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UNION CHEMICAL TRIAL BURN
SAMPLING AND ANALYSIS

Final Report

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CONTENTS

Appendix

FIGURES

TABLES

TABLES (continued)

SECTION 1

SUMMARY

A trial burn was conducted on Union Chemical's hazardous waste incinerator on 3 and 4 November 1983. The incinerator is briefly described in Section 2 of this report. Sampling and field analysis procedures are presented in Section 3. Laboratory analysis protocols and quality assurance results are presented in Sections 4 and 5, respectively. The program results are discussed in Section 6. Additional details on sampling and analytical procedures are presented in the Quality Assurance Project Plan which is included as Appendix A. The purpose of this section is to present a brief summary of the program results.

During the three replicate test runs, the average combustible waste feed rate was 610 lb/hr (8.1 x 10^6 Btu/hr) and the contaminated water feed rate was 800 lb/hr. The combustible waste feed contained 13.8 percent chlorine and 4.7 percent ash. The viscosity of the combustible waste could not be measured by standard procedures because residues in the feed interfered with the analysis.

The average temperature in the freeboard or combustion zone above the bed was 2201°F. At the inlet to the ash knockout chamber, the temperature averaged 2146°F.

The average destruction/removal efficiency for five difficult to incinerate chlorinated organic compounds ranged from greater than 99.9915 percent to 99.99949 percent as shown in Table 1-1. Concentrations of these five compounds in the flue gas, based on the gas chromatography/mass spectrometry (GC/MS) analysis of volatile organic sampling train samples, were in the 1-9 ppb (volume/volume) range. The results of the primary sampling and analytical methods, presented in Table 1-1, demonstrate that the destruction/removal efficiency was greater than 99.99 percent.

During the test burn, field analyses were conducted to yield an immediate indication of incinerator performance. The field analyses using gas chromatography with electron capture detection (GC/ECD) tended to indicate slightly lower destruction/removal efficiencies but only 1,1,2-trichloro-1,2,2-trifluoroethane (which is not an Appendix VIII hazardous pollutant) appeared to be below 99.99 percent. Definitive program results and conclusions should be based on the primary laboratory GC/MS results presented in Table 1-1.

TABLE 1-1. SUMMARY OF DESTRUCTION/REMOVAL EFFICIENCY RESULTS[a]

	Concentration in Combustible Waste Feed, Percent by Weight	Concentration in Flue Gas		Destruction/ Removal Efficiency, Percent
		$\mu g/m^3$	ppb[b]	
1,1,2-trichloro-1,2,2-trifluoroethane	0.29	<10	<1.2	>99.9915
trichloromonofluoromethane	1.5	48	7.8	99.9916
tetrachloroethene	3.9	64	8.6	99.9958
trichloroethene	0.76	11	1.5	99.9967
1,1,1-trichloroethane	5.1	10	1.7	99.99949

[a] All results are based on gas chromatography/mass spectrometry analysis of samples collected with a volatile organic sampling train. The average result for three runs is presented.

[b] Parts per billion (volume/volume).

Measurement of products of incomplete combustion (PICs) in the flue gas was planned. For this program, PICs were defined as compounds at concentrations above 100 µg/m^3 that could be identified by the planned gas chromatography/mass spectrometry procedures. Data were not obtained due to a computer malfunction.

However, we believe there would have been no PICs at the observed destruction efficiencies. The above conclusion is based on tests, for EPA, of six small boilers that were burning waste oil. The waste oil was spiked with chloroform, 1,1,1-trichloroethane, trichloroethene, and tetrachloroethene at concentrations of 0.2 to 0.5 percent. The destruction/removal efficiency was typically only 99.8 percent compared to greater than 99.99 percent at Union Chemical. Test compound emission concentrations averaged about 100 µg/m^3 compared to about 50 µg/m^3 at Union Chemical. Under these circumstances, there were no PICs present at concentrations above 100 µg/m^3. Because the destruction/removal efficiencies for the test compounds were much higher and the emission concentrations were lower at Union Chemical, it is unlikely that any PICs were emitted.

The scrubber water and scrubber water supply were analyzed for 31 volatile organic priority pollutants. The only compound detected in the scrubber water was methylene chloride, a common laboratory contaminant, at concentrations of 8.7, 13, and 8.5 µg/l. The scrubber water supply also contained methylene chloride at similar concentrations (10, 9.7, and 7.6 µg/l). Low concentrations (<10 µg/l) of 1,1,1-trichloroethane, trichloroethene, tetrachloroethene, toluene, and ethylbenzene were detected in one or more of the scrubber water supply samples.

Ash from the cyclone was analyzed for the five volatile chlorinated organic test compounds. None of these compounds were detected at a detection limit of 10 µg/g. The scrubber sludge sample was not analyzed because the sample contained a significant amount of head space. Because the scrubber water did not contain any of the five test compounds (detection limit 1 µg/l), it is very unlikely that any of the compounds would have been detected in the sludge.

Potential hydrogen chloride (HCl) emissions, based on analysis of the combustible waste feed, averaged 86.6 lb/hr. Actual emissions averaged 0.51 lb/hr which demonstrates a control efficiency of 99.42 percent. The HCl concentration in the flue gas averaged 51-60 ppm (volume/volume based on dry flue gas) when corrected to 7 percent oxygen (O_2). The higher value is based on the measured O_2 concentrations in each run while the lower value is based on the average O_2 concentrations for runs 2 and 3. Because the high O_2 concentration in run 1 was inconsistent with the other runs and the process data, there may be some justification for using the lower number.

Particulate emissions, corrected to 7 percent O_2, averaged 0.50 to 0.60 gr/dscf. The range reflects the same variations in O_2 concentrations that were noted in the previous paragraph.

The concentration of carbon monoxide (CO) in the flue gas averaged 2 ppm, 11 ppm and not detectable (probably less than 1 ppm) for runs 1, 2, and 3, respectively. Brief peak concentrations of 30 ppm were observed in run 2.

The higher CO concentrations in run 2 did not appear to reflect any decrease in the destruction/removal efficiency of the chlorinated organic test compounds. The destruction/removal efficiency for run 2 was actually similar to or greater than runs 1 and 3.

Leachates were generated from an ash sample and a scrubber sludge sample in accordance with Federal Resource Conservation and Recovery Act EP Toxicity procedures. The leachates were analyzed for arsenic, barium, beryllium, cadmium, chromium (VI), iron, lead, mercury, selenium and silver. The concentrations of each of these metals in both samples were well below applicable hazardous waste criteria.

The combustible waste feed and particulate emissions were analyzed for the trace metals noted in the previous paragraph. Iron and lead were found in the waste feed and the emissions at higher concentrations than the other metals.

SECTION 2

FACILITY DESCRIPTION

Union Chemical has submitted a detailed engineering description of the incinerator as part of its Part B permit application. A brief description of the facility is presented in this section as background to the sampling and analysis program. A schematic of the Union Chemical fluidized bed incineration system is presented in Figure 2-1.

Combustible wastes consist of residues from the solvent reprocessing operations and other organic chemicals not suitable for reprocessing. These wastes are mixed in either of two continuously stirred 1500 gal storage tanks to achieve the proper viscosity, chlorine content, solid content, and heating value. Typically, one tank contains nonchlorinated wastes and the other contains chlorinated materials. For this test program, drums of selected wastes were mixed in one of the tanks to meet the program specifications.

Potentially contaminated water is collected and stored to prevent site run-off. This water, containing trace organics, is also fed to the incinerator.

The fluidized-bed incinerator is a refractory lined cylindrical vessel with a height of 24 feet and an inside diameter that varies from 32 in. in the bed area, to 42 in. in the freeboard area. Overfire air is injected into the freeboard area at five different heights. Primary combustion air, for bed fluidization, is supplied by a forced draft fan to the plenum below the air distribution plate. The distribution plate supports the silica sand bed and provides openings for injection of the fluidizing air.

The duct from the incinerator to the ash-drop-out chamber and the chamber itself are refractory lined. The ash knockout chamber is essentially a settling chamber that collects small quantities of sand elutriated from the bed and coarse particulates from the waste feed.

The reactor was originally designed for dry neutralization of acid gases and cooling by air dilution, but is no longer used for these purposes.

A refractory lined Fisher Kostermann XQ cyclone removes most of the particulate matter from the flue gas. Solids are removed from the cyclone by a continuously operating rotary valve.

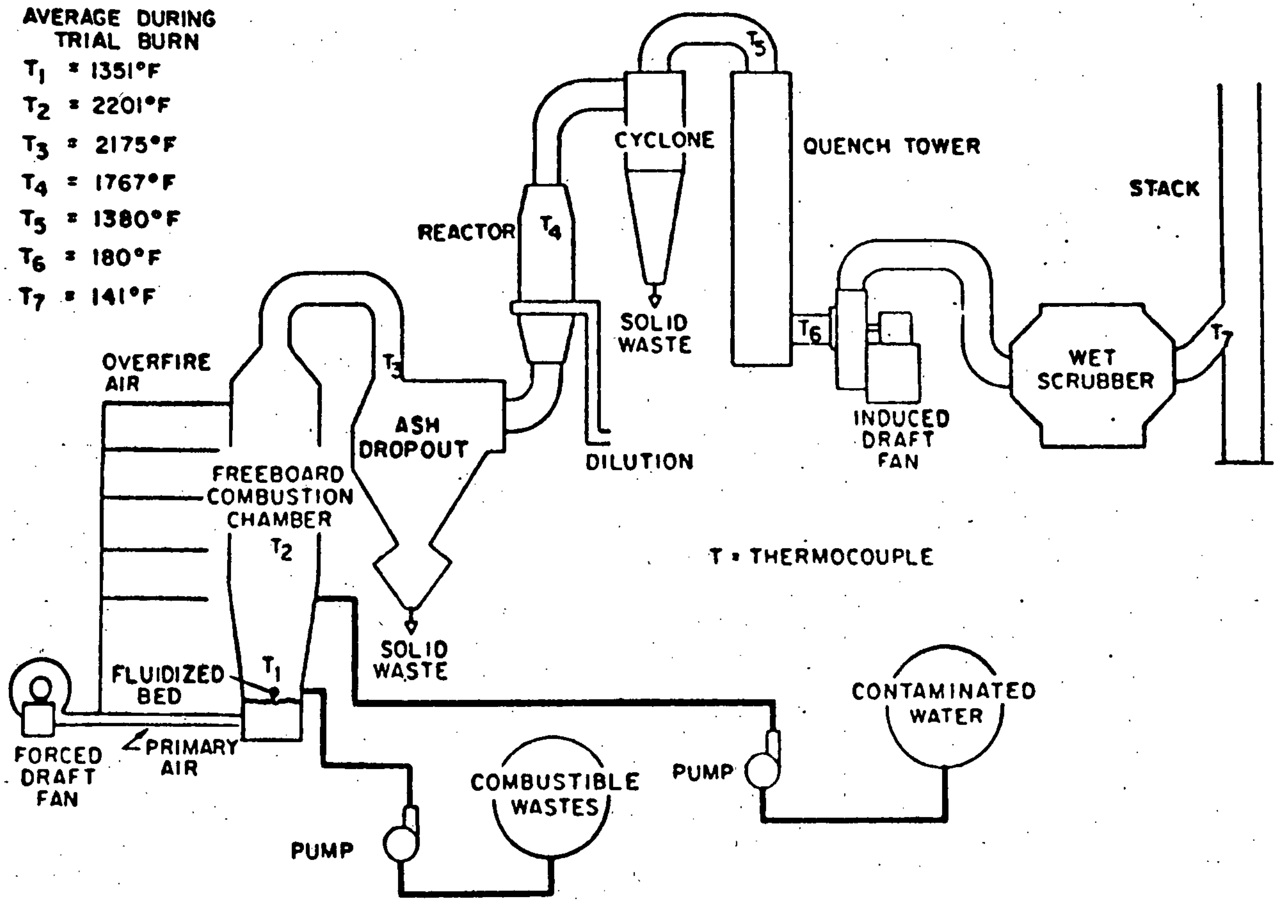


Figure 2-1. Schematic of Union Chemical fluidized bed incineration system.

In the quench tower, flue gases are cooled from about 1300°F - 1400°F to about 180°F. Lime slurry is fed to the quench tower for removal of HCl from the gas stream. Quenching liquid is recycled after the solids are removed in a settling vessel.

The final control device is a horizontal cross flow packed tower manufactured by Celicote. Lime slurry is used to adjust the pH of the scrubber liquid and to insure that HCl emissions meet the regulatory requirements.

The 2 foot diameter stack is 60 feet high. A sampling platform is located 30 feet above the ground and 10 stack diameters downstream from the transition joining the scrubber with the stack. The sampling platform is 15 stack diameters upstream from the stack exit.

Operating data were recorded by the incinerator operators and are presented in Appendix B.

SECTION 3

FIELD SAMPLING AND ANALYSIS

GCA/Technology Division conducted a trial burn at the Union Chemical facility on November 3 and 4, 1984 in order to demonstrate compliance with applicable hazardous waste incineration performance standards. The trial burn consisted of three replicate test runs during which representative samples were collected from seven process streams.

SOLID AND LIQUID STREAMS

A list of six solid and liquid streams and the total number of samples collected and analyzed is presented in Table 3-1. The specific sampling and measurement techniques for each of these streams are addressed below.

Combustible Waste Feed

Liquid combustible waste feed samples were obtained from a tap located in the line connecting the liquid waste holding tank and the burners. Two volatile organic analysis (VOA) samples and one 50 ml grab sample were collected at 20 minute intervals throughout each 2-hour incineration burn. The VOA samples were collected for POHC analysis while the individual grab samples were composited into a single sample from each run for subsequent inorganic and physical characterization.

The average waste feed rate was determined from the change in the level in the feed tank and the density of the feed (see Appendix B).

Contaminated Water Feed

Union Chemical Co. introduces on-site contaminated run-off water into the incinerator as required to maintain a combustion chamber temperature of 2000 - 2200°F. Duplicate VOA samples of this stream were collected during each test run from a tap located on line between the holding tank and the injection point. These samples were maintained for POHC and inorganic analysis. The contaminated water feed rate was determined from the change in the level in the feed tank (see Appendix B).

Scrubber Water Supply

Duplicate samples of clean scrubber water were collected once during each run. These samples were extracted from an on-line tap into VOA vials for subsequent POHC analysis.

TABLE 3-1. SAMPLING AND ANALYSIS SUMMARY FOR LIQUID AND SOLID STREAMS

Sample type	No. of samples collected	No. of samples analyzed[b]	Analysis	Method
1. Combustible Waste feed	36 VOA vials 3-500 ml	3 composites 3-500 ml	POHCs[a] Chlorine Metals	Tetraglyme--GC/MS Parr Bomb--Ion chromatography ICAP[c]
2. Contaminated water	6 VOA vials	3 composites	POHCs Chloride	Purge and trap--GC/MS Ion chromatography
3. Ash	3-1 gal	1 composite	POHCs EP toxicity	GC/MS EP toxicity
4. Scrubber water	18 VOA vials	3 composites	POHCs	Purge and trap--GC/MS
5. Scrubber sludge	3-1 gal	1 composite	POHCs EP toxicity	GC/MS EP toxicity
6. Scrubber water supply	6 VOA vials	3 samples	POHCs	Purge and trap--GC/MS

[a] Trichloroethene, tetrachloroethene, 1,1,1-trichloroethane, trichloromonofluoromethane and 1,1,2-trichloro-1,2,2-trifluoroethane.

[b] Not including additional quality control analyses.

[c] Inductively coupled argon plasma spectrometry.

Scrubber Water

Process water is recirculated from a contaminated water holding tank through a quench tower and packed bed scrubber for cooling and pH adjustment of the flue gas. Three sets of duplicate VOA samples were collected from this closed system during each sample run for POHC analysis.

Scrubber Sludges

The scrubber water system includes a settling tank for the removal of particulate matter collected by the scrubbing liquor. A 1-gallon, dual phase solid, aqueous sample was collected from the settling tank at the conclusion of the test series and subjected to analyses for POHC content and EP Toxicity.

Ash

Solid waste combustion material was collected from both the ash dropout chamber and cyclone hopper at the conclusion of each test run. Ash samples from these two streams were composited in the field in approximate proportion to the stream flow rates. The 1-gallon composite samples were retained for POHC and EP Toxicity analysis.

FLUE GAS STREAM

Five separate sampling systems were used to characterize the atmospheric emissions from the incinerator. A summary of the samples collected during each trial burn test run is presented in Table 3-2. The field data sheets resulting from these tests are presented in Appendix C.

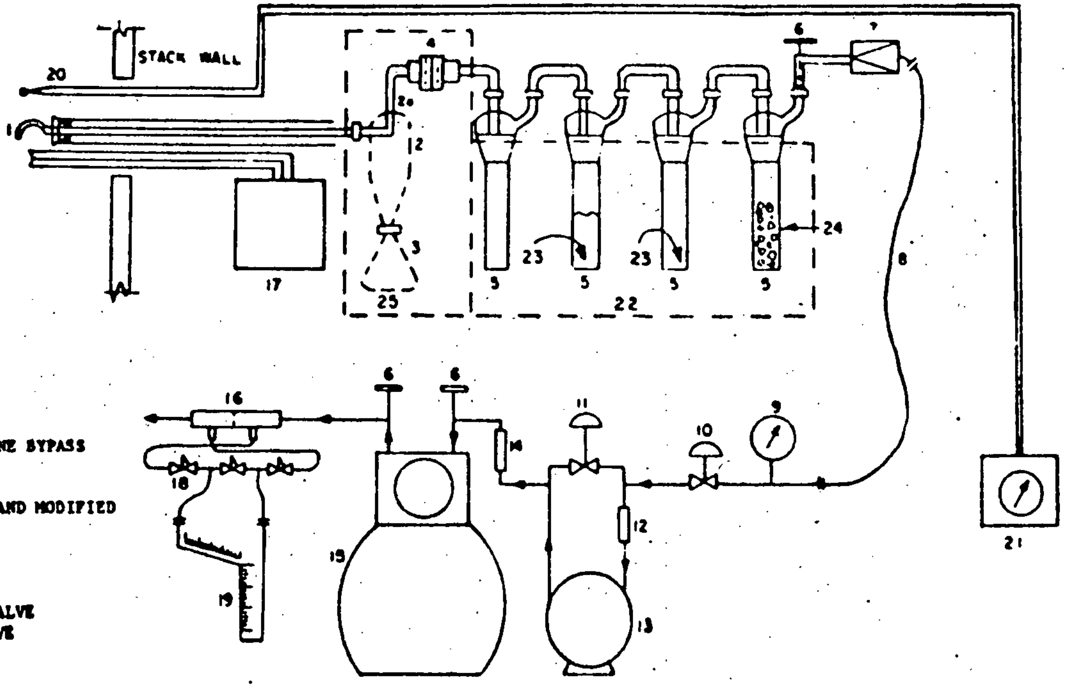
Particulate and HCl Emissions

A modified EPA Method 5 Train was used to simultaneously collect particulate and HCl from the flue gas. A velocity traverse was performed along two diameters of the stack prior to the initiation of sampling. Twenty four sampling points, as determined by EPA Method 1, were sampled during each of the three test runs. The proposed sampling time for each of the points was 5 minutes providing a total sampling time of 120 minutes. The sampling time for the final run was reduced to 60 minutes when a previous test run was voided due to the presence of a significant leak in the sampling train. Sampling for each of the three test runs was determined to be isokinetic (±10 percent). A schematic of the modified Method 5 train is presented in Figure 3-1. The sampling train consisted of a heated stainless steel probe with a stainless steel button hook nozzle and attached thermocouple and pitot tubes. The sampled gas passed through the probe assembly to a heated glass fiber filter (Reeve Angel 934 AH). The filter holder was maintained at 248°F ±25 throughout the test period. Downstream of the heated filter, the gas passed through a series of four ice-cooled impingers to effect the removal of entrained moisture. The first impinger was left empty to provide for the collection of the flue gas condensate. The recovered sample of condensate was retained for HCl analysis. The second and third impingers each contained

TABLE 3-2. SUMMARY OF FLUE GAS SAMPLES

Sample description	Analysis	Number collected per run	Container type	Comments
Modified Method 5				
Particulate filter	Gravimetric	1	Petri dish	
Front half	Gravimetric	1	500-ml LPE	
Condensate	Chlorine	1	1-liter LPE	a
Impingers 2 and 3	Chlorine	1	500-ml LPE	a
VOST				
Tenax cartridge	Volatile organic	3	Self contained	a
Tenax:charcoal cartridge	Volatile organic	3	Self contained	a
Condensate	Volatile organic	1	VOA vial	a
Tenax cartridge, field-biased blank	Volatile organic	1	Self contained	a
Tenax:charcoal cartridge, field-biased blank	Volatile organic	1	Self contained	a
Condensate, field-biased blank	Volatile organic	1	VOA vial	a
Tenax, method blank	Volatile organic	1	Glass culture tube	a
Tenax:charcoal, method blank	Volatile organic	1	Glass culture tube	a
Gas Bag Analysis				
Tedlar bag sample	Volatile organic	2	25-l Tedlar bag	
Condensate	Volatile organic	1	VOA vial	a
Tedlar bag, field blank	Volatile organic	1	25-l Tedlar bag	
Condensate, field blank	Volatile organic	1	VOA vial	a
Tedlar bag for fixed gases	Fixed gases	2	25-l Tedlar bag	
Continuous Emission Monitoring				
Carbon monoxide	NDIR		None	

[a] Stored at 4°C.



1. PROBE
2. CYCLONE 2a. CYCLONE BYPASS
3. FLASK
4. PARTICULATE FILTER
5. IMPINGERS, STANDARD AND MODIFIED
6. THERMOMETER
7. CHECK VALVE
8. UMBILICAL CORD
9. VACUUM GAUGE
10. COARSE FLOW ADJUST VALVE
11. FINE FLOW ADJUST VALVE
12. OILER
13. VACUUM PUMP
14. FILTER
15. DRY GAS METER
16. ORIFICE TUBE
17. HASTINGS METER
18. SOLENOID VALVES
19. MONOMETER
20. THERMOCOUPLE
21. PYROMETER
22. ICE BATH
23. 1 N NaOH
24. SILICA GEL DESICCANT
25. HOT BOX

Figure 3-1. Schematic of modified Method 5 sampling train.

100 ml of 1 N NaOH to ensure the complete collection of HCl. The final impinger contained a known amount of desiccant. The impingers were followed by a pump, gas meter and calibrated orifice.

A Hastings Air Velocity Meter was substituted for the traditional water manometer in the determination of the flue gas velocity and volumetric flow rate of the unit. This technique eliminated the potential interferences in making flow determinations in saturated gas streams.

An additional integrated sample of flue gas was collected in conjunction with the modified Method 3 test for fixed gas analysis. The samples were analyzed for CO_2 and O_2 using a Orsat Gas Analyser.

Carbon Monoxide Monitoring

A continuous monitoring system was used to measure carbon monoxide concentrations throughout each test run in accordance with EPA Method 10. The monitoring system was equipped with a gas conditioning system and continuous chart recorders. The flue gas was 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). Carbon monoxide concentrations were determined using a Horiba Model PIR 2000 nondispersive infrared CO analyzer. Centified gases containing 0 ppm, 50 ppm and 950 ppm were used to calibrate the monitor.

VOLATILE ORGANIC COMPOUNDS

VOST

The volatile organic sampling train was used as the primary method to quantify the concentrations of the POHCs in the flue gas. This method employs Tenax, an organic sorbent resin, to collect the organic species of interest.

The train consisted of a heated glass-lined probe with a glass wool plug to remove particulate, followed by an assembly of condensers and organic resin traps as illustrated in Figure 3-2. The first condenser was used to cool the gas stream and condense the water vapor present. The flue gas and condensed moisture were then passed through a cartridge containing 1.5 grams of Tenax resin (60 to 80 mesh). The condensate was collected in the first impinger which is continually purged by the gas stream itself. The second condenser and trap containing Tenax/charcoal (50/50) served as a backup for low volume breakthrough compounds. A series of impingers and drying tubes was placed downstream of the second Tenax trap for residual moisture removal.

Sample temperatures were monitored at the outlet of the sample probe and the inlet to the Tenax cartridge through the use of thermocouples. The gas temperature through the probe was maintained above 130°C to prevent the premature condensation of the volatile components. The temperature of the gas through the resin cartridges was maintained at less than 20°C. The sample gas volume through the resin traps was maintained at approximately 1/2 liter per minute. The total sample volume for each set of tubes did not exceed 20 liters.

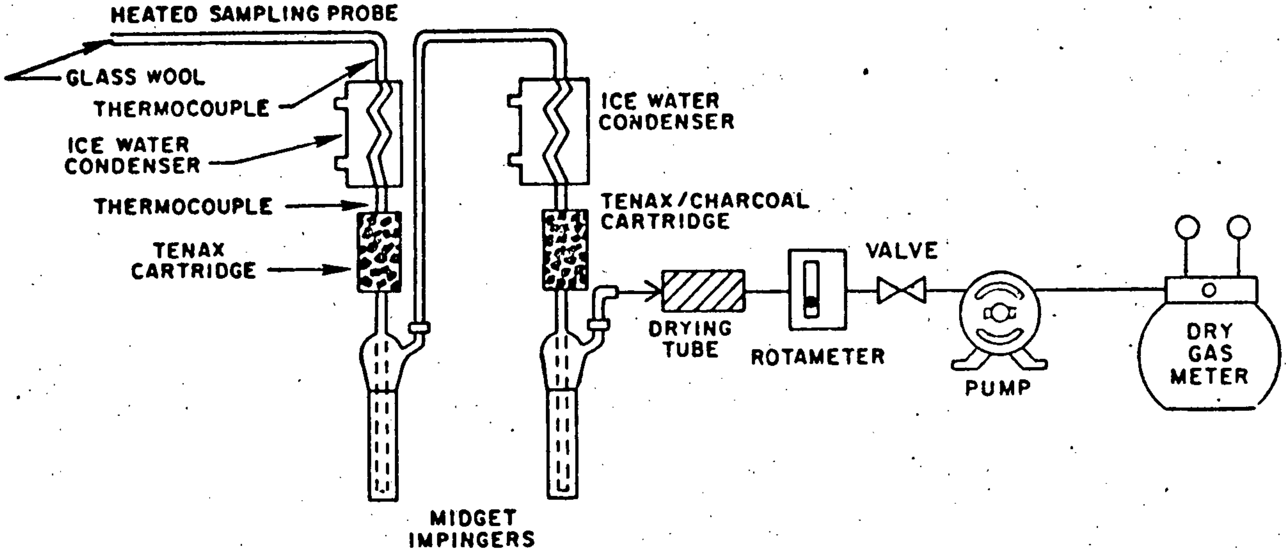


Figure 3-2. Schematic of volatile organic sampling train.

Three VOST runs were conducted during each 2-hour test period. The results of the three runs were averaged to yield one result for each test. Method and field blanks of the sorbent resins were collected in conjunction with each of the three tests. During the sampling program, the reagents and sorbent resin samples associated with this train were maintained offsite to minimize the potential for sample contamination from the ambient air.

Tedlar Bag Samples

Additional samples of the flue gas were collected for POHC analysis by GC/ECD in the field. Duplicate samples of flue gas were collected through the use of an integrated gas sampling train as illustrated in Figure 3-3.

The sample was extracted from the stack through a stainless steel probe containing pre-extracted glass wool to remove particulate. A condenser was used to remove excess moisture from the gas stream. Prior to their use in the field, the Tedlar gas bags were baked in an oven at 130°C for a period of 1 hour and purged with prepurified nitrogen. The sample was collected at a rate of 0.3 liters per minute in conjunction with the VOST tests. At the conclusion of each test, the bags were sealed and removed to a designated area for chromatographic analysis. The condensate samples were collected in VOA vials and maintained at 4°C pending analysis. Blank samples of the condensate and bag samples (conditioned bags, inflated with prepurified nitrogen) were collected in conjunction with each sample run.

The collected samples were subjected to GC/ECD analysis for the volatile components of interest. Sample gas was aspirated from each bag through a heated gas sampling valve and then injected onto the GC by diverting carrier flow through the valve sample loop. Instrumental conditions used for this analysis are presented in Table 3-3.

Calibration standards were prepared daily by injecting microliter quantities of commercially available solvents into a 500 ml gas sampling bulb which had been previously rinsed with hexane, heated at 120°C and prepurged with nitrogen. Serial dilutions were then performed using a gas-tight syringe and several 1-liter prepurged Tedlar bags in order to provide a four-point calibration curve between 20 and 240 $\mu g/m^3$. Calibration standards were analyzed under the same operating conditions as samples. A minimum of two of the four calibration points were analyzed in duplicate with an acceptance criterion of ± 20 percent. Calibration curves were prepared from a linear regression analysis of the integrated area response from the injections of calibration standards. The calibration curves were all determined to have correlation coefficients greater than or equal to 0.99. All samples were quantified by entering the sample area response into the appropriate calibration curve. The reported results represent the mean of two determinations.

FIELD SAMPLING QUALITY ASSURANCE

A detailed Quality Assurance Project Plan was written for this project. This document was used as a guide for use during the field, lab and data handling segments of this project. The following items are highlights of some

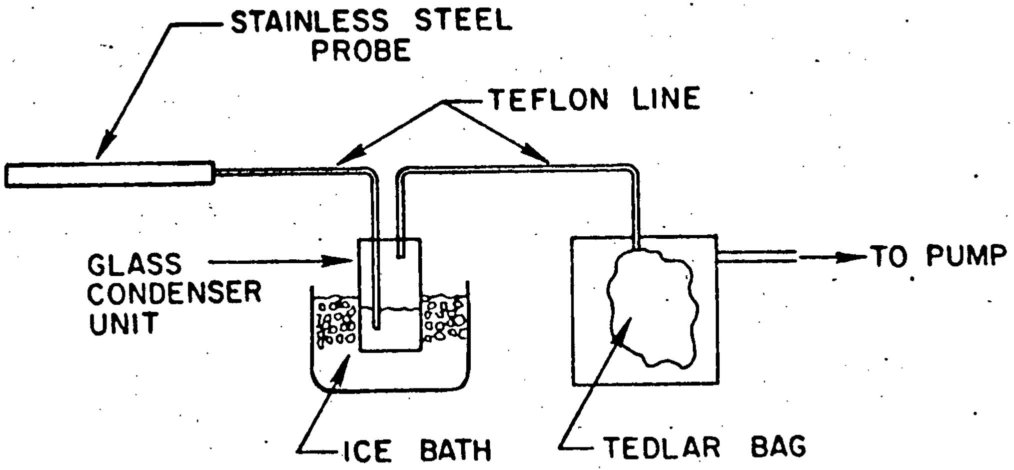


Figure 3-3. Integrated gas sampling train.

TABLE 3-3. GC/ECD OPERATING CONDITIONS FOR TEDLAR BAG ANALYSIS

Instrument	Perkin Elmer 3920 with Ni^{63} electron capture detector and Spectra Physics Minigrator
GC Conditions	
Column	20% SP-2100/0.1% Carbopack 1500 on 100/120 mesh Supelcoport, 10 ft x 1/8 in. SS column
Temperature program	Isothermal at 50°C
Injector temperature	110°C
ECD temperature	325°C
Carrier flow	Argon/methane, 25 ml/min
Sampling Valve Conditions	
Loop volume	1 ml
Loop temperature	125°C

of the procedures implemented during the field sampling portion of this project to assure the generation of quality data.

- Maintenance of the proper sample chain of custody.
- Use of properly calibrated equipment.
- Field calculation to assure isokinetic ± 10 percent.
- Collection of the appropriate field sample blanks.
- Use of standardized forms.

There were very few deviations from the sampling procedures outlined in the QAPP. The following is a summary of QAPP deviations.

- Use of GC/TCD for determination of fixed gases; at a pretest meeting it was agreed the EPA Reference Method 3 would be used instead of the GC.
- There were intermittent problems with the Hasting flowmeter. During these instances the pitot lines were connected to the inclined manometer for Δp determination. The pitot lines were blown out to assure moisture was not accumulating in the system. Manometer readings were consistent with the Hasting flowmeter readings.

The carbon monoxide concentrations were very low (in the range of near zero to 30 ppm). This created a problem in that the lowest calibration gas was 50 ppm and the instrument output voltage were two low to overcome noise associated with the strip chart recorder. Instrument outputs were accurately recorded at 5-minute intervals by observing a digital voltmeter.

The sample custody procedures used for the project followed EPA recommended procedures. A detailed description of the Quality Assurance procedures followed in this program are presented in the Quality Assurance Project Plan (see Appendix A).

SECTION 4

LABORATORY ANALYSES

INTRODUCTION

The following section presents a discussion of the procedures used for sample preparation and instrumental analysis for the following analytes and matrices:

• Total chlorine (as chloride) and chloride	- Combustible waste feed - Contaminated water feed - Flue gas particulates - Flue gas condensates - Flue gas impinger catches
• Trace metals	- Combustible waste feed - Solid waste leachates (ash, sludge) - Flue gas particulates
• POHCs	- Combustible waste feed - VOST tubes and condensates - Composited ash - Contaminated water
• Priority pollutant volatile organics	- Scrubber water supply - Contaminated scrubber water

Quality control protocols for the above analyses are presented in Section 5. Analytical results are provided on Data Report Sheets in Appendix E.

CHLORIDE ANALYSES

Combustible Waste Feed

The total chlorine/chloride content of the fuel feed was determined as total chloride by Parr bomb combustion followed by ion chromatography (IC) analysis. The procedures used for this analysis are detailed on p. 49 of the Quality Assurance Project Plan (QAPP) which is presented in Appendix A.

Contaminated Water Feed

Chloride analysis of the contaminated water feed was accomplished via the direct injection ion chromatographic techniques detailed on p. 48 of the QAPP (see Appendix A).

Flue Gas Particulates

Equivalent aliquots of the particulate filter and probe rinse residue were combined and subjected to hot aqueous extraction, followed by sonication. Subsequent analysis for chloride was performed using the direct injection ion chromatography procedure referenced above for water feed samples and presented on p. 48 of the QAPP (see Appendix A).

Flue Gas Condensates/Impinger Catches

Direct injection ion chromatography analysis for chloride was implemented for these samples. The instrumental procedure was identical to the one used for water feed samples (see p. 48 of QAPP in Appendix A).

TRACE METALS ANALYSES

Waste Feed

The combustible waste feed samples were initially prepared for analysis of silver, barium, beryllium, cadmium, chromium, iron and selenium by controlled dry ashing. Sample aliquots (2-5 g) in platinum crucibles were placed beneath an array of adjustable heat lamps which were lowered at a rate such that samples neither boiled nor ignited. Samples were exposed for an approximate 8 hour period or until they achieved a tar-like appearance. The range of the exposure varied from ambient temperature to approximately 180°C. The samples were subsequently transferred to a cold muffle furnace where the temperature was gradually elevated to 600°C over a period of approximately 6 hours. Each sample was maintained at 600°C until ashing was complete and the resultant ash dissolved by means of 1:1 Ultrex hydrochloric acid. Metals concentrations were subsequently determined via Inductively Coupled Argon Plasma Emission Spectroscopy (ICAP) utilizing a Jarrell Ash Model 855 Atom Comp. Instrumental analysis followed the procedures outlined in Section 10 of Method 200.7 (Reference 1).

The samples were prepared for arsenic, lead and mercury analyses following the Parr bomb procedure described in Reference 2 (ASTM D3684-78). A weighed sample (~1.0 g) was combusted in an oxygen bomb containing dilute nitric acid absorbing solution. Quantitation of mercury was accomplished by the cold vapor technique utilizing a Perkin Elmer Model 460 Atomic Absorption Spectrophotometer (AAS) in conjunction with a MHS 20 Hydride Generator. These solutions were also analyzed for lead and arsenic by ICAP.

Solid Wastes

Ash and sludge samples were subjected to the Extraction Procedure (EP) leachate generation and analyzed for trace metals as detailed on p. 50 of the QAPP (see Appendix A).

Flue Gas Particulates

Equal aliquots of the particulate filter and probe rinse were combined for analysis of arsenic, barium, beryllium, cadmium, chromium, iron, lead, silver and selenium. Initially the samples wre digested in 3m nitric acid and the digestates filtered through a 0.45 µm Millipore filtering apparatus. The filter plus the insoluble particulates were subsequently digested with hydrofluoric acid in order to achieve total dissolution of silicate material. The resulting digestate as well as the 3m nitric filtrate were than individually analyzed for metals using a Jarrell-Ash Model 855 Atom Comp./ICAP. Instrumental analysis was conducted using the same procedures referenced for waste feed analysis. Measured silver concentrations were verified using flame atomic absorption spectrophotometry utilizing a Perkin Elmer Model 2380.

A second aliquot of the particulate filter and probe rinse were combined and analyzed for mercury according to the cold vapor technique described in Method 245.1 (Reference 1). Quantitation was accomplished on a Perkin Elmer Model 460 Atomic Absorption Spectrophotometer in conjunction with a MHS-20 Hydride Generator.

POHC ANALYSIS

Combustible Waste Feed

An aliquot of the combustible waste feed was diluted in tetraglyme for subsequent purge and trap GC/MS analysis using the procedures discussed on pp. 43-46 of the QAPP (see Appendix A). It should be noted that a Finnigan MAT OWA 30B CC/MS system was used in place of the Hewlett-Packard 5985 specified in the QAPP.

VOST Tubes and Condensates

The Tenax and Tenax/charcoal tubes generated from the VOST runs were analyzed individually using the protocol detailed in pp. 46-48 of the QAPP. Some changes were made in the protocol in order to accommodate equipment replacement and advances in the analytical methodology. The changes are as follows:

- Sample tubes were analyzed using a heating tape desorption unit. Flow from that unit was directed through the purge chamber of a Tekmar LSC-2 purge and trap sample concentrator.
- Internal standards used for this analysis were bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane. These components were added to the Tekmar purge tube prior to sample desorption.
- Surrogate spikes were also utilized for these analyses. A known quantity of d_4-dichloroethane, d_6-benzene and d_8-toluene was injected directly onto the upstream end of the sample tube prior to desorption. The use of surrogate spikes was added to the program as a means of obtaining an indication of efficiency of tube desorption.

All other calibration and tuning procedures were implemented as specified in the QAPP.

Condensates from the VOST trains were analyzed for POHCs using the purge and trap GC/MS procedure provided on p. 43 of the QAPP (see Appendix A).

Composited Ash

An aliquot of the ash composite was extracted with tetraglyme and analyzed for POHCs via GC/MS techniques as detailed above for combustible waste feed.

Contaminated Water

These samples were analyzed for POHCs using the purge and trap GC/MS procedure as described for VOST condensates.

PRIORITY POLLUTANT VOLATILE ORGANICS

Samples of the scrubber water supply and composited scrubber water were analyzed for priority pollutant volatile organics using GC/MS purge and trap protocols as detailed for VOST condensates.

REFERENCES

1. 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. Revised March 1983.

2. Total Mercury in Coal by the Oxygen Bomb Combustion/Atomic Absorption Method." (Method D3684-78), 1982. Annual Book of ASTM Standards, Part 26.

SECTION 5

LABORATORY QUALITY ASSURANCE

Quality control procedures for the analysis of project samples included analysis of laboratory quality control samples, replicate aliquots of project samples and NBS Standard Reference Materials (SRM) and, for organic analyses, the use of surrogate spikes. Quality control data for each of the major analytical categories are presented in this section.

TOTAL CHLORINE AND CHLORIDES

Waste Feed

Quality control protocols for the determination of total chlorine in the waste feed included analysis of replicate aliquots of a project sample and a reference sample (Alpha Resources, Inc., Stevensville, Michigan) certified for chlorine content. Instrument calibration was verified, prior to analysis of project samples by analysis of a quality control sample certified to contain 281 µg/ml chloride; the reported value was 270 µg/ml, a recovery of 96 percent. Replicate aliquots of the composite waste feed from Run 1 were carried through the entire preparation and analysis procedure; the reported values were 13.3 and 13.8 percent total chlorine. A sample of oil certified to contain 1.93 percent chlorine was obtained from Alpha Resources and analyzed in the same manner as project samples. The reported value was 1.83 percent chlorine, a recovery of 95 percent.

Water

Quality control procedures for the determination of chloride in water samples included analysis of an EPA quality control sample. The reported value for WP 478-3 was 21.3 mg/l, a recovery of 104 percent.

Particulate Filters

Quality control procedures for the determination of chloride on particulate filters included preparation of a spiked filter. A blank filter was spiked with 0.5 ml of a 1000 µg/ml chloride standard and analyzed in the same manner as project samples. The expected value was 500 mg Cl^-; the reported value was 610 mg Cl^-, a 122 percent recovery.

Impingers

Quality control procedures for the determination of chloride in impinger samples included analysis of an EPA quality control sample. The reported value for WP 478-3 was 18.7 µg/ml, a recovery of 91 percent.

