:			Date/Time:	Received by:	
Relinquished by:			Date/Time:	Received by:	





CHAIN OF CUSTODY RECORD  
WATER & WASTEWATER

5

CONTRACT NO: 1-619-099		ANALYSES				
Sample Code	Seq. No.	Date	Source Description	Container		Comments
				Size	G/P	
<p>SAMPLERS (Signatures)</p> <p><i>[Signature]</i></p>						
NB-PI-20			ALAN INDEPENDENT			500ml Amber
-21						
-22						
-23						
-24						
-25						
-26						
-27						
NB-SF-28			INDEPENDENT			500ml Amber
29						
30						
31						
32						
33						

Relinquished by:	Date/Time	Received by:	Date/Time	Relinquished by:	Date/Time	Received by:	Date/Time
<i>[Signature]</i>			9/8/13				

Relinquished by:	REMARKS:





