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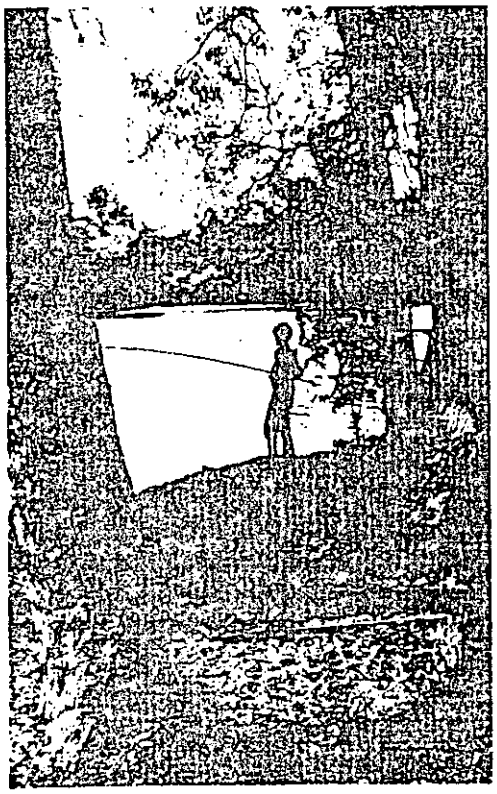

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EVALUATION OF A COATED BAGHOUSE AT A SECONDARY ALUMINUM SMELTER

Contract No. 68-02-1402

OCTOBER 1976

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REFERENCE 73-153-015

February 11, 1977

Mr. John Bools
United States Environmental
Protection Agency
I E R L
Metals and Inorganic Chemicals Branch
Cincinnati, Ohio 45268

Dear John:

Attached please find 5 copies of our draft report on the assessment of the performance and reliability of the coated baghouse at Rochester Smelting and Refining, Inc.

If I can be of any assistance please let me know.

Sincerely,

Robert A Baker

Robert A. Baker, P.E.
Group Leader, Industrial Studies

RAB/dc

Enclosures

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EVALUATION OF A COATED BAGHOUSE AT A SECONDARY ALUMINUM SMELTER

by

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Contract No. 68-02-1402

EPA Project Officer: M. Stasikawski

Industrial Environmental Research Laboratory
Metals and Inorganic Chemicals Branch
Cincinnati, Ohio 45268

Prepared for

U.S. Environmental Protection Agency
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Washington, D.C. 20460

October 1976

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ABSTRACT

This report assesses the use of a coated baghouse to control emissions from charging and demagging operations at a secondary aluminum smelter. On-site sampling was conducted for characterization of the emissions generated by specific tests for total particulates, particle sizing, elemental analysis of particulates, polynuclear aromatics, sulfur dioxide, sulfur trioxide, total methane hydrocarbons, total chlorides, and total fluorides. Operating data supplied by the smelter is evaluated as to the system's reliability.

The coated bahouse system was found to be reliable and very effective for reducing emissions of particulate matter, sulfur trioxide, chlorides, and fluorides. It had no effect on the total methane hydrocarbon emissions. Particulate emissions from the baghouse are characterized as containing small quantities of copper, aluminum, sodium, potassium, and zinc.

This report was submitted in fulfillment of Contract No. 68-02-1402 by Environmental Science and Engineering, Inc., under the sponsorship of the U.S. Environmental Protection Agency. This report covers a period from 10/17/76 to 10/22/76, and work was completed as of 10/22/76.

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SECTION 1

INTRODUCTION

In 1973, Rochester Smelting and Refining Co. installed a coated baghouse, designed by Teller Environmental Systems, Inc., in order to reduce particulate and gaseous emissions from the charging wells on their three reverbatory furnaces. Subsequently, a U.S. EPA demonstration contract was obtained by the smelter to show the effectiveness of this control facility during their charging and demagging operations. In 1974, Clayton Environmental Consultants, Inc., was contracted by EPA to conduct a series of emission tests on the facility in order to characterize its major particulate and gaseous emissions during operations of fluoride fluxing and chlorine demagging. However, the chlorine demagging equipment was not installed, therefore, that aspect of the testing program could not be completed.

In August of 1976, Environmental Science and Engineering, Inc. (ESE) was assigned Task Order #015 of EPA Contract No. 68-02-1402 to perform a testing and evaluation program of the coated baghouse. The sampling was to be performed during demagging operations using chlorine and to include the following parameters:

1) Particle size distribution at the inlet and outlet of the baghouse,
2) Simultaneous measurement of average particulate loading at the baghouse inlet and outlet,
3) Velocity, temperature, moisture, flue gas composition, sulfur dioxide and sulfur trioxide determinations as required,
4) Characterization of the particulate and gas composition (chlorides, polynuclear aromatics, chlorine, etc.), and
5) Analyze and evaluate the testing and operating data (compiled by Rochester Smelting and Refining Co.) and prepare a final report of the systems performance and reliability.

During ESE's preparation of the work plan, the scope of work was extended to include possible sampling during charging and fluorine demagging periods if chlorine demagging could not be performed continuously. The field testing work was to be performed during the week of October 10th, 1976. However, it was postponed until the week of October 17th due to a malfunction of an electrical transformer at the plant.

SECTION 2

CONCLUSIONS

The results of the testing program have demonstrated that the use of a coated baghouse is very effective for reducing certain emissions from a charging well of an aluminum reverbatory furnace. As shown in Table 2-1, the total particulate emissions were reduced 84 percent, sulfur trioxide emissions were reduced 83 percent, chlorine emissions reduced 84 percent, hydrogen chloride emissions were reduced 97.4 percent and total fluoride emissions were reduced 75.4 percent.

The particulate emissions were sized with an in-stack impactor and found to have an average particle size of .4 microns at both the inlet and outlet locations. The particulate control efficiency of 84 percent is within the expected range for a typical baghouse when the exhaust gases have particles of this size range. Therefore, the coating on the bags does not appear to affect the overall collection efficiency.

Surprisingly, the records of Rochester Smelting and Refining do not report a significant weight gain when collecting the used coating material. This is interesting since the average inlet emission rate was 11.75 lbs/hr and the bags are used from several days to more than a week without recoating.

The sulfur dioxide emissions at the inlet and outlet were consistently below the test method's stated detection limits. So, the sulfur dioxide collection efficiency of the coated baghouse was questionable.

The outlet levels of sulfur trioxide were found to be below the test method's detectable limit while inlet levels were much higher. Therefore, the calculated efficiency of 83 percent may be lower than actual. In any case, the control system appears to be effectively reducing sulfur trioxide emissions.

The efficiencies determined for chlorine and hydrogen chloride were 84 percent and 97.4 percent, respectively. These results show that the coated baghouse is very effective for emissions during chlorine demagging. It was noted that the charging well hoods and chlorination well designs were inadequate to prevent severe in-plant ventilation problems during chlorine demagging. The "bell" would leak some emissions around the sides of the well where the heavier-than-air gases would not be captured by the hoods' inlet flow.

A reduction of 75.4 percent for total fluorides was demonstrated by ESE's tests. While this is not as high as some efficiencies for other contaminants, it does show a significant reduction by passing through the coated baghouse.

Other than sulfur dioxide, the only other contaminant not positively shown to be effectively controlled by the system was total methane hydro-

Table 2-1. Summary of Testing Results

Parameters	Results* (Averages)
Total Particulate	Baghouse is 84% efficient [11.75 lbs/hr in vs. 9.95 lbs/hr out]
Particle Size	Size distribution is approximately the same at inlet and outlet [D50 In = 0.4μ vs. D50 Out = 0.4μ]
Sulfur Dioxide	Baghouse is 54% efficient [0.71 lbs/hr in vs. 0.43 lbs/hr out]
Sulfur Trioxide	Baghouse is 83% efficient [1.81 lbs/hr in vs. 0.63 lbs/hr out]
Total Hydrocarbons (as Methane)	Baghouse has little or no effect on gaseous hydrocarbons [46 ppm in vs. 40 ppm out]
Chlorine	Baghouse is 84% efficient [7.66 lbs/hr in vs. 1.29 lbs/hr out]
Hydrogen chloride	Baghouse is 97.4% efficient [5.06 lbs/hr in vs. 0.28 lbs/hr out]
Polynuclear Aromatic Hydrocarbons	None found in the particulate samples.
Total Fluoride	Baghouse is 75.4% efficient [1.23 lbs/hr in vs. 0.23 lbs/hr out]
Elemental Analysis of Outlet Dust	SiO 50%, Fe 20%**, Sr 0.0% Na 2.0%, Cu 7.5%, Cd 0.5% K 2.0%, Pb 0.3%, Mg 0.6% Al 3.5%, Zn 1.0%, Ca 0.3%

* The average emission rates are from all test runs while the average efficiencies are only from those runs performed simultaneously.

** Suspect rust contamination.

carbon. A comparison on strip-chart data shows no difference in peak magnitudes or frequency between the inlet and the outlet.

The elemental analysis of the outlet dust reported quantities of Iron (20%), Copper (7.5%), Aluminum (3.5%), Sodium (2%), Potassium (2%), Zinc (1%), and all other metals (less than 1%). The high quantity of Iron is believed to be from contamination from the sampler.