METALS

Waste Feed

Quality control procedures for the determination of metals in combustible waste feed samples included analysis of an NBS Standard Reference Material and analysis of replicate aliquots of a project sample. An aliquot of NBS SRM 1085, Wear Metals in Lubricating Oil, was prepared and analyzed with project samples, the results are presented in Table 5-1. Replicate aliquots of the combustible waste feed composite from Run 1 were carried through the entire preparation and analysis procedure in order to assess analytical precision, these results are presented in Table 5-2.

Solid Wastes

Quality control procedures for the determination of metals in the solid waste leachate included analysis of an EPA quality control sample for the metals of interest; these results are presented in Table 5-3.

Particulate Filters

Quality control procedures for the determination of metals on particulate filter samples included analysis of blank filter spiked with an EPA quality control sample and analysis of NBS SRM 1648, Urban Particulate; these results are presented in Tables 5-4 and 5-5, respectively. Recoveries for the urban particulate are somewhat lower than expected. The low recoveries may be due to difficulties encountered in solubilizing this sample and may not reflect recoveries of these elements in project samples. No difficulties were noted in solubilizing project samples.

Initial data exhibited high levels of silver in the field blank; the laboratory method blank did not have high silver levels. Analysis of the field blank and samples by atomic absorption spectrophotometry (AAS) confirmed the original data generated using ICAP. Aliquots of several lots of acetone were placed in a beaker, evaporated to dryness and extracted with HF. This resulting extract was found to contain silver. While an adequate data base does not exist at this time, it appears that the acetone used in the front half rinse may have contributed a significant quantity of silver to the blank and samples.

ORGANIC ANALYSIS

Waste Feed

Aliquots of tetraglyme spiked with a series of volatile compounds were analyzed with project samples; these results are presented in Table 5-6. Surrogate recoveries for the waste feed samples are included in Table 5-7.

TABLE 5-1. QUALITY CONTROL DATA: ANALYSIS OF NBS SRM 1085, WEAR METALS IN LUBRICATING OIL

Element	Concentration (μg/g) Reported	Expected	Percent recovery
Aluminum	278	296	94
Chromium	263	298	88
Copper	284	295	96
Iron	274	300	91
Lead	314	305	103
Magnesium	206	297	69
Molybdenum	285	292	98
Nickel	296	303	98

TABLE 5-2. QUALITY CONTROL DATA: ANALYSIS OF REPLICATE ALIQUOTS OF RUN 1 COMPOSITE WASTE FEED (GCA 35263)

	Reported (μg/g)	
Element	35263A	35263B
Barium	120	119
Beryllium	4.42	4.08
Cadmium	3.83	3.93
Chromium	157	152
Iron	21,800	20,100
Selenium	<0.5	<0.5
Silver	<0.02	<0.03

TABLE 5-3. QUALITY CONTROL DATA: ANALYSIS OF EPA AQUEOUS QUALITY CONTROL SAMPLE FOR METALS

	Concentration (µg/ml)		
Element	Reported	Expected	Percent recovery
Aluminum	127	120	106
Cadmium	27	26	104
Chromium	136	160	85
Lead	237	240	99
Mercury	4.2	3.5	120
Selenium	64	60	107

TABLE 5-4. QUALITY CONTROL DATA: ANALYSIS OF SPIKED FILTER FOR TRACE ELEMENTS

Element	Concentration (µg/ml)[a] Reported	Expected	Percent recovery
Barium	496	478	104
Cadmium	4.55	5.2	88
Chromium	23.3	26	90
Lead	32.8	32	103
Mercury	8.24	8.7	95
Silver	9.95	24	41

[a] Reported as concentration in solution.

TABLE 5-5. QUALITY CONTROL DATA: ANALYSIS OF NBS 1648, URBAN PARTICULATE

Element	Concentration (ug/g)		
	Reported	Expected	Percent recovery
Arsenic	129	115	112
Barium	428	737	58
Cadmium	72.8	75	97
Chromium	121	403	30
Iron	25,800	39,100	66
Lead	5,300	6,550	81
Selenium	ND[a]	27	-
Silver	ND[a]	6	-

[a] Not detected.

TABLE 5-6. QUALITY CONTROL DATA: ANALYSIS OF TETRAGLYME SPIKES FOR VOLATILE ORGANICS

Parameter	Concentration (mg/kg)				Average recovery (%)
	Reported				
	1/18/84	1/25/84	$\overline{X}$	Expected	
1,1,1-trichloroethane	2,200	2,000	2,100	2,000	105
Trichloroethene	4,800	4,800	4,800	4,000	120
Tetrachloroethene	1,900	1,800	1,900	1,600	119

TABLE 5-7. QUALITY CONTROL DATA: ANALYSES OF SAMPLES FOR VOLATILE ORGANICS SURROGATE RECOVERIES

Sample	GCA Control	Sample I.D.	% Recovery		
			d_4-1,2-dichloroethane	d_8-toluene	Bromofluorobenzene
Combustible Waste Feed					
	QC 835	Blank	100	90	90
	35257	UC-A-V-1	84	88	89
	35263-1	UC-1-CWF-1	100	65	70
	35263-2	UC-1-CWF-1	88	72	70
	35269	UC-2-CWF-2	120	80	90
	35273	UC-3-CWF-1	90	70	85
Contaminated Water					
	35254	UC-CW-1	110	70	74
	35255	UC-CW-2	100	80	100
	35256	UC-CW-3	140	79	93
Scrubber Water					
	35287	UC-1-SW-1,2,3	110	110	110
	35290	UC-2-SW-1,2,3	120	110	120
	35293	UC-3-SW-1,2,3	110	110	110
Scrubber Water Supply					
	35284	UC-SWS-1	97	100	100
	35285	UC-SWS-2	100	90	93
	35286	UC-SWS-3	99	130	120
Condensates					
	35225	UC-GC-CD-FBB	110	100	100
	35228	UC-GB-CD-1	100	100	100
	35229	UC-GB-CD-2	120	110	120
	35230	UC-GB-CD-3	86	90	91
	35231	UC-V-CD-B	110	110	120
	35232	UC-V-CD-FBB	100	100	100
	35233	UC-V-CD-1	110	110	110
	35234	UC-V-CD-2	130	130	130
	35235	UC-V-CD-3	86	88	88

Waters

Quality control procedures for the determination of volatile organics in water samples included analysis of an EPA quality control sample; these results are presented in Table 5-8. Surrogate recoveries for the water samples are included in Table 5-7.

VOLATILE ORGANIC SAMPLING TRAIN

Quality control procedures for the analysis of Tenax and condensate samples included analysis of a spiked sample and the use of surrogate spikes. An aqueous sample spiked with the parameters of interest was prepared and analyzed with the condensate samples; results from this analysis are presented in Table 5-9. Surrogate recoveries for the condensate samples are included in Table 5-7.

A field-biased blank Tenax and Tenax/Charcoal tube was analyzed for each run; these results are shown in Table 5-10. Each tube was spiked with a series of surrogate compounds which were intended to provide an indication of the efficiency associated with the tube desorption procedure; these results are presented in Table 5-11. In some cases, however, it appears that the low recoveries obtained may be related to the difficulty associated with the addition of the surrogates to the tube rather than a reflection of the desorption efficiency. A single spiked tube was also analyzed with project samples; these results are presented in Table 5-12.

PRECISION, ACCURACY AND COMPLETENESS

Precision and accuracy goals presented in Table 3-2 of the QA Plan have generally been achieved. Completeness, defined as the percentage of generated data points judged valid, is considered to be 100 percent; data on products of incomplete combustion were lost due to a computer malfunction.

DEVIATIONS FROM THE QA PLAN

Minor changes in the analytical procedures stated in the QA Plan have been noted in Section 4 of this report. The analysis of particulate filters and front half rinses for chloride and metals was not addressed in the QA Plan. This represents additional work which was requested after completion of the QA Plan.

Data on products of incomplete combustion (PICs) were lost due to a computer malfunction. VOST tube desorption was initiated immediately upon receipt of samples from the field. While data on one tube were being acquired, the data acquired for the previous tube - including POHC, surrogate and internal standard responses were reduced. Data for PICs were stored on the disc for subsequent reduction. This approach was used because the POHC, surrogate and internal standard data were of primary importance to the program and because evaluation of the generated data was essential for assessment of instrument performance. Normal protocol used in the laboratory includes the transfer of data onto 9-track tapes as soon as possible after acquisition. For these samples, a computer malfunction occurred shortly after completion of

TABLE 5-8. QUALITY CONTROL DATA: ANALYSIS OF EPA QUALITY CONTROL SAMPLE FOR VOLATILE ORGANICS IN WATER

Parameter	Concentration (μg/g) Reported	Concentration (μg/g) Expected	Percent recovery
Methylene chloride	23	18	128
1,1-dichloroethane	12	11	109
Trans-1,2-dichloroethylene	14	11	127
Chloroform	27	22	123
1,2-Dichloroethane	12	11	109
1,1,1-trichloroethane	11	10	110
Carbon tetrachloride	23	20	115
Trichloroethylene	25	20	125
Dibromochloromethane	13	12	108
Benzene	25	25	100
Tetrachloroethene	8.8	8.0	110
Toluene	70	74	95
Chlorobenzene	19	17	112
Ethylbenzene	62	66	94

TABLE 5-9. QUALITY CONTROL DATA: ANALYSIS OF AQUEOUS SAMPLE SPIKED WITH VOLATILE ORGANIC COMPOUNDS

Parameter	Concentration (μg/l) Reported	Concentration (μg/l) Expected	Concentration (μg/l) Percent recovery
1,1,1-trichloroethane	11	10	110
Trichloroethene	26	20	130
Tetrachloroethene	10	8.0	125

TABLE 5-10. QUALITY CONTROL DATA: RESULTS OF ANALYSIS OF VOST FIELD-BIASED BLANKS

		Quantity Detected (ng)				
Run no.	Tube ID	Freon 11	Trichloro-trifluoro-ethane	1,1,1-Trichloro-ethane	Trichloro-ethylene	Tetrachloro-ethylene
1	T	ND	ND	78	ND	27
	T/C	ND	ND	41	ND	17
2	T	ND	ND	53	ND	22
	T/C	ND	ND	41	ND	20
3	T	ND	ND	46	ND	54
	T/C	ND	ND	46	ND	19
Average quantity detected (ng)		ND	ND	51	ND	26

ND = <15 ng

T = Tenax

T/C = Tenax/charcoal

TABLE 5-11. QUALITY CONTROL DATA: SURROGATE RECOVERY-TENAX (VOST) SAMPLES

		Percent recovery (spiked at 200 ng)		
Run no.	Tube type	d_4-1,2-dichloroethane	d_6-benzene	d_8-toluene
1A	T	89	96	98
	T/C	110	110	130
1B	T	a	a	a
	T/C	140	130	150
1C	T	86	90	28[b]
	T/C	100	99	100
FBB (run 1)	T	85	81	49[b]
	T/C	120	100	130
2A	T	54[b]	55[b]	2.9[b]
	T/C	130	120	130
2B	T	66	83	43[b]
	T/C	12[b]	5.5[b]	2.0[b]
FBB (run 2)	T	92	110	130
	T/C	56[b]	66	74
3A	T	95	99	160
	T/C	110	110	130
3B	T	100	140	98
	T/C	110	90	81
FBB (run 3)	T	110	120	120
	T/C	150	110	140

[a] Sample data lost due to computer malfunction.

[b] Low recovery of surrogate compound indicates incomplete desorption of the tube and/or a problem with the addition of surrogate compounds to the sample.

T = Tenax tube

T/C = Tenax/charcoal tube

FBB = Field biased blank

TABLE 5-12. QUALITY CONTROL DATA: ANALYSIS OF SPIKED TENAX TUBE

Parameter	Concentration (ng) Reported	Concentration (ng) Expected	Percent Recovery
Trichloromonofluoromethane	660	800	83
1,1,1-trichloroethane	670	800	84
Trichloroethylene	760	800	95
Tetrachloroethylene	880	800	110

acquisition, but prior to data reduction or routine taping. The computer malfunction caused irreparable destruction of the disc directory for the area in which the VOST data were stored.

PERFORMANCE AND SYSTEM AUDITS AND CORRECTIVE ACTION

As stated in the QA Plan, EPA quality control concentrates and NBS Standard Reference Materials were used in assessing the quality of the analytical work. A system audit was not conducted by the QA Manager during this program nor were any external audits performed.

No formal corrective actions were initiated during this program. An informal corrective action was initiated by laboratory personnel in order to address the high levels of silver found in particulate field biased blanks as discussed earlier in this section.

SECTION 6

PROGRAM RESULTS

PROCESS RESULTS

The Union Chemical facility was briefly described in Section 2. Operating data collected during the test program are presented in Appendix B. Some of the key operating parameters are summarized in Table 6-1.

The average combustible waste feed rate was 4,610 g/min (610 lb/hr). Based on the measured heating value of 310,000 J/g (13,343 Btu/lb), the average heat input was 2.35 MW (8.1 x 10^6 Btu/hr). Ash content of the waste feed averaged 4.7 percent. Residues in the samples interfered with the measurement of viscosity. The contaminated water feed rate exceeded the waste feed rate by about 30 percent. Large amounts of dilution air were added after the quench tower causing the total stack gas flow to be much higher than the combustion air input rate. The carbon monoxide (CO) concentrations in the flue gas were measured by EPA Method 10 and were very low.

Temperatures in the combustion system averaged 1205°C (2201°F) in the freeboard or combustion zone above the bed and 1191°C (2146°F) at the inlet to the ash knockout chamber. After the ash knockout chamber, the flue gases pass through the "reactor" (no longer used for any reactions) where the temperature falls to 964°C (1767°F).

Details on the variations in temperature, combustion air flow, waste feed rates, and CO concentrations during each run are presented in Tables 6-2, 6-3, and 6-4. During each run, the above parameters were relatively constant. Also, each run was similar except for the higher CO concentrations during the second run.

PARTICULATE EMISSIONS

Results of the EPA Method 5 particulate emission tests are presented in Table 6-5. The particulate emission concentrations, corrected to 7 percent O_2 averaged 1,150 mg/dscm (0.50 gr/dscf). The mass emission rate averaged 41 g/min (5.4 lb/hr). The first run had the highest concentration, corrected to 7 percent O_2, in part due to the very high measured oxygen concentration of 17.6 percent. This high oxygen concentration is not consistent with the other two runs and the available data on combustion air flow, waste feed rate, and flue gas flow rate. If the same O_2 concentration measured in Runs 2 and 3 was assumed to apply to Run 1, then the particulate concentration for

TABLE 6-1. SUMMARY OF TEST RUN TIMES, FEED RATES, EXIT STREAM FLOWS, AND SELECTED PROCESS DATA

	Run 1	Run 2	Run 3	Average	Average (English units)
Date	11/3/83	11/3/83	11/4/83	-	
Time	9:34-12:00	14:24-16:41	10:41-13:11[a] 13:10-14:10[b]	-	
Feed rates					
Combustible waste, g/min	4350[c]	4780	4700	4610	610 lb/hr 8.1×10^6 Btu/hr[d]
Contaminated water, g/min	6370	6160	5670	6070	800 lb/hr
Combustion air, m^3/min	48	49	49	49	1750 ft^3/min
Stack gas flow, m^3/min[e]	108	116	116	113	4000 ft^3/min
Average CO concentration, ppm	2	11	ND[f]	4	
Temperatures, °C					
Bed	738	731	729	733	1350°F
Freeboard	1202	1185	1228	1205	2201°F
Inlet to ash knockout	1186	1198	1188	1191	2146°F
Reactor	928	1037	926	964	1767°F
Inlet to quench tower	707	801	707	738	1360°F

[a] VOST samples.

[b] Method 5 samples.

[c] 1.12 gal/min with a density of 8.56 lb/gal = 9.59 lb/min.

[d] Heating value was 13,343 Btu/lb.

[e] Dry standard cubic meters per minute.

[f] ND = not detected.

TABLE 6-2. SELECTED PROCESS DATA FOR TEST RUN NUMBER 1

Time	T_1 Bed (°F)	T_2 Freeboard (°F)	T_3 Inlet to ash knockout (°F)	T_4 Reactor (°F)	T_5 Inlet to quench tower (°F)	Combustion air flow (ft^3/min)	Combustible waste feed flow[a] (gal/min)	Contaminated water flow[a] (percent of full scale)	CO concentration[b] (ppm)
9:30	1356	2194	2146	1639	1259	1700	1.80	48	2
9:45	1361	2209	2176	1653	1268	1700	1.87	50	2
10:00	1371	2144	2182	1653	1251	1700	1.79	40	ND
10:15	1353	2166	2184	1684	1277	1700	1.75	48	2
10:30	1359	2143	2153	1690	1293	1700	1.79	48	5
10:45	1358	2148	2163	1708	1306	1700	1.81	48	ND
11:00	1363	2142	2137	1711	1318	1700	1.82	48	3
11:15	1362	2143	2175	1735	1331	1700	1.83	48	1
11:30	1360	2149	2156	1739	1341	1700	1.80	48	2
11:45	1359	2176	2178	1755	1348	1700	1.87	48	ND
12:00	1365	2139	2172	1771	1362	1700	1.82	48	2
Average, °F	1361	2196	2166	1703	1305	-	-	-	-
°C	738	1202	1186	928	707	-	-	-	-

[a] Meter readings indicating variations in real time flow. Average flows for each test were calculated from measured changes in storage tank levels.

[b] CO readings from a continuous monitor operated according to EPA Method 10 were recorded every 5 minutes. The reported value is a 15-minute average ending at the indicated time.

TABLE 6-3. SELECTED PROCESS DATA FOR TEST RUN NUMBER 2

	T_1	T_2	T_3	T_4	T_5				
Time	Bed (°F)	Freeboard (°F)	Inlet to ash knockout (°F)	Reactor (°F)	Inlet to quench tower (°F)	Combustion air flow (ft^3/min)	Combustible waste feed flow[a] (gal/min)	Contaminated water flow[a] (percent of full scale)	CO concentration[b] (ppm)
14:20	1376	2128	2193	1878	1458	1725	1.99	54	-
14:35	1387	2151	2224	1889	1463	1725	1.94	54	-
14:50	1379	2186	2224	1901	1472	1750	1.93	54	-
15:05	1381	2197	2208	1902	1476	1750	1.97	54	11
15:20	1333	2207	2191	1909	1482	1750	1.83	42	11
15:35	1334	2208	2154	1887	1472	1750	1.88	54	8
15:50	1311	2173	2179	1898	1471	1750	1.98	54	5
16:05	1304	2109	2192	1909	1477	1750	1.95	54	19
16:20	1332	2141	2197	1916	1486	1750	1.92	54	9
16:35	1332	2149	2132	1887	1479	1750	1.89	54	17
Average, °F	1347	2165	2189	1898	1474	-	-	-	-
°C	731	1185	1198	1037	801	-	-	-	-

[a] Meter readings indicating variations in real time flow. Average flows for each test were calculated from measured changes in storage tank levels.

[b] CO readings from a continuous monitor operated according to EPA Method 10 were recorded every 5 minutes. The reported value is a 15-minute average ending at the indicated time.

TABLE 6-4. SELECTED PROCESS DATA FOR TEST RUN NUMBER 3

	T_1	T_2	T_3	T_4	T_5				
Time	Bed (°F)	Freeboard (°F)	Inlet to ash knockout (°F)	Reactor (°F)	Inlet to quench tower (°F)	Combustion air flow (ft^3/min)	Combustible waste feed flow[a] (gal/min)	Contaminated water flow[a] (percent of full scale)	CO concentration[b] (ppm)
10:40	1336	2246	2273	1689	1282	1725	2.00	64	ND
10:55	1336	2236	2192	1673	1285	1725	1.94	64	ND
11:10	1332	2224	2189	1680	1290	1750	1.91	65	ND
11:25	1333	2233	2206	1694	1300	1750	1.89	65	-
11:40	1351	2234	2144	1690	1308	1750	1.94	65	-
11:55	1350	2241	2147	1693	1307	1750	1.89	65	ND
12:10	1354	2245	2155	1702	1313	1750	1.81	65	ND
12:25	1351	2267	2182	1717	1307	1750	1.91	65	ND
12:40	1352	2243	2138	1720	1316	1750	1.72	65	ND
12:55	1351	2258	2148	1725	1317	1750	1.85	65	ND
13:10	1340	2239	2105	1716	1325	1750	1.80	65	1
Average, °F	1344	2242	2171	1699	1305	-	-	-	-
°C	729	1228	1188	926	707	-	-	-	-

[a] Meter readings indicating variations in real time flow. Average flows for each test were calculated from measured changes in storage tank levels.

[b] CO readings from a continuous monitor operated according to EPA Method 10 were recorded every 5 minutes. The reported value is a 15-minute average ending at the indicated time.

TABLE 6-5. RESULTS OF PARTICULATE SAMPLING PROGRAM

	Run number		
	1	2	3
Date	11/3/83	11/3/83	11/4/83
Time	9:34-12:00	14:24-16:41	13:05-14:15
Nozzle diameter, in.	0.300	0.300	0.300
Barometric pressure, in. Hg	29.78	29.78	29.72
Time of test, min	120	120	60
Sampled gas volume, ft^3	74.356	82.841	38.590
Average gas meter temperature, °F	83	86	68
Average ΔP, in. H_2O	0.27	0.32	0.31
Average orifice pressure drop, in. H_2O	1.26	1.6	1.47
Standard gas volume, dscf	72.058	79.823	38.403
Volume of water collected, ml	642.8	727.6	328.2
Moisture content, %	30	30	29
CO_2 content, %	3.2	4.5	4.4
O_2 content, %	17.6	15.4	15.2
CO content, ppm	2	11	ND
Average stack temperature, °F	151	158	154
Pitot tube coefficient	0.84	0.84	0.84
Stack gas velocity, afpm	2000	2200	2100
Stack area, sq. in.	452	452	452
Volumetric flow rate, dscfm	3800	4100	4100
Isokinetic ratio, %	101	104	100
Particulate collected, mg	941.74	682.63	366.94
Particulate emissions, gr/dscf	0.201	0.132	0.147
Particulate emissions corrected to 7% O_2, gr/dscf	0.829	0.329	0.355

Run 1 would be 1,150 mg/dscm (0.50 gr/dscf) instead of 1,910 mg/dscm (0.83 gr/dscf), and the three-run average would be reduced to 920 mg/dscm (0.40 gr/dscf).

The particulate collected by the Method 5 probe and by the Method 5 filter were analyzed for chloride as an indication of liquid carryover from the scrubber. The results for Runs 1, 2, and 3, were 14.6 percent, 34.0 percent, and 16.6 percent, respectively. Assuming the chloride was present as $CaCl_2$, this compound would represent 23, 53, and 26 percent of the particulate catch for Runs 1, 2, and 3, respectively. These chloride results indicate a significant amount of liquid carryover from the scrubber. The liquid carryover also probably contains other elements contributing to the total particulate emissions.

The particulate emissions were also analyzed for selected metals. The results are shown in Table 6-6. Iron and lead were present at much higher concentrations than the other elements. The measured level of silver was much higher in the field blank than in the samples. These data indicate a possible sampling and/or analytical problem despite the rarity of silver as a laboratory or field contaminant. Presented barium data should also be considered as maximum values due to the relative contribution measured in the field blank (12 to 21 percent).

The combustible waste feed was also analyzed as the expected source of the emissions of metals. The results are presented in Table 6-7. Iron was present in the feed at an average concentration of 2.08 percent.

The ratio between the emissions of each element and the feed rate from the combustible waste is also presented in Table 6-7. These numbers should be used with some caution because relatively small deviations can cause significantly different results. For example, if the cadmium waste feed rate were 10 percent higher than indicated and the emissions were 10 percent lower, then the ratio would be 0.96 instead of 1.3. Further indication of the problem with measuring silver emissions is evident from the very high ratio between the emissions and the feed. It appears that high percentages of the arsenic, cadmium, and lead in the feed are emitted. Low percentages of barium, beryllium, chromium, iron, and mercury are emitted.

HYDROGEN CHLORIDE (HCl) EMISSIONS

Emissions of HCl are controlled by a quench tower and a cross-flow scrubber. The alkalinity of the scrubbing solution in each tower is controlled by the addition of lime slurry. During the three test runs, Union Chemical monitored the pH; data are presented in Table 6-8.

The results of the HCl sampling and analysis program are presented in Table 6-9. The combustible waste feed contained an average of 13.8 percent total chlorine as chloride. At the observed feed rate, potential HCl emissions averaged 655 g/min or 86.6 lb/hr. The quench tower and scrubber were very

TABLE 6-6. CONCENTRATIONS OF METALS IN PARTICULATE EMISSIONS[a]

	Run 1		Run 2		Run 3		Average	
	μg/g[b]	μg/m³[c]	μg/g	μg/m³	μg/g	μg/m³	μg/m³	mg/min[d]
Arsenic	764	351	1,320	399	932	314	355	40.1
Barium	1,094	503	1,480	447	1,605	541	497	56.2
Beryllium	23.7	10.9	39.4	11.9	36.0	12.1	11.6	1.31
Cadmium	424	195	753	227	616	208	210	23.7
Chromium	509	234	1,160	350	962	324	303	34.2
Iron	99,400	45,700	158,000	47,700	146,000	49,200	47,500	5,370
Lead	46,200	21,300	77,400	23,400	51,800	17,500	20,700	2,340
Mercury	0.35	0.16	0.75	0.23	0.65	0.22	0.20	0.023
Selenium	<2	<0.92	<2	<0.60	<2	<0.67	<0.73	<0.082
Silver	11.0[e]	5.1[e]	29.6[e]	8.9[e]	24.5[e]	8.3[e]	7.4[e]	0.840[e]

[a] Results have been corrected for laboratory method blanks but not for field blanks. With the exception of barium and silver, the field blank values were insignificant compared to the sample values.

[b] μg per g of particulate.

[c] μg per dry standard m³ of flue gas.

[d] Derived from the flue gas flow rate in Table 6-1 and the metal concentrations in this table.

[e] The field blank exceeded the sample by a factor of 2.5 to 6. Thus, there may not be any measurable silver in the emissions.

TABLE 6-7. CONCENTRATIONS OF METALS IN COMBUSTIBLE WASTE FEED AND COMPARISON OF INPUT RATES TO EMISSION RATES

	Average concentration in combustible waste (μg/g)	Average feed rate[a] (mg/min)	Average emission rate[b] (mg/min)	Ratio of emission to input from combustible waste
Arsenic	19.3	88.9	40.1	0.45
Barium	121	558	56.2	0.10
Beryllium	4.67	21.5	1.31	0.061
Cadmium	4.06	18.7	23.7	1.3
Chromium	166	765	34.2	0.045
Iron	20,800	95,900	5,370	0.056
Lead	458	2,110	2,340	1.1
Mercury	0.52	2.40	0.023	0.0096
Selenium	<0.5	<2.3	<0.82	-
Silver	<0.02	<0.092	0.840[c]	>9.1[c]

[a] Based on the waste feed rates reported in Table 6-1 and the average metal concentrations reported in this table.

[b] From Table 6-6.

[c] Since the measured silver in the field blank exceeded that in the sample by a factor of 2.5 to 6, the reported silver emissions are questionable.

TABLE 6-8. QUENCH TOWER AND SCRUBBER pH DATA

	Time	Quench tower pH	Scrubber pH
Run 1	9:30	6.37	6.50
	9:45	8.10	8.20
	10:00	7.87	7.65
	10:15	7.52	7.07
	10:30	7.80	6.53
	10:45	6.26	7.13
	11:00	7.39	7.34
	11:15	7.03	7.20
	11:30	7.60	7.58
	11:45	8.15	7.47
	12:00	7.33	7.64
	Average	7.40	7.30
Run 2	14:20	7.51	7.60
	14:35	7.40	7.74
	14:50	5.72	5.89
	15:05	7.25	7.08
	15:20	8.00	6.32
	15:35	7.34	7.67
	15:50	7.58	8.13
	16:05	5.60	6.73
	16:20	5.69	6.59
	16:35	6.72	5.82
	16:42	2.95	6.53
	Average	6.52	6.92
Run 3	13:10	7.82	8.31
	13:25	7.55	7.62
	13:40	6.82	6.45
	13:55	7.67	7.57
	14:10	8.12	6.74
	14:24	7.71	6.76
	Average	7.62	7.24

TABLE 6-9. RESULTS OF HCl SAMPLING AND ANALYSIS

	Run 1	Run 2	Run 3	Average
Feed				
Combustible waste, g/min	4,350	4,780	4,700	4,610
Total chloride concentration, %	13.6	13.8	14.0	13.8
Total chloride input, g/min	592	660	660	637
Potential HCl emissions, g/min	609	679	679	655
Emission measurements				
Volume sampled, m^3	2.04	2.26	1.09	-
Total chloride measured, mg	55.9	104.2	26.7	-
HCl concentration, mg/m^3	28.2	47.4	25.2	33.6
Flue gas flow, m^3/min	108	116	116	113
HCl emissions, g/min	3.04	5.50	2.92	3.82
lb/hr	0.40	0.73	0.39	0.51
HCl collection efficiency, %	99.50	99.19	99.57	99.42
HCl concentration, ppm[a]	17.3	29.1	15.5	-
O_2 concentration, %	17.6	15.4	15.2	-
HCl concentration corrected to 7% O_2, ppm	71.3[b]	72.5	37.4	60[b]
Average quench tower pH	7.40	6.52	7.62	7.18
Average scrubber pH	7.30	6.92	7.24	7.15

[a] ppm by volume based on dry flue gas.

[b] If the O_2 concentration for Run 1 is assumed to be 15.3 percent, the corrected HCl concentration would be 44.0 ppm and the average concentration would be 51 ppm.

effective in reducing HCl emissions as indicated by the average efficiency of 99.42 percent. The mass emission rate was 3.82 g/min (0.51 lb/hr). The emission concentrations, on a dry basis, when corrected to 7 percent O_2, averaged 60 ppm (volume/volume). As has been previously suggested, there are some indications that the measured flue gas O_2 concentration (17.6 percent) in the first test run may have been high. If the O_2 concentration was actually 15.3 percent (the average of runs 2 and 3), then the average HCl concentration would be 51 ppm.

CARBON MONOXIDE

During the test runs, carbon monoxide concentrations in the stack gas were continuously monitored in accordance with EPA Method 10. The concentrations were too low to be recorded on available strip charts. Therefore, the monitor signal output was monitored with a digital voltmeter at 5-minute intervals. These voltage readings were converted to concentrations based on the calibration curve. The results are presented in Table 6-10.

The average CO concentrations were 2, 11, and not detectable (probably <1 ppm) for Runs 1, 2, and 3, respectively. Numerous readings below the detection limit were recorded during Runs 1 and 3. The CO concentrations were higher during Run 2 for unknown reasons. Two readings of 30 ppm were recorded during Run 2.

DESTRUCTION OF ORGANIC COMPOUNDS

One of the primary objectives of the program was to measure the destruction/removal efficiency for difficult to incinerate chlorinated volatile organic compounds. In order to obtain an immediate indication of incinerator performance, flue gas samples were collected with an integrated gas sampling train and immediately analyzed near the test site with a gas chromatograph equipped with an electron capture detector. The primary method used to identify and quantitate flue gas emissions consisted of sample collection with a volatile organic sampling train and analysis by thermal desorption gas chromatography/mass spectrometry.

Waste Feed Analysis

The combustible waste feed and the contaminated water feed were analyzed by gas chromatography/mass spectrometry (GC/MS) for five chlorinated organic compounds. The amounts of organic compounds in the contaminated water were not significant when compared to the combustible waste. Average concentrations in the combustible waste were 0.29 percent 1,1,2-trichloro-1,2,2-trifluoroethane, 1.5 percent trichloromonofluoromethane, 3.9 percent tetrachloroethene, 0.76 percent trichloroethene, and 5.1 percent 1,1,1-trichloroethane as shown in Table 6-11. Also shown in Table 6-11 are the feed rates for each compound that are used in later calculations of destruction/removal efficiencies.

TABLE 6-10. CARBON MONOXIDE MONITORING DATA

Run 1		Run 2		Run 3	
Time	CO concentration, dry basis (ppm)	Time	CO concentration, dry basis (ppm)	Time	CO concentration, dry basis (ppm)
9:30	ND	15:00	5.4	10:00	ND
9:35	ND	15:05	16.5	10:05	ND
9:40	5.4	15:10	5.4	10:10	ND
9:45	ND	15:15	10.9	10:15	ND
9:50	ND	15:20	16.5	10:20	ND
9:55	ND	15:25	5.4	10:25	ND
10:00	ND	15:30	10.9	10:30	ND
10:05	ND	15:35	8.2	10:35	ND
10:10	5.4	15:40	5.4	10:40	ND
10:15	ND	15:45	10.9	10:45	ND
10:20	5.4	15:50	ND	10:50	ND
10:25	8.2	15:55	30.3	10:55	ND
10:30	2.6	16:00	13.7	11:00	ND
10:35	ND	16:05	13.7	11:05	ND
10:40	ND	16:10	8.2	[a]	-
10:45	ND	16:15	8.2	11:55	ND
10:50	2.6	16:20	10.9	12:00	ND
10:55	ND	16:25	30.3	12:05	ND
11:00	5.4	16:30	2.6	12:10	ND
11:05	ND	16:35	19.2	12:15	ND
11:10	ND	16:40	5.4	12:20	ND
11:15	2.6	16:45	10.9	12:25	ND
11:20	2.6			12:30	ND
11:25	2.6			12:35	ND
11:30	ND			12:40	ND
11:35	ND			12:45	ND
11:40	ND			12:50	ND
11:45	ND			12:55	ND
11:50	2.6			13:00	[b]
11:55	2.6			13:05	ND
12:00	ND			13:10	2.6
12:05	ND			13:15	ND
12:10	ND				
12:15	5.4				

[a] Readings not recorded between 11:05 and 11:55 due to condenser malfunction.

[b] Missed reading.

TABLE 6-11. ANALYTICAL RESULTS FOR POHCs IN COMBUSTIBLE WASTE FEED AND CONTAMINATED WATER

Compound	Run 1		Run 2		Run 3		Average	
	Concentration (μg/g)	Feed rate[a] (g/min)	Concentration (μg/g)	Feed rate[a] (g/min)	Concentration (μg/g)	Feed rate[a] (g/min)	Concentration (μg/g)	Feed rate (g/min)
Combustible waste feed								
1,1,2-Trichloro-1,2,2-trifluoroethane	3,000	13.1	2,900	13.9	2,800	13.2	2,900	13.4
Trichloro-monofluoromethane	15,000	65.3	12,000	57.3	19,000	89.3	15,000	70.7
Tetrachloroethene	43,000	187	38,000	182	36,000	169	39,000	179
Trichloroethene	7,200	31.3	6,800	32.5	8,900	41.8	7,600	35.2
1,1,1-Trichloroethane	53,000	231	50,000	239	49,000	230	51,000	233
Contaminated water feed								
1,1,2-trichloro-1,2,2-trifluoroethane	0.18	0.001	0.25	0.002	0.33	0.002	0.25	0.002
Trichloro-monofluoromethane	0.084	0.0005	0.075	0.0005	0.096	0.0005	0.085	0.0005
Tetrachloroethene	4.2	0.03	4.7	0.03	4.6	0.03	4.5	0.03
Trichloroethene	4.4	0.03	4.8	0.03	4.6	0.03	4.6	0.03
1,1,1-Trichloroethane	15	0.1	18	0.1	17	0.1	17	0.1
Total feed								
1,1,2-Trichloro-1,2,2-trifluoroethane	-	13.1	-	13.9	-	13.2	-	13.4
Trichloro-monofluoroethane	-	65.3	-	57.4	-	89.3	-	70.7
Tetrachloroethene	-	187	-	182	-	169	-	179
Trichloroethene	-	31.3	-	32.5	-	41.8	-	35.2
1,1,1-Trichloroethane	-	231	-	239	-	230	-	233

[a] Based on the waste feed rates reported in Table 6-1 and the concentrations reported in the table.

Volatile Organic Sampling Train (VOST) and Gas Chromatography/Mass Spectrometry Analysis

Each of the three runs included two or three sets of Tenax and Tenax/charcoal cartridges and two impingers. The cartridges were each analyzed separately by thermal desorption GC/MS, and the impingers were analyzed by purge and trap GC/MS. VOST blank corrections were based on field-biased blanks. Detailed analytical data are presented in an Appendix E.

The VOST results are summarized in Table 6-12. One compound (1,1,2-trichloro-1,2,2-trifluoroethane) was not detected in any of the VOST samples and is therefore reported as below the detection limit of 10 μg/m^3. A concentration of 10 μg/m^3 of 1,1,2-trichloro-1,2,2-trifluoroethane is equivalent to 1.2 ppb (volume per volume basis). Concentrations of the other compounds were in the 10 to 135 μg/m^3 range.

Destruction/removal efficiencies for each run and the average of the three runs are reported in Table 6-13. The compounds are listed in the order of their incinerability as published by EPA for Appendix VIII constituents with the exception that the first compound is not an Appendix VIII pollutant. The destruction/removal efficiency for each compound exceeded 99.99 percent, and for one compound the destruction/removal efficiency exceeded 99.999 percent.

Integrated Gas Sampling Train With Analysis by Gas Chromatography/Electron Capture Detection

The GC/ECD analyses were conducted in the field to obtain an immediate indication of incinerator performance. The GC/ECD analyses were not intended to be the primary quantitative measurement of emissions. The concentrations measured by this method are compared to the VOST GC/MS results in Table 6-14. In general, the GC/ECD results show reasonable agreement with the GC/MS results. It should be noted that the GC/ECD results were not blank corrected because the blanks were below the instrument calibration range and thus difficult to quantify.

Destruction/removal efficiencies based on the GC/ECD analyses are shown in Table 6-15. One compound appears to be below 99.99 percent, while the others are above 99.99 percent. The definitive program results should be based on the VOST GC/MS analyses conducted in the laboratory, not on the Tedlar bag GC/ECD analyses conducted in the field.

Products of Incomplete Combustion

The program plan called for analysis of the VOST samples for products of incomplete combustion (PICs). It was agreed that these would be defined as identifiable compounds exceeding 100 μg/m^3. No data are available on products of incomplete combustion due to a computer failure. A detailed discussion of the problems encountered is provided in Section 5.

TABLE 6-12. AVERAGE EMISSIONS OF ORGANIC CONSTITUENTS BASED ON GC/MS ANALYSIS OF VOST SAMPLES

Compound	Run 1		Run 2		Run 3		Average emissions (g/min)
	Concentration ($\mu g/m^3$)	Emissions[a] (g/min)	Concentration ($\mu g/m^3$)	Emissions[a] (g/min)	Concentration ($\mu g/m^3$)	Emissions[a] (g/min)	
1,1,2-Trichloro-1,2,2-trifluoroethane	<10	<0.00108	<10[b]	<0.00116[b]	<10	<0.00116	<0.00113
Trichloro-monofluoromethane	57	0.00616	29[b]	0.00336[b]	59	0.00684	0.00239
Tetrachloroethene	42	0.00453	13-18[b,c]	0.00164[b]	135	0.0157	0.00115
Trichloroethene	<10	<0.00108	<10[b]	<0.00116[b]	23	0.00267	0.00086-0.0016[c]
1,1,1-Trichloroethane	<10	<0.00108	5.5 - 11[b,c]	0.00096[b]	10 - 15[c]	0.00145	0.00098

[a] Based on the indicated concentrations and the flue gas flow rates reported in Table 6-1.

[b] Low recovery of surrogate compounds indicates incomplete desorption of the tube and/or a problem with the addition of surrogate compounds to the sample.

[c] The range is derived by averaging the two or three results which included one or more below the detection limit.

TABLE 6-13. DESTRUCTION/REMOVAL EFFICIENCY (DRE) RESULTS BASED ON GC/MS ANALYSIS OF VOST SAMPLES

Compound	Run 1			Run 2			Run 3			Average DRE (percent)
	Input (g/min)	Emissions (g/min)	DRE (percent)	Input (g/min)	Emissions (g/min)	DRE (percent)	Input (g/min)	Emissions (g/min)	DRE (percent)	
1,1,2-Trichloro-1,2,2-trifluoroethane	13.1	<0.00108	>99.9918	13.9	<0.00116[a]	>99.9917	13.2	<0.00116	>99.9912	>99.9915
Trichloro-monofluoromethane	65.3	0.00616	99.9906	57.4	0.00336[a]	99.9941	89.3	0.00684	99.9923	99.9916
Tetrachloroethene	187	0.00453[a]	99.9976	182	0.00164[a]	99.99910	169	0.0157	99.9907	99.9958
Trichloroethene	31.3	<0.00108	>99.9965	32.5	<0.00116[a]	>99.9964	41.8	0.00267	99.9936	99.9955
1,1,1-Trichloroethane	231	<0.00108	>99.99953	239	0.00096[a]	99.99960	230	0.00145	99.99937	99.99949

[a] Low recovery of surrogate compounds indicates incomplete desorption of the tube and/or a problem with the addition of surrogate compounds to the sample.

TABLE 6-14. COMPARISON OF EMISSION CONCENTRATIONS MEASURED BY TEDLAR BAGS--GC/ECD AND VOLATILE ORGANIC SAMPLING TRAIN--GC/MS ($\mu g/m^3$)

Compound	Run 1		Run 2		Run 3		Average	
	GC/ECD	GC/MS	GC/ECD	GC/MS	GC/ECD	GC/MS	GC/ECD	GC/MS
1,1,2-Trichloro-1,2,2-trifluoroethane	22	< 10	13	< 10	14	< 10	16	< 10
Trichloro-monofluoromethane	55	57	43	29	60	59	53	48
Tetrachloroethene	42	68	58	13 - 18[a]	255	135	127	64
Trichloroethene	18	< 10	16	< 10	< 15	23	11 - 16[a]	8 - 14[a]
1,1,1-Trichloroethane	33	< 10	29	5.5 - 11[a]	70	10 - 15[a]	44	10

[a] The range is derived by averaging two or three results which included one or more below the detection limit.

TABLE 6-15. DESTRUCTION/REMOVAL EFFICIENCY (DRE) RESULTS BASED ON GC/ECD ANALYSIS OF TEDLAR BAG SAMPLES[a]

	Run 1			Run 2			Run 3			
Compound	Input (g/min)	Emissions (g/min)	DRE (percent)	Input (g/min)	Emissions (g/min)	DRE (percent)	Input (g/min)	Emissions (g/min)	DRE (percent)	Average DRE (percent)
1,1,2-Trichloro-1,2,2-trifluoroethane	13.1	0.00237	99.982	13.9	0.00151	99.989	13.2	0.00162	99.988	99.986
Trichloro-monofluoromethane	65.3	0.00591	99.9909	57.4	0.00498	99.9913	89.3	0.00696	99.9922	99.9915
Tetrachloroethene	187	0.00720	99.9961	182	0.00672	99.9963	169	0.0296	99.982	99.9915
Trichloroethene	31.3	0.00194	99.9938	32.5	0.00185	99.9943	41.8	< 0.00174	> 99.9958	99.9946
1,1,1-Trichloroethane	231	0.00355	99.9985	239	0.00336	99.9986	230	0.00812	99.9965	99.9979

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[a] Field analyses conducted to provide an immediate indication of emissions. Conclusions regarding destruction/removal efficiency should be based on GC/MS results in Tables 6-12 and 6-13.

However, we believe there would have been no PICs at the observed destruction efficiencies. The above conclusion is based on tests, for EPA, of six small boilers that were burning waste oil. The waste oil was spiked with chloroform, 1,1,1-trichloroethane, trichloroethene, and tetrachloroethene at concentrations of 0.2 to 0.5 percent. The destruction/removal efficiency was typically only 99.8 percent compared to greater than 99.99 percent at Union Chemical. Emission concentrations averaged about 100 $\mu g/m^3$ compared to about 50 $\mu g/m^3$ at Union Chemical. Under these circumstances, there were no PICs present at concentrations above 100 $\mu g/m^3$. Because the destruction removal efficiencies were much higher and the emission concentrations were lower at Union Chemical, it is unlikely that any PICs were emitted.

OTHER ANALYSES

RCRA EP Toxicity

The ash collected in the cyclone and the scrubber sludge were extracted and analyzed in accordance with RCRA EP toxicity procedures. The results are presented in Table 6-16. The concentrations of all the trace metals were far below EPA hazardous waste criteria.

Scrubber Water and Scrubber Water Supply

Three samples of the scrubber water and scrubber water supply were analyzed for 31 volatile organic priority pollutants. The only compound detected in the scrubber water was methylene chloride, a common laboratory contaminant, at concentrations of 8.7, 13, and 8.5 $\mu g/l$. The detection limits for acrolein and acrylonitrile were 40 $\mu g/l$, and for the other compounds the detection limits were 1 $\mu g/l$.

The scrubber water supply also contained methylene chloride at similar concentrations (10, 9.7, and 7.6 $\mu g/l$). Low concentrations (<10 $\mu g/l$) of 1,1,1-trichloroethane, trichloroethene, tetrachloroethene, toluene, and ethylbenzene were detected in one or more of the scrubber water supply samples.

Principal Organic Hazardous Constituents in Ash and Sludge Samples

Three samples of the ash from the cyclones were analyzed for the five test compounds. None of these compounds were detected at a detection limit of 10 $\mu g/g$. The scrubber sludge sample was not analyzed because the sample contained a significant amount of head space. Because the scrubber water did not contain any of the five test compounds (detection limit 1 $\mu g/l$ on 1 ppb), it is very unlikely that any of the compounds would have been detected in the sludge.

TABLE 6-16. RCRA EP TOXICITY ANALYSES

	Concentration in Aqueous Leachate		
	Ash (μg/l)	Scrubber sludge (μg/l)	Hazardous waste criteria (μg/l)
Arsenic	<30	< 30	5,000
Barium	72	305	100,000
Beryllium	< 1	< 1	Not established
Cadmium	12	71	1,000
Chromium(VI)	< 3	< 4	5,000
Iron	648	< 4	Not established
Lead	< 20	456	5,000
Mercury	< 0.5	< 0.5	200*
Selenium	< 20	<20	1,000
Silver	< 1	<1	5,000

APPENDIX A

QUALITY ASSURANCE PROJECT PLAN

The enclosed Quality Assurance Project Plan was prepared in August 1983. An amendment, also included in this Appendix was prepared in September 1983. Prior to the field program in November 1983, the analytical program was modified to include trace metal analyses of the Method 5 filters. In addition, it was decided to use EPA Method 10 to measure CO. These and other changes are reviewed in Section 1-6 of this report. Subsequent to the field program, it was decided to analyze the filters for chloride as a possible contribution to the high particulate loading.

Union Chemical P.O. Number 5412

GCA Project Number 7-468-001

Amendment

Quality Assurance Project Plan
Union Chemical Trial Burn
Sampling and Analysis

September 1983

The Quality Assurance Project Plan is amended to provide for the analysis of flue gas samples for volatile products of incomplete combustion and the analysis of waste feed samples for specific metals of interest. Additions to the text of the QAPP are as follows:

QAPP Section 7.2.4 Volatile Products of Incomplete Combustion

(Additional Section)

The collected VOST samples will also be analyzed for volatile products of incomplete combustion (PICs). Mass spectral data will be obtained for up to 10 peaks noted on the total ion chromatogram at levels greater than 100 µg/m^3. The concentration levels for these peaks will be obtained by comparison of peak areas to that of the closest eluting internal standard. Where necessary, background correction of the mass spectrium will be achieved 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; positive identification will be made when the following criteria are met:

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

QAPP Section 1.3.7 Flue Gas

(Existing Section)

Include: The collected VOST samples will also provide for the analysis of up to 10 products of incomplete combustion (PIC) by GC/MS techniques. Data will be reported for those confirmed components exhibiting concentrations greater than 100 µg/m^3.

Table 1-3 Summary of Flue Gas Sampling and Analysis

(Existing Table)

	Test Parameter	Sampling Method	Analysis Method
Include:	PIC	Volatile organic train	Thermal desorption-GC/MS

QAPP Section 7.5 Combustible Waste Feed-Metals Analyses

(Additional Section)

The combustible waste feed samples will be analyzed for a number of metals including arsenic, barium, beryllium, cadmium, chromium, lead, mercury, selenium and silver. All of these metals with the exception of mercury will be prepared for analysis by means of controlled dry ashing. This technique basically involves the placing of sample aliquota (2-5g) in platinum crucibles beneath an adjustable array of IR lamps. The lamps are gradually lowered to achieve temperature ramping thereby precluding the actual combustion of the oil and resultant loss of volatile metals. Samples are heated via the IR lamps until they resemble tar. They are then transferred to a cold muffle furnace where the temperature is gradually elevated to 600°C. Previous analysis of fuel samples using this procedure indicate that the volatile elements, such as arsenic, are not lost during the ashing procedure. This temperature is maintained until the samples are at constant weight. The resultant ash is dissolved by means of hot 1:1 nitric/hydrochloric acid. Metals concentrations are subsequently determined by means of Inductively Coupled Argon Plasma Emission Spectroscopy (ICAP).

The analysis of mercury in the waste feed samples presents a problem in that the mercury may be present in volatile organometallic compounds. Consequently, a digestion procedure designed to recover the total mercury is required. We propose using ASTM Method D-3684-78 "Total Mercury in Coal by the Oxygen Bomb Combustion/Atomic Absorption Method." This procedure combusts the sample in an oxygen rich atmosphere with the mercury vapors collected in a dilute nitric acid solution. The acid solutions and the solution used to rinse the bowl are combined and subsequently analyzed by cold vpor atomic absorption.

QAPP Section 1.3.1 Combustible Waste Feed

(Existing Section)

Include: The samples will also be analyzed for a number of metals including arsenic, barium, beryllium, cadmium, chromium, lead, mercury, selenium and silver by either Inductively Coupled Argon Plasma Emission Spectroscopy (ICAP) or cold vapor atomic absorption techniques.

Table 1-2 Summary of Sampling and Analysis Plans for Liquid and Solid Streams

Sample type	Number of samples collected	Number of samples analyzed	Analysis	Method
Include:				
Combustible Waste feed	36 VOA vials	3 composites	Metals	ICAP/AA

Union Chemical P.O. Number 5412

GCA Project Number 7-468-001

QUALITY ASSURANCE PROJECT PLAN

UNION CHEMICAL TRIAL BURN
SAMPLING AND ANALYSIS

August 1983

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CONTENTS

CONTENTS (continued)

Appendices

FIGURES

TABLES

1.0 PROJECT DESCRIPTION

1.1 OVERVIEW

Union Chemical Company, Inc. operates a fluidized bed hazardous waste incinerator in South Hope, Maine. Permit applications have been submitted to the Maine Department of Environmental Protection and the U.S. Environmental Protection Agency's Region I Office. The purpose of this document is to complete the required submittals by addressing the sampling and analysis portions of the required trial burn plan. The overall objective of this project is to conduct a trial burn to demonstrate compliance with the hazardous waste incinerator performance standards. The results of the this program will include:

- A quantitative analysis of the trial principal organic hazardous compounds (POHCs) in the waste feed to the incinerator.
- A quantitative analysis of the exhaust gas for the concentration and mass emissions of the trial POHCs and hydrogen chloride (HCl) and the concentration of oxygen (O_2).
- If the HCl emission rate exceeds 1.8 kilograms of HCl per hour (4 lb per hour), a computation of HCl removal efficiency will also be performed.
- A quantitative analysis of the scrubber water, scrubber sludge and ash residues for the purpose of estimating the fate of the trial POHCs.
- A computation of destruction and removal efficiency (DRE).
- A computation of particulate emissions.
- A continuous measurement of carbon monoxide (CO) in the exhaust gas.

Required process data will be supplied by Union Chemical. In addition, Union Chemical will test the emergency shutoff systems immediately prior to or after the sampling program.

Three replicate test runs over a 2-day period are planned. Two methods will be used to address the most important question; the concentration of POHCs in the incineration flue gas. A portable gas chromatograph equipped with an electron capture detector will be set up in a clean area near the site and used to measure POHCs in flue gas samples collected in Tedlar bags. Flue gas samples will also be obtained with a volatile organic sampling train (VOST). The Tenax and Tenax/charcoal cartridges from the VOST will be returned to GCA's laboratory in Bedford, MA for analysis of POHCs by thermal desorption-gas chromatography/mass spectrometry.

The POHCs that have been selected for analysis are tetrachloromethane, tetrachloroethene, trichloroethene, and 1,1,1-trichloroethane. In addition, although 1,1,2-trichloro-1,2,2-trifluorethane is not on EPA's Appendix VIII hazardous constituent list, it will be measured as an additional compound. These five compounds have been selected for analysis because they will be present in the waste feed at concentrations in the 2 to 5 percent range and because they rank very high on EPA's hierarchy of waste incinerability. EPA's hierarchy of waste incinerability, which is based on heat of combustion, includes 271 organic compounds with the number one ranked compound being the most difficult to incinerate. Tetrachloromethane is ranked second, tetrachloroethene is eleventh, trichloroethene is twenty-second and 1,1,1-trichloroethane is twenty-fifth. Demonstration of the incinerator's ability to destroy these difficult to incinerate compounds should be adequate to conclude that it can destroy other compounds on EPA's hierarchy with the exception of the top-ranked compound.

Additional discussions of the incineration facility, sampling, and analysis are presented in this section in order to completely describe the project. Details of the project organization, sampling methods, analytical methods and the associated quality control procedures are presented in Sections 2.0 through 14.0.

1.2 FACILITY DESCRIPTION

Union Chemical has submitted a detailed engineering description of the incinerator as part of its Part B permit application. A brief description of the facility is presented in this section as background to the sampling and analysis strategy. A schematic of the Union Chemical fluidized bed incineration system is presented in Figure 1-1.

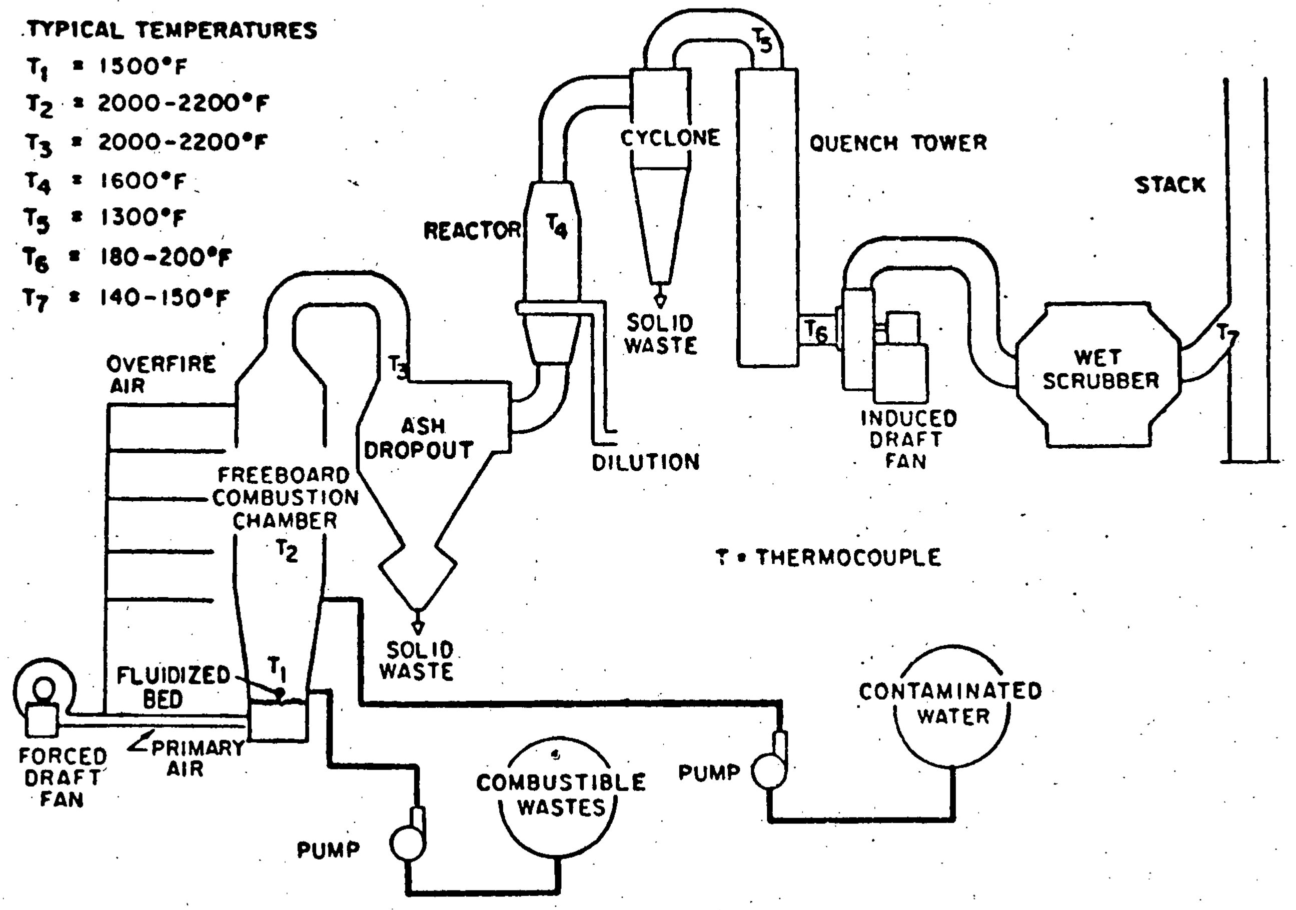

Figure 1-1. Schematic of Union Chemical fluidized bed incineration system.

Combustible wastes consist of residues from the solvent reprocessing operations and other organic chemicals not suitable for reprocessing. These wastes are mixed in either of two continuously stirred 1500 gal storage tanks to achieve the proper viscosity, chlorine content, solid content, and heating value. Typically, one tank contains nonchlorinated wastes and the other contains chlorinated materials. For this test program, drums of selected wastes will be mixed in one of the tanks to meet the program specificatons. One tank of waste is sufficient for 24 hours of operation and will be sufficient for all three test runs.

Potentially contaminated water is collected and stored to prevent site run-off. This water, containing trace organics, is also fed to the incinerator.

The fluidized-bed incinerator is a refractory lined cylindrical vessel with a height of 24 ft and an inside diameter that varies from 32 in. in the bed area, to 42 in. in the freeboard area. Overfire air is injected into the freeboard area at five different heights. Primary combustion air, for bed fluidization, is supplied by a forced draft fan to the plenum below the air distribution plate. The distribution plate supports the silica sand bed and provides openings for injection of the fluidizing air.

The duct from the incinerator to the ash-drop-out chamber and the chamber itself are refractory lined. The ash knockout chamber is essentially a settling chamber that collects small quantities of sand elutriated from the bed and coarse particulates from the waste feed. Typically, the temperature at the inlet to the ash-dropout chamber is 2000-2200°F.

Flue gases are cooled, after the ash dropout chamber by dilution in the reactor. The reactor was originally designed for dry neutralization of acid gases, but is no longer used for this purpose.

A refractory lined Fisher Kostermann XQ cyclone removes most of the particulate matter from the flue gas. Solids are removed by a continuously operating rotary valve.

In the quench tower, flue gases are cooled from about 1300°F to 180-200°F. Lime slurry is fed to the quench tower for removal of HCl from the gas stream. Quenching liquid is recycled after the solids are removed in a settling vessel.

The final control device is horizontal cross flow packed tower manufactured by Celicote. Lime slurry is used to adjust the pH of the scrubber liquid and to insure that HCl emissions meet the regulatory requirements.

The 2 foot diameter stack is 60 feet high. A sampling platform is located 30 feet above the ground and 10 stack diameters downstream from the transition joining the scrubber with the stack. The sampling platform is 15 stack diameters upstream from the stack exit.

1.3 SAMPLING AND ANALYSIS

A list of the seven-sample streams and the planned measurements is presented in Table 1-1. Additional information on the plans for the six liquid and solid streams is presented in Table 1-2 and discussed below. The plans for flue gas sampling are discussed later.

1.3.1 Combustible Waste Feed

Six sets of duplicate combustible waste feed samples will be collected during each of the three 2-hour test runs. The samples will be collected from a tap on the feed pipe to the incinerator. The samples will be collected in Volatile Organic Analysis (VOA) vials and stored at 4°C pending analysis. One set of samples per run will be composited for analysis of POHCs and chlorine. The other set will be stored in GCA's sample bank.

The combustible waste feed samples will be prepared for analysis of POHCs following the general approach outlined in Method A101.[1] The actual method as developed and verified in the GCA laboratory will employ a tetraglyme dispersion technique in lieu of polyethylene glycol. A small portion of the tetraglyme dispersion will be mixed with water and analyzed by purge and trap GC/MS procedures similar to EPA Method 624. The analyses will be conducted using a Hewlett-Packard 5985 computerized GC/MS system.

Total chlorine analysis of the combustible waste feed will be performed using Parr Bomb combustion (ASTMD808-63) with quantitation by ion chromatography. The samples will be oxidized by combustion in a Parr oxygen

TABLE 1-1. SUMMARY OF SAMPLE STREAMS AND REQUIRED MEASUREMENTS

Stream type	Measurements
Combustible waste feed	POHCs,[a] chlorine, ash, Btu
Contaminated water feed	POHCs, chlorine
Ash	POHCs, EP toxicity[b]
Scrubber water	POHCs
Scrubber sludge	POHCs, EP toxicity[b]
Scrubber water supply	POHCs
Flue gas	Moisture, CO_2, CO, O_2, flow, particulate, POHCs, HCl

[a] Trichloroethylene, tetrachloroethylene, 1,1,1-trichloroethane, tetrachloromethane and 1,1,2-trichloro-1,2,2-trifluoroethane.

[b] Not required for trial burn.

TABLE 1-2. SUMMARY OF SAMPLING AND ANALYSIS PLANS FOR LIQUID AND SOLID STREAMS

	Sample type	No. of samples collected	No. of samples analyzed[b]	Analysis	Method
1.	Combustible Waste feed	36 VOA vials	3 composites	POHCs[a] Chlorine	Tetraglyme--GC/MS Parr Bomb--Ion chromatography
2.	Contaminated water	6 VOA vials	3 composites	POHCs Chloride	Purge and trap--GC/MS Ion chromatography
3.	Ash	3-1 gal	1 composite	POHCs EP toxicity	GC/MS EP toxicity
4.	Scrubber water	18 VOA vials	3 composites	POHCs	Purge and trap--GC/MS
5.	Scrubber sludge	3-1 gal	1 composite	POHCs EP toxicity	GC/MS EP toxicity
6.	Scrubber water supply	6 VOA vials	3 samples	POHCs	Purge and trap--GC/MS

[a] Trichloroethylene, tetrachlorethylene, 1,1,1-trichloroethane, tetrachloromethane and 1,1,2-trichloro-1,2,2-trifluoroethane.

[b] Not including additional quality control analyses.

bomb containing a sodium carbonate absorbing solution. The absorbing solution and washings will be combined, diluted to volume and analyzed for chloride using a Dionex Model 14 ion chromatograph.

Ash content and heating value will be determined by ASTM Methods D-1888 and D-3286 respectively.

1.3.2 Contaminated Water Feed

The contaminated water feed contains POHCs and chlorine in trace quantities. Past experience has shown that the contribution of the contaminated water to the total POHC feed is not significant and that the contribution to the total chlorine is minor.[2] Therefore, one set of duplicate samples, in VOA vials, will be collected per run. The samples will be stored at 4°C and analyzed within 14 days after completion of the sampling program. POHCs will be analyzed by an EPA Method 624 protocol with modifications approved for use in the GCA laboratory.

Contaminated water samples will be analyzed for chloride by means of ion chromatography. Samples will be analyzed directly with no prior treatment other than dilution where necessary.

1.3.3 Ash

Ash collected by the knockout chamber is periodically discharged to a 55-gallon drum while ash from the cyclone is continuously discharged to a 55-gallon drum. At the end of each run, a composite sample of both ashes will be collected.

Because the ashes are collected at temperatures above 1000°F, they should not contain any of the POHCs that have been selected for this program. However, one of the three composites will be selected for analysis of POHCs. The analytical method will be similar to the procedure described for the combustible waste field; dispersion in tetraglyme followed by purge and trap GC/MS.

One ash sample will be subjected to the Extraction Procedure Toxicity test as outlined in §261.24 and Test Methods for Evaluating Solid Waste.[3]

Samples of the ash will be extracted with deionized water which is kept at a pH of 5 using acetic acid. The resulting extract will be analyzed for arsenic, barium, cadmium, chrome VI^{+}, lead, mercury, selenium, and silver Analyses for pesticides are not appropriate and are not planned. The Extraction Procedure Toxicity test is not required by the incinerator regulations but will be conducted to determine whether or not the ash is hazardous.

1.3.4 Scrubber Water

The water that is recirculated through the packed bed scrubber and the quench tower will be sampled to determine whether or not the POHCs are accumulating in the water. It should be noted that this water is not discharged from the plant. During each run, three samples will be collected in duplicate VOA vials. Samples will be stored at 4°C and analyzed within 14 days by purge and trap GC/MS procedures (GCA's approved version of EPA Method 624). One composite sample will be analyzed for each run.

1.3.5 Scrubber Sludge

The sludge that is removed from the recirculated water will be sampled and analyzed for POHCs and EP Toxicity. At the end of each run, one grab sample will be obtained. One of these three samples will be selected for analysis of POHCs and EP toxicity. One sample should be sufficient because POHCs will be measured in the water phase during each run and EP toxicity is not required by the incineration regulations.

1.3.6 Scrubber Water Supply

One duplicate set of samples of the scrubber water supply will be collected during each run. Sampling and analysis procedures will be similar to those discussed for the scrubber water. The results will be used as blank corrections for any POHCs found in the scrubber water.

1.3.7 Flue Gas

The most intensive sampling and analysis efforts will be devoted to quantifying the atmospheric emissions from the incinerator. The planned sampling and analysis procedures for this stream are summarized in Table 1-3. The particulate and HCL emissions will be determined using a standard EPA Method 5 sampling train. GCA will utilize a Hastings Air Velocity Meter for the determination of the flue gas velocity and volumetric flow rate of the unit. This device eliminates the interference encountered while making flow determinations in particulate laden or saturated gas streams. The concentration of fixed gases (CO_2, O_2, CO) will be determined during each of the three sampling runs. Integrated gas samples will be collected in Tedlar bags and analyzed by gas chromatography with a thermal conductivity detector. This method meets the requirements of EPA Method 3 and provides for better accuracy than traditional methods with Orsat analyzers. In accordance with the requirements of §270.62(b)(6)(ix) the concentration of CO in the flue gas will also be continuously measured throughout the tests. The CO levels will be monitored using a Horiba PIR 2000 detector.

The determination of selected POHC concentrations in the flue gas will be the primary focus of the flue gas sampling and analysis program. The five compounds that will be measured are tetrachloromethane, trichloroethylene, tetrachloroethylene, 1,1,1-trichloroethane and 1,1,2-trichloro-1,2,2-trifluorethane. These compounds have been selected because they will be present in the combustible waste feed at concentrations in the 2 to 5 percent range and because they are difficult to incinerate as previously discussed. The flue gas concentrations of these compounds that can be expected at different destruction/removal efficiencies are summarized in Table 1-4. Two methods will be used to sample the flue gas for POHCs, the volatile organic sampling train (VOST) with GC/MS analysis and integrated gas sampling train with GC/ECD analysis.

TABLE 1-3. SUMMARY OF FLUE GAS SAMPLING AND ANALYSIS

Test parameter	Sampling method	Analysis method
Moisture	EPA Method 4	Volumetric
Flow	EPA Method 2	Hastings Air Velocity Meter
CO_2, CO, O_2	EPA Method 3 equivalent	Gas chromatography/thermal conductivity detection
CO	Continuous extraction	Continuous monitor--Horiba PIR 2000
Particulate	EPA Method 5	Gravimetric
HCl	EPA Method 5 impingers	Ion chromatography
POHC	Volatile organic sampling train	Thermal desorption--GC/MS
	Integrated gas sampling train	Gas chromatography/electron capture detection

TABLE 1-4. EXAMPLE POHC CONCENTRATIONS IN THE STACK GAS AT SELECTED WASTE FEED CONCENTRATIONS AND DESTRUCTION EFFICIENCIES

Waste feed composition, percent by weight	Destruction/removal efficiency			
	99.99%	99.995%	99.9975%	99.999%
2	120[a]	60	30	12
4	240	120	60	24
5	300	150	75	30

[a] All concentrations are in $\mu g/m^3$. A concentration of 30 $\mu g/m^3$ is approximately equivalent to 5 ppb (volume/volume) depending on the molecular weight of the compound.

With the VOST, Tenax and Tenax/charcoal cartridges are used to collect POHCs. Although this method is still being developed and validated by EPA, it is expected to be adopted as a standard method in the future. Although it offers increased sensitivity over the GC/ECD configuration, the demonstrated precision to date has not been as good as achieved with GC/ECD. Therefore, each run will consist of three samples which will be averaged. Contamination problems initially experienced by several users of the sampling train and encountered by GCA during the tests at Union Chemical in 1982 have been largely eliminated by incorporating more stringent storage and quality assurance measures into the handling of the VOST samples. For example, the sorbent cartridges will be stored and transported under ice water. However, contamination by 1,1,1-trichloroethane may still present a problem when flue gas concentrations are very low. The sorbent cartridges will be thermally desorbed and analyzed by a GC/MS procedure similar to EPA Method 624.

The VOST provides the capability to measure POHCs at concentrations well below 30 $\mu g/m^3$ (e.g., 1 $\mu g/m^3$). Therefore, if the incinerator is achieving a destruction removal/efficiency well above 99.99 percent, the VOST results should unequivocally demonstrate that the destruction removal efficiency is greater than 99.99 percent (see Table 1-4).

Prior to the initiation of the VOST sampling and as a part of each of the test runs GCA will determine the concentrations of individual POHCs in the stack gas using gas chromatographic techniques. Samples of flue gas will be collected in Tedlar bags with an integrated gas sampling train. The samples will be analyzed by gas chromatography with electron capture detection. A field transportable gas chromatograph will be set up in a nearby clean area for this program.

1.4 SCHEDULE AND REPORTING

The field sampling program will be initiated approximately 3 weeks after approval of the test plan. The schedule for the field tests is shown in Table 1-5.

TABLE 1-5. SAMPLING SCHEDULE

Day	Activity
1	Travel and set up
2	Complete set up, calibrate instrumentation (GC's and continuous monitors) Screen stack emissions to determine approximate POHC emissions
3	Conduct test run 1
4	Conduct test runs 2 and 3
5	Pack and travel

The Federal regulations for hazardous waste incinerator permits require that the results of the trial burn be submitted to EPA within 90 days of completion of the trial burn, or later if approved by the administrator (§270.62(b)(7). To meet the schedule, GCA plans to complete the laboratory analyses within 50 days, submit the draft report to Union Chemical within 70 days, and prepare a final report incorporating comments from Union Chemical within 85 days.

The final report will contain discussions of the field sampling procedures, laboratory procedures, quality control procedures and results, appropriate raw data sheets and the results of the program.

2.0 PROJECT ORGANIZATION

The project organization chart for this project is presented in Figure 2-1.

The Division QA Manager is the responsible Quality Assurance Officer for this project. She will review and approve the QA Project Plan before it is submitted to EPA. 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 control problems found and corrective actions taken will be described.

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

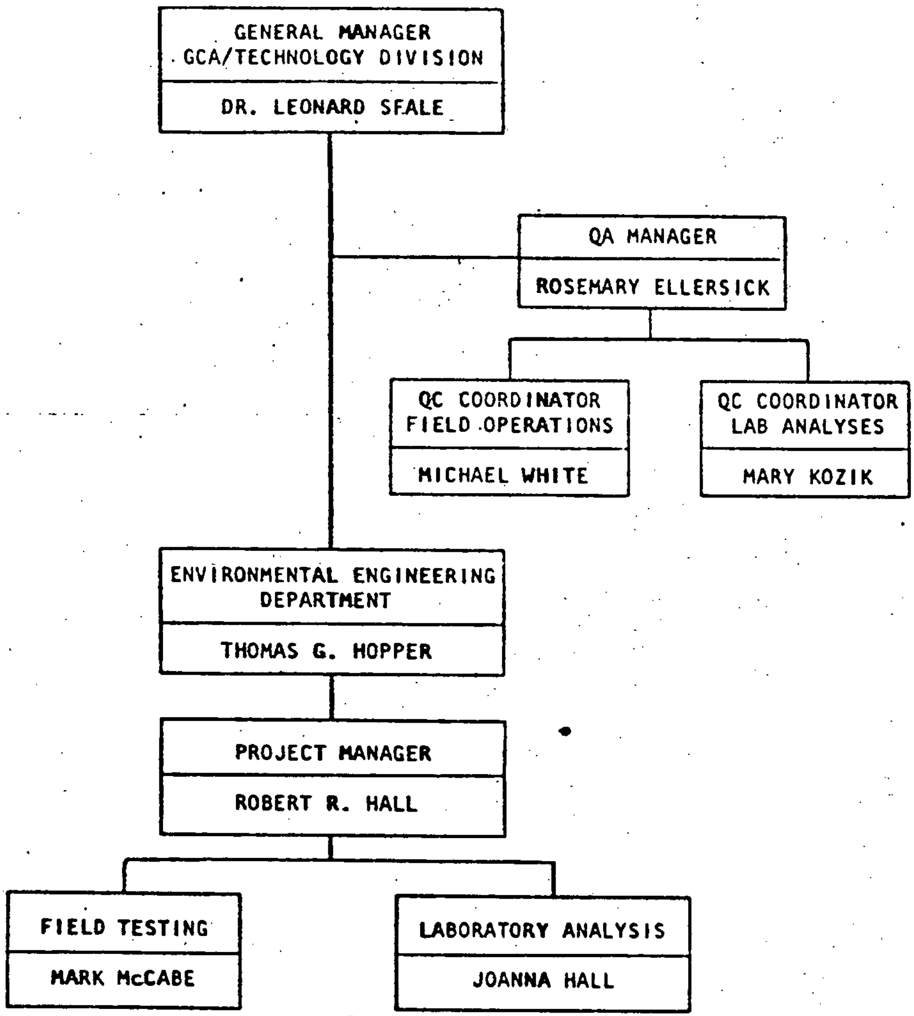

Figure 2-1. Project organization and responsibility.

3.0 QA OBJECTIVES FOR PRECISION, ACCURACY AND COMPLETENESS

The collection of data that can be used to successfully accomplish the goals outlined in Section 1.0 of this Quality Assurance Project Plan requires that the sampling and analytical procedures be conducted with properly operated and calibrated equipment by trained, experienced personnel.

Precision, accuracy and completeness objectives for this project are given in Tables 3-1 and 3-2. The formulas used to calculate precision, accuracy and completeness are given in Section 12.0 of this Plan.

It is recognized that the usefulness of the data is contingent upon meeting criteria for representativeness and comparability. Every effort will be made to assure representativeness by adhering strictly to the sampling and analytical protocols outlined. The QA objective is that all measurements be representative of the different streams sampled and of the incinerator operation being tested. The corresponding QA objective is that all the data being generated be comparable with measurements made under similar process conditions by GCA or another organization.

TABLE 3-1. GOALS FOR PRECISION, ACCURACY AND COMPLETENESS--SAMPLING

Measurement (method)	Precision (standard deviation)	Accuracy	Completeness
Particulate (EPA M-5)	12% RSD	Not determinable	95%
Carbon Monoxide (NDIR)	±2.5%	±2.5%	95%
HCl Emissions (impinger train)	Untested	Untested	Untested
Volatile organics	Not documented	Not documented	Not documented

RSD = Relative Standard Deviation

TABLE 3-2. GOALS FOR PRECISION, ACCURACY AND COMPLETENESS--ANALYTICAL

Measurement method	Matrix	Precision (relative standard deviation)	Accuracy	Completeness
Organic Analyses				
GC/ECD	Stack gas in Tedlar bags	< 30%[b]	±50%[b]	95%
GC/MS (purge and trap	Water	< 25%	±25%	95%
GC/MS (tetraglyme)	Waste feed	< 25%	±25%	95%
GC/MS	Sludge, ash	< 30%	±30	95%
GC/MS	Tenax	< 50%	±50	95%
Inorganic Analyses				
IC	Waste Feed	<20%	±20%	95%
	Impinger	<10%	±10%	95%
ICAP	Ash/Sludge[a]	<15%	±15%	95%
AA	Ash/Sludge	<15%	±15%	95%

[a] Precision and accuracy goals represent ICAP analysis only. Precision and accuracy estimates for the EPA Toxicity method are not available.

[b] Precision and accuracy of this method has been demonstrated at ±10% under laboratory conditions. These goals are stated considering the analysis will be performed in the field.

4.0 SAMPLING PROCEDURES

Three replicate 2-hour sample tests will be conducted for this program. The identification of six separate process streams and the sampling techniques are discussed below. A sampling scheme is presented in Table 4-1.

4.1 LIQUID AND SOLID SAMPLE STREAM IDENTIFICATION

4.1.1 Combustible Waste Feed (CWF)

Liquid combustible waste feed samples will be obtained by means of a tap located in the line connecting the liquid waste holding tank and the burners. Two VOA vial samples will be taken at 20-minute intervals during each 2-hour incineration burn.

An NP Industries ultra-scan single unit doppler is used to continuously monitor the liquid combustible feed ratio. This instrument will be calibrated prior to each test by means of a 1 gallon bucket and a stop watch. A calibration chart will be prepared by Union Chemical personnel and provided to GCA. In addition, the level in the waste feed holding tank will be dip checked manually every 1/2 hour.

4.1.2 Contaminated Water Feed (WF)

Onsite contaminated run-off water is used to control combustion chamber temperatures. Duplicate VOA vial samples of this stream will be collected during each 2-hour burn from a tap located in line between the holding tank and spray nozzles. Prior to each test burn, a Fisher & Porter meter which is used to monitor the contaminated water will be calibrated by means of a 1-gallon bucket and a stop watch. The contaminated water holding tank will also be manually dip checked every 1/2 hour.

TABLE 4-1. SOLID AND LIQUID SAMPLING SCHEME

Sampling location	Sample code	Method	Analysis	Total number collected during program[b]	Container type	Volume required
Combustible waste feed	CWF	Discrete grab	POHCs[a] Chloride	36	VOA vials	40 ml
Contaminated water	CWF	Discrete grab	POHCs Chloride	6	VOA vials	40 ml
Scrubber water supply	SWS	Discrete grab	POHCs	6	VOA vials	40 ml
Scrubber water	SW	Integrated sample	POHCs	18	VOA vials	40 ml
Scrubber sludge	SS	Grab composite	POHCs EP toxicity	3	Amber glass	1 gal
Ash	A	Grab composite	POHCs EP toxicity	3	Amber glass	1 gal

[a] Trichloroethylene, tetrachloroethylene, 1,1,1-trichloroethane, tetrachloromethane, and 1,1,2-trichloro-1,2,2-trifluoroethane.

[b] Not including additional quality control analysis.

4.1.3 Scrubber Water Supply (SWS)

Clean scrubber water samples will be collected into 2-VOA vials once per run. These samples will be extracted from a tap located in the scrubber water supply line, which connects the supply well to the FBC scrubber unit. There is no monitoring device in place to measure the total clean well water input.

4.1.4 Scrubber Water (SW)

Three sets of duplicate VOA vials per run will be used to collect samples of process water from the scrubber effluent stream. Samples will be obtained from a tap located in the line leading to the contaminated water holding tank. There is no process monitoring device in place to measure the flow rate of the scrubber water; there is no discharge of scrubber water from the plant.

4.1.5 Scrubber Sludge (SS)

The entrapped particulates from the wet scrubber are conveyed to a scrubber sludge tank for settling and the water is sent back to the wet scrubber. A 1-gallon scrubber sludge sample will be collected from this tank at the conclusion of each trial burn. There is no process monitoring device that measures the total sludge collected.

4.1.6 Ash (A)

Solid waste combustion material will be collected from both the ash dropout chamber and cyclone hopper at the conclusion of each trial burn. Ash samples from these two sites will be composited in proportion to the stream flow rates. A 1-gallon ash composite sample will be retained from each run. The monitoring of the ash dropout flow will be accomplished by preweighing 55-gallon drums. Upon completion of each test run, the drum(s) will be reweighed on the floor model scale used daily by Union Chemical. The scale will be calibrated with a known weight during each test burn.

4.2 FLUE GAS

4.2.1 Particuate and HCl Emissions

A modified EPA Method 5 Train will be used to simultaneously collect particulate and HCl in the flue gas. A sampling and velocity traverse will be performed along two diameters of the stack. Twenty-four sampling points, determined in accordance with EPA Method 1, will be sampled at 5 minutes per point yielding a total sample time of 120 minutes. Sampling will be isokinetic (±10 percent) with readings of the flue gas parameters recorded at every sampling point. A schematic of the modified Method 5 train is presented in Figure 4-1. The sampling train consists of a heated stainless steel probe with a stainless steel button hook nozzle and attached thermocouple and pitot tubes. The sampled gas passes through the probe assembly to a heated glass fiber filber (Reeve Angel 934 AH). The filter holder will be maintained at 248°F ±25 throughout the test period. Downstream of the heated filter, the gas is passed through a series of four ice-cooled impingers to effect the removal of entrained moisture. The first impinger is empty to provide for the collection of the flue gas condensate. The recovered sample of condensate will be analyzed for HCl determinations. The impinger is modified to have a short stem so that sample gas does not bubble through the collected sample. The second impinger contains 100 ml of 1 N NaOH to ensure the complete collection of HCl. The third impinger is empty, and the final impinger contains a known amount of desiccant. The impingers are followed by a pump by gas meter and calibrated orifice.