Samples of the particulate catch were analyzed for polynuclear aromatics, however none were detectable.

Table 2-2 is a summary of the operation log books of the four furnaces during the testing program. Each furnace was operated on its own schedule due to specific product requirements. Except for the time during fluoride and chloride testing, no attempt was made to adjust production with sampling. In fact, fluoride demagging, charging and idle periods of operation were randomly occurring during all sampling periods.

A review of the monthly operating reports showed no serious maintenance problems with the baghouse system. Some bags have been replaced over the last year but this is expected with any baghouse system.

A problem of in-leakage was apparent during our testing period since flows were up to 16,000 DSCM greater at the outlet. This is only significant in that it reduces the effective capture zone of the exhaust hood at charging wells.

In summary, the coated baghouse system proved to have very effective collection efficiencies for particulate emissions, sulfur trioxides, chlorides, and fluorides. It was not shown to have good collection characteristics for total methane hydrocarbons. Particualte emissions contained small quantities of Copper, Aluminum, Sodium, Potassium, and Zinc. An overall assessment of the system's reliability is that it is very good and its effectiveness on reducing the emissions of most test contaminants is also very good.

Table 2-2. Summary of Process Data

Furnace	10/18/76	10/19/76	10/20/76	10/21/76	10/22/76
No. 1					
Clean Scraps & Material (lbs)	13,018	15,386	25,121	27,278	27,822
Dirty Scraps (lbs)	24,411	31,851	17,321	15,352	15,927
Flux (lbs)	6,000	6,000	6,000	6,000	6,000
Skimmed (lbs)	10,300	5,405	3,274	9,090	7,274
Finished Product (lbs)	38,554	39,374	39,948	40,024	29,716
Demagging Type	Fluorine	Fluorine	None	None	None
No. 2	----------Not operating----------				
No. 3					
Clean Scraps & Material (lbs)	10,496	5,393*	6,642	11,474	9,745
Dirty Scraps (lbs)	6,018	11,982	7,976	6,516	5,010
Flux (lbs)	4,000	4,000	4,000	4,000	4,000
Skimmed (lbs)	1,736	2,330	5,394	5,599	2,294
Finished Product (lbs)	16,650	13,440	15,740	14,280	19,970
Demagging Type	Fluorine	Fluorine Chlorine	Fluorine Chlorine	Fluorine	Fluorine Chlorine
No. 4					
Clean Scraps & Material (lbs)	52,292	46,629	47,622	39,411	43,319
Dirty Scraps (lbs)	8,546	11,254	5,842	14,420	15,453
Flux (lbs)	6,000	6,000	------	6,000	6,000
Skimmed (lbs)	7,341	8,842	------	9,718	5,531
Finished Product (lbs)	46,780	54,084	44,268	51,784	56,182
Demagging Type	Fluorine	Fluorine	Fluorine	Fluorine	Fluorine

*All added during night shift

SECTION 3

PROCESS DESCRIPTION

General

The operation of a typical secondary aluminum reverbatory furnace, similar to those at Rochester Smelter and Refining, Inc., consists of charging scrap metals with a high aluminum content; melting the scrap by firing fuel oil; fluxing for removal of impurities; alloying, mixing, demagging and degassing to achieve specifications, and skimming of floating impurities. Finally, the furnace is tapped to obtain molten aluminum. A secondary smelter utilizes metal wastes which contain a high percentage of aluminum and impurities of magnesium, copper, silicon, zinc, iron, and other metals. These are classified as clean or dirty depending on their physical state; oil coated, painted, etc. The dirty scraps are not usually pretreated on-site, except that some of the oily scraps are degreased by passage through a direct fired rotary kiln.

The charge is introduced into the furnace through a charging well, where it melts and flows through a bottom connection into the combustion zone of the furnace. A flux is applied to float some of the impurities and also to create a cover to help prevent oxidation with air. The molten metal is analyzed for composition and is adjusted to specifications by alloying, mixing, demagging and degassing. The demagging process which reduces the content of magnesium in the melt is of interest for this testing project. Essentially, there are two primary methods of demagging in use. The more common demagging procedure is to add fluorine, in the form of aluminum fluoride, to form magnesium fluoride which floats to the top of the molten metal. This is a relatively simple operation which requires no additional process equipment.

The other demagging operation is performed by lancing gaseous chlorine into the lower portion of the charging well to form magnesium chloride. Chlorine demagging is susceptible to over-chlorination which creates ventilation problems within the plant as well as odors outside the plant. Due to the toxic nature of chlorine gas, a special charging well bell and scrubber system is required to try to contain the excess chlorine gas and chloride emissions, as shown in Figure 3-1.

The semi-solid impurities formed by the above mentioned steps are manually skimmed with ladles and put into containers. The final step is to tap the furnace and to pour the molten metal into a "shot" machine (vibration disc) or a billet log machine or a refractory lined container for transfer to a nearby foundry. Production records are maintained to indicate the amount and type of materials in the charge as well as the amount of flux, skimmings, and the molten metal tapped.

Specific

Rochester Smelting and Refining Company operates four reverbatory furnaces for smelting of aluminum scraps. Each furnace has two separate

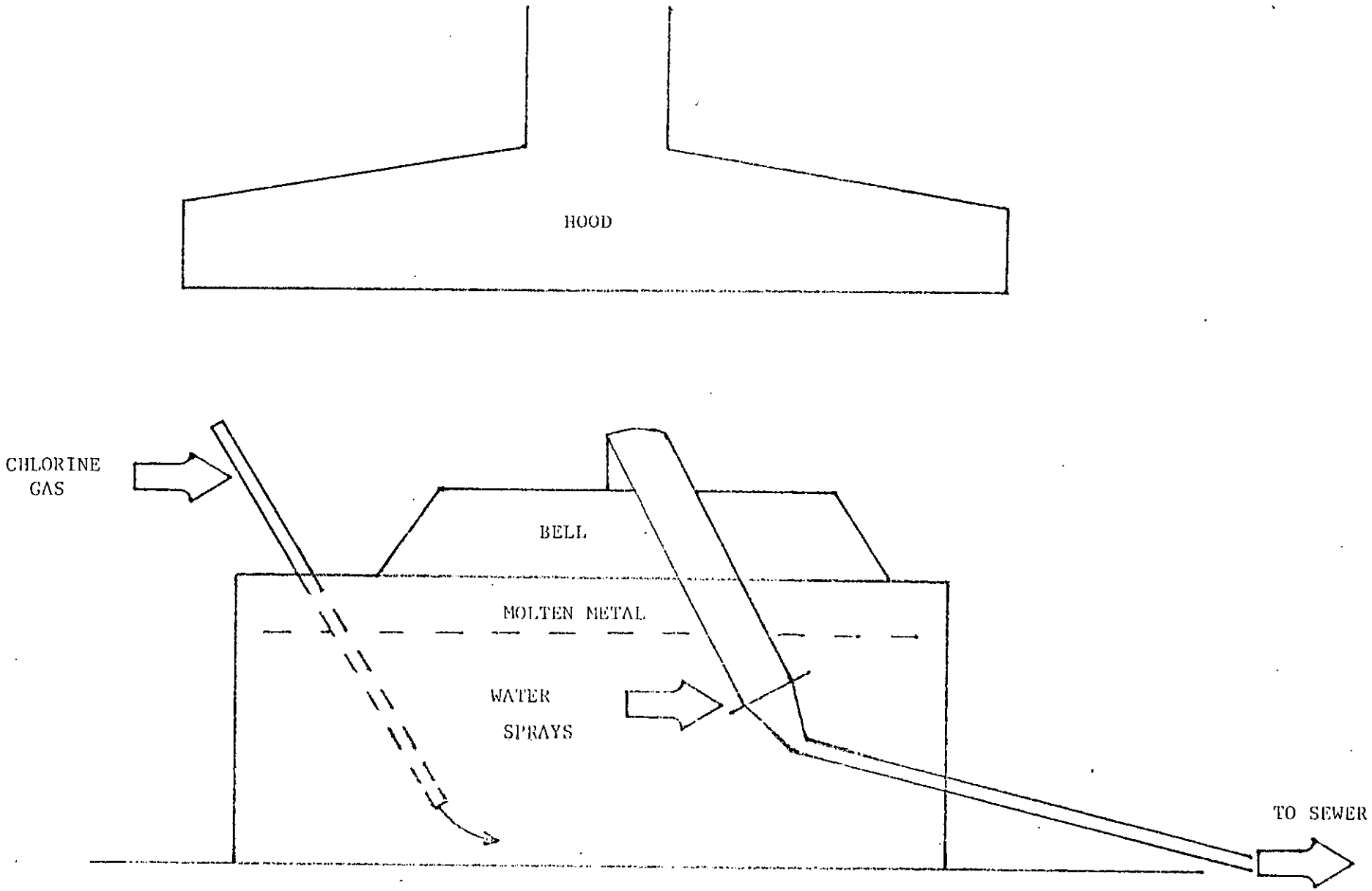


Figure 3-1. Chlorination "Bell" In-Place at Charging Well

exhausts as shown in Figure 3-2. The combustion gases from firing fuel oil are exhausted through the combustion zone stack. Due to the impingement of the flame through part of the stack, these exhaust gases do not appear to present a significant particulate emission problem. The charging well is separated from the combustion zone as are its emissions which are vented through an adjacent hood. The major emissions from the charging well are caused during the charging and demagging phases of the operation. Based on recent EPA contracts studying this process, emissions during charging are generally related to aerosols of unburned hydrocarbons, while emissions caused by demagging can be a mixture of many different gases and aerosols. A summary of the expected emissions is presented in Table 3-1.