A Hastings Air Velocity Meter will be substituted for the traditional water manometer in the determination of the flue gas velocity and volumetric flow rate of the unit. This technique eliminates this potential interference in making flow determinations in saturated gas streams.

Recovery activities for this stream will be:

1. Remove sample train to a predetermined recovery area.
2. Note the condition of the train (e.g., desiccant color, filter condition, etc.).

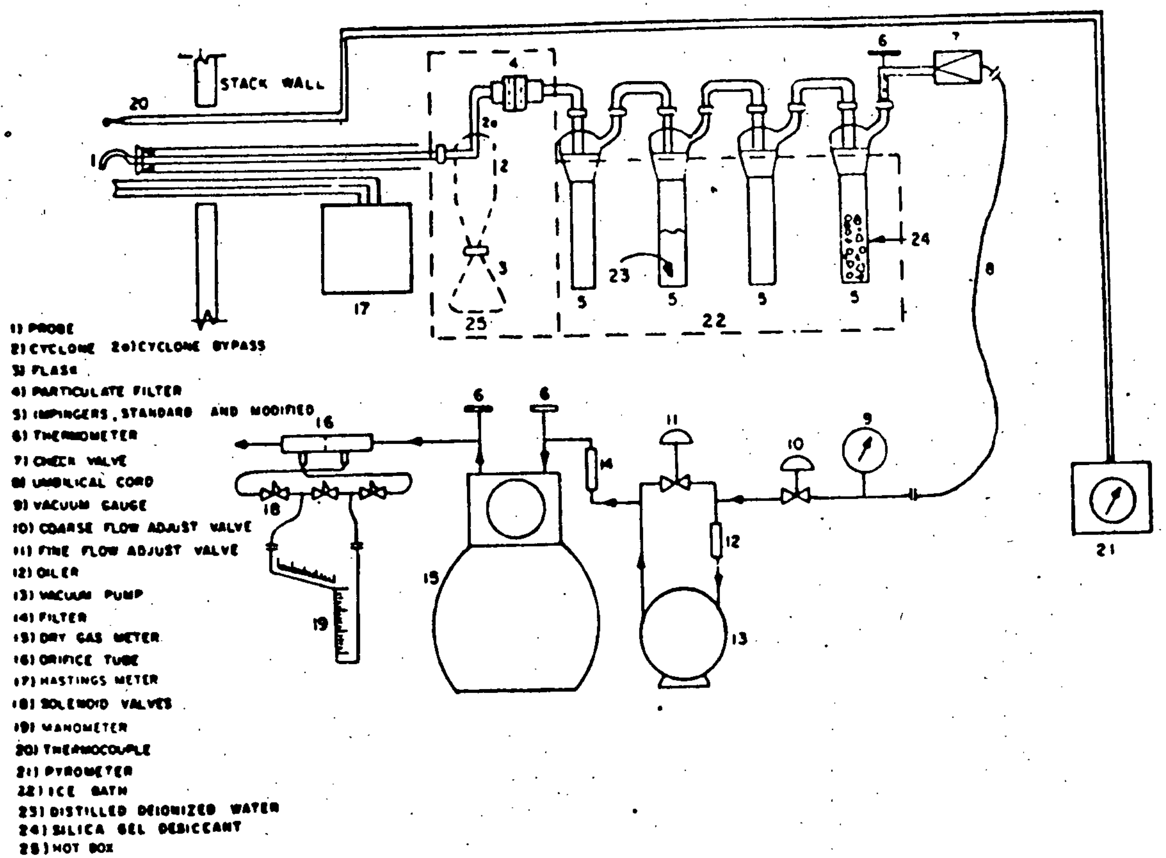

Figure 4-1. Schematic of RAC Staksamplr^{TM}.

3. Disassemble the filter housing and transfer the filter to its original petri dish. Seal the container and lable the sample as; -M5-PF

4. Rinse the front half of the train (nozzle, liner, and filter assembly) three times with acetone. Seal the linear polyethylene container and label the samples as -M5-FH.

5. Measure the volume of condensate in the first impinger in a precleaned glass graduated cylinder. Record the volume and transfer to a linear polyethylene container. Rinse and recover the impinger with known amounts of DI water. Seal the container and label the sample as -M5-CD.

6. Combine the contents of the second and third impingers. Record the volume and transfer to a linear polyethylene container. Rinse and recover the impinger with known amounts of DI water. Seal the contained label and label the sample as -M5-IMP.

7. Record the weight gained by the silica gel impinger.

8. Examine all containers to ensure that they are properly sealed and labeled and that the liquid levels are marked.

4.2.2 Volatile Organic Compounds

VOST

The volatile organic sampling train will be the primary method used to quantify the concentrations of the POHCs in the flue gas. This method uses Tenax, an organic sorbent resin, to collect the organic species of interest.

The train consists of a heated glass-lined probe with a glass wool plug to remove particulate, followed by an assembly of condensers and organic resin traps as illustrated in Figure 4-2. The first condenser cools the gas stream and condenses the water vapor present. The flue gas and condensed moisture then pass through a cartridge containing 1.5 grams of Tenax resin (60 to 80 mesh). The condensate is collected in the first impinger which is continually purged by the gas stream itself. The second condenser and trap containing Tenax/charcoal (50/50) serve as a backup for low volume breakthrough compounds. Following the second Tenax trap is a series of impingers and drying tubes for residual moisture removal.

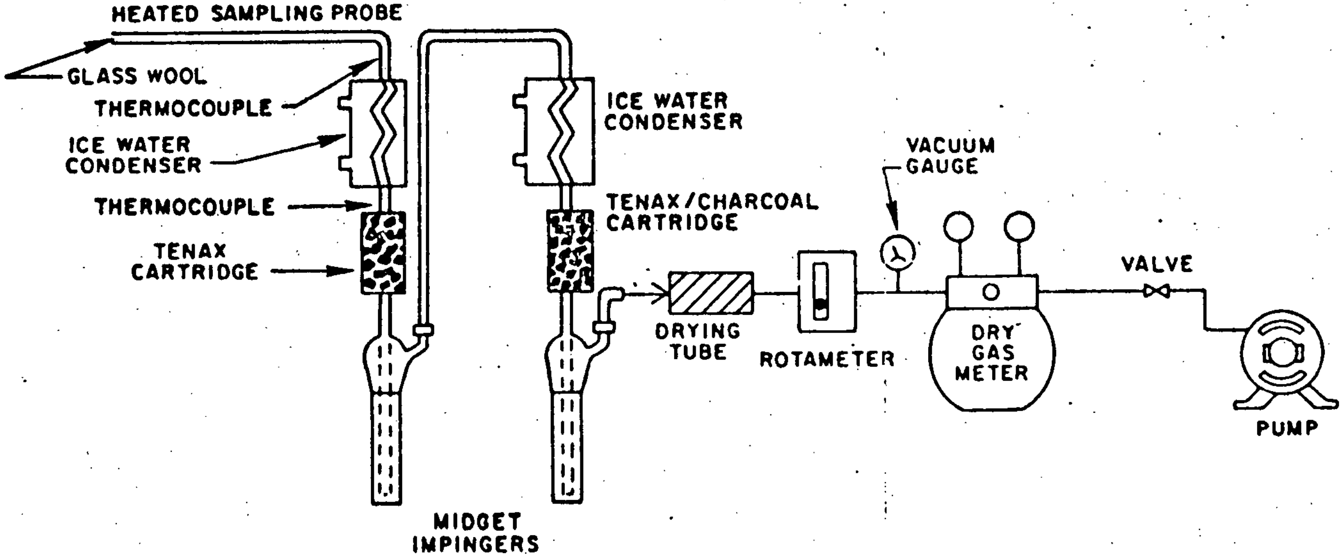

Figure 4-2. Schematic of volatile organic sampling train.

Sample temperatures will be monitored at the outlet of the sample probe and the inlet to the Tenax cartridge through the use of thermocouples. The gas temperature through the probe will be maintained above 130°C to prevent the premature condensation of the volatile components. The temperature of the gas through the resin cartridges will be maintained at less than 20°C. The sample gas volume through the resin traps will be maintained at 1/2 liter per minute. The total sample volume for each set of tubes will not exceed 20 liters.

Three VOST runs will be conducted during each 2-hour test period. The results of the three runs will be averaged to yield one result for each test. The samples collected from each VOST run will consist of a Tenax cartridge, a backup cartridge containing Tenax and charcoal, and a flue gas condensate. The sealed sorbent cartridges will be stored in containers packed with activated charcoal. The contents of the condensate impinger will be transferred to 40 ml VOA vials and brought up to volume with DI water.

Extensive sorbent preparation and quality assurance procedures will be instituted to ensure the preparation of these samples. All components of the system coming into contact with the samples will be rinsed with methanol and dried in an oven at 130°C for a period of 1 hour.

Method and field blanks of the sorbent resins will be collected in conjunction with each of the three tests. During the sampling program, the reagents and sorbent resin samples associated with this train will be maintained offsite to minimize the potential for sample contamination from the ambient air. All of the resin cartridges and collected samples associated with this train will be stored and transported at a temperature of 4°C to prevent contamination and minimize the formation of naturally occurring Tenax degradation products such as benzene and toluene.

Tedlar Bag Samples

Additional samples of the flue gas will be collected for POHC analysis by GC/ECD in the field. Duplicate samples of flue gas will be collected through the use of an integrated gas sampling train as illustrated in Figure 4-3.

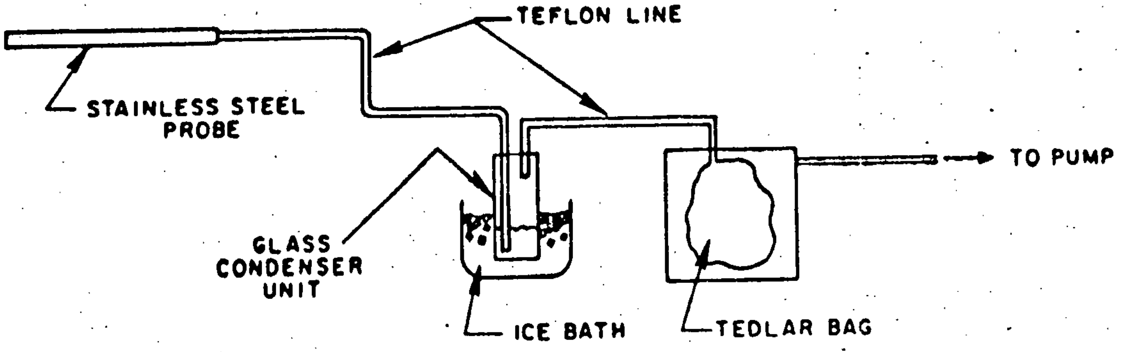

Figure 4-3. Integrated gas sampling train.

The sample will be extracted from the stack through a stainless steel probe containing pre-extracted glass wool to remove particulate. A condenser will be used to remove excess moisture from the gas stream. Prior to their use in the field, the Tedlar gas bags will initially be baked in an oven at 130°C for a period of 1 hour and purged with prepurified nitrogen. This sequence will be repeated three times. The remaining components of the train coming into contact with the sample will be rinsed with methanol and baked for a period of an hour at 130°C. The sample will be collected at a rate of 0.3 liters per minute in conjunction with the VOST tests. At the conclusion of each test, the bags will be sealed and removed to a designated area for chromatographic analysis. The condensate samples will be collected in VOA vials and maintained at 4°C pending analysis. Blank samples of the condensate and bag samples (conditioned bags, inflated with prepurified nitrogen) will be collected in conjunction with each sample run.

A summary of the flue gas samples to be collected for each of the three test runs is presented in Table 4-2.

4.2.3 Bag Samples for Fixed Gases

Additional integrated bag samples will be collected for the determination of fixed gases (O_2, CO_2, N_2, and CO). These samples will be obtained through the use of the sampling system depicted in Figure 4-3. The samples will be analyzed on a gas chromatograph equipped with a thermal conductivity detector in accordance with procedures outlined in Section 7.0.

4.2.4 Carbon Monoxide Monitoring

A continuous monitoring system will be used to measure carbon monoxide concentrations throughout each test run. The 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).

TABLE 4-2. FLUE GAS SAMPLING SUMMARY

Sample description	Sample code	Analysis	Number collected per run	Container type	Comments
Modified Method 5					
Particulate filter	-M5-PF	Gravimetric	Process dependent	Petri dish	
Front half	-M5-FH	Gravimetric	1	500-ml LPE	
Condensate	-M5-CD	Chlorine	1	1-liter LPE	a
Impingers 2 and 3	-M5-IMP	Chlorine	1	500-ml LPE	a
VOST					
Tenax cartridge	-VOST-T	Volatile organic	3	Self contained	a
Tenax:charcoal cartridge	-VOST-TC	Volatile organic	3	Self contained	a
Condensate	-VOST-CD	Volatile organic	1	VOA vial	a
Tenax cartridge, field-biased blank	-VOST-T-FBB	Volatile organic	1	Self contained	a
Tenax:charcoal cartridge, field-biased blank	-VOST-TC-FBB	Volatile organic	1	Self contained	a
Condensate, field-biased blank	-VOST-CD-FBB	Volatile organic	1	VOA vial	a
Tenax, method blank	-VOST-T-B	Volatile organic	1	Glass culture tube	a
Tenax:charcoal, method blank	-VOST-TC-B	Volatile organic	1	Glass culture tube	a
Gas Bag Analysis					
Tedlar bag sample	-GB	Volatile organic	2	25-l Tedlar bag	
Condensate	-GB-CD	Volatile organic	1	VOA vial	a
Tedlar bag, field blank	-GB-B	Volatile organic	1	25-l Tedlar bag	
Condensate, field blank	-GB-CDB	Volatile organic	1	VOA vial	a
Tedlar bag for fixed gases	-GB-FG	Fixed gases	2	25-l Tedlar bag	
Continuous Emission Monitoring					
Carbon monoxide		NDIR		None	

[a] Store at 4°C.

Carbon monoxide concentrations will be determined using a Horiba Model PIR 2000 NDIR CO analyzer. The monitor will be calibrated with zero and two span gases before and after each test period. The data will be corrected for instrument drift (if any) and reduced to 15 minute averages. Maximum and minimum values for each test period will be determined.

5.0 SAMPLE CUSTODY

GCA follows sample custody procedures based on the EPA recommended source sampling procedures.[4] Appendix A presents custody record sheets.

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.

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 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. The level of contents is marked on the container. All samples are logged in the field sample log whether they are analyzed onsite or in the GCA laboratories.

Customized sample identification tags are frequently used and can be quickly designed and printed.

5.2 LABORATORY OPERATIONS

All samples received at CCA are submitted to the CCA Sample Bank Manager. Each sample is logged into the large bound master log and assigned a CCA Control Number which is unique to that sample, identifies it and follows it through all operations. The Sample Bank Manager initiates a page for each sample in the Custody Notebook and ensures that each handling of the sample is documented. Each analyst working with the sample provides a record of such actions in the custody book, thereby maintaining the chain of custody on the original sample.

When preparation and analysis procedures necessitate the transfer of samples between two analysts within the laboratory, or between two laboratories a Sample Custody Transfer Form is required. This document becomes part of the permanent project file and serves as a supplement to the Custody Notebook record of sample handling.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

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 calibration sheet will be submitted to 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."[4]

- Sample meter system--leak checked, then compared against a wet test meter to a ratio average of Y ± 0.01Y.
- Thermocouples--compared to mercury in glass thermometer to accuracy of ± 1.5%, or the use of a constant correction factor.
- Field Barometer--calibrated initially vs. mercury in glass barometer to ± 0.01 in. Hg. 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 ± 1 mg.
- Rotameters--calibrated with a bubble tube or spirometer.

Hastings Velocity Meter

The Hastings Stack Gas Velocity Meter is calibrated in a wind tunnel against a standard pitot tube as described in EPA Method 2. A four point calibration curve will be generated plotting Δp inches of water to output in volts.

Zero and full scale response will be validated prior to each run.

6.2 ANALYTICAL INSTRUMENTATION

6.2.1 GC/TCD

The flue gas samples collected for fixed gases will be analyzed using an Analytical Instrument Development, Inc., Model 511-17 field gas chromatograph equipped with a thermal conductivity detector. The instrument will be calibrated daily using commercially obtained, certified (±2 percent) standard gases. A three-point calibration curve of instrument response vs. concentration will be developed for each of the fixed gases, O_2, CO_2, CO and N_2 in the anticipated concentration ranges for the stack gas. Previous tests at Union Chemical have indicated values of 6 percent CO_2, 12 percent O_2 and 82 percent N_2. Each of the standards will be analyzed in duplicate with ± 10 percent replication used as the criteria for acceptance. The validity of the calibration curve will be checked with duplicate injections of a single standard prior to the analysis of samples from each run.

6.2.2 Horiba Model PIR-2000 CO Analyzer

A three-point calibration curve will be generated for the Horiba PIR_2000 Analyzer, by introducing zero, mid and high range span gases into the instrument. This three-point calibration will be conducted prior to and at the conclusion of each trial burn.

6.2.3 Hewlett-Packard 3920 Gas Chromatograph with Ni^{63} Electron Capture Detector

Calibration Standards

1. Prepare a stock solution of chlorinated solvents by combining equal volumes of the components of interest in a septum sealed vial. The solvents are reagent grade or better, used as obtained from local distributors. Stock solutions are prepared weekly.
2. Add 1.0 µl of the stock solution to a nitrogen - purged Supelco 500 ml gas sampling bulb and allow a 10 minute equilibration period. The concentration of this standard is approximately 1 g/m^3.

3. Prepare the working standard by serially diluting the 1 g/m^3 gas standard into the 10 to 500 μg/m^3 range using Hamilton 1001-LT gas tight syringes and several nitrogen purged Tedlar bags. Prepare working standards daily.

4. Verify working standards by analysis of certified gas standards obtained from Scott Environmental Technology, or equivalent.

Calibration Procedures

1. Calibrate the instrument daily using 4 to 5 calibration (working) standards.

2. 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.

6.2.4 Hewlett-Packard 5993 GC/MS--Thermal Desorption

Calibration Standards

Prepare standard Tenax cartridges using the following procedure:

1. Pipet a known volume of a standard mixture into a 250 ml sampling bulb and allow the bulb to equilibrate in a 35°C water bath.

2. Withdraw a known quantity of the vapor using a gas tight 100 μl syringe and inject onto a Tenax cartridge which is under negative pressure.

3. Prepare a minimum of three standard cartridges by varying the volume of vapor withdrawn from the sampling bulb. The standards should be prepared to bracket the expected concentration ranges for the compounds of interest.

Calibration Procedure

1. Check and adjust mass calibration, if necessary, at the beginning of each 8-hour shift to meet the PFTBA criteria shown in Table 6-1. To perform the MS calibration, the following instrumental specifications are required:

   Electron Energy - 70 eV
   Mass Range - 35 to 350 m/z
   Scan Time - 1 sec/scan

If the performance criteria listed in Table 6-1 are not met, the analyst must retune the instrument and repeat the performance check. The performance criteria must be met before any standards, blanks, or samples are analyzed.

TABLE 6-1. PFTBA KEY ION ABUNDANCE CRITERIA

Mass	Ion abundance criteria
69	100 percent
131	25 to 30 percent of mass 69
219	25 to 25 percent of mass 69

2. Thermal Desorption System Check: Proper operating temperatures and gas flow rates for the thermal desorption system must be verified for every 8 hours of analysis time.

3. After all system criteria have been met, the GC/MS must be initially calibrated to determine response by generating a four-point calibration curve in triplicate. System calibration will be accomplished by the analysis of aDsorbent tubes spiked with the compounds of interest. Spiked tubes will be prepared at at least four levels and responses of the compounds at these levels recorded to form a calibration curve.

4. On every day that samples are to be analyzed, verification of the four-point calibration curve is necessary. Prepare a standard trap with concentrations of compounds of interest between the low and high points of the calibration curve and analyze as a sample. If the calculated concentrations of the measured components fall outside the ± 20 percent of expected acceptance range, repeat the calibration check and repeat the procedure. A second failure indicates the calibration curve is invalid and the instrument must be recalibrated. Calibration should be performed a minimum of once per calendar week.

5. Program the GC/MS data system to operate in the Extracted Ion Current Profile (EICP) mode collecting the major ion of each of the compounds of interest. Measure the peak area for the major ion of each compound versus concentration at three levels of calibration. These measurements are collected in triplicate.

6.2.5 Dionex Model 14 Ion Chromatograph

Calibration Standards

1. Prepare the 1000 ppm stock chloride solution using sodium chloride.
2. Prepare working standards in the range of 0.5 to 5.0 ppm by dilution of the stock solution.
3. Verify the working standards by analysis of a QC sample prepared from an EPA minerals concentrate.

Calibration Procedure

1. Set up the instrument according to the manufacturer's instructions.
2. Inject the blank and calibration standards and record the peak height of each.
3. Analyze a QC sample prepared from an EPA minerals concentrate; if the reported value is within 5 percent of the expected value, sample analysis may begin. Reanalyze the sample at the end of the analysis period or after every 10 to 15 samples if the instrument is running for an extended period.
4. Enter all information regarding instrument operating parameters and analysis of laboratory control samples in the instrument logbook; sign and date the entry. Standard preparation must be documented in the analyst's notebook.

6.2.6 Jarrell Ash Model 855 Inductively Coupled Plasma Spectrometer

Calibration Standards

1. Prepare the 1000 ppm stock solution from the high purity metal or an appropriate salt; if the salt is used, it must be dried at 105°C for 1 hour unless otherwise specified.
2. Prepare the mixed working standards daily by dilution of the 1000 ppm stock solution.
3. Verify the working standards by analyzing against a sample prepared from an EPA Trace Metals concentrate.

Calibration Procedure

1. Profile and calibrate the instrument according to the procedures outlined in the instrument operating manual using a minimum of three standards. Flush the system with the calibration blank between each standard.

2. Analyze a quality control sample prior to beginning sample analysis. Enter the reported values for the QC sample in the instrument logbook and sign and date the entry. If the reported values are acceptable, generally within 5 percent of the expected value, sample analysis may begin.

3. Flush the system with the calibration blank between each sample.

4. Reanalyze the quality control sample at the end of the analysis session or after every 10 to 15 samples if the instrument is running for an extended period.

5. Standard preparation must be documented in the analyst's notebook. All instrument operating parameters must be noted in the instrument logbook; the logbook entry must be signed and dated by the analyst.

6.2.7 Hewlett Packard 5985 GC/MS--Purge and Trap

Calibration Standards--

1. Purchase or prepare the stock solutions from the pure compound. Store at 4°C, prepare fresh every 2 weeks or as needed.

2. Prepare the working standard by dilution of the stock standard using the appropriate solvent.

3. Verify the standards by analysis of an EPA QC sample or other appropriate Laboratory Control Sample.

Calibration Procedure

1. Tune the instrument daily to the criteria stated in the analytical method.

2. Prepare a three- to five-point calibration curve every 2 weeks; the standard curve must be verified daily.

3. Analyze an EPA QC sample or other appropriate Laboratory Control Sample. If the results are within the established control limits, analysis may proceed.

4. Document standard preparation in the analyst's notebook. Enter the required information in the instrument logbook; sign and date the entry.

6.2.8 Perkin Elmer 2380 Atomic Absorption Spectrophotometer: MHS-20 Mercury-Hydride System

Calibration Standards

1. Prepare the 1000 ppm stock mercury solution by dissolving 1.080 g Mercury (II) Oxide in a minimum volume of (1+1) HCl. Dilute to 1 liter with deionized water.

2. Prepare working standards in the range of 25-100 ng/ml, daily, by dilution of the 1000 ppm stock solution.

3. Verify the working standards by analysis of EPA Trace Metals concentrate.

Calibration Procedures

1. The MHS-20 System is microprocessor controlled and should be set up according to the manufacturer's specifications. The following instrument parameters are normally used.

Wavelength	253.7 nm
Slit	0.7 nm
Lamp Setting	EDL - 5 watts
Mode	$SnCl_2$
Purge I	50S
Reaction	15S
Purge II	30S
Cell Temperature	200°C

2. Analyze the blank and calibration standards and record the absorbance of each. Prepare a calibration curve by linear regression analysis of the absorbance vs. concentration data.

3. Verify instrument calibration by analysis of a quality control sample. If the reported value is within 5 percent of the expected value sample analysis may begin. Enter QC sample data in the instrument logbook; sign and date the entry.

4. Reanalyze the QC sample at the end of the analysis session or after every 10-15 samples if the instrument is running for an extended period.

5. Standard preparation must be documented in the analyst's notebook. All instrument operating parameters must be noted in the instrument logbook; the entry must be signed and dated.

7.0 ANALYTICAL PROCEDURES

7.1 FIELD MEASUREMENTS

7.1.1 Fixed Gases by GC/TCD

Samples for the determination of the fixed gas composition, CO_2, O_2, N_2 and CO, of the flue gas will be obtained from the stack in conjunction with the particulate tests. An integrated gas sample will be collected using the train presented in Figure 4-3. The train consists of a stainless steel probe containing a glass wool plug, a glass condenser unit placed in an ice bath, a Tedlar bag, a pump and a rotometer. The procedures for this sampling technique will be as specified in Appendix A of 40 CFR 60 (EPA Reference Method 3). The sample will be collected at a rate of 0.25 l/m during the 2-hour test period. Analysis will be performed onsite by direct injection into an Analytical Instrument Development, Inc., Model 511-17 portable chromatograph equipped with a Thermal Conductivity Detector.

The samples will be introduced into appropriate chromatographic columns, packed with Chromosorb 102 and 13X molecular sieve, by a gas sampling valve. The components of interest, CO_2, O_2, CO, and N_2, will be identified by retention time comparison with standard chromatograms. Calibration curves (response vs. concentration) for these compounds will be developed daily using commercially available certified gas mixtures. The accuracy of these curves will be verified prior to the analysis of any samples. Each sample will be analyzed in duplicate with a required precision of ±10 percent. The results of these analyses will be reported as percent (%) for CO_2, O_2, and N_2 and as parts per million (ppm) for CO. The operating conditions for the instrument are presented in Table 7-1.

7.1.2 Continuous Carbon Monoxide Measurements

In accordance with the requirements of §270.62(b)(6), the concentration of CO in the flue gas will be measured using continuous emissions monitoring techniques. A Horiba PIR 2000 monitor will be used to quantify these levels.

Prior to its introduction into the instrument, the sample stream will pass through a Balston Filter and Horiba GC-12 conditioning system for the removal of entrained particulate and moisture. The monitor will be calibrated prior to and at the conclusion of each of the sampling runs. A three-point (zero, mid range, and span) calibration check will be performed using commercially obtained, certified (±2 percent) gas standards. The concentration of CO in the flue gas will be determined by a comparison of the instrument's response for samples and the calibration standards. The output from the instrument will be recorded on a strip chart to provide a continuous record of the CO levels.

TABLE 7-1. GC/TCD OPERATING CONDITIONS FOR THE ANALYSIS OF FIXED GASES

Instrument	AID 511-17
GC Conditions	
Column	Chromosorb 102 and 13X molecular sieve
Temperature program	40°C
Carrier flow	Helium, 10 ml/min

7.1.3 Onsite GC/ECD Analysis

Flue gas samples, collected in Tedlar bags, will be subjected to onsite GC/ECD analysis for the volatile components of interest. Sample gas will be aspirated from each bag through a heated gas sampling valve and then injected onto the GC by diverting carrier flow through the valve sample loop. Instrumental conditions to be used for this analysis are presented in Table 7-2.

Calibration standards will be prepared by injecting microliter quantities of commercially available solvents into a 500 ml gas sampling bulb which has been previously rinsed with hexane, heated at 120°C and prepurged with nitrogen. Serial dilutions will then be performed using a gas-tight syringe and several 1-liter prepurged Tedlar bags in order to provide a four-point

TABLE 7-2. GC/ECD OPERATING CONDITIONS FOR TEDLAR BAG ANALYSIS

Instrument	Perkin Elmer 3920 with Ni^{63} electron capture detector and Spectra Physics Minigrator
GC Conditions	
Column	20% SP-2100/0.1% Carbopack 1500 on 100/120 mesh Supelcoport, 10 ft x 1/8 in. SS column
Temperature program	Isothermal at 50°C
Injector temperature	110°C
ECD temperature	325°C
Carrier flow	Argon/methane, 25 ml/min
Sampling Valve Conditions	
Loop Volume	1 ml
Loop temperature	125°C

calibration curve between 40 and 1500 $\mu g/m^3$. Calibration standards will be analyzed under the same operating conditions as samples. A minimum of two of the four calibration points will be analyzed in duplicate with an acceptance criterion of ±30 percent. Calibration curves will be prepared from a linear regression analysis of the integrated area response from the injections of calibration standards. Calibration curves will be rejected if the correlation coefficient of the linear regression analysis is less than 0.95. All samples will be quantified by entering the sample area response into the appropriate calibration curve. Reported results will represent the mean of two determinations.

Detection limits for the five components of interest generally range from 20 to 50 $\mu g/m^3$ under controlled laboratory conditions. The effect of the environment on program samples will be determined through the analysis of laboratory method blanks and field-biased blanks.

7.2 ORGANIC LABORATORY ANALYSIS PROCEDURES

7.2.1 Aqueous Samples

Four types of aqueous samples will be collected: VOST condensates, contaminated water feed, scrubber effluent, and scrubber water supply. These samples will be analyzed for volatile organics using purge and trap GC/MS techniques as outlined in EPA Method 624 with modification approved for use in the GCA laboratory.[5] Mass spectral tuning will be performed with PFTBA in place of DFTPP or BFB as specified in that procedure. Instrumental operating conditions are presented in Table 7-3. Anticipated detection limits for this analysis are approximately 10 $\mu g/l$ with a precision of ±25 percent (expressed as relative standard deviation) for replicate analysis of spiked samples.

7.2.2 Solid Samples

Analysis of combustible waste feed, scrubber sludge, and fly ash for volatile organics will be accomplished by extraction followed by purge and trap GC/MS procedures. Sample preparation will follow procedures as given in Method A101b[5] with the substitution of tetraglyme for the polyethylene glycol

TABLE 7-3. GC/MS CONDITIONS FOR VOLATILE ORGANICS ANALYSES

Instrument	Hewlett-Packard 5985
GC Conditions	
Column	1% SP-1000 on Carbopack B, 6 ft x 2 mm ID column
Temperature program	60°C held for 4 min, then 10°/min to 220°C and held
Injector temperature	225°C
Carrier flow	UHP helium, 30 ml/min
Purge and Trap Conditions	
Purge gas	UHP helium, 40 ml/min
Desorption temperature	180°C
Desorption time	4 min
Oven temperature	200°C
MS Conditions	
Emission	300 μa
Electron energy	70 eV
Scan rate	133.3 amu/sec
Mass interval	45-350 amu

specified. Tetraglyme (tetraethylene glycol dimethyl ether) is similar in chemical and physical properties to polyethylene glycol and is commercially available with fewer interfering contaminants. It has been recommended for extraction of a variety of nonaqueous matrices for volatile organics.[6]

A weighed aliquot (nominally 1 gram) of each waste feed sample will be mixed with 20 ml of precleaned tetraglyme. The tetraglyme/sample mixture will be allowed to equilibrate until phase separation is complete, generally for a period of 12 to 18 hours.

An aliquot of the tetraglyme extract will then be added to 25 ml of deionized water for analysis by purge and trap GC/MS techniques. The size of the tetraglyme aliquot will be determined by calculating the amount required, based on the nominal concentration, to produce a concentration of approximately 100 μg/l of the components of interest in the final tetraglyme/water sample. This concentration represents the midpoint of the linear dynamic range of the mass spectrometer. Each sample will then be spiked with bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane internal standards.

Analysis will be conducted on a Hewlett-Packard 5985 quadrupole mass spectrometer interfaced to a Tekmar LS3 liquid sample concentrator. Instrumental operating conditions are presented in Table 7-3 for these analyses. All analyses will be performed according to Method 624 (see Reference 1) with the following modifications, as routinely implemented at GCA/Technology Division:

- A 25-ml sample will be added to the purging chamber with a 30-ml syringe.
- The concentration of internal standard in each sample will be reduced by a factor of five in order to prevent detector saturation.
- PFTBA, in place of DFTPP or BFB, will be used for mass spectral tuning.

Standard reference materials of all analytes will be used to prepare instrument calibration mixes. Serial dilutions of the components in tetraglyme solution will be spiked into deionized water and analyzed to establish a calibration curve for program samples. The curves will be verified with EMSL standards mixed with tetraglyme on each day of sample analysis. In addition, tetraglyme spiked into deionized water will be analyzed daily as a laboratory blank.

Previously conducted analyses using the above techniques demonstrated method recoveries greater than 80 percent for trichloroethylene and 1,1,2-trichloro-1,2,2-trifluoroethane. Replicate analyses of waste feed samples demonstrated precision of 10 to 20 percent for chlorinated volatile organics. Detection limits for this analysis are generally 10 ppm (mg/kg).

7.2.3 Volatile Organic Sampling Train (VOST)--Flue Gas Samples

As previously mentioned, sampling for the volatile POHCs will be accomplished, primarily, via the VOST. The Tenax and Tenax/charcoal sample generated from each VOST run will be analyzed by thermal desorption GC/MS procedures using a Nutech Model 320 desorption unit. Flow from the desorption unit will be directed through the purge chamber of a Spex/Chromalytics purge and trap sample concentrator onto a Hewlett-Packard 5993 quadrupole mass spectrometer operating under the conditions listed in Table 7-4. Internal standards for this analysis, d_6-benzene and d_8-toluene, will be injected directly onto the adsorbent trap at the midpoint of the desorption period.

Calibration for the volatile POHCs will be achieved by injection, via gas-tight syringe, of varying amounts of a vapor phase stock standard onto blank Tenax tubes under negative pressure. Vapor phase stock standards will be prepared by injecting a known volume of a standard mixture into a 250-ml sampling bulb and allowing the bulb to equilibrate in a 35°C water bath. A minimum of five calibration standards will be analyzed on each day of analysis. Linear regression plots of total nanograms per tube versus response will be made to calculate sample concentrations.

Standard operating procedures in the GCA laboratory require that the HP 5993 GC/MS be tuned daily to criteria established for PFTBA rather than for BFB as specified in EPA Method 624.[5] PFTBA will be continuously bled into the source during instrument parameter adjustment to meet the following ion abundances:

TABLE 7-4. GC/MS INSTRUMENT OPERATING CONDITIONS FOR VOST ANALYSIS

Thermal Desorption Conditions	
Instrument	Nutech Model 320
Cartridge desorption temperature	250°C
Cartridge desorption time	10 min
Desorption flow rate	40 ml/min
GC/MS Conditions	
Instrument	Hewlett-Packard 5993 equipped with a Spex/Chromalytics purge and trap device
Trap Packing	Tenax (60/80 mesh), 3% OV-1 on Chromosorb W (60/80 mesh), silica gel (Davison Grade 15, 35/60 mesh)
Purge and Trap Conditions	
Desorption temperature	180°C
Desorption time	4 min
GC Conditions	
Column	1% SP-1000 on Carbopack B, 6 ft x 2 mm ID column
Temperature program	80°C held for 4 min, then 10°/min to 200°C and held
Injector temperature	225°C
Carrier flow	UHP helium, 30 ml/min
MS Conditions	
Emission	300 μa
Ionization energy	70 eV
Scan rate	133.3 amu/sec
Mass interval	41-350 amu

Mass	Ion Abundance Criteria
69	100 percent
131	25 to 30 percent of mass 69
219	20 to 25 percent of mass 69

Anticipated detection limits for this analysis are approximately 100 ng per train for each component. Precision of ±50 percent as measured by analysis of replicate spiked Tenax tubes is generally attainable for this analysis.

Field-biased blanks and laboratory method blanks will be analyzed with program samples. All Tenax tubes will be subjected to the following preparation procedure prior to use:

- sequential 16-hour extraction with methanol and pentane
- heat treatment of extracted bulk Tenax
- soap/water wash of glass tubes
- 105°C bakeout of glass tubes for 16 hours
- pentane extraction and overnight bakeout (105°C) of glass wool
- two 2-hour heat treatments (250°C) of packed tubes with 15 ml/min helium flow

7.3 CHLORIDE LABORATORY ANALYSIS PROCEDURES

7.3.1 Aqueous Samples

Chloride analysis of Method 5 train impinger/condensate solutions and contaminated water feed will be accomplished via direct injection onto a Dionex Model 14 ion chromatograph (IC). Sample concentrations will be determined by means of standard additions and by comparison of calibration and sample chromatograms. Working standards for chloride (1 to 20 ppm) will be prepared daily from a 1000-ppm stock solution utilizing deionized water as a diluent. Table 7-5 presents the operating conditions of the IC for chloride analysis.

TABLE 7-5. INSTRUMENT OPERATING PARAMETERS FOR IC ANALYSES

Instrument	Dionex Model 14
Conditions	
Columns	Anion precolumn Anion separator column Fiber suppressor column
Injection loop	100 µl
Eluent	0.003M $NaHCO_3$/0.0024M Na_2CO_3
Suppressor regenerating solution	0.025N H_2SO_4
Flow rate	138 ml/hr

Those samples exhibiting a matrix interference for chloride on the IC will be quantified utilizing a Technicon AutoAnalyzer II according to the protocol specified in Method 325.2.[7] 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 will be prepared daily from a 1000-ppm chloride stock solution. Sample concentrations will be determined by comparison of sample percent scale to the established standard curve.

7.3.2 Solid Samples

The total chlorine/chloride content of the fuel feed will be determined as total chloride by Parr bomb combustion followed by ion chromatography (IC) analysis. A 1-gram aliquot of waste oil fuel feed will be oxidized for chlorine/chloride analysis by combustion in a bomb containing oxygen under pressure.[8] The resulting solutions will then be analyzed on a Dionex Model 14 IC using the instrumental conditions presented in Table 7-5 and the calibration procedures outlined for aqueous samples.

7.4 EP TOXICITY--TRACE METALS LABORATORY ANALYSIS PROCEDURES

Ash samples will be subjected to Extraction Procedure (EP) leachate generation as outlined in Reference 3. Sample aliquots will be extracted for a period of 24 hours in an aqueous medium whose pH is maintained at or below 5 using 0.5N acetic acid. The generated leachate will subsequently be analyzed for arsenic, barium, cadmium, hexavalent chromium, lead, mercury, selenium and silver. All metals except hexavalent chromium and mercury will be directly determined using inductively coupled argon plasma emission spectroscopy (ICAP) utilizing a Jarrell-Ash Model 855 Atom Comp.

The Model 855 has a background correction system that compensates for continuum background interference. Spectral overlap is compensated for by established interelement correction factors entered into a PDP-8 computer. The PDP-8 minicomputer performs all management functions, data collection, and analysis. Communication with the ICAP is conducted through a Texas Instrument Silent 733 electronic printer. The printer uses cassette magnetic tapes for loading programs and storing data. In addition, the generated data and pertinent sample statistics are fed to another in-house computer, via the Texas Instrument printer, which is programmed to perform all data reduction functions and final data formatting and printing.

Hexavalent chromium in the leachates will be determined using the coprecipitation method contained in SW-846.[9] This method is based on the separation of Cr (VI) from solution by co-precipitation of lead chromate with lead sulfate in a solution of acetic acid. After separation, the supernatant liquid containing Cr (III) is drawn off and the precipitate is washed to remove any remaining Cr (III). The Cr (VI) is then reduced and resolubilized in nitric acid and quantified as Cr (III) by ICAP.

Mercury analysis of the leachates will be accomplished by means of Atomic Absorption (AA) utilizing the cold vapor technique defined in Reference 7. The Perkin-Elmer MHS-20 hydride generation system in conjunction with a Perkin Elmer Model 2380 AA will be utilized for this analysis. The MHS-20 is a microprocessor controlled system which automatically delivers reductant to the sample reservoir and sweeps the liberated gaseous mercury into a heated quartz cell for quantitation. The typical detection limit utilizing this technique is 0.0005 mg/l mercury.

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 this project.

8.1 DATA REDUCTION

Appendix A of this QA Plan presents the standardized forms that will be used to record sampling and analysis data. All forms will be filled in completely by the technician performing the work then the information will be checked and initialed by at least two other project participants. Figure 8-1 shows the data flow scheme for this project.

8.1.1 Field Data Reduction

Data reduction to be performed in the field is limited to the following information:

- Modified EPA Method 5—Check run for sample volume, moisture, and associated parameters to determine percent isokinetic. These values are hand calculated on standardized calculation forms (Appendix B) or with a TI-59 Programmable Desktop Calculator with Printer.

8.2 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.

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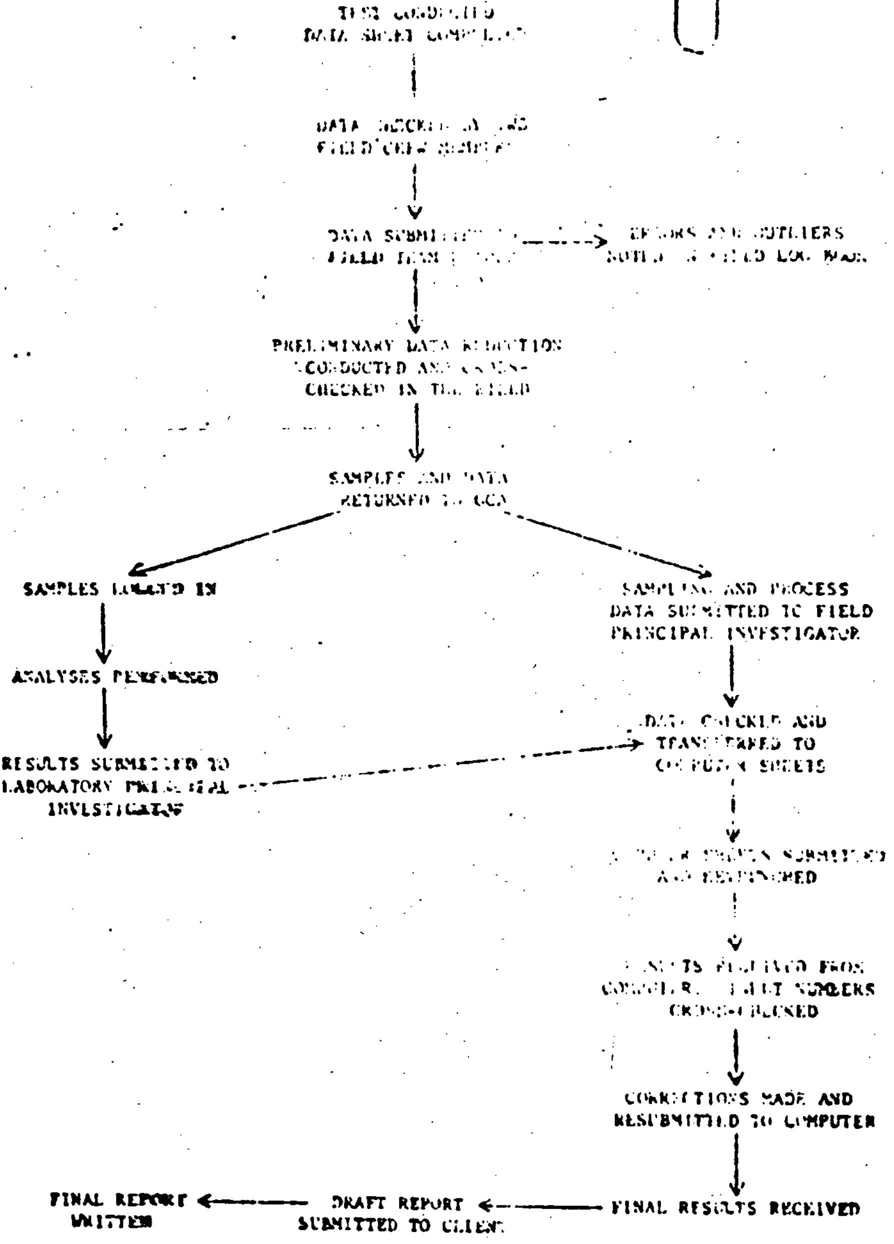

IV-1
Figure ~~I-1~~. Data flow scheme.

8.2.1 Field Data

The following criteria will be used to evaluate sampling data:

- Use of approved test procedures
- Steady state operation of the process being tested
- Use of properly operating and calibrated equipment
- Use of reagents that have passed QC checks
- Leak checks conducted before and after tests
- Proper chain of custody maintained
- VOST Train--Check to assure proper sample gas volume collected.

8.1.2 Laboratory Data

The following criteria will be used to validate laboratory data:

- Use of approved analytical procedure.
- Use of properly operating and calibrated instrumentation.
- Precision and accuracy achieved comparable to that achieved in similar analytical programs.

8.3 DATA REPORTING

All data will be reported in standard units depending on the measurement and the ultimate use of the data. The bulk of the data will be computer processed and reported as follows:

- Particulate emissions
  - Nanograms per joule heat input
  - Grains per standard cubic foot
  - Grains per actual cubic foot

  - Pounds per million Btu heat input
  - Pounds per hour
- HCl emissions
  - Micrograms per cubic meter
  - ppm, actual
  - Pounds per hour
- Organic emissions
  - Micrograms per cubic meter
  - ppm, actual
  - Pounds per hour
- Volumetric flow rate
  - Cubic meters per second, dry basis, standard conditions
  - Cubic feet per minute, dry basis, standard conditions
  - Cubic meters per second, actual
  - Cubic feet per minute, actual
- Gas analyses for each run
  - Carbon dioxide--to nearest 0.1 percent CO_2
  - Oxygen--to nearest 0.1 percent O_2
  - Carbon monoxide--to nearest 1.0 ppm CO
  - Nitrogen--to nearest 0.1 percent N_2
  - Dry molecular weight--to nearest tenth gram
- Liquid grab samples
  - Micrograms per unit volume
- Solid grab samples
  - Weight per unit weight

This data will be used to calculate the destruction removal efficiency, then will be organized into a comprehensive draft final report and submitted to the client for review.

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 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[10] 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 collection, those persons involved in the analysis and collection 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.

9.0 INTERNAL QUALITY CONTROL CHECKS

Quality control checks are performed to ensure the collection of representative samples by using the proper sampling techniques and the generation of valid analytical results on these samples. These checks are performed by project participants throughout the program under the guidance of the QA Manager and the Project QC Coordinator.

GCA's QC program for the sampling aspects of this program will include the following:

1. Equipment Calibration--All sampling equipment (dry gas meters, pitot tubes, thermocouples, etc.) will be calibrated as previously described in this QA Plan.
2. Use of Designated Sampling Forms--Sample data forms are developed for all methods and are completed by personnel collecting the sample to ensure that all pertinent information is recorded.

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. Field-biased blanks are routinely used when sampling for volatile organics.
    - b. Method Blanks--Blanks which are processed through the sample preparation procedures to account for contamination introduced in the laboratory. One method blank is prepared with each batch of 20 or fewer samples processed.
    - 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. At least one sample in each analysis batch of 20 or fewer samples is analyzed in duplicate.

3. Laboratory Control Samples--At least one sample in each analysis batch of 20 or fewer samples will be a laboratory control sample (LCS). The LCS may be an NBS Standard Reference Material, an EPA/EMSL quality control sample, or a project sample spiked with the parameters of interest at a level two to three times the detection limit. These samples are carried through the entire preparation and analysis procedure with program samples..

4. Surrogate Spikes--Samples requiring organic analysis are routinely surrogate spiked using a series of deuterated analogues of the components of interest. This is designed to assess the behavior of actual components in individual program samples during the entire preparative and analysis scheme.

The duplicate and spiked samples or reference materials may also be submitted as "blind" QC samples, those which are not recognizable to the analyst. Blind QC samples are prepared by the laboratory QC Coordinator and inserted through the Sample Bank Manager at the time project samples are received.

- 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

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 continuously monitored through the use of an in-line conductivity meter.

10.0 PERFORMANCE AND SYSTEM AUDITS

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 correction action.

10.1 PERFORMANCE AUDITS

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

In this program, 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 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.

Performance audits of the field sampling equipment are made with the assistance of EPA/EMSL who provide the necessary audit materials and devices during regularly scheduled interlaboratory performance audits. Specific to this project will be a performance audit of the dry gas meters scheduled during the project. Results will be included in the final report.

10.2 SYSTEM AUDITS

System audits are indepth qualitative checks of the program to ensure elements outlined in the QA Plan are functioning. Whether a system audit is made, and how detailed it is, is determined by the size and scope of the project. It is not anticipated that a system audit, performed by the QA Manager, will be required during this program. Should difficulties arise during the program, however, this type of system audit may be conducted.

Smaller, less formal, system audits may be made by the Field and Laboratory QC Coordinators. For example, before the field crew leaves GCA, the Field QC Coordinator will conduct an audit to check on:

- Selection and use of properly calibrated equipment
- Use of filters that have been weighed and clearly identified
- Use of required safety equipment
- Availability of the standardized data forms

10.3 EXTERNAL AUDITS

GCA will cooperate fully in any system or performance audits conducted or arranged by the client, the State of Maine, or EPA Region I.

11.0 PREVENTIVE MAINTENANCE

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 help to assure accurate measurements from field and laboratory instruments.

The Sampling and Field Measurements QC Manual covers equipment such as Hi-Vol Samplers, impinger sampling trains, wet and dry gas meters, and ambient and source monitors. Operational checks, maintenance and calibration procedures are given in this volume.

In the analytical laboratories, preventive maintenance includes attention to glassware, water supply, reagents, analytical balances as well as more complex instrumentation. Technology Division's quality control procedures for these components are detailed in The Analytical QC Manual. Instrument maintenance and calibration procedures are included.

Specific preventive measures to be employed for this program include the use of maintenance procedures from GCA's QC Manuals, and EPA's QA Handbook[4] for Reference Method equipment, and from the operating manual for continuous monitors. Steps will be taken to ensure an adequate inventory of spare parts for reference method testing equipment, continuous monitoring equipment, and the field gas chromatographs. Tables 11-1 and 11-2 summarize the preventive maintenance performed on major equipment used during this program.

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

Equipment	Maintenance procedure/frequency	Spare parts
Vacuum system	• Before and after each sample trip: 1. Check oil and oiler jar 2. Leak check 3. Vacuum gauge functional	Additional meter box
	• Yearly or as needed: 1. Replace valves in pump	
Manometer	• Before and after each sample trip: 1. Leak check 2. Check fluid for discoloration or visible matter	Spare fluid
	• Yearly or as needed: 1. Disassemble and clean 2. Replace fluid	
Dry gas meter	• Before and after each sample trip: 1. Check meter dial for erratic rotation	
	• Every 3 months: 1. Remove top plate and check for excessive oil or corrosion 2. Disassemble and clean	
Nozzles	• Before and after each test: 1. No dents, corrosion or other damage	
Diaphragm pump	• Before and after each test: 1. Leak check; change diaphragm if needed	
	• Yearly or as needed: 1. Disassemble and clean	
Rotameter	• Before and after each test: 1. Observe for erratic behavior; clean if needed	
	• Every 3 months: 1. Clean according to manufacturer's instructions 2. Recalibrate	
Miscellaneous		Fuses, fittings Variable transformers

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

Instrument	Maintenance procedure/schedule	Spare parts
Jarrell-Ash Model 855 Inductively Coupled Plasma Spectrometer	1. Clean optical surfaces--weekly or as needed. 2. Clean torch assembly when discolored or after 8 hours of running high dissolved solids samples.	Spare torch
Perkin-Elmer 460 Atomic Absorption Spectrophotometer with MHS 20 Mercury-Hydride System	1. Clean optical surfaces including cell windows weekly. 2. Periodically rinse immersion tube in dilute HCL; rinse reaction flask with dilute HNO^3. 3. Purge reductant transport system daily. 4. Clean flashback arrestor as needed.	1. Fuses 2. Flange gasket 3. Transfer hose.
Dionex Model 14 Ion Chromatograph	1. Check all valves and column fittings for leaks daily. 2. Check all air and liquid lines for crimping or discoloration weekly. 3. Oil instrument pumps monthly.	1. Millipore filter unit for syringes 2. Anion precolumn 3. Plastic syringes
Hewlett Packard 3920B	1. Change septa daily. 2. Check syringe for burrs daily. 3. Change gas line dryers quarterly. 4. Leak check when installing new analytical column. 5. Periodically check inlet system for residue buildup.	1. 10 µl syringes 2. Inlet septa
Hewlett-Packard 5993 GC/MS	1. Replace pump oils annually. 2. Change septa daily. 3. Change gas line dryers quarterly. 4. Replace electron multiplier as needed.	1. Syringes 2. Septa

12.0 ASSESSMENT OF DATA PRECISION, ACCURACY AND COMPLETENESS

12.1 PRECISION ESTIMATES

12.1.1 Reference Method Tests, Process Parameter and Analytical Measurements

Replicate samples will be collected and analyzed for each pollutant or process parameter measurement system. The standard deviation of these replicate measurements will be used to estimate their precision. The following equation will be used:

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

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:

$$RSD = 100 \frac{S}{\overline{X}}$$

where

RSD = relative standard deviation, expressed in percent

S = standard deviation

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

12.1.2 Continuous Monitor Measurements

Precision will be estimated from replicate measurements of the monitor response to zero calibration gas and the calibration gas concentration closest to that pollutant concentration in the gas stream being measured.

The difference between the current response and the previous value of each calibration gas will be calculated using the following equation:

$$X_i = C_m - C_s$$

where

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

C_m is the current concentration in metric units

C_s is the previous concentration in metric units.

Next, the arithmetic mean of the individual differences will be calculated for the zero difference and the span difference columns maintaining the positive or negative signs.

$$\bar{X} = \frac{\Sigma \; X_i}{n}$$

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:

$$CI_{95} = \frac{t_{.975}}{n(n-i)} \sqrt{n \; \Sigma\left(x_i^2\right) - \; \Sigma\left(x_i\right)^2}$$

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

Precision will be calculated as follows:

$$\% \text{ precision} = \left(\frac{\bar{X} + CI_{95}}{R}\right) \times 100$$

where

R is the instrument range setting in engineering units for the zero precision calculation, and equal to the calibration gas concentration when calculating the precision near the gas stream pollutant concentration.

12.2 ACCURACY ESTIMATES

For each pollution measurement system used in this program, measurements will be made on samples whose true values are known to QA/QC personnel but not to the person making the measurement. Examples of this activity include:

- Analyses of audit samples of unknown concentration
- Collection and analysis of blanks
- Weighing of filters and Class S standards of known weight

Accuracy will be expressed as percent recovery or as relative error. The formulas given below will be used to calculate these values.

$$\text{Percent Recovery} = 100 \; \frac{\text{Measured Value}}{\text{True Value}}$$

$$\text{Relative Error} = 100 \; \frac{\text{Measured Value} - \text{True Value}}{\text{True Value}}$$

12.3 COMPLETENESS

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

$$C = 100 \frac{V}{T}$$

where

C = Percent completeness

V = Number of measurements judged valid

T = Total number of measurements

13.0 CORRECTIVE ACTION

Perhaps the single most important part of any quality assurance program is a well-defined, effective policy for correcting quality problems. GCA/Technology Division maintains a closed-loop corrective action system under the direction of the QA Manager with full management support. While the entire quality assurance program operates to prevent problems, it also serves to identify and correct those that may exist. Usually these quality problems require either on-the-spot, immediate corrective action or long-term corrective action.

Specific QC procedures and checklists are designed to help field technicians and analysts detect the need for corrective action. Often the person's experience will be most valuable in alerting the operator to suspicious data or malfunctioning equipment. Planned corrective actions taken as a part of standard QC procedures are summarized in Section 9.0.

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 most 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.

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

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 this 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.

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.

Figure 13-1 shows the sequence of activities in the loop which is designed to assure action and follow-up on all reported problems.

Documentation of the problem is important to the system. A Corrective Action Request Form is filled out by the person finding the quality problem. This form identifies the problem, possible causes and the person responsible for action on the problem. The responsible person may be an analyst, field team leader, project QA 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 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.

The QA manager maintains an active follow-up file, filing the QA forms in date order. If the follow-up on the indicated date shows a need for other action or continued follow-up, the action to be taken is identified on the form and it is filed under the date for the next follow-up.

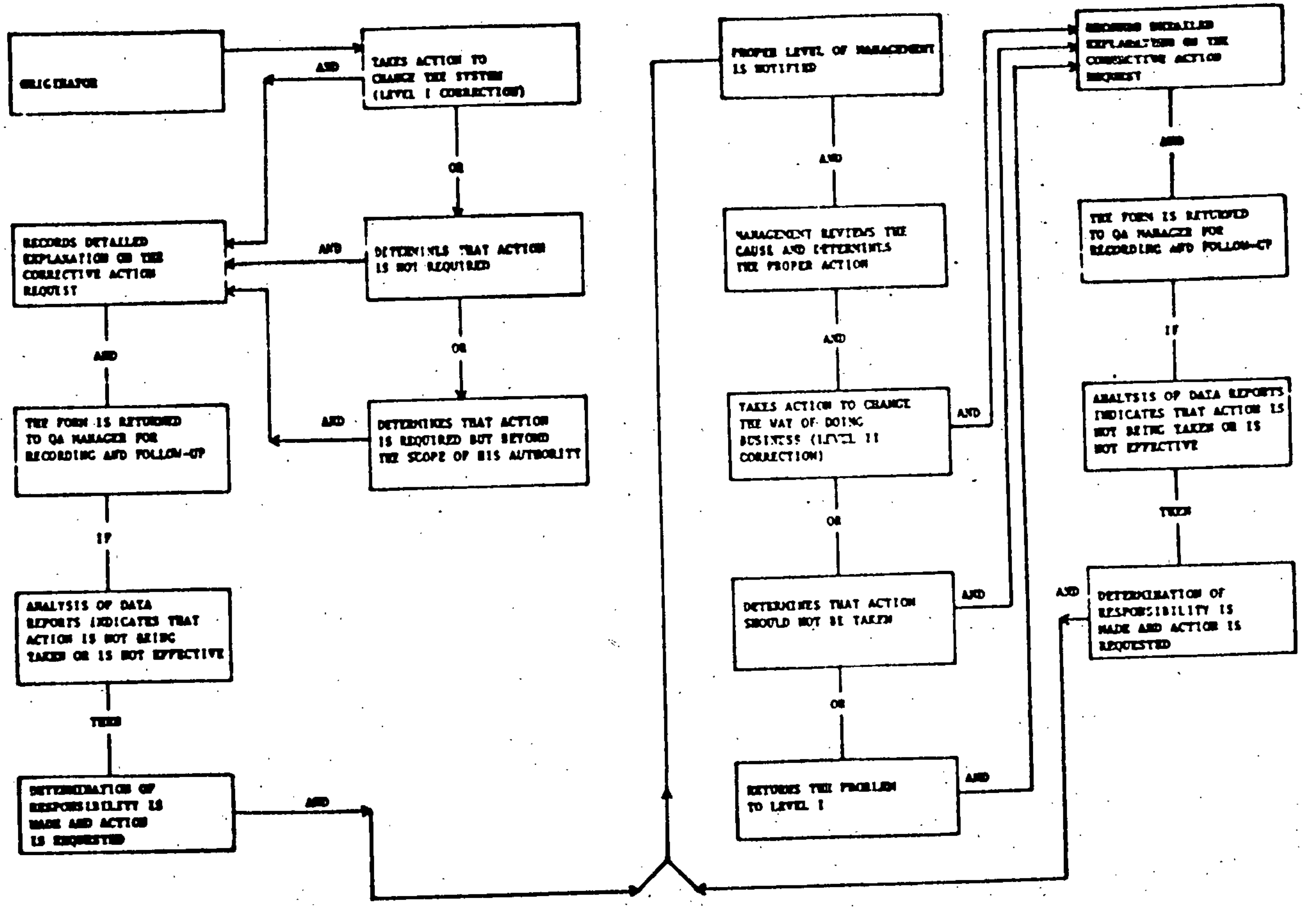

Figure 13-1. A closed-loop corrective action system.

This system has proved quite effective in handling sequential types of corrective action since it brings the QA form to the QA Manager's attention at a time appropriate to check on the next stage of corrective action. The same form can follow a problem until it has been solved.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

14.1 INTERNAL REPORTING

Each department QC Coordinator reports to his department manager and the QA Manager routinely, and on any quality problem or need. The QA Manager will check with the QC Coordinators involved in this project on an ad-hoc basis in addition to the regular monthly review conferences she holds with them individually to discuss department concerns.

The Environmental Measurements Department (Field) QC Coordinator maintains QC records such as instrument calibration records, results on known and blind QC samples and interlaboratory studies.

The Laboratory Analysis Department QC Coordinator maintains control charts on instrument calibration, and performance on known and blind QC samples. She directs the preparation and inclusion of QC samples, blanks, and duplicates in the project sample load in a fashion unrecognizable to the analyst. She will review all routine laboratory QC results germane to this project and initiate corrective actions as needed. These results, problems found, and actions taken may be included in her monthly written report to the Department and QA Managers.

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 THE CLIENT

The Field and Laboratory QC Coordinators will prepare sections for the final report summarizing QC activities on this project. This section may address any or all of the following topics:

- Estimates of precision, accuracy and completeness of reported data
- Results of performance audits, including pertinent interlaboratory studies

- System audit reports
- Quality problems found
- Corrective actions taken:

The QA Manager will aid in preparation and review of the Final Report summarizing QA/QC activities appropriate to this program.

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APPENDIX A

DATA SHEETS

GCA TECHNOLOGY DIVISION

PRELIMINARY FIELD DATA

STACK GEOMETRY & GAS VELOCITY DATA

Page 1 of

Job No.

Client

Plant

Sample Location

Date

Test Type

Operating Conditions

Operator(s)

Pitot Type, No. /Calib=

Flue Draft, In. H_2O

Barometer, In. Hg.

Flue Dimensions, Dia.

Length

Width

Wall Thickness, inches

Area Sq Ft, inches

Time (24 hr. clock)	Sample point	Stack temp. °F or °C	Manom. reading in. H_2O	Cyclonic flow null angle

LOCATION OF SAMPLING POINTS

Pt	% from wall	Dist from wall	Nipple inches	Total dist. from wall inches
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

GCA/TECHNOLOGY DIVISION

NOMOGRAPH SETUP DATA SHEET

CLIENT ____________________

GCA WORK ORDER NO. ____________________ DATE: __________

PLANT ____________________

SAMPLING LOCATION ____________________

METER BOX NO. ________ METER BOX ΔH@ ________

MOISTURE VOL.% ________ BAR. PRESSURE (PB)(PM) ________ in Hg

STATIC PRESSURE SAMPLING POINT (PST) ________ in. $H_2O \times \frac{1}{13.6}$ = ________ in. Hg

PRESSURE STACK (PS) PB±(PST) = ______ ______ = ________ in. Hg

$PS/_{PM}$ = ______ = ______ PITOT TUBE NO. ______

C_p = ________ ; $(C_p/0.85)^2 = (\quad)^2$ = ______ Cpc ; Cc = C x cpc

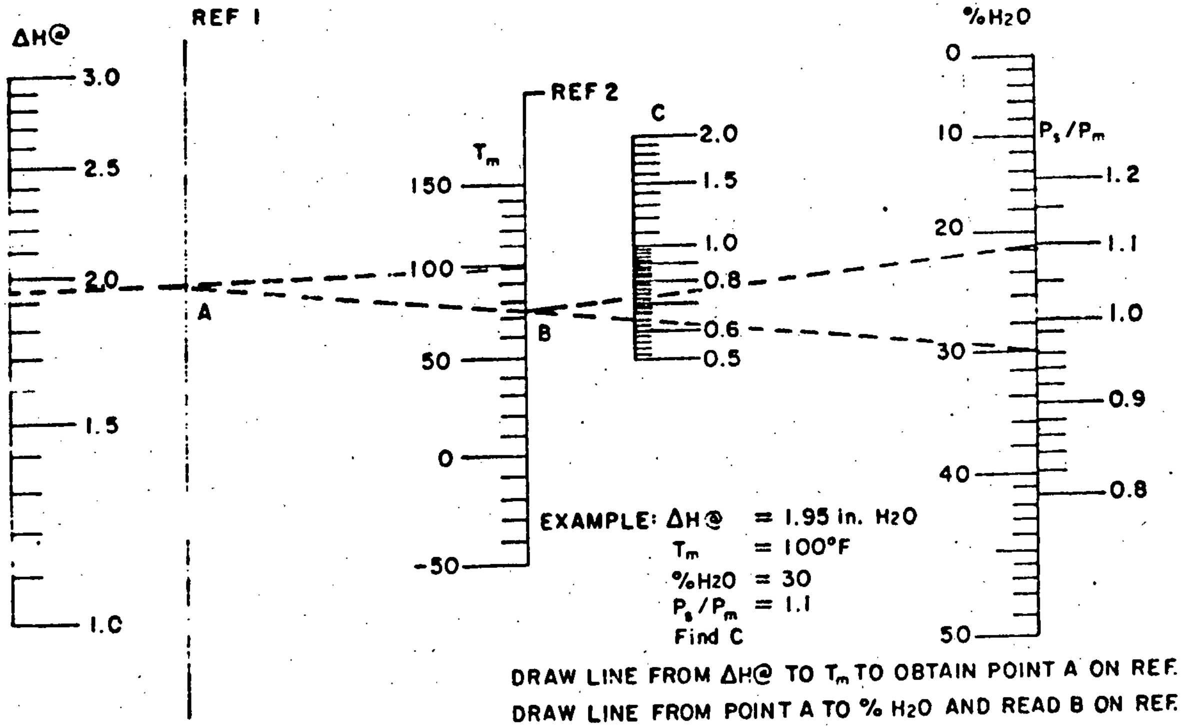
Δp LOW ________ ; Δp AVERAGE ________ ; Δp HIGH ________

NOZZLE DIAMETER ________ in. ; TS min ______ TS avg ______ TS high ______

K FACTOR CHART

C										
CC										
TM TS										

K FACTOR REFERENCE ON Δp SCALE



EXAMPLE: ΔH@ = 1.95 in. H_2O
T_m = 100°F
%H_2O = 30
P_s/P_m = 1.1
Find C

DRAW LINE FROM ΔH@ TO T_m TO OBTAIN POINT A ON REF. 1.
DRAW LINE FROM POINT A TO % H_2O AND READ B ON REF. 2.
DRAW LINE FROM POINT B TO P_s/P_m, AND OBTAIN ANSWER OF 0.74 FOR C.

GCA TECHNOLOGY DIVISION GCA

PARTICULATE/NONPARTICULATE FIELD DATA CODING FORM
PG 1 of 2

VERY IMPORTANT – FILL IN ALL BLANKS

READ AND RECORD AT THE START OF EACH TEST POINT

SKETCH

CLIENT ____________
PROJECT No ____________
PLANT ____________
RUN No ____________
LOCATION ____________
DATE ____________
OPERATOR ____________
SAMPLE BOX NO. ____________
METER BOX NO. ____________
METER ΔH@ ____________
Y FACTOR ____________

SHEET ____ OF ____
PREPARED BY ____________
PITOT NUMBER and SIDE ____________
PITOT TUBE CP ____________
FILTER No./THIMBLE No ______/______
AMBIENT TEMP °F ____________
BAR PRESS, in. Hg ____________
ASSUMED MOISTURE, % ____________
HEATER BOX SETTING, °F ____________
NOZZLE #/DIA., in. ______/______
PROBE LENGTH ____________
PROBE HEATER SETTING ____________

* FIELDS FOR NONPARTICULATE RUN

1*	5*	11*	21	31	41*		46	51	56				61*	66*	71* 75
POINT	DISTANCE IN INCHES	CLOCK TIME		DRY GAS METER, CF	PITOT in. H_2O ΔP	ORIFICE ΔH in. H_2O		DRY GAS TEMP. °F		PUMP VACUUM IN Hg GAUGE	BOX TEMP °F	IMPINGER TEMP °F	STACK PRESS in H_2O in Hg	STACK TEMP °F °C	NULL ANGLE (DEGREES)
		ACTUAL	RUN			DESIRED	ACTUAL	INLET	OUTLET						
				TOTAL											

COMMENT

METER LEAK CHECK:
BEFORE TEST: ______ CF ______ SEC ______ in. Hg
AFTER TEST: ______ CF ______ SEC ______ in. Hg

PITOT LEAK CHECK ______
ORSAT LEAK CHECK ______

STATIC PRESSURE
PORT ______ ______ ______
in. H_2O ______ ______ ______
in. Hg ______ ______ ______

DRS-1
(REVISED 8/31/79)

GCA/TECHNOLOGY DIVISION GCA

PARTICULATE/NONPARTICULATE FIELD DATA CODING FORM
PG. 2 of 2

SHEET _____ OF _____

PREPARED BY ____________________

CLIENT ____________________

PROJECT NO. ____________________

PLANT ____________________

LOCATION ____________________ ________

RUN NO. ____________________

DATE ____________________

1*	5*	11*	21	31	41*	46		51	56				61*	66*	71* 75
POINT	DISTANCE IN INCHES	CLOCK TIME		DRY GAS METER, CF	PITOT in. H_2O ΔP	ORIFICE ΔH in. H_2O		DRY GAS TEMP. °F		PUMP VACUUM IN. Hg GAUGE	BOX TEMP. °F	IMPINGER TEMP. °F	STACK PRESS in. H_2O in. Hg	STACK TEMP. °F °C	NULL ANGLE (DEGREES)
		ACTUAL	RUN			DESIRED	ACTUAL	INLET	OUTLET						
				TOTAL											

METER LEAK CHECK DURING TEST:		METER READING STOP	METER READING START
____ CF ____ SEC ____	in. Hg	____	____
____ CF ____ SEC ____	in. Hg	____	____

STATIC PRESSURE				
	PORT	____	____	____
	in. H_2O	____	____	____
	in. Hg	____	____	____

COMMENTS:

* FIELDS FOR NONPARTICULATE RUN

DRS-5
(REVISED 8/31/79)

GCA/TECHNOLOGY DIVISION

SOURCE PARTICULATE SAMPLING
TRAIN ASSEMBLY, RECOVERY AND ANALYTICAL REPORT SHEET

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

Thimble No.	Lab No.	Weight Results
		mg
		mg
		mg
		mg

Thimble particulate weight____________mg

Filter No.	Lab No.	Weight Results
		mg
		mg
		mg
		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 0.22u 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 Analyzed by:____________

GCA/TECHNOLOGY DIVISION

PARTICULATE ANALYSIS

Lab No. ____________ WO No. ____________

Client ____________ Date Received ____________

Description ____________

A. SAMPLE VOLUME

______ 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		g
Filter		g
Thimble		g
Total		g

CONTAINER NO. ______

0.22/0.45 μ FILTER NO. ______

E. CROSS WEIGHTS

RH/°F	Date/Time			RH/°F	Date/Time		
/	/	(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: ____________

Analyst ____________

5/79

GCA/TECHNOLOGY DIVISION GCA

GASEOUS SAMPLING IMPINGER TRAIN

Client: ______________________ Personnel: ______________________

Location: ______________________ Sample location: ______________________

W.O. No. ______________________ Date: ______________________

Barometric Pressure: ______________________

Pollutant and Run No.						
Time	F.					
	S.					
	T.					
Gas Meter	F.					
	S.					
Total ft.³						
Vacuum In. Hg.	F.					
	S.					
	Av.					
Inlet Dry Gas Meter Temp. °F.	F.					
	S.					
	Av.					
Outlet Dry Gas Meter Temp. °F	F					
	S					
	Av.					
Impinger Volumes Sol'n A	F					
	S					
	Net.					
Impinger Volumes Sol'n B	F					
	S					
	Net.					
Silica Gel gms	F					
	S					
	N					
Filter or Thimble #						

NOTES: ______________________

____

____

____

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: ____________

Sampling Task: ____________

Contract Number: ____________

Anticipated Sampling Date ____________

Site Identification ____________

Field Sample Code	Sample Description	Container	Preservative	Reagent/Grade (Manufacturer and Lot Number)	Laboratory Comment

Submitted by ____________

Date ____________

Page ______ Of ______

Follow-up Dates ____________

Final Laboratory Approval ____________

Date ____________

GCA, Tech. Div. 2/80

SOLID AND GASEOUS SAMPLES

Project No.	**Sample Site:**
Field Team Leader: (signature)	

Sample Code	**Date**	**Sample Description**	**Volume, ml**	**Filter Number**	**pH**					**Remarks**

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

GCA CORPORATION
Technology Division

SAMPLE IDENTIFICATION LABEL

GCA CORPORATION
Technology Division

Field Log No. ☐

Run No. ______

Client ______

Pollutant ______

Project No. ______

Sample Type and Ident ______

Plant ______

LAB NO. ☐

Sampling Location ______

Dry down Container No. ☐

Preservative (if any ______

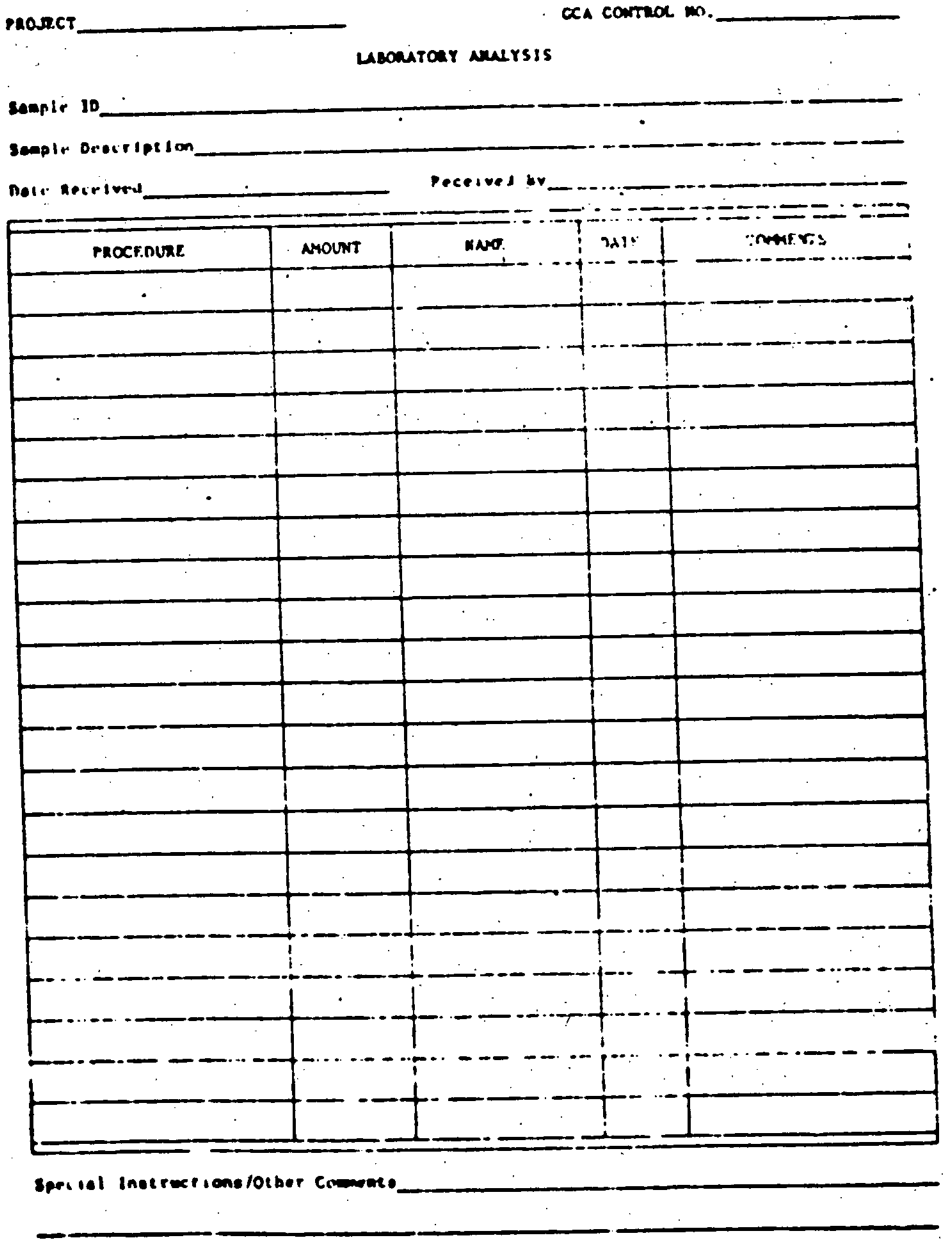
Date ______

Volume ml
Final ______
Initial ______
Net ______

Time ______

Recovered By ______

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



PROJECT__________________ GCA CONTROL NO.______________

LABORATORY ANALYSIS

Sample ID______________________________________

Sample Description___________________________________

Date Received________________ Received by___________________

PROCEDURE	AMOUNT	NAME	DATE	COMMENTS

Special Instructions/Other Comments________________________________

____

Laboratory custody record.

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 CCA 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, receipient, and Task Manager.

RETURN ORIGINAL TO PROJECT FILE

RECEIPT OF SAMPLES FOR ANALYSIS

Received from ______________________________

of GCA/Technology Division, Bedford, Massachusetts, on ____________________.

the following samples for analysis as specified:

GCA Control No.	Analysis	GCA Control No.	Analysis

Signed ____________________

Title ____________________

Laboratory ____________________

Date ____________________

GCA Reference ____________________

GCA Purchase Order No. __________

Rev. 10/81

Interlaboratory custody transfer.

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, notify QA Manager immediately. Submit all CA forms to QA Manager for initial approval of CA.)

State Date, Person, and Action Planned:

CA Initially Approved By: ______________ Date: ______________

Follow-up Dates ______________

Final CA Approval By: ______________ Date: ______________

Information copies to:

RESPONSIBLE PERSON/DEPARTMENT QC COORDINATOR: ______________

QA MANAGER: ______________

DEPARTMENT MANAGER: ______________

ANALYZERS

DAILY 3-POINT CALIBRATION CHECK

I. Analyzer Data

A. O_2 Analyzer MFG ______________ S/N ______________

Zero Setting ______________ Span ______________

B. SO_2 Analyzer MFG ______________ S/N ______________

Coarse Zero Setting ______________ Fine Zero ________ Span ________

C. CO_2 Analyzer MFG ______________ S/N ______________

Zero Setting ______________ Span ______________

D. H_2O Analyzer MFG ______________ S/N ______________

Zero Setting ______________ Span ______________

II. Data

Run No.	O_2		CO		CO_2		SO_2		Moisture	
	Conc	Reading	Conc	Reading	Conc	Reading	Conc	Reading	Conc	Reading
1										
2										
3										

III. Data Reduction

Calculate calibration equation using least square linear regression.

$Y = mx + b$ where:

Y = concentration v/v

m = slope conc/mv

x = reading in mv

b = intercept concentration v/v

	O_2	SO_2	CO_2	CO	Moisture
m =					
b =					
Corr. coeff. r^2 =					

Analyzers, daily calibration check.

2-HOUR ZERO AND CALIBRATION DRIFT

Analyzer: ______________________ Location: ______________________

Serial No.: ______________________

Measurement Range: ______________________

Data set no	Date	Time		Zero Rd.		Zero drift X_L	Hi-Range Rdg.		Span drift	Calib. drift
		Begin	End	Init.	Fin.		Init.	Fin.		
				A	B	C=B-A	D	E	F=E-D	G=F-C X_L

Arithmetic Mean	$\bar{X}$		$\bar{X}$	
Confidence Interval .	CI_{95}		CI_{95}	
Zero drift			Calibration drift	

Calibrated By ____________________ Barometric Pressure, Pb= _____ in.Hg

Date ____________________ Dry Gas Meter No. ____________

Control Box No. ____________

Orifice manometer setting, ΔH, in. H_2O	Gas volume wet test meter V_w, ft^3	Gas volume dry gas meter V_d, ft^3	Temperature				Time θ, min	γ	ΔH@	Deviation	
			Wet test	Dry gas meter							
			Meter t_w, °F	Inlet t_{di}, °F	Outlet t_{do}, °F	Average t_d, °F				γ	ΔH@
0.5	5										
1.0	5										
2.0	10										
							Average				

Calculations

γ	ΔH@
$\dfrac{V_w P_b (t_d + 460)}{V_d\left(P_b + \frac{\Delta H}{13.6}\right)(t_w + 460)}$	$\dfrac{0.0317\ \Delta H}{P_b\ (t_d + 460)}\left[\dfrac{(t_w + 460)\ \theta}{V_w}\right]^2$

γ = Ratio of accuracy of wet test meter to dry test meter. Tolerance = ± 0.01

ΔH@ = Orifice pressure differential that gives 0.75 cfm of air at 70° F and 29.92 inches of mercury, in. H_2O. Tolerance - ± 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 XFormer _______(Check Operation with Probe or Light)

(All Items Must Be Checked and Initialed)

GCA CORPORATION
Technology Division
GCA

QC Okayed ____________

Dry Gas Meter Calibration form

UNI004

0483

PRETEST NOZZLE CALIBRATION

Date____________ Set No. ______ Calibrated by______________

Nozzle identification number	D_1 mm, (in.)	D_2 mm, (in.)	D_3 mm, (in.)	ΔD, mm, (in.)	D_{avg}

POST-TEST NOZZLE CALIBRATION

Date ____________ Set No. ______ Calibrated by ______________

where:

$D_{1,2,3}$, = 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_{avg} = average of D_1, D_2, D_3.