In 1973, a coated baghouse system, designed by Teller Environmental Systems, Inc., was installed. The bag coating known as TESISORB was reported by Teller to act as an acid gas absorber and neutralizer as well as a precoat filter. Once the coating is applied, the baghouse is operated without a shaking cycle to clean the bags until the pressure drop across the baghouse approaches 18" W.C. Depending on specific operational parameters such as type of charge (dirty or clean), number of furnaces on-line and type and amount of demagging operations, it may take from several days to more than a week before a cleaning cycle is needed.

The entire system is a very unique arrangement of charging hoods, flow combiners, flow splitters, dampers, water sprays, an evaporator chamber, baghouse modules and exhaust fans. Figues 3-3 and 3-4 show the essential components.

The charging hoods of the four furnaces are ducted to above the roof, where the original stacks are dampered closed and a horizontal manifold connects all the stacks. However, the present capacity of the cleaning system allows for only three furnaces to be vented at a time. The combined exhaust gases are passed through a water quench duct (sprays are only used when gas temperature is above 260°F) and a large baffled evaporator chamber for water evaporation and larger particulate matter removal. A 2-foot diameter damper is mounted in the side of the evaporator for emergency temperature reduction by mixing ambient air with the exhaust gases. Next the flow is divided for entry into three separate baghouse modules; each has separate inlet and outlet dampers for flow control and contains 504 Dacron tubular bags. When coated, the bags are reported to have an operating range up to 250°F. A hopper collects the spent coating and larger particles during normal operation and the remainder of the coating plus captured particulates and absorbed gases during the bag shaking phase. This material is passed through a rotary valve into a flexible discharge hose for collection into containers for weighing and removal. During coating operations, the module inlet damper is closed and the coating is pulled up a flexible hose and through a special inlet duct into the dirty side of the baghouse by the fan system. The three modules are usually coated with a total of 3,000 pounds of material. It has been reported by Rochester Smelting and Refining Co. that material collected after the cleaning cycle is approximately the same weight.

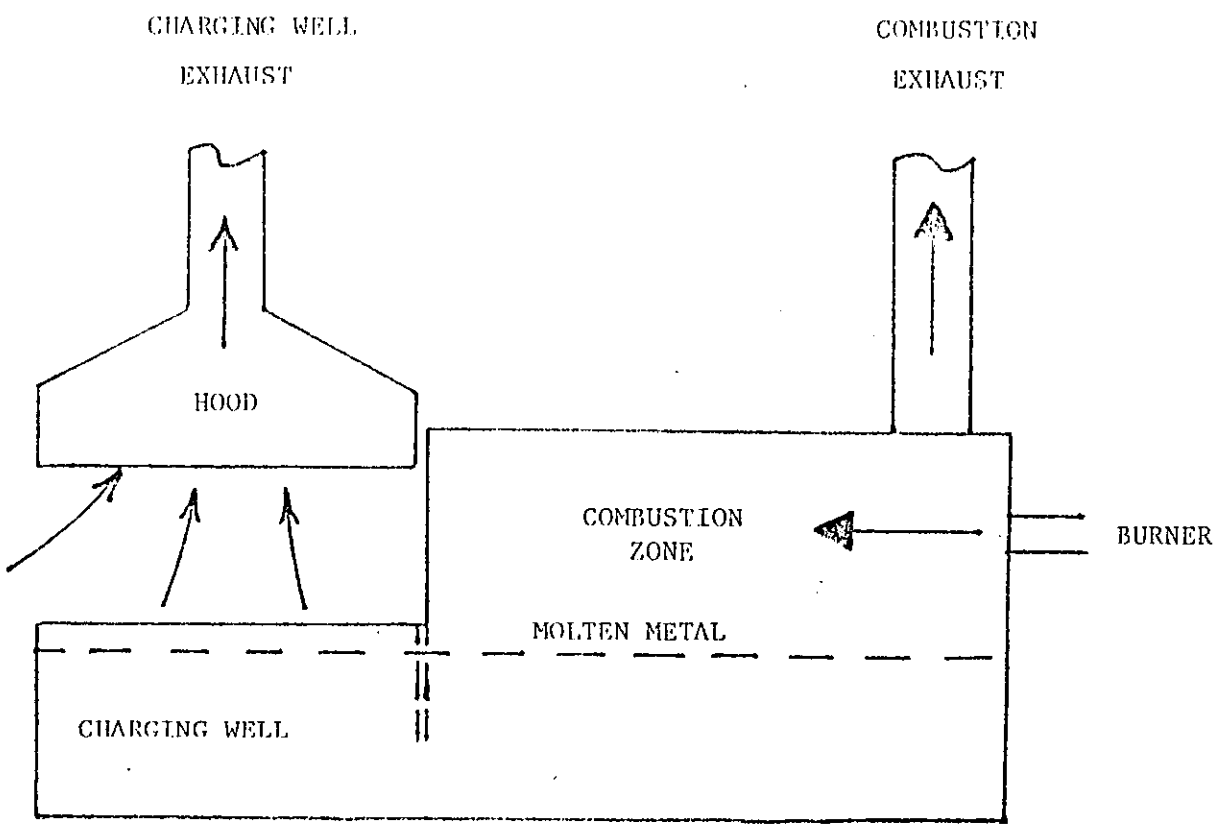
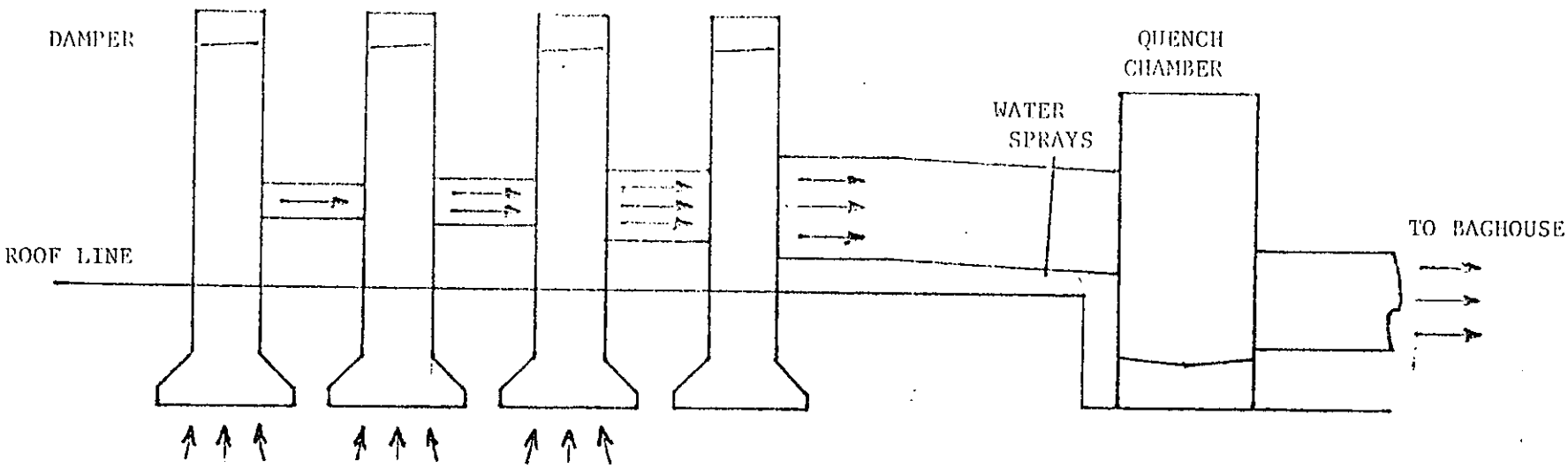


Figure 3-2. Typical Reverbatory Furnace at Rochester Smetling and Refining

Table 3-1. Summary of Expected Emissions from a Secondary Aluminum Reverbatory Furnace.

Process Step	Composition	Remarks
Charging	Fumes and smoke from volatization and/or combustion of scrap contaminants.	
Fluxing, alloying and mixing (N_2)	Gases: Nitrogen, hydrogen fluoride, hydrogen chloride, combustion gases, and other volatile components. Particulate: Major constituent when fluxing with salt-cryolite mixture is sodium chloride with minor amounts of aluminum and magnesium compounds. Additional compounds of Na, Al, Mg, Ca, Fe, Pb, Mn, K, Cr, Zn, and Ni may also be presented.	Fluxing fume samples collected by thermal precipitation were found to contain particles smaller than 2 microns with most of the particles 0.1 micron. The fumes were characterized as corrosive particularly when collected wet. These emissions are converted to air pollution control residuals (either wastewater, sludge, or solid waste) at plants where such systems are implemented. See gas cleaning processes below.
Demagging	Gases: Chlorine, hydrogen halides, volatile metal chlorides, combustion gases (if there is not a separate chlorination chamber). Particulate: Aluminum chloride, aluminum fluoride, aluminum oxide, magnesium chloride, magnesium fluoride, magnesium oxide, calcium chloride, calcium fluoride, sodium chloride, plus additional compounds of Cu, Ni, Cd, and other metals.	The parameters which determine quantities of emissions from demagging are: - metal temperature - chlorine flow rate - magnesium content of alloy - depth of chlorine injection - thickness and composition of dross Air pollution and control systems are usually used to process demagging effluent gases. See below.
Degassing (N_2, Cl_2, or N_2 -Cl_2 mixture)	Severe fuming results only with Cl_2 or N_2-Cl_2 mixtures. Emissions are similar to those from demagging.	The results of one study showed that all fume particles from degassing with chlorine were under 2 microns and 90-95% were less than 1 micron. Mean particle size appeared to be 0.7 microns. Refer to gas cleaning processes below.
Skimming	Minor fugitive emissions.	Can be controlled by hood.
Pouring	Minor atmospheric emissions.	Can be controlled by hood.