DUPONT PUMPS

CALIBRATION AND SAMPLING DATA SHEET

Instrument Model ______________________

Pump Serial No. ______________________

Calibration Device ______________________

Date ______________________

Location ______________________

Clock Time Start ______________ Stop ______________

Temperature Initial ______________ °C Final ______________ °C

Atmospheric Pressure Initial ______________ mm Hg Final ______________ mm Hg

Relative Humidity Initial ______________ % Final ______________ %

Vapor Pressure ______________________

Collection Media ______________________

Calibrated by ______________________

A. INITIAL READINGS

Reading No.	Time (sec)	Distance Traveled (ml)	Actual Flowrate, Q_i (ml/min)	Standard Flowrate, Q_i(std) (ml/min)
1				
2				
3				
Average				

B. FINAL READINGS

Reading No.	Time (sec)	Distance Traveled (ml)	Actual Flowrate, Q_f	Standard Flowrate, Q_f(std) (ml/min)
1				
2				
3				
Average				

C. % DIFFERENCE - Initial versus Final Flowrates

% Difference = ______________________ %

D. VOLUME SAMPLED

Volume = ______________________ L @ 25°C and 760 mmHg ______________

Notes: ______________________

APPENDIX B

SAMPLE CALCULATIONS

- According to 40 CFR Part 264, the DRE for each POHC is calculated as:

$$DRE = \frac{W_{in} - W_{out}}{W_{in}} \times 100\%$$

Where:

W_{in} = mass feed rate of one POHC in the waste stream feeding the incinerator, and

W_{out} = mass emission rate of the same POHC present in stack exhaust emissions.

a. Calculations of W_{in} (lb/hr)

$$W_{in} = \frac{C_w \times FR_w}{100}$$

Where:

C_w = Concentration one POHC in the waste, %

FR_w = Mass Feed Rate of waste to the incinerator, lb/hr.

- The stack emission rate of HCl can be calculated from:

$$HCl_{out} = C_{in} \times ER_s \times 1.32 \times 10^{-4}$$

Where: C_{in} = concentration of HCl (as Cl^-) in the stack gas effluent and collected in the impingers.

ER_s = volumetric flow rate of the stack gas in m^3/min.

1.32×10^{-4} = conversion factor from mg/min to lb/hr.

Facility ______________________ Job No. ______________________

Source ______________________ Date ______________________

Run ______________________ Calc/Review ______________________

SAMPLE CALCULATIONS

Particulate Isokinetic Sampling

I. Calculations for stack volume and Isokinetic Ratio

Time	Dry Gas Meter ft^3	Pitot ΔP, in. H_2O	Orifice ΔH, in. H_2O	Dry Gas Temp °F In	Dry Gas Temp °F Out	Stack Static Pressure in. H_2O	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^3 ______________ ft^3

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.

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

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

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

$$= (\qquad) - (\qquad) = \underline{\qquad\qquad} ft^3$$

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

$$TM = \frac{\text{Avg. TMI} + \text{Avg. TMO}}{2} = \underline{\qquad\qquad} {}^{o}F$$

6. PM = Average Orifice Pressure Drop, inches H_2O

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

7. Volume of dry gas sampled at standard conditions,^{a} dscf

$$VMSTD = \frac{528\,(Y)(VM)\left(PB + \frac{PM}{13.6}\right)}{29.92\,(TM + 460)}$$

Y = dry gas meter calibration factor ______________

$$= \frac{528\,(\qquad)(\qquad)(\qquad)}{29.92\,(\qquad)} = \underline{\qquad\qquad} ft^3$$

Job No. ____________________

Run No. ____________________

8. VW = Total Water Collected = gm H_2O Silica gel + ml Imp. H_2O = ml

$$(\qquad) + (\qquad) = \underline{\hspace{3cm}}$$

9. Volume of water vapor at standard conditions, scf

$$VW\ gas = 0.04715 \times VW = scf = 0.04715\ (\qquad) = \underline{\hspace{3cm}}\ ft^3$$

10. Percent moisture in stack gas

$$\%\ M = \frac{100 \times VW\ gas}{VMSTD + VW\ gas} = \frac{100\ (\qquad)}{(\qquad) + (\qquad)} = \frac{(\qquad)}{(\qquad)} =$$

$$= \underline{\hspace{3cm}}\ \%$$

11. Mole fraction of dry gas

$$MD = \frac{100 - \%M}{100} = \frac{100 - (\qquad)}{100} = \underline{\hspace{3cm}}$$

12. Molecular weight of dry stack gas

$$MWD = (\%CO_2 \times \frac{44}{100}) + (\%O_2 \times \frac{32}{100} + \left[(\%CO + \%N_2) \times \frac{28}{100}\right]$$

$$= (\qquad \times 0.44) + (\qquad \times 0.32) + (\qquad \times 0.28)$$

$$= (\qquad) + (\qquad) + (\qquad) = \underline{\hspace{2cm}}\ lb/lb\ mole\ dry$$

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

$$= \frac{(\qquad - \qquad) \times 100}{[(0.264\ (\qquad)] - (\qquad) + 0.5\ (\qquad)}$$

$$= \frac{(\qquad) \times 100}{(\qquad) - (\qquad) + (\qquad)} = \underline{\hspace{2cm}}\ \%$$

13. Molecular weight of wet stack gas

$$MW = MWD \times MD + 18\ (1 - MD)$$

$$= (\qquad)\ (\qquad) + 18(1 - \qquad)$$

$$= (\qquad) + (\qquad) = \underline{\hspace{3cm}}\ lb/lb\ mole\ wet$$

14. AS = Stack Area, square inches

$$Circular, = \left(\frac{Stack\ diameter}{2}\right)^2 \pi = \left(\frac{\qquad}{2}\right)^2 \pi = \underline{\hspace{4cm}}\ sq.\ in.$$

$$Rectangular, = Length \times width = (\qquad)(\qquad) = \underline{\hspace{2.5cm}}\ sq.\ in.$$

15. PS = Stack Pressure, absolute, inches Hg = PB ± AV PST

PST = Stack static pressure

Job No. ____________________

Run No. ____________________

$PST\ in.\ Hg = \frac{PST\ in.\ H_2O}{13.6} = \frac{\qquad}{13.6} = \underline{\qquad\qquad}$ in. Hg

$PS = PB \pm Avg.\ PST = (\qquad)(\qquad) = \underline{\qquad\qquad}$ in. Hg

16. TS = Average Stack Temperature, $\underline{\qquad}\ ^{\circ}F + 460 = \underline{\qquad}\ ^{\circ}R$

$TS = Average\ TS \quad \sqrt{TS_{AV} + 460} = \underline{\qquad\qquad}$

17. $SDE_{AV} = (\sqrt{\Delta P})_{AV} \times \sqrt{TS_{AV} + 460} = (\qquad)(\qquad) = \underline{\qquad\qquad}$

18. Stack gas velocity at stack conditions, afpm

$VS = 5130^{(e)} \times Cp \times Avg.\ (SDE) \times \left[\frac{1}{PS \times MW}\right]^{\frac{1}{2}} = afpm$

Cp = pitot tube coefficient

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

19. Stack gas volumetric flow rate at standard conditions,[c] dscfm

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

$= \frac{528\ (\qquad) \times (\qquad) \times (\qquad) \times (\qquad)}{29.92 \times 144\ (\qquad)} = \underline{\qquad\qquad}$ dscfm

20. Stack gas volumetric flow rate at stack conditions, acfm[d]

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

$= \frac{29.92\ (\qquad)(\qquad)}{528\ (\qquad)(\qquad)} = \underline{\qquad\qquad}$ acfm

21. Percent isokinetics

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

---

[a] Dry standard cubic feet at 68°F (528R) and 29.92 in. Hg.

[b] Standard conditions at 68°F (528R) and 29.92 in. Hg.

[c] Dry standard cubic feet per minute at 68°F (528) and 29.92 in. Hg.

[d] Actual cubic feet per minute

[e] $5130 = 85.5\ \frac{ft}{sec}\left[\frac{(lb/lb\ mole)(in.\ Hg)}{(^{\circ}R)(in.\ H_2O)}\right]^{\frac{1}{2}} \times 60\ sec/min$

[f] $1039 = \frac{29.92\ in.\ Hg}{528\ DegR} \times \frac{144\ in.^2}{ft^2} \times \frac{4}{\pi} \times 100$

Job No. ____________________

Run No. ____________________

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

$= \underline{\hspace{3cm}}\ \%$

II. Calculations for grain loading and emission rates

22. Particulate, gr/dscf

$$\text{gr/dscf} = 0.0154 \times \frac{\text{mg}}{\text{VMSTD}} = \frac{0.0154\ (\qquad)}{(\qquad)} = \underline{\hspace{3cm}}\ \text{gr/dscf}$$

23. Particulate at stack conditions, gr/acf

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

$$= \frac{528\ (\qquad) \times (\qquad) \times (\qquad)}{29.92\ (\qquad)} = \underline{\hspace{3cm}}\ \text{gr/acf}$$

24. Particulate, lb/hr conc. method

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

$= \underline{\hspace{2.5cm}}$ lb/hr

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

$$= \frac{0.132 \times (\qquad) \times (\qquad)}{\pi\left(\frac{\qquad}{2}\right)^2 \times (\qquad)} = \underline{\hspace{4cm}} = \underline{\hspace{2.5cm}}\ \text{lb/hr}$$

26. $\dfrac{\text{lb/hr area} \times 100}{\text{lb/hr conc.}} = \dfrac{(\qquad)}{(\qquad)} \times 100 = \underline{\hspace{2.5cm}}\ \%\ I$

27. Particulate combustion lb/10^6 Btu heat input method

lb/hr = avg. of area and conc. method = ________________

10^6 Btu from fuel flow, steam generation or heat rate = ________________

$$\frac{\text{lb/hr}}{10^6\ \text{Btu hr}} = \frac{(\qquad)}{(\qquad)} = \underline{\hspace{3cm}}\ \text{lb/}10^6\ \text{Btu}$$

28. lb/10^5 Btu F Factor method =

$$\frac{\text{gr/dscf}}{7000} \times F \times \frac{20.9}{(20.9 - \%\ O_2)} = \frac{\qquad}{7000} \times (\qquad) \times \frac{20.9}{[20.9 - (\qquad)]}$$

$$= \frac{\cdot}{7000} \times (\qquad) \times \frac{(20.9)}{(\qquad)} =$$

$= \underline{\hspace{3cm}}$ lb/10^6 Btu

Job No. ____________________

Run No. ____________________

29. Density of stack gas

a. Wet at stack condition $= \text{MW lb/lb mol} \Big/ \left[21.85 \cdot \left(\frac{\text{TS} + 460}{\text{PS}}\right)\right]$

$$= (\qquad) \Big/ \left[21.85 \times \frac{(\qquad)}{(\qquad)}\right] = (\qquad) / (\qquad) = \underline{\qquad}\ \text{lb/lb mole wet}$$

b. Dry at 68°F (528R) and 29.92 in. Hg = MWD/385.6

$$= (\qquad)/385.6 = \underline{\qquad\qquad}\ \text{lb/lb mole dry}$$

30. Exhaust gas flow rate

a. lb/hr dry = QS × 60 × density dry

$$\text{FRS} \quad = (\qquad) \times 60 \times (\qquad) = \underline{\qquad\qquad}\ \text{lb/hr}$$

b. lb/hr wet = QA × 60 × density wet

$$\text{FRA} \quad = (\qquad) \times 60 \times (\qquad) = \underline{\qquad\qquad}\ \text{lb/hr}$$

31. gr/dscf at 12% CO_2 $= \text{gr/dscf} \times \frac{12}{\%\ CO_2}$

$$= (\qquad) \times \frac{12}{(\qquad)} = \underline{\qquad\qquad}\ \text{gr/dscf}$$

32. gr/dscf at 50% excess air $= \frac{100 + \text{EA}}{150} \times \text{gr/dscf}$

$$= \frac{(\qquad) + 100}{150} \times (\qquad) = \underline{\qquad\qquad}\ \text{gr/dscf}$$

33. lb pollutant/1000 lb flue gas at 12% CO_2

wet or dry $= \frac{\text{lb pollutant/hr}}{\text{FRA or FRS}} \times \frac{12}{\%\ CO_2} \times 1000$

$$= \frac{(\qquad)}{(\qquad)}\ \frac{12}{(\qquad)} \times 1000$$

$$= \underline{\qquad\qquad}\ \text{lb/1000 lb}$$

APPENDIX B

PROCESS DATA AND CALIBRATION DATA FOR WASTE FEED AND CONTAMINATED WATER TANKS

Time	Bed	(Upper)	(Lower)	Free Bd	Vapor Space	Reactor	Cyclone	Tower	Stack	Under Bed	Over Bed	[illegible]	[illegible]	Scrubber	Cyclone 1 & 2	Inlet	O₂	Main Fire	OF 1	OF 2	OF 3	OF 4	OF 5	[illegible]
[illegible]	°F	°F	°F	°F	°F	°F	°F	°F	°F	Inches of Water							%	%	—	—	—	—	-	
[illegible]	175	[illegible]	1201	2132	255	1402	1277	192	152	25	-1[illegible]	27	4.2	.5	7½	3½	[illegible]	95	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	196	[illegible]	[illegible]	[illegible]	27	4.6	1.6	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5	10	7	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	1217	[illegible]	[illegible]	[illegible]	1434	190	143	[illegible]	-1[illegible]	30½	4.6	1.5	8	4	[illegible]	[illegible]	[illegible]	5	[illegible]	7	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1457	180	140	25[illegible]	-15	30½	4.6	1.5	8	4	49	[illegible]						
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1575	1455	175	143	25½	-1½	30½	4.6	1.5	8	4	[illegible]	95	5	5	10	7	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	1216	[illegible]	[illegible]	[illegible]	1452	150	144	25½	-13	30½	4.6	1.5	8	4	45	95	5	5	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	2224	1291	1472	150	154	25½	-13	[illegible]	4.6	1.5	8	4	[illegible]	95	5	[illegible]	10	7	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1292	[illegible]	171	154	25	-13	[illegible]	4.8	1.5	8	4	45	95	[illegible]	5	[illegible]	7	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1309	1452	173	[illegible]	[illegible]	-1[illegible]	[illegible]	4.9	1.6	8	4	45	95	5	5	10	7	[illegible]	[illegible]
[illegible]	72	[illegible]	129	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	-13	27	4.7	1.6	[illegible]	4	45	95	5	5	10	7	[illegible]	[illegible]
[illegible]	72	1211	112	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	-1[illegible]	[illegible]	4.7	1.6	8[illegible]	4	47	95	5	5	10	7	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1422	176	[illegible]	21	[illegible]	26	4.9	1.7	[illegible]	4	[illegible]	95	5	5	10	7	[illegible]	[illegible]
[illegible]	7	125	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	154	20½	-1[illegible]	[illegible]	4.9	1.7	[illegible]	4	[illegible]	95	5	[illegible]	10	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	125	[illegible]	[illegible]	[illegible]	1429	165	145	145	-14	24	4.7	1.6	[illegible]	4	[illegible]	[illegible]	5	5	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	70	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1490	159	[illegible]	[illegible]	-17	27	4.7	1.6	7½	4	44	95	5	5	10	7	[illegible]	[illegible]

Fuel Type: #1 Test 3

Time	Water Flow			Scrubber Flush		Tank Levels				Fuel Pump	Fuel Flow	[illegible]	[illegible]	[illegible]	Air Total		Every Two Hours					
[illegible]	[illegible]	GPM	%	PSI	GPM	Inches from Top				—	GPM	[illegible]	[illegible]	%	SCFM	FPM	[illegible]	[illegible]	[illegible]	[illegible]	%	%
[illegible]	[illegible]	[illegible]	54	16	—	—	—	—	—	[illegible]	1.81	[illegible]	14		1700	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—
[illegible]	7	[illegible]	[illegible]	[illegible]	—	16	[illegible]	—	4[illegible]	8	1.92	[illegible]	13½	—	1775	1500	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—
[illegible]	[illegible]	[illegible]	54	16	—	—	—	—	—	8½	1.90	[illegible]	14	—	1725	1500	[illegible]	[illegible]	-0[illegible]	[illegible]	0[illegible]	—
[illegible]	44	[illegible]	54	16	—	17	41½	—	43½	8½	1.90	W	14	—	1730	1500	[illegible]	[illegible]	-00[illegible]	001	034	—
[illegible]	[illegible]	[illegible]	54	16	—	[illegible]	43½	—	[illegible]	[illegible]	[illegible]	W	14	126	[illegible]	1500						
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	[illegible]	—	—	—	9	[illegible]	[illegible]	13		[illegible]	1500						
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	[illegible]	[illegible]	—	45	79	1.98	[illegible]	13[illegible]		[illegible]	[illegible]						
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	[illegible]	—	—	—	[illegible]	1.77	[illegible]	[illegible]		1750	[illegible]						
[illegible]	[illegible]	114	[illegible]	[illegible]	—	17½	57½	—	44½	9	[illegible]	[illegible]	13		1750	1500						
[illegible]	04	[illegible]	54	16	—	—	—	—	—	9½	1.82	W	13½	[illegible]	1750	1500						
[illegible]	[illegible]	[illegible]	54	16	—	18½	57½	—	44	[illegible]	[illegible]	W	13	[illegible]	1750	[illegible]						
[illegible]	[illegible]	[illegible]	54	16	—	—	—	—	—	9½	1.95	[illegible]	13½		1750	1500						
[illegible]	[illegible]	[illegible]	54	[illegible]	—	18	[illegible]	—	[illegible]	9½	1.97	[illegible]	13½	125	1750	1500						
[illegible]	32	17	54	16	—	—	—	—	—	10	1.89	W	13		1750	1500						
[illegible]	52	114	54	16	—	20	58	—	44	10	1.72	W	14	12[illegible]	1750	1500						

CONT. ON BACK PAGE

End of Shift Data:

Total Hours = hours

Total Fuel = gallons

Lime Slurry Conc. = %

CaCl₂ Tank Level = inches from top

RL RSC

TEST 2

Date: 11-2-57

Operator: Dilday

Notes

12:57 2 [illegible] 1:17 [illegible] 1:39 [illegible] fuel tank 139 2:09 [illegible] 2:36 [illegible] fuel nozzle [illegible] 3:09 [illegible] 3:24 [illegible] H2O [illegible] [illegible] 4:01 [illegible] fuel nozzle [illegible] 4:22 [illegible] After test we [illegible] 4 emergency shut down procedures #1 was low [illegible] #2 was [illegible] #4 was low pressure (water) in [illegible] combustion chamber and #3 was low pressure (water) in Quench Tower.

Richard Bowman Head Operator

Union Chemical Co Inc Raymond Esposito Cne

TIME	[illegible]	[illegible] PH	[illegible]	CO	CO2
2:50	5.?3	5.22	-.020	0.01	02.6
3:05	7.07	7.25	-.000	0.03	01.9
3:20	5.32	5.00	.00	0.02	01.2
3:35	7.67	7.34	.000	0.03	00.7
3:50	5.13	7.25	.000	0.02	00.4
4:05	[illegible]	5.66	.000	0.02	00.2
4:20	5.59	5.69	.001	0.03	00.0
4:35	5.42	6.72	.001	0.02	00.1
4:45	5.53	7.75	.001	0.01	00.1

Fuel Type: #1 Test #3

	Under Bed	[illegible]	[illegible]	Bed	[illegible]	Vapor Space	Reactor	Cyclone	Quench Tower	Stack	Under Bed	Over Bed	[illegible]	Scrubber	Cyclone	[illegible]	Inlet	I.D. [illegible]	Main Damper	O.F. 1	O.F. 2	O.F. 3	O.F. 4	O.F. 5	Total
	°F	°F	°F	°F	°F	°F	°F	°F	°F	°F	Inches of Water							%	%	—	—	—	—	—	
[illegible]	[illegible]	[illegible]	1355	[illegible]	[illegible]	1359	1252	150	142	[illegible]	22½	-1½	[illegible]	4.4	1.5	0	2½	44	95	5	5	10	7	[illegible]	[illegible]
.5	2	[illegible]	1372	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	27½	-1½	32	4.5	1.5	8	2½	[illegible]	95	5	5	[illegible]	7	[illegible]	1.14
2	1.2	[illegible]	[illegible]	[illegible]	2159	[illegible]	[illegible]	150	[illegible]	[illegible]	22½	-1½	32	4.5	1.7	8	2½	44	95	5	5	[illegible]	7	[illegible]	[illegible]
1.5	[illegible]	1355	1541	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	24	-1½	32	4.5	1.7	8	2¼	44	95	5	5	[illegible]	7	[illegible]	1.14
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	190	[illegible]	[illegible]	27	-1½	31½	4.5	1.7	[illegible]	[illegible]	42	95	5	5	–	–	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	180	141	[illegible]	27	-1½	31	4.5	1.2	[illegible]	2½	[illegible]	95	5	5	10	7	[illegible]	[illegible]
[illegible]	[illegible]	1354	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	182	141	[illegible]	26	-1½	[illegible]	[illegible]	1.7	[illegible]	2¼	42	95	5	5	10	7	[illegible]	1.14
[illegible]	[illegible]	[illegible]	1372	2262	[illegible]	[illegible]	[illegible]	[illegible]	141	[illegible]	26	-1½	30	4.5	1.7	8	2½	46	95	5	5	10	7	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	2252	[illegible]	[illegible]	[illegible]	[illegible]	140	[illegible]	25	-1½	30	4.5	1.7	8	2½	[illegible]	95	5	5	[illegible]	7	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1310	[illegible]	[illegible]	[illegible]	25	-1½	30	4.5	1.7	0	2½	[illegible]	[illegible]	5	[illegible]	[illegible]	–	[illegible]	[illegible]
[illegible]	1.	1347	1363	[illegible]	2155	[illegible]	[illegible]	[illegible]	140	[illegible]	25	-1½	30	4.5	1.7	0	2½	[illegible]	[illegible]	5	[illegible]	[illegible]	–	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1722	[illegible]	[illegible]	[illegible]	[illegible]	25	-1½	30	4.5	1.7	0	2½	[illegible]	[illegible]	5	5	10	7	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	2247	[illegible]	[illegible]	1327	[illegible]	141	[illegible]	25	-1½	30	4.5	1.7	0	2½	[illegible]	[illegible]	5	5	[illegible]	7	[illegible]	1.1
[illegible]	27	[illegible]	[illegible]	[illegible]	2100	1604	[illegible]	[illegible]	[illegible]	[illegible]	25	-1½	[illegible]	6.5	1.7	8	2½	42	[illegible]	5	5	[illegible]	7	[illegible]	[illegible]
[illegible]	22	1027	[illegible]	2262	2097	1151	1308	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4.5	1.7	[illegible]	[illegible]	[illegible]	[illegible]	5	5	[illegible]	7	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	22	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	25	-1½	30	16.5	1.7	8	2	[illegible]	[illegible]	[illegible]	[illegible]	–	7	[illegible]	[illegible]

Date: 11-1-[illegible]

Operators: [illegible]

Time	Water Flow			Scrubber Flush		Tank levels				Fuel Flow		[illegible]				
	[illegible]	[illegible]	[illegible]			[illegible]	Fuel Tank #1	Fuel Tank #2	Cooling Water							
	[illegible]	GPM	%	PSI	GPM	Inches from top				—	GPM	[illegible]	[illegible]	%	SPM	FPM
[illegible]	71	[illegible]	64	16	—	20½	71½	—	[illegible]	[illegible]	[illegible]	W	[illegible]	[illegible]	1725	[illegible]
[illegible]	94	–	64	12	—	—	—	—	—	[illegible]	1.91	W	17½	–	17[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	65	[illegible]	—	[illegible]	74½	—	[illegible]	[illegible]	[illegible]	W	13	—	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	65	1	—	—	—	—	—	[illegible]	1.59	W	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	[illegible]	77	—	[illegible]	[illegible]	[illegible]	W	12	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	—	—	—	—	[illegible]	[illegible]	W	14	[illegible]	175[illegible]	[illegible]
[illegible]	22	[illegible]	[illegible]	15	—	22	[illegible]	—	27	–	1.51	W	12	1.6	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	—	—	—	—	1.	[illegible]	W	12½	—	[illegible]	[illegible]
[illegible]	[illegible]	10	[illegible]	16	—	[illegible]	[illegible]	—	21	4	[illegible]	W	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	24	[illegible]	1	—	—	—	—	—	[illegible]	[illegible]	[illegible]	[illegible]	12.6	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	16	—	[illegible]	[illegible]	—	35	[illegible]	[illegible]	W	12	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	—	—	—	—	[illegible]	1.79	W	12	[illegible]	2[illegible]	[illegible]
[illegible]	38	12	[illegible]	16	—	[illegible]	74	—	31	4½	1.8	W	14	18.6	[illegible]	[illegible]
[illegible]	[illegible]	12	[illegible]	16	—	—	—	—	—	4½	1.80	W	14	[illegible]	1750	[illegible]
[illegible]	95	[illegible]	95	16	—	24	89½	—	23	4½	1.65	W	14		1750	1500
2227	75	1395	[illegible]	[illegible]	—	24¼	90½	—	31½	4½	1.49	W	13½	17.6	1750	1500

Emission Instruments

[illegible]

Total Hours = [illegible]

Total Fuel = [illegible]

Limestone [illegible] = [illegible]

CaCl₂ Tank Level = [illegible]

RB.

TEST #3

Date: 11-4-[illegible] Operators: R. [illegible] and D. [illegible]

Notes

[illegible] 11:30 added [illegible]

[illegible] 11:04 2 [illegible] 11:15 [illegible] 11:45 added [illegible] 12:36 [illegible] 1:07 [illegible] after testing did 4 emergency shutdowns, same as yesterday [illegible] 1:50 added 1 bag of [illegible]

[illegible] Head Operator

Union Chemical Co. Inc. [illegible]

	pH				
Time	[illegible]	ST	[illegible]	CO	CO_2
[illegible]	7.25	[illegible]	.025	0.07	01.5
[illegible]	[illegible]	7.73	[illegible]	[illegible]	01.5
[illegible]	[illegible]	6.0	[illegible]	[illegible]	01.1
[illegible]	[illegible]	[illegible]	[illegible]	0.05	00.7
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	00.7
[illegible]	7.01	[illegible]	.005	[illegible]	00.4
1.0	[illegible]	[illegible]	[illegible]	[illegible]	00.3
[illegible]	[illegible]	[illegible]	[illegible]	.10	00.2
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	00.1
[illegible]	7.57	[illegible]	.015	[illegible]	00.0
[illegible]	6.74	[illegible]	.015	0.19	[illegible]
[illegible] 24	[illegible]	[illegible]	.025	0.19	[illegible]

TEST 1

Fuel Type: #[illegible]

Time	Under Grid	In Bed (Upper)	In Bed (Lower)	Combust. Chamber	Vapor Space	Reactor	Cyclone	Quench Tower	Stack	Under Bed	Over Bed	Differential	Scrubber	Cyclone Differential	Quench Inlet	Inlet	I.D. Fan	Main Air	O.F. 1	O.F. 2	O.F. 3	O.F. 4	O.F. 5	Pilot
[illegible]	°F	°F	°F	°F	°F	°F	°F	°F	°F	Inches of Water							%	%	—		—	—	—	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1272	277	147	[illegible]	223	-1	[illegible]	1.2	1	5	2	[illegible]	[illegible]	[illegible]		[illegible]	—	—	[illegible]
[illegible]	1972	1236	1230	[illegible]	2011	1277	247	156	147	22	-1	22	1.3	1.5	7	3	[illegible]	45	5	[illegible]	12	[illegible]	[illegible]	[illegible]
[illegible]	175	1255	1230	[illegible]	[illegible]	[illegible]	1140	164	131	27	-13	31	2	1.6	8	4	[illegible]	75	5	2	12	5	[illegible]	[illegible]
[illegible]	15	1257	121	227	[illegible]	1113	1210	169	[illegible]	27	-13	31	0.2	1.6	9	4	[illegible]	[illegible]	5	[illegible]	11	[illegible]	[illegible]	[illegible]
2:30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	177	[illegible]	27	-1	31	0.2	1.6	9	4	[illegible]	[illegible]	5	[illegible]	10	2	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	2229	[illegible]	1652	1265	153	141	27	-1	31	0.2	1.6	8	4	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1251	171	147	27	-1	31	0.1	1.5	7	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	1255	[illegible]	[illegible]	[illegible]	1694	1277	173	151	27	-1	31	0.2	1.5	7	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	1257	1222	[illegible]	2158	[illegible]	1273	175	[illegible]	[illegible]	-1	31	0.2	1.5	[illegible]	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1725	[illegible]	[illegible]	[illegible]	26	-1	[illegible]	0.2	1.5	7	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	171	[illegible]	[illegible]	[illegible]	[illegible]	-13	31	0.3	1.5	7	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1231	175	140	[illegible]	-13	30	0.2	1.5	7	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	179	142	25	-14	29	0.2	1.5	7	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	2176	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	25	[illegible]	[illegible]	0.2	1.5	7	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	25	-13	29	0.2	1.5	7	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	25	[illegible]	29	0.2	1.5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

Date 11/3/83

TIME	Water Flow			Scrubber Flush		Tank Levels: Dirty Water	Fuel Tank	Fuel Tank	Cooling Water	Fuel Feed: Pump Setting	Rate	[illegible]	[illegible]	[illegible]	Air Total		Elapsed Time Hours
[illegible]	[illegible]	GPM	[illegible]	PSI	GPM	Inches from Top				—	GPM	[illegible]	[illegible]	%	CFM	FPM	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	35	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	—	—	—	—	[illegible]	1.7	[illegible]	12	—	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	[illegible]	[illegible]	—	[illegible]	[illegible]	1.7	[illegible]	[illegible]	12	1700	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	—	—	—	—	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]		
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	[illegible]	[illegible]	—	[illegible]	[illegible]	1.8	[illegible]	[illegible]	12	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	1	—	12	[illegible]	—	[illegible]	[illegible]	1.87	[illegible]	[illegible]	12.4	1700	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	[illegible]	[illegible]	—	[illegible]	[illegible]	1.71	[illegible]	14	12.8	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	[illegible]	[illegible]	—	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	[illegible]	[illegible]	—	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	—	—	—	—	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	—	[illegible]	[illegible]	—	[illegible]	7	1.52	[illegible]	[illegible]	12.6	[illegible]	[illegible]	
[illegible]	[illegible]	12	[illegible]	16	—	—	—	—	—	[illegible]	1.9	[illegible]	[illegible]	12.6	1700	1500	
[illegible]	[illegible]	[illegible]	25	16	—	[illegible]	31	—	[illegible]	7.3	1.50	W	[illegible]	12.6	1700	1500	
[illegible]	[illegible]	13	27	16	—	[illegible]	—	—	[illegible]	7½	1.57	W	13½	12.6	1700	1500	
12:[illegible]	[illegible]	[illegible]	45	16	—	15	33½	—	44½	7	1.82	W	CONT. ON NEXT				
[illegible]	[illegible]	13	48	16	—	15½	[illegible]	—	45	7	1.83	W	14 PAGES		1700	1500	

Total Hours = [illegible] hours

Total Fuel = [illegible] gallons

Limestone [illegible] = 1.1 %

Oil Tank level = [illegible] inches

	[illegible]	O.T. [illegible]	[illegible]	CO	CO_2
10:15	2.?7	7.52	-.000	[illegible]	[illegible]
10:30	6.53	2.?0	-.000	[illegible]	[illegible]
10:45	7.1?	6.2?	-.000	[illegible]	[illegible]
11:00	[illegible]	[illegible]	-.000	[illegible]	[illegible]
11:15	7.87	[illegible]	-.000	[illegible]	[illegible]

11/3/83

Date: [illegible]

Operators: [illegible] and [illegible]

TEST

NOTES

[illegible] 7:51

7:30 [illegible] 7:3[illegible] [illegible] 8:27 [illegible]

7:3[illegible] [illegible] [illegible] stopped [illegible] 8:10 [illegible] 8:5[illegible] [illegible]

[illegible] 8:50 [illegible] 9:50 [illegible]

[illegible] 10:09 [illegible] 10:15 [illegible]

[illegible] 10:20 [illegible] 10:30 [illegible]

9:5[illegible] 10:[illegible] [illegible] 12:0[illegible] [illegible] 12:24 [illegible] 3 [illegible]

12:[illegible] Manometer reading [illegible] 2.6 – 2.00 neg

reading on Fisher – Porter Indicator 1.5" – 2" neg

[illegible] Ericson Lead Operator

[illegible] Chemical Co. [illegible] [illegible]

	Sample Ph	QT Ph	[illegible]	CO	CO₂
[illegible]	7.2[illegible]	7.[illegible]	-.001	0.04	00.7
[illegible]	7.[illegible]	7.15	-.001	0.05	00.7
[illegible]	7.27	7.11	-.001	0.05	00.7
[illegible]	7.2[illegible]	7.27	-.001	0.05	00.7

Dirty H2O

Dirty Water...

MASSACHUSETTS ENGINEERING CO., INC.

North Quincy, Massachusetts 02171

Tel. 773-7777
Area Code 617

June 1, 1964

CALIBRATION OF AN 8050 GALLON TANK
8'-0" dia. x 21' [illegible]" long
TANK NO. B-8A

Depth Inches	Capacity Gallons	Depth Inches	Capacity Gallons	Depth Inches	Capacity Gallons
1	15	33	2445	65	5806
2	41	34	2546	66	5904
3	75	35	2649	67	6002
4	115	36	2752	68	6098
5	159	37	2857	69	6193
6	206	38	2961	70	6288
7	261	39	3065	71	6382
8	318	40	3170	72	6475
9	380	41	3274	73	6567
10	444	42	3380	74	6658
11	511	43	3487	75	6748
12	581	44	3594	76	6836
13	654	45	3702	77	6921
14	729	46	3809	78	7005
15	807	47	3918	79	7087
16	884	48	4025	80	7166
17	963	49	4132	81	7243
18	1045	50	4241	82	7321
19	1129	51	4348	83	7396
20	1214	52	4456	84	7469
21	1302	53	4563	85	7539
22	1392	54	4670	86	7606
23	1483	55	4776	87	7670
24	1575	56	4880	88	7732
25	1668	57	4985	89	7789
26	1762	58	5089	90	7844
27	1857	59	5193	91	7891
28	1952	60	5298	92	7935
29	2048	61	5401	93	7975
30	2146	62	5504	94	8009
31	2244	63	5605	95	8035
32	2344	64	5706	96	8050

UNI004

04949

APPENDIX C

FIELD DATA AND CALIBRATION SHEETS

GCA TECHNOLOGY DIVISION GCA

PARTICULATE/NONPARTICULATE FIELD DATA CODING FORM

PG 1 of 2

VERY IMPORTANT – FILL IN ALL BLANKS

READ AND RECORD AT THE START OF EACH TEST POINT

SKETCH

SHEET ____ OF ____

PREPARED BY [illegible]

PITOT NUMBER and SIDE ____

PITOT TUBE CP ____

FILTER No / THIMBLE No GC [illegible] 1

AMBIENT TEMP °F [illegible]

BAR. PRESS., in. Hg [illegible]

ASSUMED MOISTURE, % [illegible]

HEATER BOX SETTING, °F [illegible]

NOZZLE #/DIA., in. [illegible] 1

PROBE LENGTH ____

PROBE HEATER SETTING ____

CLIENT ____

PROJECT No 2468-001

PLANT [illegible]

RUN No 1

LOCATION [illegible]

DATE [illegible]

OPERATOR [illegible]

SAMPLE BOX NO [illegible]

METER BOX NO ____

METER ΔH@ ____

Y FACTOR ____

* FIELDS FOR NONPARTICULATE RUN

*	5*	11*	21	31	41*	46		51	56				61*	66*	71* 75
POINT	DISTANCE IN INCHES	CLOCK TIME		DRY GAS METER CF	PITOT in. H_2O ΔP	ORIFICE ΔH in. H_2O		DRY GAS TEMP. °F		PUMP VACUUM IN Hg. GAUGE	BOX TEMP °F	IMPINGER TEMP °F	STACK PRESS in H_2O in Hg	STACK TEMP °F	NULL ANGLE DEGREES
		ACTUAL	RUN			DESIRED	ACTUAL	INLET	OUTLET						
1		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4	[illegible]	[illegible]		[illegible]	
[illegible]				[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	6	[illegible]	[illegible]		[illegible]	
						[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	[illegible]		[illegible]	
				[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			[illegible]	
				[illegible]			2	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	
				[illegible]	[illegible]		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	
[illegible]		[illegible]		[illegible]											
[illegible]															
			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	6	[illegible]	[illegible]		[illegible]	
								[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	
1					[illegible]	[illegible]	1.[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	
			TOTAL												

COMMENT

METER LEAK CHECK:

BEFORE TEST: ____ CF ____ SEC ____ in. Hg

AFTER TEST: ____ CF ____ SEC ____ in. Hg

PITOT LEAK CHECK ✓

ORSAT LEAK CHECK ✓

STATIC PRESSURE

PORT			
in. H_2O			
in. Hg			

DRS-4

(REVISED 8/31/79)

NOTICE: If the film image is less clear than this notice, it is due to the quality of the document being filmed

UNI004

1050

ECHN OGY DIVISION

PARTICULATE/NONPARTICULATE FIELD DATA CODING FORM
PG. 2 of 2

SHEET 2 OF 2

PREPARED BY AW

CLIENT U.S.

PROJECT NO.

PLANT

LOCATION [illegible]

RUN NO.

DATE [illegible]

1* POINT	5* DISTANCE IN INCHES	11* CLOCK TIME ACTUAL	21 CLOCK TIME RUN	31 DRY GAS METER, CF	41* PITOT in. H_2O ΔP	46 ORIFICE ΔH in. H_2O DESIRED	ORIFICE ΔH in. H_2O ACTUAL	51 DRY GAS TEMP. °F INLET	56 DRY GAS TEMP. °F OUTLET	PUMP VACUUM IN. Hg GAUGE	BOX TEMP. °F	IMPINGER TEMP. °F	61* STACK PRESS in. H_2O in. Hg	66* STACK TEMP. °F °C	71* 75 NULL ANGLE (DEGREES)
-			[illegible]	11[illegible]	[illegible]	[illegible]	[illegible]			[illegible]				[illegible]	
	[illegible]	[illegible]	55	[illegible]											
10	[illegible]			[illegible]	[illegible]		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	59		[illegible]	
11			[illegible]	[illegible]	[illegible]	[illegible]	1.3	[illegible]		1[illegible]	[illegible]	[illegible]		[illegible]	
12					[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	53		[illegible]	
	[illegible]			127.5[illegible]											
	[illegible]														
														1969	
				~~24.356~~	6.7535		16.33								
					13		13							13	
				73.623	=0.5216									=151	
							=1.26								
				[illegible]											
				TOTAL	[illegible]	[illegible]									

METER LEAK CHECK DURING TEST: METER READING STOP START

_____ CF _____ SEC _____ in. Hg _____ _____

_____ CF [illegible] SEC _____ in. Hg _____ _____

STATIC PRESSURE PORT _____ _____ _____

in. H_2O _____ _____ _____

in. Hg _____ _____ _____

* FIELDS FOR NONPARTICULATE RUN

COMMENTS:

DRS-5
(REVISED 8/31/79)

2050

UNI004

GCA/TECHNOLOGY DIVISION

SOURCE PARTICULATE SAMPLING
TRAIN ASSEMBLY, RECOVERY AND ANALYTICAL REPORT SHEET

Run Date: 3 Nov 83
Run No.: 2 1
Sample Box No.:
Operator: A.W.

Client: Union Chem
W. O. No.:
Plant:
Sampling Location: M.W. STACK

FRONT HALF

Laboratory Results

Nozzle and Probe (Cyclone Bypass)-Acetone Wash, Lab No.: 35111 Residue 31.7 mg

Cyclone and Flask-Acetone Wash Lab No.: Residue mg

Thimble No.	Lab No.	Weight Results
		mg
		mg
		mg
		mg

Thimble particulate weight — mg

Filter No.	Lab No.	Weight Results
GE-22	35110	910.04 mg
		mg
		mg
		mg

Filter particulate weight 910.04 mg

FRONT HALF Sub Total 941.74 mg

BACK HALF

Impinger WATER and Water Wash of Impingers Connectors and Back Half of Filter Holder

Lab No.:

Collected on 0.22u 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) 941.74 mg

238
295
533

213
166
379

174
166
08

MOISTURE

Impingers: IMP 1 379ml +28ml rinse
2+3 533 + 98ml rinse

			Silica Gel			
Weight after test:	428.7		266.2	[illegible]
Weight before test:	324.8 A		339.3 D	[illegible]
Net Weight:				
Container No.:	1.	2.	3.	4.

Final Volume Total

Initial Volume Total

Net Volume

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 Analyzed by:

ORSAT DATA SHEET

PLANT [illegible]
SAMPLE LOCATION [illegible]
DATE [illegible]
OPERATOR Clark
CO. NO.

COMMENTS: [illegible]

SAMPLE POINT	TIME	(CO$_2$) READING 1	(O$_2$) READING 2		(CO) READING 3	
			ACTUAL	NET	ACTUAL	NET
	1335	3.6	16.4	12.8		
	[illegible]	[illegible]	[illegible]	14.0		
	[illegible]	3.0	[illegible]	13.4		

O$_2$ reading = Reading 2 - Reading 1 (Actual)

CO reading = Reading 3 - Reading 2 (Actual)

EMD-24(1)

1-3-[illegible]

VOST DATA SHEET

[illegible]

Job No. 2 [illegible]

Source UNION [illegible]

Run No. ~~[illegible]~~ 1

Barometric Pressure ("Hg) 25.64

	Actual Time (hr)	Run Time (min)	Tube Set No.	Leak Check	DGM Volume (ft³)	ΔH "H₂O	Stack Temp °F	Condenser Inlet Temp (°F)	Tenax Inlet Temp (°F)	DGM Inlet Temp (°F)	DGM Outlet Temp (°F)
START	0932	00	2	0.001	504.2[illegible]		275	124	51	63	[illegible]
		5			[illegible]		266	117	50	63	65
		10			[illegible]		26[illegible]	[illegible]	47	64	[illegible]
					[illegible]		[illegible]		47	[illegible]	65
STOP	[illegible]	20	2	0.001	505.177	0.854		VMSTD	15.83	64	
START	[illegible]41	00	3	0.001	505.770		257	[illegible]	5[illegible]	64	66
					505.[illegible]		24[illegible]	[illegible]	47	[illegible]	64
					506.074		258	134	47	64	65
					506.220		26[illegible]	134	49	65	66
	1041	[illegible]	3		506.[illegible]	0.715		VMSTD	20.4 l	64	
START	1120	[illegible]	4		506.950		26[illegible]	[illegible]	[illegible]	65	67
		5			507.105		263	[illegible]	[illegible]	[illegible]	61
		10									
		[illegible]			507.401		264	128	49	[illegible]	
STOP	1149	20			507.452	0.503		VMSTD	14.13 l	67	
	[illegible]		5								

GCA/TECHNOLOGY DIVISION

PARTICULATE ANALYSIS

Lab No. 35107 WO No. 7-465-001

Client Union Chemical Date Received 11/8/83

Description Acetone Blank UC-3-M5-FHB

A. SAMPLE VOLUME

200 mL

B. WASH VOLUME

mL

mL

mL

mL

Total 200 mL

C. BLANK CORRECTION

Sample: mL × g/mL = gm

+

Wash: mL × g/mL = gm

=

TOTAL gm

D. TARE WEIGHTS

	No.	Wt.
Container	WO-102	94.2503 g
Filter	GE-25	.62759 g
Thimble		g
Total		g

CONTAINER NO. WO-102

0.22/0.45 μ FILTER NO. GE-25

E. CROSS WEIGHTS

RH/°F	Date/Time		RH/°F	Date/Time	
/	/	(1) 94.2514 g	/	/	(4) g
/	/	(2) .62769 g	/	/	(5) g
/	/	(3) g	/	/	(6) g

		Filter GE-25
Final Gross Weight	94.2514 g	.62769
Total Tare Weight	- 94.2503 g	- .62759
Residue Weight	.0011 g	.00010
Blank Weight	- — g	—
	.0011 g	.00010

E. NET WEIGHT

Remarks:

Analyst Renée Arrigo

5/79

M White 11/22/83

NOTICE: If the film image is less clear than this notice, it is due to the quality of the document being filmed

UNI004

0506

GCA/TECHNOLOGY DIVISION

PARTICULATE ANALYSIS

Lab No. 35115.　　WO No. 7-468-001

Client Union Chemical　　Date Received 11/8/83

Description Acetone Rinse
UC-1-M5-FH

A. SAMPLE VOLUME

200 mL

B. WASH VOLUME

mL
mL
mL
mL
Total 200 mL

C. BLANK CORRECTION

Sample: ____ mL × ____ g/mL = ____ gm
+
Wash: ____ mL × ____ g/mL = ____ gm
=
TOTAL ____ gm

D. TARE WEIGHTS

	No.	Wt.
Container	WO-101	95.7448 g
Filter	GE-22	~~1.53524~~ g
Thimble		.62510 g
Total		g

CONTAINER NO.
WO-101

0.22/0.45 µ FILTER NO.
GE-22

E. GROSS WEIGHTS

RH/°F	Date/Time		RH/°F	Date/Time	
/	/	(1) 95.7776 g	/	/	(4) g
/	/	(2) 1.53524 g	/	/	(5) g
/	/	(3) g	/	/	(6) g

		filter GE 22
Final Gross Weight	95.7776 g	1.53524
Total Tare Weight	- 95.7448 g	- .62510
Residue Weight	.0328 g	.91014
Blank Weight	- .0011 g	- .00010
	↓ .0317 g	.91004

E. NET WEIGHT

Remarks:

Analyst Renée Arrigo

5/79

11/22

RV. 11-22-83

NOTICE: If the film image is less clear than this notice, it is due to the quality of the document being filmed

GCA TECHNOLOGY DIVISION GCA

PARTICULATE/NONPARTICULATE FIELD DATA CODING FORM

PG 1 of 2

VERY IMPORTANT - FILL IN ALL BLANKS

READ AND RECORD AT THE START OF EACH TEST POINT

SKETCH

CLIENT U.C.
PROJECT No
PLANT [illegible]
RUN No 2
LOCATION
DATE [illegible]
OPERATOR [illegible]
SAMPLE BOX NO
METER BOX NO. [illegible]
METER ΔH@ [illegible]
Y FACTOR [illegible]

SHEET ___ OF [illegible]
PREPARED BY [illegible]
PITOT NUMBER and SIDE
PITOT TUBE CP
FILTER No / THIMBLE No GE 27 1
AMBIENT TEMP °F [illegible]
BAR PRESS., in. Hg [illegible]
ASSUMED MOISTURE, %
HEATER BOX SETTING, °F [illegible]
NOZZLE #/DIA., in. [illegible] 1
PROBE LENGTH [illegible]
PROBE HEATER SETTING

* FIELDS FOR NONPARTICULATE RUN

1* 5* 11* 21 31 41* 46 51 56 61* 66* 71* 75

POINT	DISTANCE IN INCHES	CLOCK TIME ACTUAL	CLOCK TIME RUN	DRY GAS METER, CF	PITOT in. H_2O ΔP	ORIFICE ΔH in H_2O DESIRED	ORIFICE ΔH in H_2O ACTUAL	DRY GAS TEMP. °F INLET	DRY GAS TEMP. °F OUTLET	PUMP VACUUM in Hg GAUGE	BOX TEMP °F	IMPINGER TEMP °F	STACK PRESS in H_2O in Hg	STACK TEMP °F	NULL ANGLE DEGREES
	START	14 21	0 0	128.140	[illegible]	[illegible]	1.3	[illegible]	[illegible]	4	275.	51.		[illegible]	
			10	135.[illegible]	.44	1.7	1.7	66.	81.	5	[illegible]	[illegible]			
			20	143.9	.46	2.	2.1	76.	[illegible]	7	[illegible]				
			[illegible]	[illegible]	[illegible]	2.1	2.1	75.	104.	5	273.	[illegible]			
			40	[illegible]	[illegible]	1.7	1.7	72.	107.	[illegible]	[illegible]	[illegible]		[illegible]	
			[illegible]	[illegible]	[illegible]	1.[illegible]	1.[illegible]	81.	108.	7	[illegible]	55.		[illegible]	
	[illegible]	[illegible]	[illegible]	170.[illegible]											
	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	[illegible]	[illegible]	[illegible]		[illegible]			[illegible]	
			[illegible]	177.4	[illegible]	[illegible]	1.8	77.	94.	10	[illegible]	[illegible]		[illegible]	
				[illegible]		1.[illegible]	1.[illegible]	[illegible]	[illegible]	12	[illegible]	[illegible]		[illegible]	
			[illegible]	[illegible]	.35	1.7	1.[illegible]	[illegible]	[illegible]	12	[illegible]	[illegible]		[illegible]	
				TOTAL											

COMMENT

METER LEAK CHECK:
BEFORE TEST: .01 CF 60 SEC 15 in. Hg
AFTER TEST: ___ CF ___ SEC ___ in Hg

PITOT LEAK CHECK ___
ORSAT LEAK CHECK ___

STATIC PRESSURE

PORT			
in. H_2O			
in. Hg			

ORS-4
(REVISED 8/31/79)

ECHN OGY DIVISION

PARTICULATE/NONPARTICULATE FIELD DATA CODING FORM
PG. 2 of 2

CLIENT

SHEET OF

PREPARED BY

PROJECT NO.

PLANT

LOCATION [illegible]

RUN NO. [illegible]

DATE [illegible]

POINT 1*	DISTANCE IN INCHES 5*	CLOCK TIME 11* ACTUAL	CLOCK TIME 21 RUN	DRY GAS METER, CF 31	PITOT In. H_2O ΔP 41*	ORIFICE ΔH In. H_2O DESIRED 46	ORIFICE ΔH In. H_2O ACTUAL	DRY GAS TEMP. °F INLET 51	DRY GAS TEMP. °F OUTLET 56	PUMP VACUUM IN. Hg GAUGE	BOX TEMP. °F	IMPINGER TEMP. °F	STACK PRESS in. H_2O in. Hg 61*	STACK TEMP. °F °C 66*	NULL ANGLE (DEGREES) 71* 75
			[illegible]	[illegible]	.3	[illegible]	1.[illegible]	[illegible]	1[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	
				[illegible]		[illegible]	1.1	[illegible]	[illegible]	11	[illegible]	[illegible]		[illegible]	
		10.41		[illegible]											
	[illegible]														
	[illegible]														
														156	
				84.611	~~[illegible]~~										
					.5691										
				TOTAL											

METER LEAK CHECK DURING TEST: METER READING STOP START

____ CF ____ SEC ____ in. Hg ____ ____

____ CF ____ SEC ____ in. Hg ____ ____ Final

STATIC PRESSURE PORT ____ ____ ____

in. H_2O ____ ____ ____

in. Hg ____ ____ ____

*FIELDS FOR NONPARTICULATE RUN

COMMENTS:

DRS-5

(REVISED 8/31/79)

6050

UNI004

GCA/TECHNOLOGY DIVISION

SOURCE PARTICULATE SAMPLING
TRAIN ASSEMBLY, RECOVERY AND ANALYTICAL REPORT SHEET

Run Date: 3 Nov 83	Client: U. C.
Run No.: 2	W. O. No.:
Sample Box No.: 2180	Plant:
Operator: AW	Sampling Location:

FRONT HALF

Laboratory Results

Nozzle and Probe (Cyclone Bypass)-Acetone Wash, Lab No.: 35115 Residue 1.4 mg

Cyclone and Flask-Acetone Wash Lab No.: ____ Residue ____ mg

Thimble No.	Lab No.	Weight Results
		mg
		mg
		mg
		mg

Thimble particulate weight — mg

Filter No.	Lab No.	Weight Results
GE-27	35114	679.93 mg
		mg
		mg
		mg

Filter particulate weight 679.93 mg

FRONT HALF Sub Total 681.33 mg

BACK HALF

Impinger WATER and Water Wash of Impingers Connectors and Back Half of Filter Holder

Lab No.:

Collected on 0.22u 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

441.2
260.6
701.8

TOTAL TRAIN

TOTAL WEIGHT (Front & Back) 681.33 mg

MOISTURE

Impingers: INIT 100 1st
100 ~~[illegible]~~ 2nd
100 ~~[illegible]~~ 3rd
Final 362ml + 40ml rinse
642 + 32ml rinse

242
150

276
438
280
704

Silica Gel

Weight after test:	~~[illegible]~~			701.8
Weight before test:	~~[illegible]~~ ~~[illegible]~~	~~[illegible]~~	~~[illegible]~~	678.2g
Net Weight:				
Container No.:	1.	2.	3.	4.

Final Volume Total

Initial Volume Total

Net Volume

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 Analyzed by:

ORSAT DATA SHEET

PLANT Union Chemical

SAMPLE LOCATION 2

DATE 3 Nov 83

OPERATOR Aw

CO. NO.

COMMENTS:

SAMPLE POINT	TIME	(CO_2) READING 1	(O_2) READING 2		(CO) READING 3	
			ACTUAL	NET	ACTUAL	NET
	1705	4.6	20.0	15.4		
	1720	4.4	19.8	15.4		
	1736	4.6	20.0	15.4		

O_2 reading = Reading 2 - Reading 1 (Actual)

CO reading = Reading 3 - Reading 2 (Actual)

EMD-24(1)

[illegible] 11-3-83

VOST DATA SHEET

Flow Meter ≈ 75

Job No.

Source UNION CHEMICAL

Run No. 2

Barometric Pressure ("Hg) 29.64

	Actual Time (hr)	Run Time (min)	Tube Set No.	Leak Check	DGM Volume (CF)	ΔP "WC	Stack Temp	Condenser inlet Temp (°F)	Tenax Inlet Temp (°F)	DGM inlet Temp (°F)	DGM [illegible] Temp (°F)
START	1424	00	7	0.001	508.800		286	100	47	60	62
Explained		13			509.010		245	[illegible]	[illegible]	60	[illegible]
[illegible]		20			509.115		220	122	[illegible]	61	62
[illegible]		30			509.363		216	120	[illegible]	61	62
[illegible] →	*	40	MISSED READING		~~[illegible]~~			~~202~~			~~62~~
		50			509.545		225	124	[illegible]	[illegible]	63
STOP	1524	60	7	0.000	509.716						
					0.916			26.2		62	
[illegible]	1533	00	8	0.001	509.750		261		[illegible]	[illegible]	65
		10			509.910		[illegible]	[illegible]	[illegible]	62	64
		20			510.080		[illegible]	120	[illegible]	[illegible]	64
		30			510.250		240	120	[illegible]	62	64
		40			510.347		[illegible]	119	[illegible]	[illegible]	[illegible]
		50			510.536		[illegible]	120	[illegible]	62	[illegible]
[illegible]	1632	60	8		510.6[illegible]					63.[illegible]	
					0.948			26.5			

GCA/TECHNOLOGY DIVISION

PARTICULATE ANALYSIS

Lab No. 35111 WO No. 7-465-001
Client Union Chemical Date Received 11/3/83

Description Acetone Rinse
UC-2-M5-FH

A. SAMPLE VOLUME
250 mL

B. WASH VOLUME
___ mL
___ mL
___ mL
___ mL
Total 250 mL

C. BLANK CORRECTION
Sample: ___ mL × ___ g/mL = ___ gm
+
Wash: ___ mL × ___ g/mL = ___ gm
=
TOTAL ___ gm

D. TARE WEIGHTS

	No.	Wt.
Container	WC-103	105.3339 g
Filter	GE-27	.63574 g
Thimble		g
Total		g

CONTAINER NO.
WC-103

0.22/0.45 μ FILTER NO.
GE-27

E. GROSS WEIGHTS

RH/°F	Date/Time		RH/°F	Date/Time	
/	/	(1) 105.3380 g	/	/	(4) g
/	/	(2) 1.30577 g	/	/	(5) g
/	/	(3) g	/	/	(6) g

		Filter GE-27
Final Gross Weight	105.3380 g	1.30577
Total Tare Weight	- 105.3339 g	- .63574
Residue Weight	.0041 g	.67003
Blank Weight	- ~~.0011~~ .0014 g	- .00010
	~~.0030~~ g .0027	.66993

E. NET WEIGHT

Remarks:

5/79

Analyst Rande Arrigo

RV. 11-22-83

GCA TECHNOLOGY DIVISION GCA

PARTICULATE/NONPARTICULATE FIELD DATA CODING FORM

PG 1 of 2

VERY IMPORTANT – FILL IN ALL BLANKS

READ AND RECORD AT THE START OF EACH TEST POINT

SKETCH

CLIENT [illegible]
PROJECT No
PLANT
RUN No 3
LOCATION Main STACK
DATE 4 [illegible]
OPERATOR [illegible]
SAMPLE BOX NO
METER BOX NO 21?5
METER ΔH@ 1.[illegible]
Y FACTOR [illegible]

SHEET 1 OF
PREPARED BY [illegible]
PITOT NUMBER and SIDE
PITOT TUBE CP
FILTER No /THIMBLE No 1
AMBIENT TEMP °F 45
BAR PRESS, in. Hg 29.72
ASSUMED MOISTURE, % 30
HEATER BOX SETTING, °F 250
NOZZLE #/DIA., in. .3[illegible] 1
PROBE LENGTH [illegible]
PROBE HEATER SETTING

* FIELDS FOR NONPARTICULATE RUN

1* POINT	5* DISTANCE IN INCHES	11* CLOCK TIME ACTUAL	21 CLOCK TIME RUN	31 DRY GAS METER, CF	41* PITOT in. H_2O ΔP	ORIFICE ΔH in H_2O DESIRED	46 ORIFICE ΔH in H_2O ACTUAL	51 DRY GAS TEMP. °F INLET	56 DRY GAS TEMP. °F OUTLET	PUMP VACUUM IN Hg GAUGE	BOX TEMP °F	IMPINGER TEMP °F	61* STACK PRESS in H_2O in Hg	66* STACK TEMP °F	71* 75 NULL ANGLE DEGREES
1		13.0[illegible]	0.0	253.375	.29	1.4	1.4	51.	51.	8	22[illegible].	5[illegible].		15[illegible].	
2			5.0	256.7	.32	1.7	1.5	62.	54.	1[illegible]	[illegible]	45.		15[illegible].	
3			10.0	[illegible]	.33	1.7	1.5	71.	55.	1[illegible]	[illegible]	46.		155.	
4			15.0	[illegible]	[illegible]	1.6	[illegible]	77.	5[illegible].	16	[illegible]	[illegible]		[illegible]	
5			20.0	[illegible]	[illegible]	1.6	[illegible]	82.	56.	16	[illegible]	[illegible]		[illegible]	
6			25.0	[illegible]	[illegible]	1.8	[illegible]	84.	[illegible]	16	223.	61.		155.	
Stop		13.[illegible]	30.0	[illegible]											
Strt															
1		13 [illegible]5	0.0	273.25[illegible]	.33	1.7	1.7	73.	60.	14	220.	[illegible]		15[illegible].	
2			[illegible]	[illegible]	.31	1.6	1.5	82.	61.	1[illegible]	21[illegible].	[illegible]		15[illegible].	
3			[illegible]	[illegible]	.31	1.5	1.4	84.	61.	[illegible]	[illegible]	[illegible]		154.	
				TOTAL											

COMMENT

METER LEAK CHECK:
BEFORE TEST: .005 CF 60 SEC 15 in. Hg
AFTER TEST: .005 CF [illegible] SEC 15 in. Hg - R[illegible]

PITOT LEAK CHECK
ORSAT LEAK CHECK

STATIC PRESSURE

PORT			
in. H_2O			
in. Hg			

DRS-4
(REVISED 8/31/79)

GCA TECHN[illegible]OGY DIVISION GCA

PARTICULATE/NONPARTICULATE FIELD DATA CODING FORM
PG. 2 of 2

CLIENT Ue[illegible]

SHEET 2 OF 2

PREPARED BY [illegible]

PROJECT NO.

PLANT

LOCATION MAIN STACK

RUN NO. 3

DATE 4 Nov 83

1*	5*	11*	21	31	41*	46		51	56				61*	66*	71* 75
POINT	DISTANCE IN INCHES	CLOCK TIME		DRY GAS METER, CF	PITOT in. H_2O ΔP	ORIFICE ΔH in. H_2O		DRY GAS TEMP. °F		PUMP VACUUM IN. Hg GAUGE	BOX TEMP. °F	IMPINGER TEMP. °F	STACK PRESS in. H_2O in. Hg	STACK TEMP. °F °C	NULL ANGLE (DEGREES)
		ACTUAL	RUN			DESIRED	ACTUAL	INLET	OUTLET						
4			[illegible]	232.4	.31	1.5	1.4	[illegible]	62.	15	273	65		[illegible]	
5			[illegible]	[illegible]	.31	1.5	1.4	66.	63.	16	[illegible]	[illegible]		[illegible]	
6			[illegible]	[illegible]	.27	1.4	1.4	[illegible]	64.	16	[illegible]	[illegible]			
STOP			[illegible]	[illegible]3138											
[illegible]		[illegible]													
				21.540	0.5604									154	
			TOTAL												

METER LEAK CHECK DURING TEST:

	METER READING STOP	METER READING START
____ CF ____ SEC ____ in. Hg		
.01 CF 60 SEC ____ in. Hg		

Final

STATIC PRESSURE
PORT ____ ____ ____
in. H_2O ____ ____ ____
in. Hg ____ ____ ____

COMMENTS:

* FIELDS FOR NONPARTICULATE RUN

DRS-5
(REVISED 8/31/79)

TECHN OGY DIVISION

ORIGINAL 4NOV

CLIENT Union Chemical

PROJECT NO. 7.

PLANT

RUN NO. II 5

PARTICULATE/NONPARTICULATE FIELD DATA CODING FORM

PG. 2 of 2

LOCATION MAIN STACK

DATE 4 NOV 83

SHEET ____ OF ____

PREPARED BY AW

1* 5* 11* 21 31 41* 46 51 56 61* 66* 71* 75

POINT	DISTANCE IN INCHES	CLOCK TIME		DRY GAS METER, CF	PITOT in. H_2O ΔP	ORIFICE ΔH in. H_2O		DRY GAS TEMP. °F		PUMP VACUUM IN. Hg GAUGE	BOX TEMP. °F	IMPINGER TEMP. °F	STACK PRESS in. H_2O in. Hg	STACK TEMP. °F °C	NULL ANGLE (DEGREES)
		ACTUAL	RUN			DESIRED	ACTUAL	INLET	OUTLET						
1		1305	0.0	253 375	.29	1.4	1.4	51	51	8	224	54		157	
2			5.0	256.9	.33	1.7	1.5	65	56	14	225	45		154	
3			10.0	257.6	.33	1.7	1.5	71	55	16	226	46		155	
4			15	263.0	.32	1.6	1.5	79	55	16	225	48		155	
5			20	266.1	.32	1.6	1.5	82	56	16	237	52		154	
6			25	269.3	.32	1.6	1.4	84	58	16	273	61		155	
STOP		1335	30.	273.086											
START															
1			0.0	273.259	.33	1.7	[illegible]	73	60	14	270	[illegible]		[illegible]	
2			5.0	[illegible]6.3	.31	[illegible]	1.5	[illegible]	[illegible]	11	[illegible]	[illegible]		[illegible]	
3			10.0	277.3	.31	1.5	[illegible]	[illegible]	61	15	[illegible]	[illegible]		[illegible]	
4			15	[illegible]	[illegible]	1.5	[illegible]	[illegible]	[illegible]	14	[illegible]	[illegible]		[illegible]	
5			20	[illegible]	.31	1.6	1.4	[illegible]	63	14	[illegible]	66		154	
6			25	[illegible]3	.29	1.4	1.4	[illegible]	64		[illegible]	66		154	
		STOP	30	292.138											
			TOTAL												

METER LEAK CHECK DURING TEST:

METER READING STOP START

005 CF 60 SEC 15 in. Hg ____ ____

.004 CF 60 SEC 17 in. Hg ____ ____ Port CHANGE

.01 6C 27

STATIC PRESSURE PORT ____ ____ ____

in. H_2O ____ ____ ____

in. Hg ____ ____ ____

* FIELDS FOR NONPARTICULATE RUN

COMMENTS:

DRS-5

(REVISED 8/31/79)

GCA/TECHNOLOGY DIVISION

SOURCE PARTICULATE SAMPLING
TRAIN ASSEMBLY, RECOVERY AND ANALYTICAL REPORT SHEET

Run Date: 1 NOV 83
Run No.: 3
Sample Box No.: 2LPO
Operator: ____

Client: ____
W. O. No.: ____
Plant: ____
Sampling Location: ____

FRONT HALF

Nozzle and Probe (Cyclone Bypass)-Acetone Wash,
Cyclone and Flask-Acetone Wash

Laboratory Results
Lab No.: ____ Residue 15.4 mg
Lab No.: ____ Residue ____ mg

Thimble No.	Lab No.	Weight Results
		mg
		mg
		mg
		mg

Thimble particulate weight ____ mg

Filter No.	Lab No.	Weight Results
GE ~~27~~		mg
26		351.54 mg
		mg
		mg

Filter particulate weight 351.54 mg
FRONT HALF Sub Total 366.94 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) 366.94 mg

MOISTURE

Impingers: INIT 100ml D.I
50ml 2nd
50ml 3rd

#1 = 330
[illegible]
Final Volume Total 5[illegible]2
Initial Volume Total ____
Net Volume ____

Silica Gel

Weight after test:				
Weight before test:	~~3[illegible]~~		~~2[illegible]~~ [illegible]	
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 Analyzed by: ____

ORSAT DATA SHEET

PLANT Union Chemical

SAMPLE LOCATION outlet stack

DATE 4 Nov 83

OPERATOR Clark

CO. NO. 7-465-[illegible]

COMMENTS: Run # 3

SAMPLE POINT	TIME	(CO$_2$) READING 1	(O$_2$) READING 2		(CO) READING 3	
			ACTUAL	NET	ACTUAL	NET
	1423	4.4	19.6	15.2		
	1500	4.2	19.4	15.2		
	1518	4.8	18.2	~~[illegible]~~ 13.4		

O$_2$ reading = Reading 2 - Reading 1 (Actual)

CO reading = Reading 3 - Reading 2 (Actual)

EMD-24(1)

GCA/TECHNOLOGY DIVISION

PARTICULATE ANALYSIS

Lab No. 3519 WO No. 7-468-001

Client Union Chemical Date Received 11/5/83

Description Acetone Rinse
UC-3-M5-FH

A. SAMPLE VOLUME

250 mL

B. WASH VOLUME

___ mL
___ mL
___ mL
___ mL
Total 250 mL

C. BLANK CORRECTION

Sample: ___ mL × ___ g/mL = ___ gm
+
Wash: ___ mL × ___ g/mL = ___ gm
=
TOTAL ___ gm

D. TARE WEIGHTS

	No.	Wt.
Container	WC-100	94.5890 g
Filter	GE-26	.62548 g
Thimble		g
Total		g

CONTAINER NO. WC-100

0.22/0.45 μ FILTER NO. GE-26

E. CROSS WEIGHTS

RH/°F	Date/Time		RH/°F	Date/Time	
/	/	(1) 94.6055 g	/	/	(4) g
/	/	(2) .97712 g	/	/	(5) g
/	/	(3) g	/	/	(6) g

		Filter GE-26
Final Cross Weight	94.6055 g	.97712
Total Tare Weight	− 94.5890 g	− .62548
Residue Weight	.0165 g	.35164
Blank Weight	− ~~.0011~~ .0014	.0010
	.0157 g .0154	.35154

E. NET WEIGHT

Remarks:

Analyst

5/79

RV 11/22/83

3519

3

VOST DATA SHEET

Flow meter = .75

Ambient = 47

Job No. ________

Source Union Chemical

Run No. 4

Barometric Pressure ("Hg) 29.72

	Actual Time (hr)	Run Time (min)	Tube Set No.	Leak Check	DGM Volume (CF)	ΔP "WC	Stack Temp	Condenser Inlet Temp (°F)	Tenax Inlet Temp (°F)	DGM Inlet Temp (°F)	DGM Out Temp (°F)
Start	1041	:00	10	0.000	513.010		220	105	45	46	[illegible]
	[illegible]	10			513.130		218	112	[illegible]	47	47
	[illegible]	20									
		30			[illegible]		[illegible]	112		[illegible]	[illegible]
	[illegible]	[illegible]			513.401		210	112	[illegible]	47	47
											47
	[illegible]	50									
Stop	[illegible]	60	[illegible]		[illegible]						
					0.521	VMSTD 10.91					
Start	[illegible]	00	[illegible]		[illegible]		[illegible]				47
		10			[illegible]		[illegible]	[illegible]	[illegible]	[illegible]	47
		20									
		30			513.7[illegible]		214	110	41		47
					513.812		210	[illegible]	42	47	47
		[illegible]			513.9[illegible]		212	2	[illegible]	47	
	9	11			514.0[illegible]						
					0.488		VMSTD 14.4				
			12	Field Blank							

Calibrated By Jeff Bil[illegible]

Date 10-4-83

Barometric pressure, P_b = [illegible] in. Hg

Dry Gas Meter No.

Control Box No. 2140

Orifice manometer setting, ΔH, in. H_2O	Gas volume wet test meter V_w, ft^3	Gas volume dry gas meter V_d, ft^3	Temperature: Wet test meter t_w, °F	Temperature: Dry gas meter Inlet t_{di}, °F	Temperature: Dry gas meter Outlet t_{do}, °F	Temperature: Dry gas meter Average t_d, °F	Time θ, min	γ	ΔH@	Deviation γ	Deviation ΔH@
0.5	5	5.079	72.5	[illegible]	[illegible]	80.0	[illegible]	.798	1.772		
1.0	5	5.164	72.5	[illegible]	[illegible]	91.75	7.26	.777	1.7[illegible]		
[illegible]	10	10.429	72.5	[illegible]	114	98.5	13.40	1.000	[illegible]		
							Average	.790	1.760		

Calculation

γ	ΔH@
$\frac{V_w P_b (t_d + 460)}{V_d \left(P_b + \frac{\Delta H}{13.6}\right)(t_w + 460)}$	$\frac{0.0317\ \Delta H}{P_b (t_d + 460)} \left[\frac{(t_w + 460)\ \theta}{V_w}\right]^2$

γ = Ratio of accuracy of wet test meter to dry test meter. Tolerance = ± 0.01

ΔH@ = orifice pressure differential that gives 0.75 cfm of air at 70° F and 29.92 inches of mercury, in. H_2O. Tolerance = ± 0.15

Maintenance Checklist

[illegible] Oil Reservoir Level ✓; Check oil [illegible] ✓

Vacuum Gage ✓; Leak Chk (No Leak) 15" Hg ✓

Manometer Clean ✓ Lubricate ✓

[illegible] Check for Leaks ✓; Fluid Level ✓; Clean Surface ✓

[illegible] Valve: Check for Click ✓

[illegible] amp Probe Heater [illegible] 7 amp Pump ✓; [illegible] amp Heater ✓

[illegible] Connecter ✓ (Check with umbilical cord connected to hot box)

~~[illegible] Warmer (Check operation with Probe or [illegible])~~

(All Items Must be Checked and Initialed)

GCA Technology Division

[illegible] Okayed

THERMOCOUPLE CALIBRATION FORM

VOST TRAIN DRY GAS METER

	Thermocouple Number	Temperature Reading 1	Reference Temperature 1	Temperature Reading 2	Reference Temperature 2	Temperature Reading 3	Reference Temperature 3
HOT	INLET	193	193	193	192	192	192
WATER	OUTLET	193	193	192	192	192	192
ICE	INLET	33	32	33	32	33	32
WATER	OUTLET	32	32	32	32	32	32
AMB	INLET	74	74	[illegible]	74	74	74
	OUTLET	73	74	73	74	[illegible]	74

Signature of Person Performing Calibration

[illegible]
Calibration Date

Calibrated By A [illegible]

Date 3 Oct 85

Control Box No. ______

Barometric Pressure, Pb = [illegible] in.Hg

Dry Gas Meter No. [illegible]

Orifice manometer setting, ΔH, in. H2O	Gas volume wet test meter V_w, ft3	Gas volume dry gas meter V_d, ft3	Temperature: Wet test Meter t_w, °F	Temperature: Dry gas meter Inlet t_{di}, °F	Temperature: Dry gas meter Outlet t_{do}, °F	Temperature: Dry gas meter Average t_d, °F	Time θ, min	Y	ΔH@	Deviation Y	Deviation ΔH@
.1	1.0	[illegible]	71	[illegible]	[illegible]	[illegible]		[illegible]			
.1	1.0	[illegible]	71	70	[illegible]	[illegible]		[illegible]			
.1	1.0	[illegible]	71	[illegible]	[illegible]	71		[illegible]			
						Average		[illegible]			

Calculations

Y	ΔH@
$\frac{V_w P_b (t_d + 460)}{V_d \left(P_b + \frac{\Delta H}{13.6}\right)(t_w + 460)}$	$\frac{0.0317 \Delta H}{P_b (t_d + 460)} \left[\frac{(t_w + 460)\theta}{V_w}\right]^2$

- Ratio of accuracy of wet test meter to dry test meter. Tolerance = ± 0.01
- Orifice pressure differential that gives 0.75 cfm of air at 70° F and 29.92 inches of mercury, in. H2O. Tolerance = ± 0.15

Maintenance Checklist

[illegible] Oil Reservoir Level __ ; Checkout for [illegible]

Vacuum Gage __ ; Leak Chk (Set to [illegible]) 15" Hg

[illegible]: Clean __ Lubricate __

[illegible] ; Fluid Level __ ; Clean Surface __

[illegible]: Check for Click

2.5 amp. Probe Heater __ ; 7 amp. Pump __ ; 10 amp. Heater __

Connector __ (Check with umbilical cord connected to hot box)

[illegible] XFormer __ (Check Operation with [illegible])

(All) Items Must Be Checked and Initialed

GCA Technology Division [illegible] Bedford

THERMOCOUPLE CALIBRATION FORM

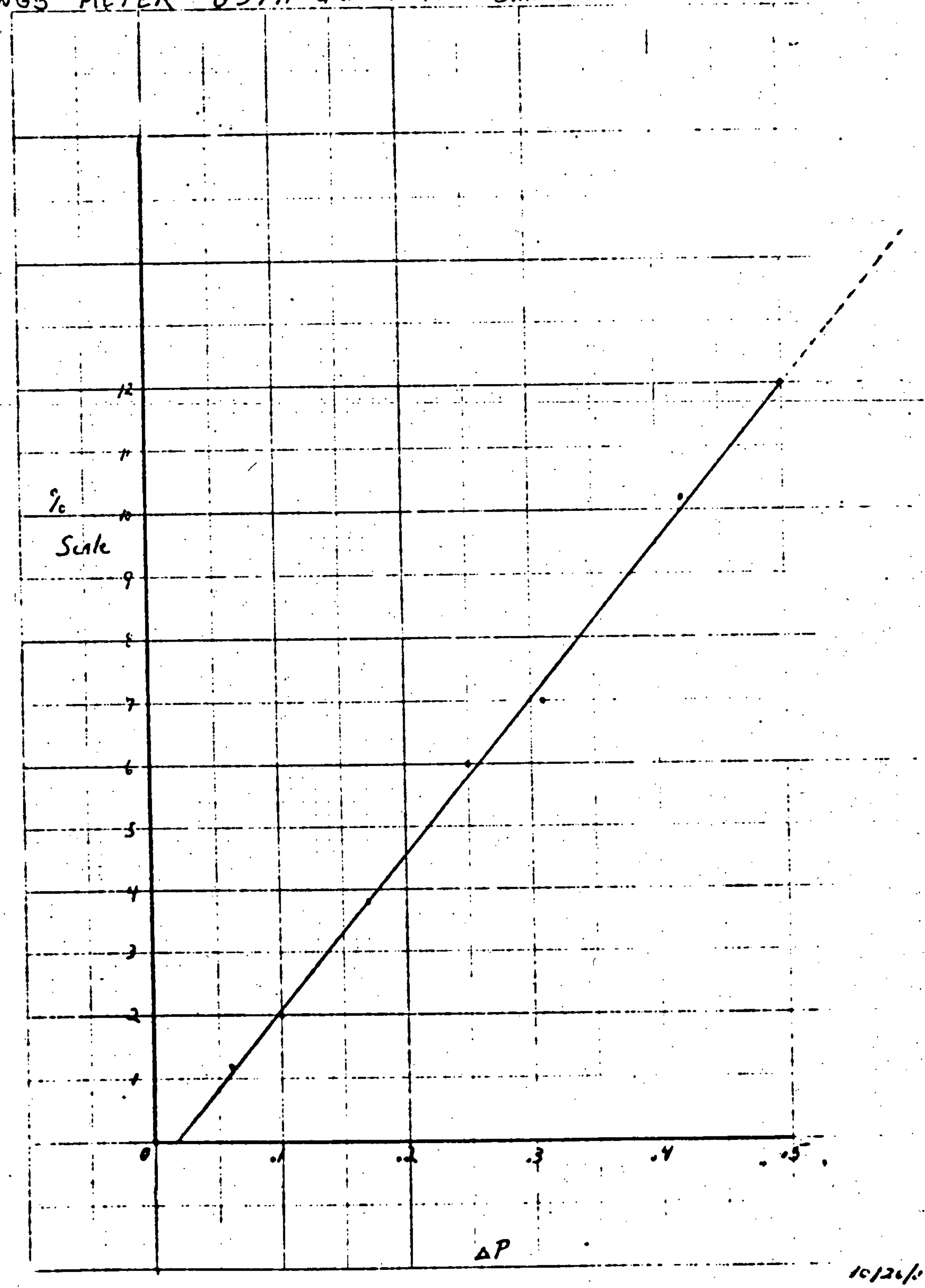
VOST TRAIN

	Thermocouple Number	Temperature Reading 1	Reference Temperature 1	Temperature Reading 2	Reference Temperature 2	Temperature Reading 3	Reference Temperature 3
[illegible]	1	188	190	[illegible]	[illegible]	[illegible]	[illegible]
	2	189	198	188	[illegible]	[illegible]	[illegible]
	3						
	1	71	71	71	71	[illegible]	71
AMBIENT	2	70	71	[illegible]	71	[illegible]	71
	3						
ICE BATH	1	32	32	32	32	32	32
	2	32	32	32	32	32	32
	3						

Signature of Person Performing Calibration

[illegible]
Calibration Date

HASTINGS METER GSM 2D5K vs Standard Plot



10/26/83
AW

GCA TECHNOLOGY DIVISION

THERMOCOUPLE CALIBRATION FORM

	Thermocouple Number	Temperature Reading 1	Reference Temperature 1	Temperature Reading 2	Reference Temperature 2	Temperature Reading 3	Reference Temperature 3
	Hot Box 2180						
ICE WATER		32°F	32°F	[illegible]	32°F	[illegible]	32°F
AMBIENT		90°F	90°F	90°F	[illegible]	[illegible]	90°F
HOT WATER		172°F	183°F	[illegible]	[illegible]	[illegible]	[illegible]
	Hot Box 1726						
ICE WATER		32°F	32°F	32°F	32°F	32°F	32°F
AMBIENT		90°F	90°F	90°F	90°F	90°F	90°F
HOT WATER		179°F	179°F	[illegible]	[illegible]	179°F	[illegible]

Signature of Person Performing Calibration

[illegible]
Calibration Date

Method 10

Plant Name: United Chemical Co Date: 3 Nov 82
Sample Location: Stack Outlet
Barometric Pressure, ~~mm~~ [✓ (in.) Hg] 29.3[illegible]
Ambient temperature: °C [✓ °F] 50 Stack Temperature: °C [✓ °F] [illegible]
Initial Leak Check 0.0 Final Leak Check
Operator: Clark Contract # 7-468-001
R. #2

Clock time, 24 h	Rotameter Setting 1/min	CO conc, ppm (dry basis)	voltage	CO_2, %	Comments
1 [illegible] /0	2 1 lpm	0	[illegible]	3.0	[illegible]
2 955 /5		0	[illegible]		[illegible]
[illegible] /10		5.4	0.003		
/15		0	[illegible]		
/[illegible]		0	[illegible]		
/25		0	0.001		
/30		0	0.001		
8 /35		0	[illegible]		
9 /40		5.4	[illegible]		
10 /[illegible]		0	[illegible]		
11 /[illegible]		[illegible]	0.001		
12		[illegible]	[illegible]		
13 [illegible]		2.6	0.002		
14		0	0.000		
15		0	[illegible]		
16		0	[illegible]		
17		2.6	[illegible]		
18		0	[illegible]		
19 [illegible]		5.4	[illegible]		
20		0	[illegible]		
21		0	[illegible]		
22		2.6	[illegible]		
23		[illegible]			

Field sampling data form for CO (continuous sample).