CHARGING WELL HOODS

(only three hood exhausts at any one time)

Figure 3-3. Sketch of Charging Well Exhaust System - Part I

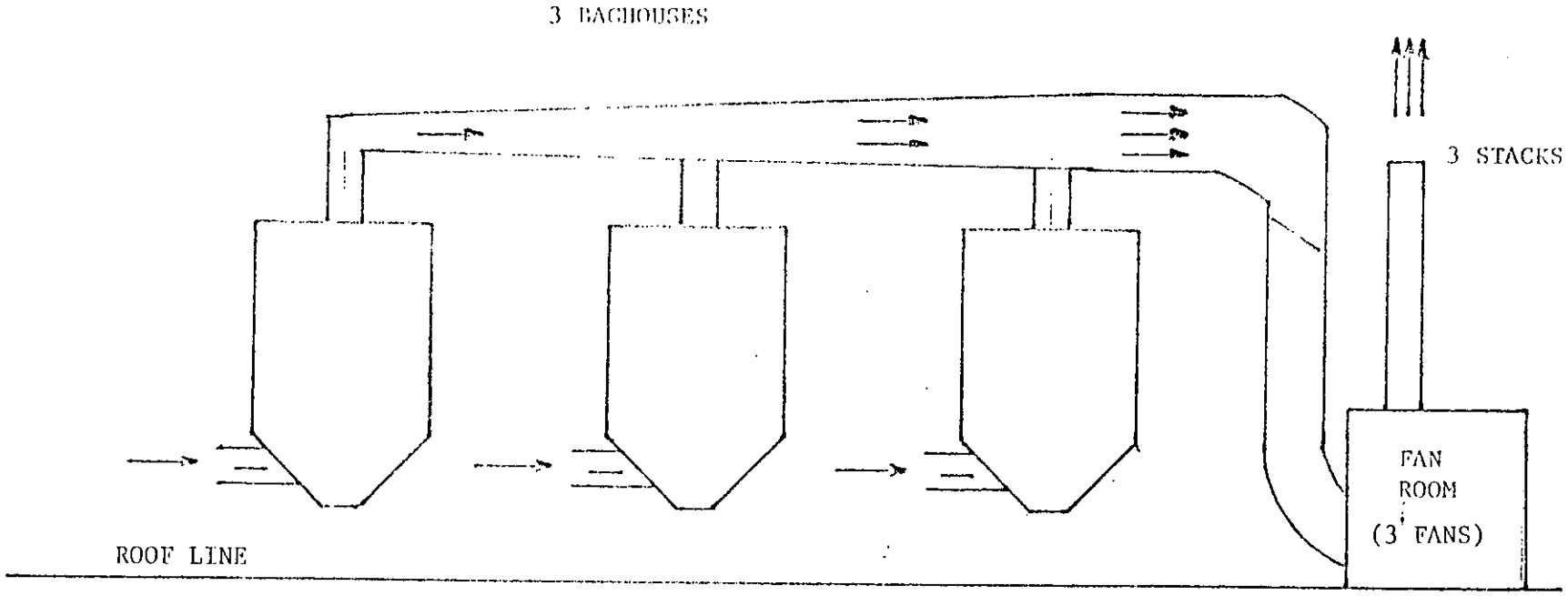


Figure 3-4. Sketch of Charging Well Exhaust System - Part II

The clean air side of the baghouse modules are manifolded together and the flow is immediately divided for entrance into the three fan exhaust system. This was designed to maintain a constant flow throughout the cycle of operation by dampening the inlet to the fans at the initially low pressure drop across the baghouse and compensating for increased pressure drops by opening the fan dampers. Each fan is followed by a stack for discharging the cleaned charging well exhaust gases into the ambient air.

SECTION 4

TEST METHODS

The specific testing methods and analytical procedures followed in this project are outlined in Table 4-1 and are individually described below.

Sampling Locations

The sampling port locations were chosen based on availability of entering locations, flow distributions and practicality of sampling. The combined inlet duct sampling location was modified with additional ports to accommodate several different sampling trains at the same time. Figure 4-1 shows the geometrics of this location. It is termed "inlet" (all exhausts from charging well hoods are combined before reaching this point) throughout this report and represents the only baghouse inlet sampling location for this testing project.

The baghouse outlet had no convenient combined sampling location, so the three outlet ducts and stacks were investigated for testing purposes. The previously established sampling locations on the rectangular ducts leading to the fans were found to be unacceptable due to a high duct vacuum and long nipples on the ports which would have interfered with our testing methods and equipment. A more convenient location was found on each of the fan exhaust stacks as shown in Figure 4-2.

Total Particulate Emissions

The total particulate emissions were sampled by following U.S. EPA [illegible] contained in Methods 1, 2, 3, and 5 of 40 CFR 60, Appendix A. [illegible] were traversed on two axes located at 90 degrees to each [illegible] and had a total of 44 sampling points. Sampling time per point [illegible] in different test runs from 1.5 to 3.0 minutes.

[illegible] of the inlet outlet stacks had two sampling ports set at 90 degrees to each other. At these locations, the ports were only 1.4 diameters [illegible] from the last bend and were not acceptable according to Method 1 [illegible] less than 2 diameters). However, it was decided to sample at these locations after an initial traverse was performed which showed the flows to be fairly uniform. Test run #1 was performed on only one outlet stack with a separate equipment train at each port. This was done in order to develop a sampling schedule which would allow for simultaneous inlet and outlet sampling, as well as obtain two particulate test runs per day. It was found that one outlet train sampling 24 points per stack at a rate of two minutes per point would be acceptable. Thereafter, simultaneous sampling across the total exhaust flow was conducted. The sampling train used is shown in Figure 4.3.

After completion of each particulate test run, the filter was recovered and the prefilter components of the test train were brushed and washed with acetone, and the effluent collected. The filters were stored in their

Table 4-1. Testing Methods and Analytical Procedures

Parameter	Test Method	Analysis
Total Particulates	USEPA Methods 1, 2, 3, 5--simultaneous inlet and outlet sampling	Gravimetric determination of filter and probe wash
Total Fluorides	USEPA Method 13A--simultaneous inlet and outlet sampling	Specific ion electrode
Chlorides (as HCl and Cl_2)	Decker and Hagar Method with alkaline arsenite absorbing reagent for Cl_2 and sodium hydroxide for HCl--simultaneous inlet and outlet sampling	Volhard titration--performed on-site
Total Methane Hydrocarbons	Sample with Teflon tubing from inlet and outlet location--performed alternately	Beckmann Model 400 Flame Ionization Detector--performed alternately
Sulfur Dioxide	USEPA Method 6--simultaneous inlet and outlet sampling	Titrations on site
Sulfur Trioxide	Modified USEPA Method 6--Isopropanol reagent is save for analysis. Simultaneous inlet and outlet sampling	Titrations on site
Particle Sizing	Andersen Mark III Cascade Impactor--performed alternately at inlet and outlet	Filters weighed on-site with electron balance
Respirable Particulates (Elemental Analysis)	Three Cyclone Particulate sampling train with backup filter--performed alternately at inlet and outlet	Spark Source Spectroscopy and Atomic Absorption Analyzer
Total Polynuclear Aromatics (in particulate catch)	Used filters from Total Particulate Sampling	Gas Chromatograph Electron Capture Detector

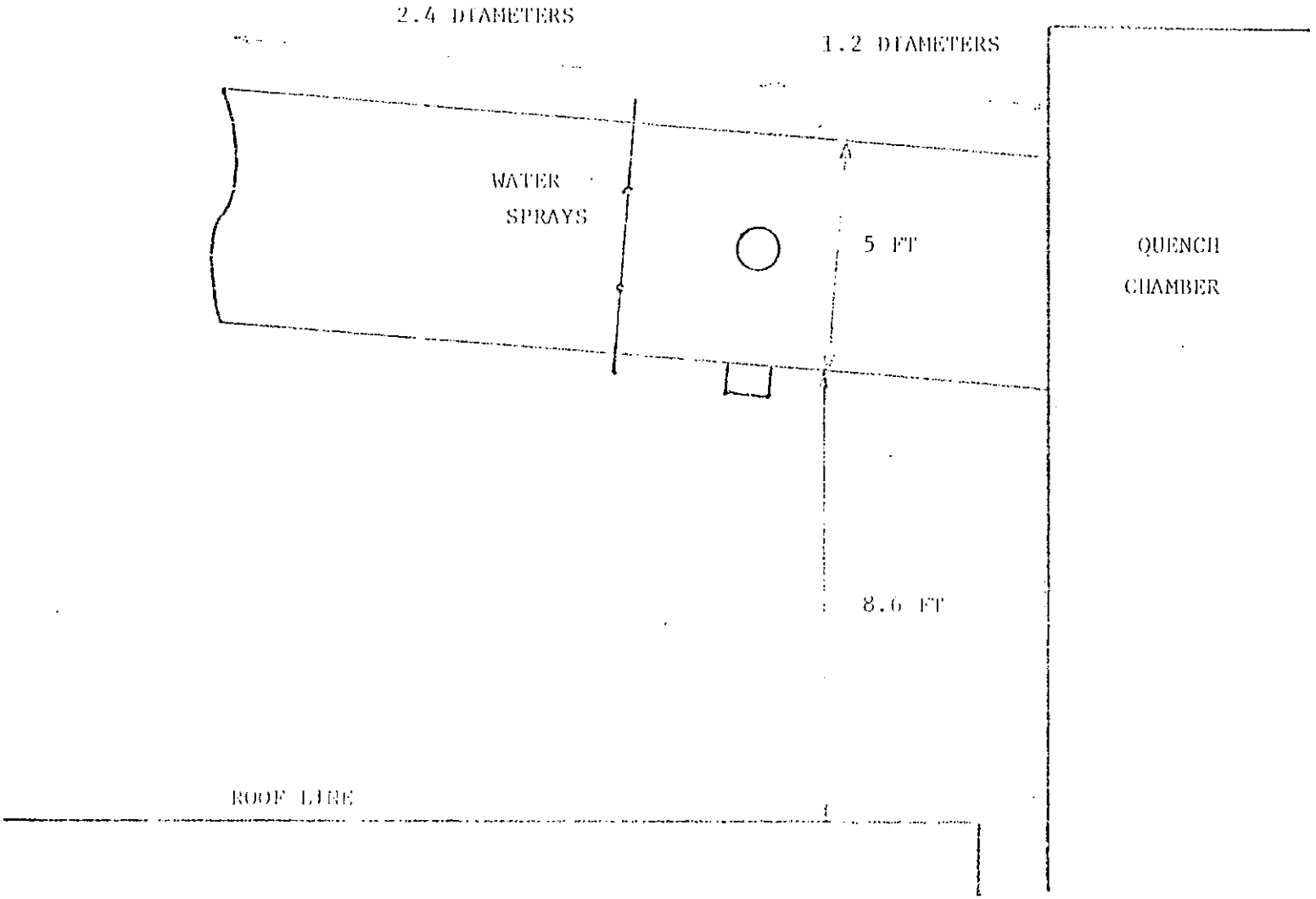
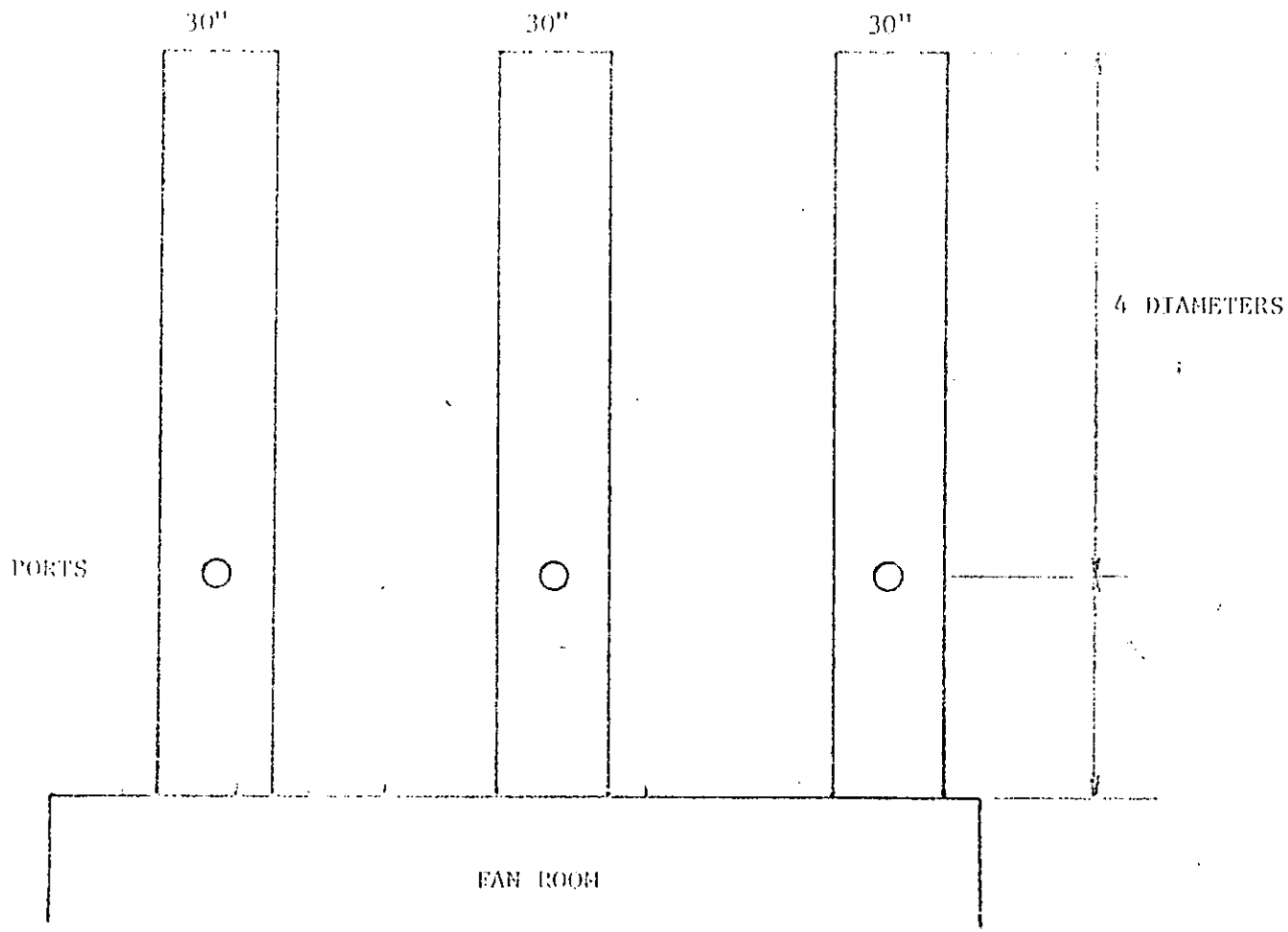
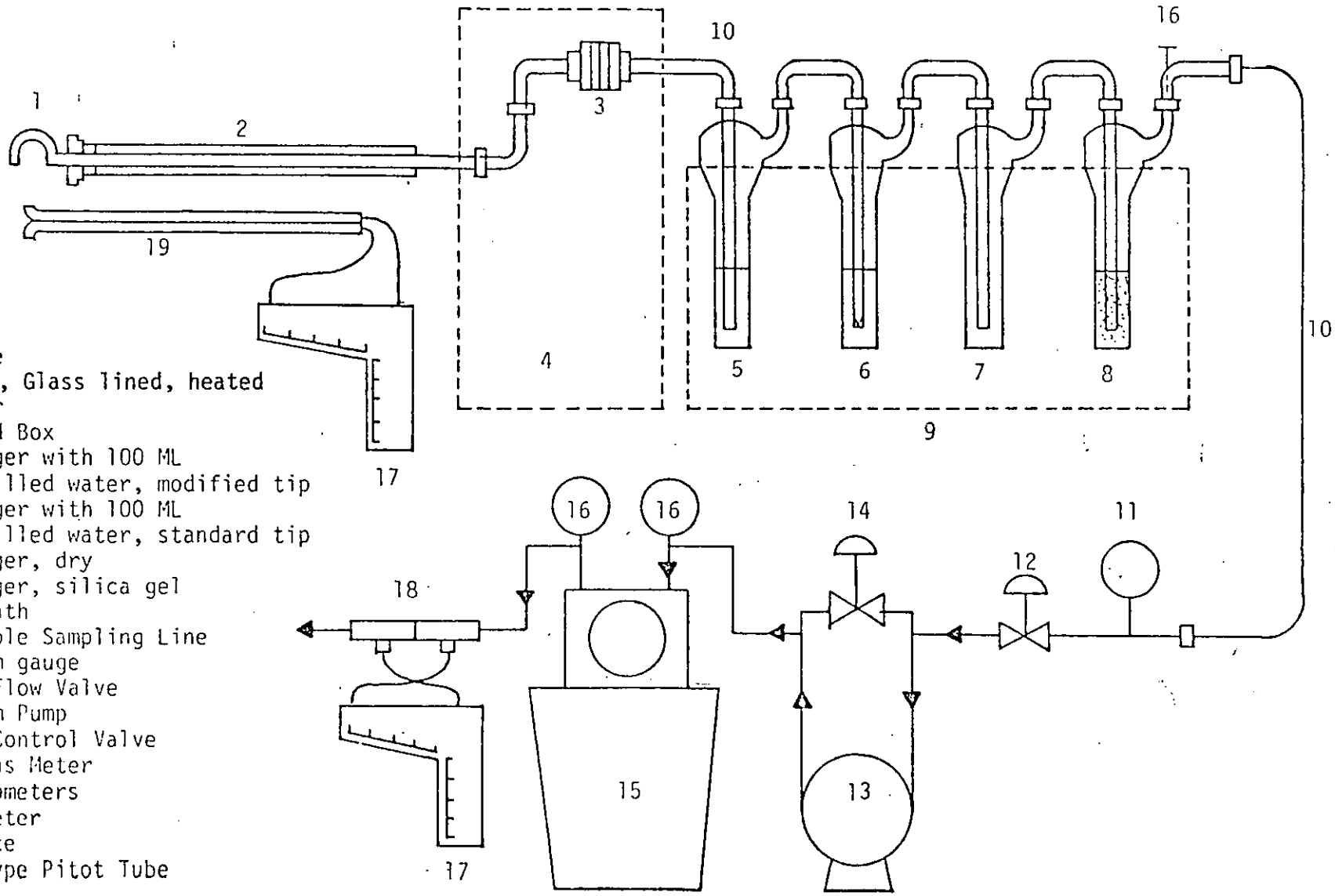