Method 10

Plant Name: ______ Date: ______

Sample Location: ______

Barometric Pressure, mm (in.) Hg ______

Ambient temperature: °C (°F) ___ Stack Temperature: °C (°F) ______

Initial Leak Check ______ Final Leak Check 0.0 lpm

Operator: ______ Contract # ______

Run # 2 [illegible]

	Clock time, 24 h	Rotameter Setting 1/min	CO conc. ppm (dry basis)		CO_2, %	Comments
24	1120/1125		1.6	[illegible]	3.0	
25	1120/1125		0	0.000		
	1130/1135		0	0.000		
	[illegible]		1	[illegible]		
28	[illegible]		0	[illegible]		
29	[illegible]		1.6	0.002		
30	[illegible]		1.6	[illegible]		
31	[illegible]		1	[illegible]		
32	[illegible]		0	[illegible]		
33	[illegible]		0	0.001		
34	[illegible]		1.6	[illegible]		

$\bar{X}$ 1.6

$$C_{CO_{stack}} = C_{CO_{NDIR}}\left(1 - \frac{\%\,CO_2}{100}\right)$$

$$= 1.6\left(1 - \frac{3}{100}\right)$$

$$C_{CO_{stack}} = 1.55 \text{ ppm Dry}$$

Field sampling data form for CO (continuous sample).

Method 10

Plant Name: U[illegible] Date:

Sample Location: C[illegible]

Barometric Pressure, mm (in.) Hg 71.[illegible]

Ambient temperature: °C (°F) [illegible] Stack Temperature °C (°F) [illegible]

Initial Leak Check 0.0 [illegible] Final Leak Check 0.0 lpm

Operator: [illegible] Contract # [illegible]

Run = 3

Clock time, 24 h	Rotameter Setting l/min	CO conc. ppm (dry basis)		O₂	Comments
1500 0	≥ 1 lpm	5.4	0.003	4.6	
1505 5		16.5	0.007		
1510 10		5.4	0.003		
[illegible]5 [illegible]		10.9	0.005		
[illegible]		16.5	0.007		
1525 25		5.4	0.003		
1530 [illegible]		10.9	0.005		
1535 [illegible]		8.2	0.004		$\bar{X} = 11.3$
1540 [illegible]		5.4	0.003		
1545 45		10.9	0.005		
1550 [illegible]		0	0.000		$C_{CO} = 11.3\left(1 - \frac{4.6}{100}\right)$
1555 55		30.3	0.012		
1600 [illegible]		13.7	0.006		$= 10.8$ ppm Dry
1605 65		13.7	0.006		
1610 [illegible]		8.2	0.004		
1615 [illegible]		8.2	0.004		
1620 [illegible]		10.9	0.005		
1625 [illegible]		30.3	0.012		
1630 [illegible]		2.6	0.002		
1635 [illegible]		19.2	0.008		
1640 [illegible]		5.4	0.003		
1645 [illegible]		10.9	0.005		
1650 [illegible]					

Field sampling data form for CO (continuous sampler).

Method 10

Plant Name: Union Carbide Date: 4 Nov 83

Sample Location: Outlet stack

Barometric Pressure, [✓ (in.) Hg] 29.63

Ambient temperature: [✓ °F] 48 Stack Temperature, [✓ °F] 155

Initial Leak Check 0.0 lpm Final Leak Check

Operator: Clark Contract # 7-468-001

Run #4

Clock time, 24 h	Rotameter Setting l/min	CO conc, ppm (dry basis)	Volts	CO_2, %	Comments
1000/0	≥1 lpm	0	.000		Readings Based on Spontaneous value (voltage) at corresponding time
1005/5		0	.000		
1010/10		0	.001		
15/15		0	.000		
1020/20		0	.000		
1025/25		0	.000		
1030/30		0	.000		
1035/35		0	.000		
1040/40		0	-.004		
1045/45		0	.000		
1050/50		0	-.001		
1055/55		0	-.001		
1100/60		0	.000		
1105/65		0	-.005		Condenser Draw Down may effect readings. Change Silica Gel
1110/70					
1115/75					
1120/80					
1125/85					
1130/90					
1135/95					
1140/100					
1145/105					
1150/110					

Field sampling data form for CO (... sample).

UNI004

0530

Method 10

Plant Name: Union Chemical Date: 4 Nov 83

Sample Location: Outlet stack

Barometric Pressure, ~~mm~~ [✓ (in.) Hg] 29.68

Ambient temperature: ~~°C~~ [✓ (°F)] 48 Stack temperature °C [✓ (°F)] 155

Initial Leak Check 0.0 Final Leak Check

Operator: Clark Contract #

Run 4 cont.

Clock time, 24 h	Rotameter Setting l/min	CO conc. ppm (dry basis)	volts	CO_2, %	Comments
1155/115	≥ 1 lpm	0	.000		
1200/120		0	.000		
5/125		0	.000		
10/130		0	-.001		
1215/135		0	.000		
1320/140		0	.000		
1225/145		0	.000		
1230/150		0	-.001		
1235/155		0	.002		
1240/160		0	.001		
1245/165		0	-.003		
1250/170		0	-.004		
1255/175		0	.000		
1300/180					Reading missed - (Lehrin)
1305/185		0	.000		
1310/190		2.6	.002		
1315/195		0	.000		
1320/200					end run #4
1325/205					
1330/210					
1335/215					
1340/220					
1345/225					

Field sampling data form for CO (continuous sample).

CO Cal
2 NOV 83
1500

ANALYZERS
DAILY 3-POINT CALIBRATION CHECK

I. Analyzer Data

A. O_2 Analyzer MFG ____________ S/N ____________

Zero Setting ____________ Span ____________

B. SO_2 Analyzer MFG ____________ S/N ____________

Coarse Zero Setting ____________ Fine Zero ________ Span ________

C. CO_2 Analyzer MFG ____________ S/N ____________

Zero Setting ____________ Span ____________

D. CO Analyzer MFG Horiba S/N ____________

Zero Setting 776 Span 755

II. Data

Run No.	O_2		CO		CO_2		SO_2		NO_x	
	Conc	Reading	Conc	Reading	Conc	Reading	Conc	Reading	Conc	Reading
1			0	0.004						
2			51	0.026						
3			948	0.348						

~~[illegible]~~

III. Data Reduction

Calculate calibration equation using least square linear regression.

$Y = mx + b$ where:

Y = concentration v/v

m = slope conc/mv

x = reading in mv

b = intercept concentration v/v

		O_2	SO_2	CO_2	CO	Moisture
m	=				−1.7	
b	=				58.6	
Corr. coeff. r^2	=				959.8	

Figure 8-14. Analyzers, daily calibration check.

3 NOV 83
0800

ANALYZERS
DAILY 3-POINT CALIBRATION CHECK

I. Analyzer Data

A. O_2 Analyzer MFG ______ S/N ______

Zero Setting ______ Span ______

B. SO_2 Analyzer MFG ______ S/N ______

Coarse Zero Setting ______ Fine Zero ______ Span ______

C. CO_2 Analyzer MFG ______ S/N ______

Zero Setting ______ Span ______

D. NO_x Analyzer MFG Horiba S/N ______

Zero Setting 776 Span 755

II. Data

Run No.	O_2		CO		CO_2		SO_2		NO_x	
	Conc	Reading	Conc	Reading	Conc	Reading	Conc	Reading	Conc	Reading
1			0	0.006						
2			51	[illegible]						
3			946	0.341						

III. Data Reduction

Calculate calibration equation using least square linear regression.

Y = mx + b where:
Y = concentration v/v
m = slope conc/mv
x = reading in mv
b = intercept concentration v/v

	O_2	SO_2	CO_2	CO	μ ~~Moisture~~
m =				-9.8	-0.1
b =				~~30.3~~ 97.1	55.2
Corr. coeff. r^2 =				928.6	7517

Figure 8-14. Analyzers, daily calibration check.

All CO Rngs w/in 3% of conc.
Except MID - new curve Drawn

GCA CORPORATION
Technology Division
GCA

0915
CO Cal #4

ANALYZERS
DAILY 3-POINT CALIBRATION CHECK

I. **Analyzer Data**

A. O_2 Analyzer MFG ____ S/N ____
Zero Setting ____ Span ____
B. SO_2 Analyzer MFG ____ S/N ____
Coarse Zero Setting ____ Fine Zero ____ Span ____
C. CO_2 Analyzer MFG ____ S/N ____
Zero Setting ____ Span ____
D. ~~NO_x~~ CO Analyzer MFG Horiba S/N ____
Zero Setting 776 Span 755

II. **Data**

Run No.	O_2		CO		CO_2		SO_2		NO_x	
	Conc	Reading	Conc	Reading	Conc	Reading	Conc	Reading	Conc	Reading
1					0	0.000				
2					51	0.021				
3										

III. **Data Reduction**

Calculate calibration equation using least square linear regression.

$Y = mx + b$ where:
Y = concentration v/v
m = slope conc/mv
x = reading in mv
b = intercept concentration v/v

	O_2	SO_2	CO_2	CO	Moisture
m =				-2.9	
b =				55.2	
Corr. coeff. r^2 =					

Figure 8-14. Analyzers, daily calibration check.

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Technology Division

1700
End of Day
0 - Span Check

ANALYZERS
DAILY 3-POINT CALIBRATION CHECK

I. **Analyzer Data**

A. O_2 Analyzer MFG ____________ S/N ____________
Zero Setting ____________ Span ____________
B. SO_2 Analyzer MFG ____________ S/N ____________
Coarse Zero Setting ____________ Fine Zero ________ Span ________
C. CO_2 Analyzer MFG ____________ S/N ____________
Zero Setting ____________ Span ____________
D. ~~NO~~ CO$_x$ Analyzer MFG Horiba S/N ____________
Zero Setting 776 Span 755

II. **Data**

Run No.	O_2		CO		CO_2		SO_2		NO_x	
	Conc	Reading	Conc	Reading	Conc	Reading	Conc	Reading	Conc	Reading
1			0	0.002						
2										
3			Out of Span Gas							

III. **Data Reduction**

Calculate calibration equation using least square linear regression.

$Y = mx + b$ where:
Y = concentration v/v
m = slope conc/mv
x = reading in mv
b = intercept concentration v/v

		O_2	SO_2	CO_2	CO	Moisture
m	=				2.6	
b	=					
Corr. coeff. r^2	=					

Figure 8-14. Analyzers, daily calibration check.

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Technology Division

APPENDIX D

GC/ECD CALIBRATION AND FIELD DATA

TABLE D-1. GC/ECD CALIBRATION DATA

	Trichloromonofluoromethane	1,1,2-trichloro-1,2,2-trifluoroethane	1,1,1-trichloroethane	Trichloroethene	Tetrachloroethene
11/3/83 - Runs 1,2					
Concentration ($\mu g/m^3$)	22	24	20	22	23
Instrument response (counts)	14662	10861	2950	1810	5614
Concentration ($\mu g/m^3$)	45	47	40	44	45
Instrument response (counts)	26358	13514	6087	3444	11215
Concentration ($\mu g/m^3$)	75	79	67	73	75
Instrument response (counts)	43744	16074	9288	5436	14743
Concentration ($\mu g/m^3$)	119	126	107	117	212
Instrument response (counts)	86817	25858	19305	11136	30738
Concentration ($\mu g/m^3$)	149	158	134	146	151
Instrument response (counts)	107681	31578	22951	14478	39953
Equation of line[a]	$y = 9.391+0.0013x$	$y = (-34.840)+0.0062x$	$y = 8.136+0.0054x$	$y = 11.657+0.0095x$	$y = 8.455+0.0036x$
Correlation coefficient	0.994	0.988	0.993	0.993	0.990
11/4/83 - Run 3					
Concentration ($\mu g/m^3$)	22	24	20	22	23
Instrument response (counts)	17895	5065	3179	1700	6172
Concentration ($\mu g/m^3$)	45	47	40	44	43
Instrument response (counts)	31153	7642	6649	4112	15770
Concentration ($\mu g/m^3$)	75	79	67	73	75
Instrument response (counts)	63353	13159	13338	8025	23660
Concentration ($\mu g/m^3$)	119	126	107	117	121
Instrument response (counts)	112478	21638	25548	15750	44000
Concentration ($\mu g/m^3$)	-	-	-	-	75341
Instrument response (counts)	-	-	-	-	242
Equation of line[a]	$y = 9.606+0.0010x$	$y = (-2.355)+0.0060x$	$y =$ [illegible]$.273+0.0037x$	$y = 14.868+0.0066$	$y = (-2.549)+0.0031x$
Correlation coefficient	0.994	0.997	0.995	0.995	0.994

[a] y = concentration, x = instrument response.

TABLE D-2. GC/ECD ANALYTICAL DATA[a]

	Blank[b]	Sample A		Sample B		
	Instrument response (counts)	Instrument response (counts)	Concentration[c] (μg/m³)	Instrument response (counts)	Concentration[c] (μg/m³)	Average concentration[c] (μg/m³)
11/3/83 - Run 1						
Trichloromonofluoromethane	327	35,073	55	34,137	54	55
1,1,2-trichloro-1,2,2-trifluoroethane	5,024	8,999	21	9,268	23	22
1,1,1-trichloroethane	319	4,337	32	4,544	33	23
Trichloroethene	<12	578	17	624	18	18
Tetrachloroethene	1,053	15,625	65	16,565	69	67
11/3/83 - Run 2						
Trichloromonofluoromethane	398	32,664	52	18,195	33	43
1,1,2-trichloro-1,2,2-trifluoroethane	2,253	7,572	12	7,686	13	13
1,1,1-trichloroethane	363	3,235	26	4,369	32	29
Trichloroethene	<12	<12	ND[d]	418	16	16
Tetrachloroethene	1,606	14,309	61	12,837	55	18
11/4/83 - Run 3						
Trichloromonofluoromethane	1,833	49,015	60	NA[e]	NA[e]	60
1,1,2-trichloro-1,2,2-trifluoroethane	1,601	2,675	14	NA[e]	NA[e]	14
1,1,1-trichloroethane	<12	17,379	70	NA[e]	NA[e]	70
Trichloroethene	<12	<12	ND[d]	NA[e]	NA[e]	<15
Tetrachloroethene	1,158	82,079	255	NA[e]	NA[e]	255

[a] Results are based on the linear regression analyses of the calibration data in Table D-1.

[b] Concentrations are not reported because the instrument responses were generally well below the lowest calibration point.

[c] Not blank corrected.

[d] Not detected.

[e] Not analyzed.

APPENDIX E

LABORATORY DATA REPORTS

Project 7-468-001 GCA Control No. 35263-1

DATA REPORT SHEET

Principal Organic Hazardous Constituents

Sample I.D. Waste Feed Composite. Run 1 Date of Analysis 1/18/84

Sample Matrix Combustible Waste Feed Instrument Finnigan MAT OWA GC/MS

Component	Quantitative Ion	Concentration (mg/kg)
trichlofluoromethane	101	15,000
1,1,1-trichloroethane	97	53,000
trichloroethene	130	7,200
tetrachloroethene	164	43,000
1,1,2-trichloro 1,2,2-trifluoroethane	101	3,000

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Project 7-468-001 GCA Control No. 35263-2

DATA REPORT SHEET

Principal Organic Hazardous Constituents

Sample I.D. Waste Feed Composite, Run 1 Date of Analysis 1/25/84

Sample Matrix Combustible Waste Feed Instrument Finnigan MAT OWA GC/MS

Component	Quantitative Ion	Concentration (mg/kg)
trichlofluoromethane	101	14,000
1,1,1-trichloroethane	97	40,000
trichloroethene	130	5,200
tetrachloroethene	164	39,000
1,1,2-trichloro 1,2,2-trifluoroethane	101	5,000

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Project 7-468-001 GCA Control No. 35269

DATA REPORT SHEET

Principal Organic Hazardous Constituents

Sample I.D. Waste Feed Composite, Run 2 Date of Analysis 1/18/84

Sample Matrix Combustible Waste Feed Instrument Finnigan MAT OWA GC/MS

Component	Quantitative Ion	Concentration (mg/kg)
trichlofluoromethane	101	12,000
1,1,1-trichloroethane	97	50,000
trichloroethene	130	6,800
tetrachloroethene	164	38,000
1,1,2-trichloro 1,2,2-trifluoroethane	101	2,900

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Project 7-468-001 GCA Control No. 35273

DATA REPORT SHEET

Principal Organic Hazardous Constituents

Sample I.D. Waste Feed Composite, Run 3 Date of Analysis 1/18/84

Sample Matrix Combustible Waste Feed Instrument Finnigan MAT OWA GC/MS

Component	Quantitative Ion	Concentration (mg/kg)
trichlofluoromethane	101	19,000
1,1,1-trichloroethane	97	49,000
trichloroethene	130	8,900
tetrachloroethene	164	36,000
1,1,2-trichloro 1,2,2-trifluoroethane	101	2,800

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RESULTS OF TOTAL CHLORINE ANALYSIS OF WASTE FEEDS

Chloride Concentration (w/w %)		
Run 1	Run 2	Run 3
13.6	13.8	14.0

RESULTS OF CHLORIDE ANALYSIS OF CONTAMINATED WATER

Chloride Concentration (mg/l)		
Run 1	Run 2	Run 3
622	585	555

Project 7-468-001 GCA Control No. 35263

DATA REPORT SHEET

Metals

Sample I.D. Waste Feed Composite, Run 1 Report Date 1/10/84

Sample Matrix Combustible Waste Feed

Element	Instrument	Concentration* (µg/g)**	Remarks
Arsenic	Jarrell-Ash 855 ICPS	16	
Barium	Jarrell-Ash 855 ICPS	119	
Cadmium	Jarrell-Ash 855 ICPS	3.88	
Chromium	Jarrell-Ash 855 ICPS	154	
Lead	Jarrell-Ash 855 ICPS	452	
Mercury	Varian AA-6 AAS	0.42	Cold Vapor Method
Selenium	Jarrell-Ash 855 ICPS	< 0.5	
Silver	Jarrell-Ash 855 ICPS	< 0.02	
Beryllium	Jarrell-Ash 855 ICPS	4.25	
Iron	Jarrell-Ash 855 ICPS	2.10%	

*Sample preparation via dry ash techniques for all elements except arsenic and lead. The latter were quantitated in the sample aliquot prepared for chloride analysis via Parr Bomb.

**Except as noted.

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Project 7-468-001 GCA Control No. 35269

DATA REPORT SHEET

Metals

Sample I.D. Waste Feed Composite, Run 2 Report Date 1/10/84

Sample Matrix Combustible Waste Feed

Element	Instrument	Concentration* (µg/g)**	Remarks
Arsenic	Jarrell-Ash 855 ICPS	22	
Barium	Jarrell-Ash 855 ICPS	110	
Cadmium	Jarrell-Ash 855 ICPS	3.72	
Chromium	Jarrell-Ash 855 ICPS	156	
Lead	Jarrell-Ash 855 ICPS	449	
Mercury	Varian AA-6 AAS	0.75	Cold Vapor Method
Selenium	Jarrell-Ash 855 ICPS	< 0.5	
Silver	Jarrell-Ash 855 ICPS	< 0.03	
Beryllium	Jarrell-Ash 855 ICPS	4.38	
Iron	Jarrell-Ash 855 ICPS	1.85%	

*Sample preparation via dry ash techniques for all elements except arsenic and lead. The latter were quantitated in the sample aliquot prepared for chloride analysis via Parr Bomb.

**Except as noted.

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GCA Control No. 35273

DATA REPORT SHEET

Metals

Sample I.D. Waste Feed Composite, Run 3

Report Date 1/10/84

Sample Matrix Combustible Waste Feed

Element	Instrument	Concentration* (µg/g)**	Remarks
Arsenic	Jarrell-Ash 855 ICPS	20	
Barium	Jarrell-Ash 855 ICPS	135	
Cadmium	Jarrell-Ash 855 ICPS	4.59	
Chromium	Jarrell-Ash 855 ICPS	188	
Lead	Jarrell-Ash 855 ICPS	472	
Mercury	Varian AA-6 AAS	0.39	Cold Vapor Method
Selenium	Jarrell-Ash 855 ICPS	< 0.5	
Silver	Jarrell-Ash 855 ICPS	< 0.02	
Beryllium	Jarrell-Ash 855 ICPS	5.38	
Iron	Jarrell-Ash 855 ICPS	2.30%	

*Sample preparation via dry ash techniques for all elements except arsenic and lead. The latter were quantitated in the sample aliquot prepared for chloride analysis via Parr Bomb.

**Except as noted.

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Project 7-468-001

RESULTS OF VOST ANALYSIS

Run #	Concentration (μg/m³)[a]				
	Freon 11	Trichlorotrifluoroethane	1,1,1-Trichloroethane[b]	Trichloroethylene	Tetrachloroethylene[b]
1A	17	ND	ND	ND	68
1B	c	c	c	c	c
1C	96	ND	ND	ND	16[d]
2A	28[d]	ND[d]	11[d]	ND[d]	ND[d]
2B	29[d]	ND[d]	ND[d]	ND[d]	25[d]
3A	47	ND	20	21	120
3B	70	ND	ND	24	150

ND = < 10 μg/m³

[a] Concentration reported represents a summation of Tenax tube, Tenax/Charcoal tube and condensate analyses.

[b] Reported results have been corrected for the average value measured in three sets of field-biased blanks.

[c] Data for this run are unavailable due to GC/MS malfunctions.

[d] Low recovery of surrogate compound indicates incomplete desorption of the tube and/or a problem with the addition of surrogate compounds to the sample.

Project 7-468-001

RESULTS OF VOST TUBE ANALYSES

GCA No.	Run No.	Tube I.D.	Quantity Detected (ng) Freon 11	Trichloro-trifluoro-ethane	1,1,1 Trichloro-ethane	Trichloro-ethylene	Tetra-chloro-ethylene	Volume Sampled (1)
35086	1A	T	210	ND	68	130	1100	15.8
35087	1A	T/C	72	ND	52	ND	48	15.8
35088	1B	T	a	a	a	a	a	20.4
35089	1B	T/C	130	ND	54	ND	31	20.4
35090	1C	T	900	ND	90	19	260	14.3
35091	1C	T/C	470	ND	54	ND	48	14.3
35092	FBB(1)	T	ND	ND	78	ND	27	NA
35093	FBB(1)	T/C	ND	ND	41	ND	17	NA
35094	2A	T	330	ND	260	66	110	26.2
35095	2A	T/C	410	ND	130	ND	51	26.2
35096	2B	T	770	ND	150	82	690	26.5
35097	2B	T/C	ND	ND	72	ND	27	26.5
35098	FBB(2)	T	ND	ND	53	ND	22	NA
35099	FBB(2)	TC	ND	ND	41	ND	20	NA
35100	3A	T	380	ND	300	370	2100	16.9
35101	3A	T/C	420	ND	130	ND	27	16.9
35102	3B	T	1000	ND	130	340	2200	14.4
35103	3B	T/C	21	ND	42	ND	19	14.4
35104	FBB(3)	T	ND	ND	46	ND	54	NA
35105	FBB(3)	T/C	ND	ND	46	ND	19	NA

ND = < 15 ng   NA = Not appropriate   T = Tenax   T/C = Tenax/Charcoal   a = Data lost due to computer malfunction.

Project 7-468-001

GCA Control No. 35106/7

DATA REPORT SHEET

Metals

Sample I.D. Field Blank

Report Date

Sample Matrix Part. filter, rinse solvent

Element	Instrument	Concentration (μg)	Remarks
Arsenic	Jarrell-Ash 855 ICPS	< 2	
Barium	Jarrell-Ash 855 ICPS	126	
Cadmium	Jarrell-Ash 855 ICPS	0.12	
Chromium	Jarrell-Ash 855 ICPS	1.3	
Lead	Jarrell-Ash 855 ICPS	< 2	
Mercury	Varian AA-6 AAS	< 0.2	Cold Vapor Method
Selenium	Jarrell-Ash 855 ICPS	< 2	
Silver	Jarrell-Ash 855 ICPS	56	
Beryllium	Jarrell-Ash 855 ICPS	< 0.09	
Iron	Jarrell-Ash 855 ICPS	59.8	

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Project 7-468-001

GCA Control No. 35110/1

DATA REPORT SHEET

Metals

Sample I.D. Run I Particulate

Report Date

Sample Matrix Part. filter, probe rinse

Element	Instrument	Concentration (µg)	Remarks
Arsenic	Jarrell-Ash 855 ICPS	719	
Barium	Jarrell-Ash 855 ICPS	1030	
Cadmium	Jarrell-Ash 855 ICPS	399	
Chromium	Jarrell-Ash 855 ICPS	479	
Lead	Jarrell-Ash 855 ICPS	43,500	
Mercury	Varian AA-6 AAS	0.33	Cold Vapor Method
Selenium	Jarrell-Ash 855 ICPS	< 2	
Silver	Jarrell-Ash 855 ICPS	10.4	
Beryllium	Jarrell-Ash 855 ICPS	22.3	
Iron	Jarrell-Ash 855 ICPS	93,600	

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Project 7-468-001

GCA Control No. 35114/5

DATA REPORT SHEET

Metals

Sample I.D. Run 2 Particulate

Report Date

Sample Matrix Part. filter, probe rinse

Element	Instrument	Concentration (µg)	Remarks
Arsenic	Jarrell-Ash 855 ICPS	904	
Barium	Jarrell-Ash 855 ICPS	1010	
Cadmium	Jarrell-Ash 855 ICPS	514	
Chromium	Jarrell-Ash 855 ICPS	795	
Lead	Jarrell-Ash 855 ICPS	52,800	
Mercury	Varian AA-6 AAS	0.51	Cold Vapor Method
Selenium	Jarrell-Ash 855 ICPS	< 2	
Silver	Jarrell-Ash 855 ICPS	20.2	
Beryllium	Jarrell-Ash 855 ICPS	26.9	
Iron	Jarrell-Ash 855 ICPS	108,000	

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Project 7-468-001 GCA Control No. 35118/9

DATA REPORT SHEET

Metals

Sample I.D. Run 3 Particulate Report Date ________

Sample Matrix Part. filter, probe rinse

Element	Instrument	Concentration (µg)	Remarks
Arsenic	Jarrell-Ash 855 ICPS	342	
Barium	Jarrell-Ash 855 ICPS	589	
Cadmium	Jarrell-Ash 855 ICPS	226	
Chromium	Jarrell-Ash 855 ICPS	353	
Lead	Jarrell-Ash 855 ICPS	19,000	
Mercury	Varian AA-6 AAS	0.24	Cold Vapor Method
Selenium	Jarrell-Ash 855 ICPS	< 2	
Silver	Jarrell-Ash 855 ICPS	9.00	
Beryllium	Jarrell-Ash 855 ICPS	13.2	
Iron	Jarrell-Ash 855 ICPS	53,600	

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Project 7-468-001

RESULTS OF CHLORIDE ANALYSES OF METHOD 5 TRAINS

Train Component	Measured Chloride (mg)*			
	Run 1	Run 2	Run 3	Field Blank
Particulate Filter/ Front Half Rinse	138	232	60.9	0.054
Condensate	42.3	75.6	26.1	< 0.3
Impingers	13.6	28.6	0.58	< 0.3

*Reported concentrations have been corrected for the laboratory method blank. No field blank corrections have been made.

Project 7-468-001

RESULTS OF PARTICULATE ANALYSIS FOR TRACE METALS

Analyte	Total µg*			
	Field Blank	Run 1	Run 2	Run 3
Silver	56.0	10.4	20.2	9.00
Arsenic	< 2	719	904	342
Barium	126	1030	1010	589
Beryllium	< 0.09	22.3	26.9	13.2
Cadmium	0.12	399	514	226
Chromium	1.3	479	795	353
Iron	59.8	93600	108000	53600
Lead	< 2	43500	52800	19000
Selenium	< 2	< 2	< 2	< 2
Mercury	< 0.2	0.33	0.51	0.24

*Results corrected for concentrations found in the laboratory method blank. No field blank corrections have been made.

Project 7-468-001 GCA Control No. 35254

DATA REPORT SHEET

Principal Organic Hazardous Constituents

Sample I.D. Contaminated Water, Run 1 Date of Analysis 1/9/84

Sample Matrix Water Instrument Finnigan MAT OWA GC/MS

Component	Quantitative Ion	Concentration (µg/l)
trichlofluoromethane	101	84
1,1,1-trichloroethane	97	15,000
trichloroethene	130	4,400
tetrachloroethene	164	4,200
1,1,2-trichloro 1,2,2-trifluoroethane	101	180

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Project 7-468-001

GCA Control No. 35255

DATA REPORT SHEET

Principal Organic Hazardous Constituents

Sample I.D. Contaminated Water, Run 2

Date of Analysis 1/9/84

Sample Matrix Water

Instrument Finnigan MAT OWA GC/MS

Component	Quantitative Ion	Concentration (µg/l)
trichlofluoromethane	101	75
1,1,1-trichloroethane	97	18,000
trichloroethene	130	4,800
tetrachloroethene	164	4,700
1,1,2-trichloro 1,2,2-trifluoroethane	101	250

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Project 7-468-001 GCA Control No. 35256

DATA REPORT SHEET

Principal Organic Hazardous Constituents

Sample I.D. Contaminated Water, Run 3 Date of Analysis 1/9/84

Sample Matrix Water Instrument Finnigan MAT OWA GC/MS

Component	Quantitative Ion	Concentration (µg/1)
trichlofluoromethane	101	96
1,1,1-trichloroethane	97	17,000
trichloroethene	130	4,600
tetrachloroethene	164	4,600
1,1,2-trichloro 1,2,2-trifluoroethane	101	330

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Project 7-468-001 GCA Control No. 35284

DATA REPORT SHEET

Purgeables

Sample I.D. Scrubber Water Supply, Run 1 Analysis Date 1/19/84

Sample Matrix Water Instrument Finnigam MAT OWA GC/MS

Parameter	Ion Used To Quantitate	Concentration (µg/1)	Remarks
chloromethane		ND	
dichlorodifluoromethane		ND	
bromomethane		ND	
vinyl chloride		ND	
chloroethane		ND	
methylene chloride	84	10	
acrolein		ND*	
acrylonitrile		ND*	
trichlorofluoromethane		ND	
1,1-dichloroethylene		ND	
1,1-dichloroethane		ND	
trans-1,2-dichloroethylene		ND	
chloroform		ND	
1,2-dichloroethane		ND	
1,1,1-trichloroethane	97	Trace	
carbon tetrachloride		ND	
bromodichloromethane		ND	
bis-chloromethyl ether		ND	
1,2-dichloropropane		ND	
trans-1,3-dichloropropene		ND	
trichloroethylene	130	Trace	
dibromochloromethane		ND	
cis-1,3-dichloropropene		ND	
1,1,2-trichloroethane		ND	
benzene		ND	
2-chloroethylvinyl ether		ND	
bromoform		ND	
tetrachloroethene	164	3.7	
1,1,2,2-tetrachloroethane		ND	
toluene	92	2.6	
chlorobenzene		ND	
ethylbenzene		ND	

ND = < 0.5 µg/1
Trace = < 2 µg/1
ND* = < 20 µg/1

GCA Technology Division

Project 7-468-001 GCA Control No. 35285

DATA REPORT SHEET

Purgeables

Sample I.D. Scrubber Water Supply, Run 2 Analysis Date 1/19/84

Sample Matrix Water Instrument Finnigan MAT OWA GC/MS

Parameter	Ion Used To Quantitate	Concentration (µg/l)	Remarks
chloromethane		ND	
dichlorodifluoromethane		ND	
bromomethane		ND	
vinyl chloride		ND	
chloroethane	84	9.7	
methylene chloride		ND*	
acrolein		ND*	
acrylonitrile		ND	
trichlorofluoromethane		ND	
1,1-dichloroethylene		ND	
1,1-dichloroethane		ND	
trans-1,2-dichloroethylene		ND	
chloroform		ND	
1,2-dichloroethane		ND	
1,1,1-trichloroethane	97	2.3	
carbon tetrachloride		ND	
bromodichloromethane		ND	
bis-chloromethyl ether		ND	
1,2-dichloropropane		ND	
trans-1,3-dichloropropene		ND	
trichloroethylene	130	2.1	
dibromochloromethane		ND	
cis-1,3-dichloropropene		ND	
1,1,2-trichloroethane		ND	
benzene		ND	
2-chloroethylvinyl ether		ND	
bromoform		ND	
tetrachloroethene	164	10	
1,1,2,2-tetrachloroethane		ND	
toluene	92	8.4	
chlorobenzene		ND	
ethylbenzene	106	Trace	

ND = < 0.5 µg/l
Trace = < 2 µg/l
ND* = < 20 µg/l

GCA CORPORATION
Technology Division

Project 7-468-001		GCA Control No. 35286	

DATA REPORT SHEET

Purgeables

Sample I.D. Scrubber Water Supply, Run 3

Analysis Date 1/19/84

Sample Matrix Water

Instrument Finnigan MAT OWA GC/MS

Parameter	Ion Used To Quantitate	Concentration (µg/1)	Remarks
chloromethane		ND	
dichlorodifluoromethane		ND	
bromomethane		ND	
vinyl chloride		ND	
chloroethane		ND	
methylene chloride	84	7.6	
acrolein		ND*	
acrylonitrile		ND*	
trichlorofluoromethane		ND	
1,1-dichloroethylene		ND	
1,1-dichloroethane		ND	
trans-1,2-dichloroethylene		ND	
chloroform		ND	
1,2-dichloroethane		ND	
1,1,1-trichloroethane		ND	
carbon tetrachloride		ND	
bromodichloromethane		ND	
bis-chloromethyl ether		ND	
1,2-dichloropropane		ND	
trans-1,3-dichloropropene		ND	
trichloroethylene		ND	
dibromochloromethane		ND	
cis-1,3-dichloropropene		ND	
1,1,2-trichloroethane		ND	
benzene		ND	
2-chloroethylvinyl ether		ND	
bromoform		ND	
tetrachloroethene	164	Trace	
1,1,2,2-tetrachloroethane		ND	
toluene		ND	
chlorobenzene		ND	
ethylbenzene		ND	

ND = < 0.5 µg/1
Trace = < 2 µg/1
ND* = < 20 µg/1

Project 7-468-001

GCA Control No. 35287

DATA REPORT SHEET

Purgeables

Sample I.D. Composite Scrubber Water(Contaminated) Run 1

Analysis Date 1/19/84

Sample Matrix Water

Instrument Finnigam MAT OWA GC/MS

Parameter	Ion Used To Quantitate	Concentration (µg/1)	Remarks
chloromethane		ND	
dichlorodifluoromethane		ND	
bromomethane		ND	
vinyl chloride		ND	
chloroethane		ND	
methylene chloride	84	8.7	
acrolein		ND*	
acrylonitrile		ND*	
trichlorofluoromethane		ND	
1,1-dichloroethylene		ND	
1,1-dichloroethane		ND	
trans-1,2-dichloroethylene		ND	
chloroform		ND	
1,2-dichloroethane		ND	
1,1,1-trichloroethane		ND	
carbon tetrachloride		ND	
bromodichloromethane		ND	
bis-chloromethyl ether		ND	
1,2-dichloropropane		ND	
trans-1,3-dichloropropene		ND	
trichloroethylene		ND	
dibromochloromethane		ND	
cis-1,3-dichloropropene		ND	
1,1,2-trichloroethane		ND	
benzene		ND	
2-chloroethylvinyl ether		ND	
bromoform		ND	
tetrachloroethene		ND	
1,1,2,2-tetrachloroethane		ND	
toluene		ND	
chlorobenzene		ND	
ethylbenzene		ND	

ND = < 1 µg/1
ND* = < 40 µg/1

Project 7-468-001 GCA Control No. 35290

DATA REPORT SHEET

Purgeables

Sample I.D. Composite Scrubber Water(Contaminated) Run 2 Analysis Date 1/19/84

Sample Matrix Water Instrument HP 5985 GC/MS

Parameter	Ion Used To Quantitate	Concentration (µg/1)	Remarks
chloromethane		ND	
dichlorodifluoromethane		ND	
bromomethane		ND	
vinyl chloride		ND	
chloroethane		ND	
methylene chloride	84	13	
acrolein		ND*	
acrylonitrile		ND*	
trichlorofluoromethane		ND	
1, dichloroethylene		ND	
1,1-dichloroethane		ND	
trans-1,2-dichloroethylene		ND	
chloroform		ND	
1,2-dichloroethane		ND	
1,1,1-trichloroethane		ND	
carbon tetrachloride		ND	
bromodichloromethane		ND	
bis-chloromethyl ether		ND	
1,2-dichloropropane		ND	
trans-1,3-dichloropropene		ND	
trichloroethylene		ND	
dibromochloromethane		ND	
cis-1,3-dichloropropene		ND	
1,1,2-trichloroethane		ND	
benzene		ND	
2-chloroethylvinyl ether		ND	
bromoform		ND	
tetrachloroethene		ND	
1,1,2,2-tetrachloroethane		ND	
toluene		ND	
chlorobenzene		ND	
ethylbenzene		ND	

ND = < 1.0 µg/1
ND* = < 40 µg/1

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Project 7-468-001 GCA Control No. 35293

DATA REPORT SHEET

Purgeables

Sample I.D. Composite Scrubber Water (Comtaminated) Run 3 Analysis Date 1/19/84

Sample Matrix Water Instrument Finnigan MAT OWA GC/MS

Parameter	Ion Used To Quantitate	Concentration (µg/l)	Remarks
chloromethane		ND	
dichlorodifluoromethane		ND	
bromomethane		ND	
vinyl chloride		ND	
chloroethane		ND	
methylene chloride	84	8.5	
acrolein		ND*	
acrylonitrile		ND*	
trichlorofluoromethane		ND	
1,1-dichloroethylene		ND	
1,1-dichloroethane		ND	
trans-1,2-dichloroethylene		ND	
chloroform		ND	
1,2-dichloroethane		ND	
1,1,1-trichloroethane		ND	
carbon tetrachloride		ND	
bromodichloromethane		ND	
bis-chloromethyl ether		ND	
1,2-dichloropropane		ND	
trans-1,3-dichloropropene		ND	
trichloroethylene		ND	
dibromochloromethane		ND	
cis-1,3-dichloropropene		ND	
1,1,2-trichloroethane		ND	
benzene		ND	
2-chloroethylvinyl ether		ND	
bromoform		ND	
tetrachloroethene		ND	
1,1,2,2-tetrachloroethane		ND	
toluene		ND	
chlorobenzene		ND	
ethylbenzene		ND	

ND = < 1 µg/l
ND* = < 40 µg/l

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Project 7-468-001

GCA Control No. 35257

DATA REPORT SHEET

Principal Organic Hazardous Constituents

Sample I.D. Ash Composite

Date of Analysis 1/25/84

Sample Matrix Ash

Instrument Finnigan MAT OWA GC/MS

Component	Quantitative Ion	Concentration (mg/kg)
trichlofluoromethane		ND
1,1,1-trichloroethane		ND
trichloroethene		ND
tetrachloroethene		ND
1,1,2-trichloro 1,2,2-trifluoroethane		ND

ND = < 10 mg/kg

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DATA REPORT SHEET

Metals

Sample I.D. Ash Composite Analysis Date 12/22/83

Sample Matrix EP Toxicity Leachate

Element	Instrument	Concentration (mg/1)	Remarks
Arsenic	Jarrell-Ash 855 ICPS	< 0.03	
Barium	Jarrell-Ash 855 ICPS	0.073	
Cadmium	Jarrell-Ash 855 ICPS	0.012	
Chromium(VI)	Jarrell-Ash 855 ICPS	< 0.003	
Lead	Jarrell-Ash 855 ICPS	< 0.02	
Mercury	Perkin-Elmer 2380 AAS	< 0.0005	
Selenium	Jarrell-Ash 855 ICPS	< 0.02	
Silver	Jarrell-Ash 855 ICPS	< 0.001	
Beryllium	Jarrell-Ash 855 ICPS	< 0.001	
Iron	Jarrell-Ash 855 ICPS	0.648	

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DATA REPORT SHEET

Metals

Sample I.D. Homogenized Sludge Analysis Date

Sample Matrix EP Toxicity Leachate

Element	Instrument	Concentration (mg/l)	Remarks
Arsenic	Jarrell-Ash 855 ICPS	< 0.03	
Barium	Jarrell-Ash 855 ICPS	0.305	
Cadmium	Jarrell-Ash 855 ICPS	0.071	
*Chromium (VI)	Varian DMS 80 UV/VIS	< 0.04	
Lead	Jarrell-Ash 855 ICPS	0.456	
Mercury	Perkin-Elmer 2380 AAS	0.0005	Cold Vapor Method
Selenium	Jarrell-Ash 855 ICPS	< 0.02	
Silver	Jarrell-Ash 855 ICPS	< 0.001	
Beryllium	Jarrell-Ash 855 ICPS	< 0.001	
Iron	Jarrell-Ash 855 ICPS	< 0.004	

*Method 307B of Standard Methods for the Examination of Water and Wastes, 15th Edition, 1980.

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