Figure 4-1. Sketch of Inlet Sampling Location

Figure 4-2. Sketch of Outlet Sampling Location





1. Nozzle
2. Probe , Glass lined, heated
3. Filter
4. Heated Box
5. Impinger with 100 ML distilled water, modified tip
6. Impinger with 100 ML distilled water, standard tip
7. Impinger, dry
8. Impinger, silica gel
9. Ice Bath
10. Flexible Sampling Line
11. Vacuum gauge
12. Main Flow Valve
13. Vacuum Pump
14. Fine Control Valve
15. Dry Gas Meter
16. Thermometers
17. Manometer
18. Orifice
19. "S"-type Pitot Tube

Figure 4-3. U.S. EPA Method 5 Particulate Collection Train

original plastic containers while the acetone washes were rinsed into clean borosilicate glass bottles with Teflon-lined tops. Samples were labeled, logged and stored for gravimetric analysis in ESE's Gainesville laboratory.

Overnight Particulate Emission

In an effort to obtain a larger catch for the elemental analysis of the baghouse's particulate emission, a series of four overnight test runs were performed. The sampling trains were started in the evening just before leaving the plant and stopped the next morning. Only one sampling point was used, however the initial sampling rate was established for correct isokinetic conditions. Samples were recovered in the normal manner. The probe and box heaters were "off" for these runs to prevent electrical complications with the inclement weather. Overnight runs 3 and 4 were conducted simultaneously; the train for run 4 did not contain a filter in order for the impingers to catch the total solid and condensed particulate matter without clogging a filter. The total impinger catch as well as probe was analyzed with the other samples.

Total Fluorides

The total particulate test runs 1 and 5 were also used for total fluorides determination per U.S. EPA Method 13B of 40 CFR 60, Appendix A. The normal Method 5 testing trains were modified on these runs by using Whatman filters instead of fiberglass filters and the recovery procedures altered by saving the impinger catch along with a preliminary water wash (no brushing) of the prefilter components (probe and filter holder). A normal brushing and an acetone wash were performed after the water wash; the filter and acetone wash were stored for analysis with the other U.S. EPA Methods test runs. After the filter had been analyzed for weight gain, it was put into the container with its water wash and impinger catch for the total fluoride determination per Method 13B. While it is recognized that some particulate matter may have been lost due to the preliminary water wash, it is not felt to be significant enough to effect the total particulate results.

Particle Sizing

Particle size distribution of the particulate matter in the inlet and outlet gases was measured with an Andersen Mark III Cascade Impactor. The apparatus used had a pre-cut cyclone, eight impaction stages, and a back-up filter. Dessicated fiberglass filters were weighed before and after sampling using a Cahn Model 4400 electrobalance (sensitivity to 0.001 mg). The testing locations were the same as those used for total particulate emission tests, however, only one sampling point at each location was used.

The assembled impactor was inserted into the stack and allowed to reach thermal equilibrium with the gases. The nozzle of the impactor was then turned into the gas stream and immediately sampling was begun. Near isokinetic sampling was achieved by sampling at a rate based on a previous velocity measurement at the single point where the sample was taken. However, changes in the sampling rate were not made to accommodate

changes in the gas velocity as this would change the cut points of the impactor stages. Figure 4-6 shows a schematic of the sampling train. After sampling, the impactor was taken back to the weighing room for sample recovery. This was performed by disassembling the impactor, removing the glass-fiber substrates and brushing the remaining particulate matter from each stage into the sample container with the substrate. Due to the difference in grain loadings between the inlet and outlet locations, the impactor was able to sample for much longer periods at the outlet (4 to 5 hours) than at the inlet (20 to 60 minutes).

Gross Sized Particulates

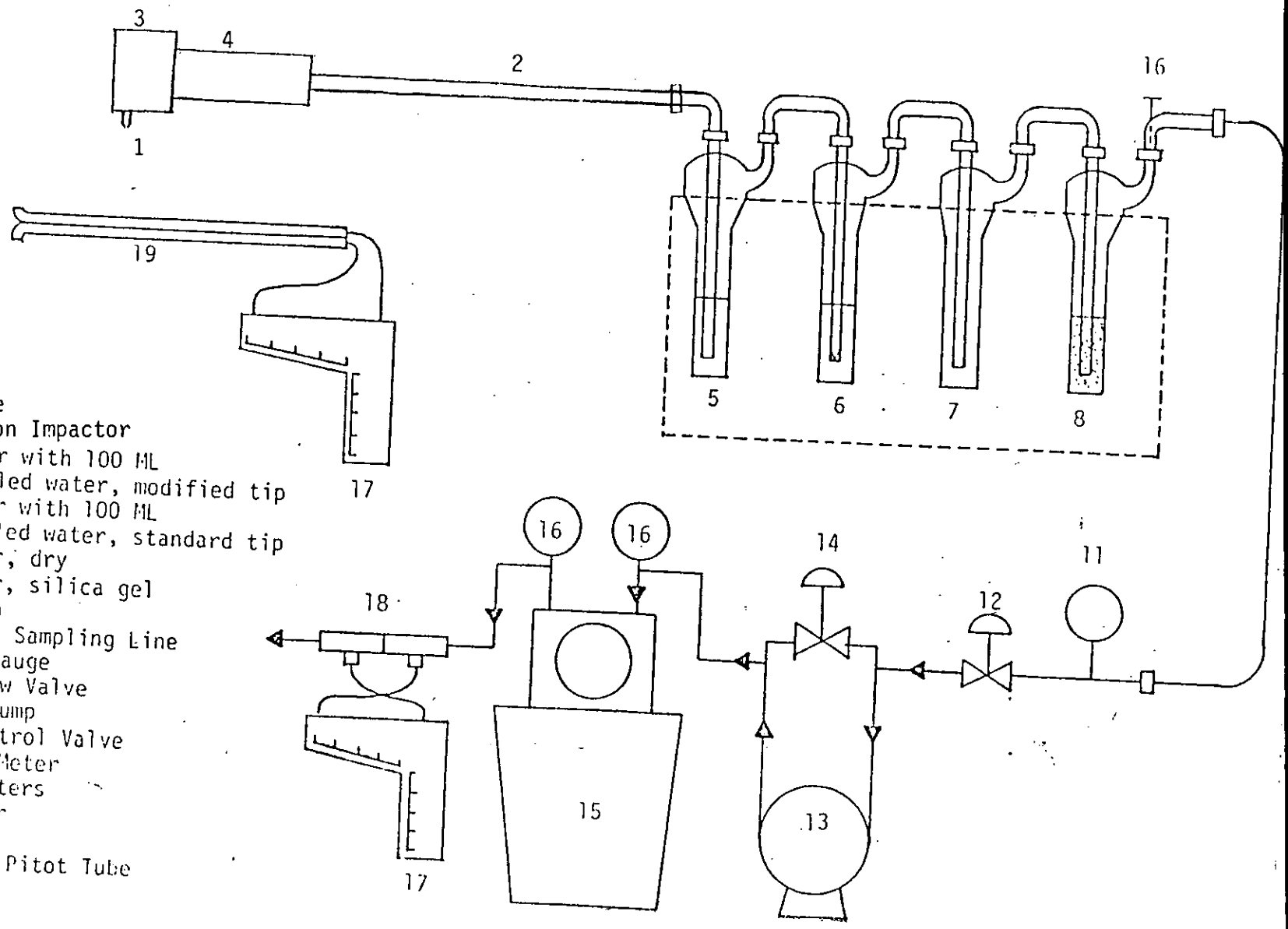
The sample weight of the particulate matter captured in the Andersen Mark III Cascade Impactor is so small as to preclude elemental analysis on the emissions from the coated baghouse. Therefore, gross samples were obtained through the use of a three-stage cyclone sampler shown in Figure 4-7. This sampler was borrowed from the U.S. EPA Industrial Environmental Research Lab in Raleigh, North Carolina. The nominal sampling rate of the cyclones is 1.0 CFM which requires sampling for fairly long periods to obtain large enough samples for analysis. At the baghouse inlet, the sampling period ranged from 20 to 60 minutes, while the outlet sampling period ranged from 360 to 899 minutes. The outlet test runs #2 and #3 were conducted overnight in order to have sufficient time to obtain an adequate sample. Tests were run during the day at the inlet location for possible comparison.

After the sampling run, the sampler was taken to the weighing room and disassembled. Each stage was brushed clean and the captured particulates placed into containers. A backup filter was used to obtain a cummulative relationship of the sample weights. These samples were dessicated and weighed on a Cahn Model 4700 electrobalance having a sensitivity of .0001 mg.

In conjunction with the elemental analysis of the emitted particulate matter, a sample of the baghouse hopper dust was obtained for analysis by spark source spectroscopy. The composition of the TESISORB was obtained from Teller Environmental Systems, Inc., and compared with that of the hopper dust. A final selection of 11 elements was made for quantification of the metals contained in the baghouse emissions. This was performed by using an Atomic Absorbtion Analyzer and using the particulate catches from the three-stage cyclone sampler's outlet runs #1 and #3. Due to problems of high blank values from fiberglass, the results are limited to samples not collected on a filter.

Sulfur Oxides

Sulfur dioxide and sulfur trioxide were samples in accordance with U.S. EPA Method 6 in CFR 60, Appendix A. The method is based on the principle that sulfur trioxide is separated from sulfur dioxide by its selective absorption in isopropyl alcohol. The sulfur dioxide is subsequently absorbed in hydrogen peroxide. Although the method was written specifically for sulfur dioxide determination, it was extended to



1. Nozzle
2. Probe
3. Cyclone
4. Anderson Impactor
5. Impinger with 100 ML distilled water, modified tip
6. Impinger with 100 ML distilled water, standard tip
7. Impinger, dry
8. Impinger, silica gel
9. Ice Bath
10. Flexible Sampling Line
11. Vacuum gauge
12. Main Flow Valve
13. Vacuum Pump
14. Fine Control Valve
15. Dry Gas Meter
16. Thermometers
17. Manometer
18. Orifice
19. "S"-type Pitot Tube

Figure 4-6 Anderson Mark III Cascade Impactor Collection Train

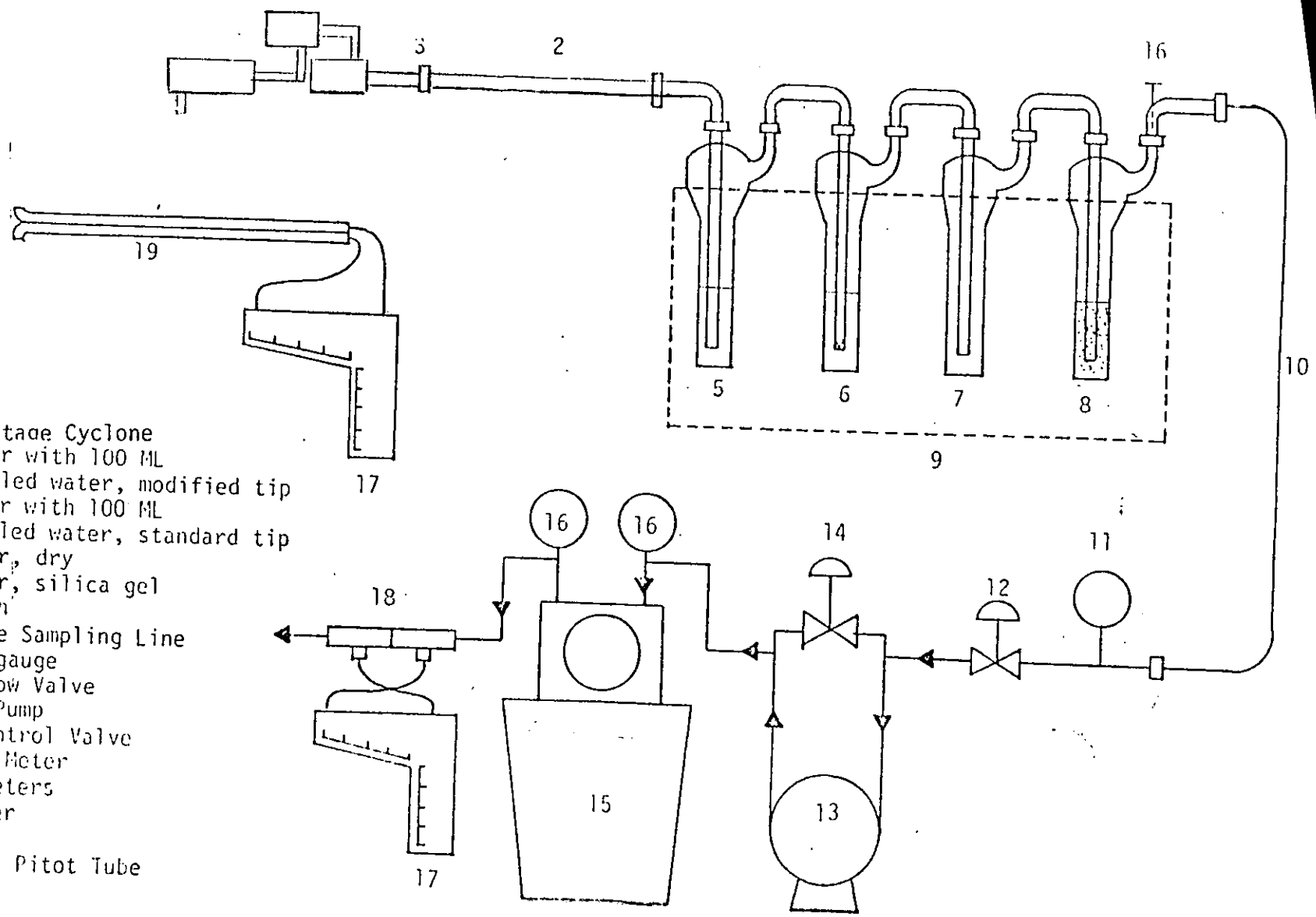


Figure 4-7 Three stage Cyclone Sampling Train

include sulfur trioxide determination by recovery of both absorbing reagents and associated washes. A schematic of the sampling apparatus is presented in Figure 4-4. Sulfur trioxide is scrubbed from the gas stream in the first impinger which contains 15 ml of 80 percent isopropanol; the sulfur dioxide is then removed in two impingers each of which contain 15 ml of three percent hydrogen peroxide. The sulfur dioxide is oxidized and subsequently analyzed by titration with barium perchlorate using thorin as an indicator. Similarly, the glass-wool mist trap, probe washings, and the first impinger's catch and wash are titrated with the barium perchlorate and thorin indicator. The samples were analyzed on-site after completion of a 15 minute purging with ambient air. Each test run was performed at the same sampling location as were the particulate determinations but only at one sampling point.

Chlorides

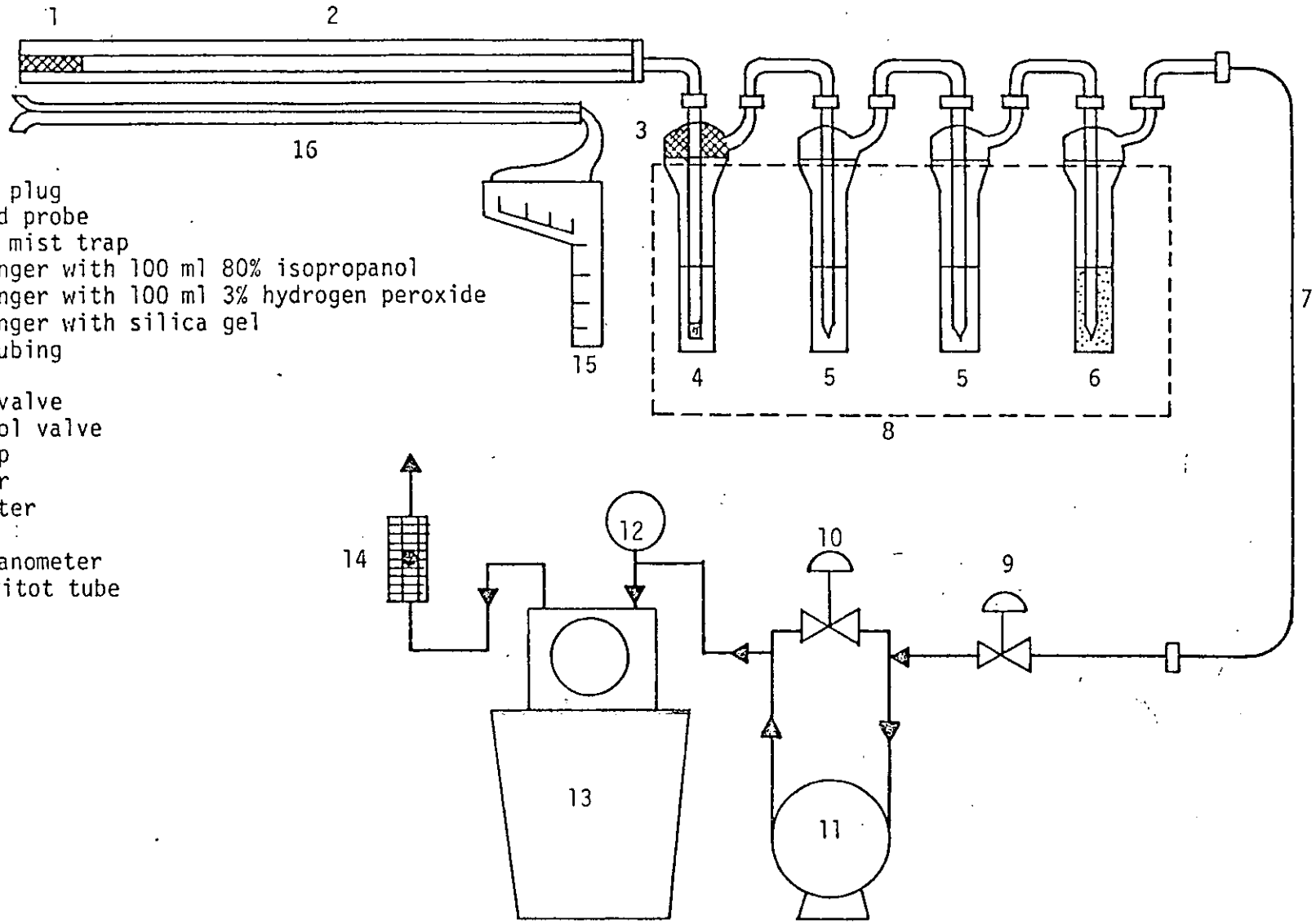
Concentrations of chlorine and hydrogen chloride were measured at both the inlet and outlet of the baghouse to determine the control efficiency of these two chemical species. The method used was that proposed by C.E. Decker and C.B. Hagar in 1968, "Determination of Hydrogen Chloride and Chlorine in Stack Gas." Gas samples were extracted from the appropriate location and passed through a midget impinger containing 15 ml of alkaline arsenite absorbing solution. The gas volume sampled was then determined by passing the clean sample gases through a vacuum pump and dry gas meter as illustrated in Figure 4-5. After a sufficient volume of gas was sampled, the impinger contents were recovered and analyzed for Cl_2/HCl per the method. Chlorine was determined by analyzing the sample for unconsumed arsenite wich reduces the Cl_2 to HCl. The total HCl was then measured using the Volhard titration. The Cl_2 determined in the first titration was then subtracted from the total chlorine found by the Volhard titration to give the free chlorine concentration. As with the sulfur oxides, each test run was conducted at the same locations as were the particulate tests but at only one sampling point.

Gaseous Methane Hydrocarbons

A Beckman Model 400 hydrocarbon analyzer was employed for Total Gaseous Hydrocarbon Determinations as Methane. Separate sampling lines (teflon) were employed for inlet and outlet sampling and were located at the same duct and stack locations as for the particulate sampling. The sample lines were alternately purged and connected to the instrument for approximately 45-minute periods. A chart recorder was connected to the instrument's electrical terminals and the data were reduced on a time weighed basis at a later time. The sampling times were essentially continuous throughout the work hours of the testing program except for periods of checking the zero and span and purging sample lines.

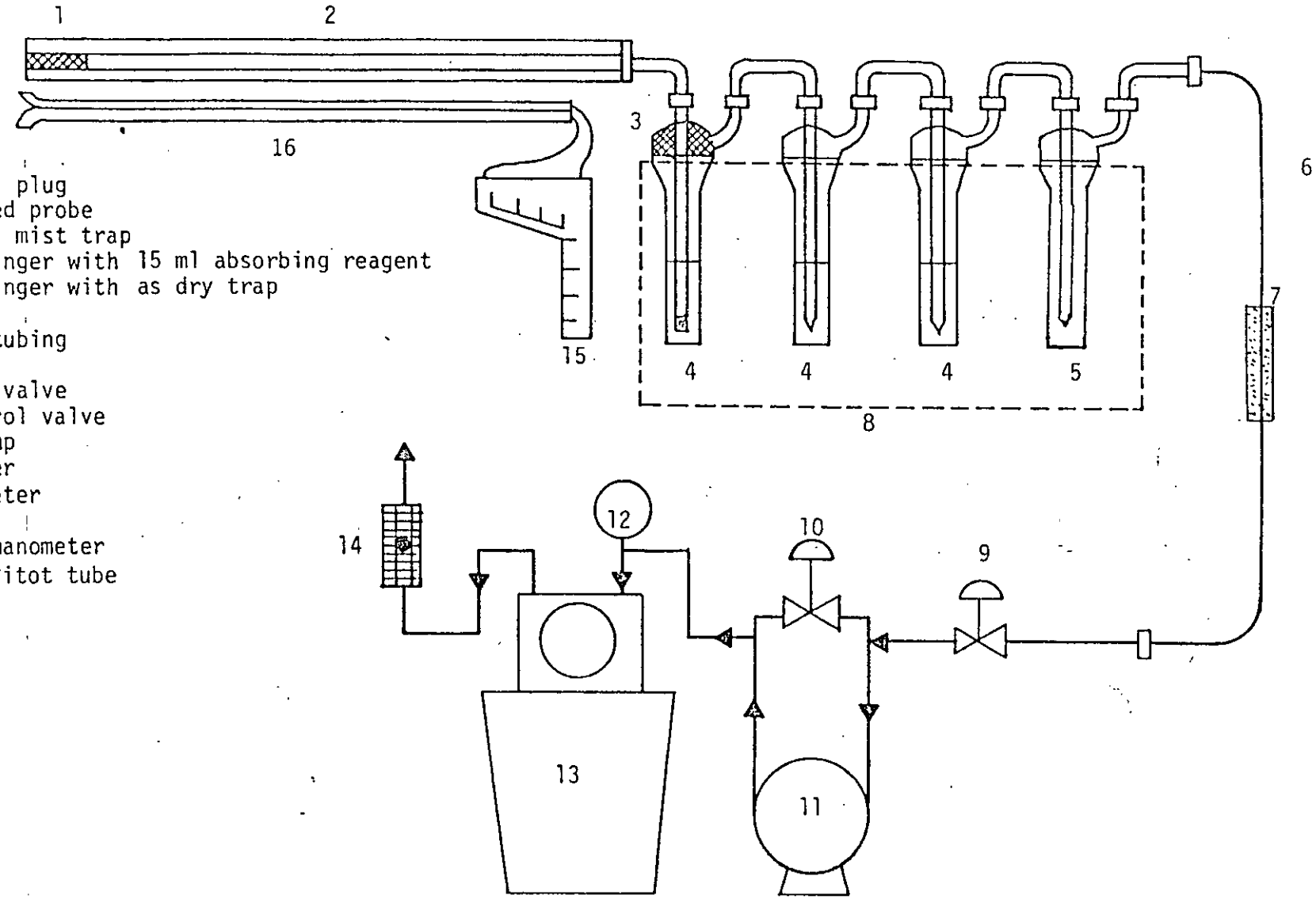
Polynuclear Aromatic Hydrocarbons

Filters from the three-stage cyclone sampler's outlet run #2 and U.S. EPA Method 5 outlet runs #4 and #7 were analyzed for polynuclear aromatic hydrocarbons (PNA). The particulate catch was extracted with methylene chloride, concentrated to 1 ml and analyzed by a gas chromatograph with a



1. Glass-wool plug
2. Glass-lined probe
3. Glass-wool mist trap
4. Glass impinger with 100 ml 80% isopropanol
5. Glass impinger with 100 ml 3% hydrogen peroxide
6. Glass impinger with silica gel
7. Flexible tubing
8. Ice bath
9. Main flow valve
10. Fine control valve
11. Vacuum pump
12. Thermometer
13. Dry gas meter
14. Rotometer
15. Inclined manometer
16. "s" type Pitot tube

Figure 4-4. U.S. EPA Method 6 Sulfur Dioxide Collection Train
(used also for Sulfur Trioxide collection)



1. Glass-wool plug
2. Glass-lined probe
3. Glass-wool mist trap
4. Glass impinger with 15 ml absorbing reagent
5. Glass impinger with as dry trap
6. Silica gel
7. Flexible tubing
8. Ice bath
9. Main flow valve
10. Fine control valve
11. Vacuum pump
12. Thermometer
13. Dry gas meter
14. Rotometer
15. Inclined manometer
16. "s" Type Pitot tube

Figure 4-5. Chlorine and Hydrogen Chloride Collection Train

flame ionization detector. The three chromatographs were compared with the chromatograph for α-Naphthyl Amine a P.N.A. hydrocarbon. This was to determine if additional analyses on a gas chromatograph-mass spectroscopy unit would be beneficial.

Copies of all the testing and/or analytical procedures are in Appendix B.

SECTION 5

RESULTS AND DISCUSSION

The testing program was conducted on October 18th, 19th, 20th, 21st, and 22nd of 1976 with the assistance of personnel from Rochester Smelting and Refining Company. The scheduled work plan was modified in the field to obtain that which is shown in Table 5-1. The overall consideration was to perform the various tests (except for Total Chlorides) on a regular basis regardless of the furnace operations. A review of the process data presented in Section 2, shows that only furnaces #1, #3, and #4 were operated during our testing schedule. However, for production reasons, a definite pattern of all furnaces performing the same operation could not be established. For example, on the second and fifth day, chlorine demagging was performed on furnace #3 while the other two furnaces were charging, using fluorine for fluxing and demagging, and were idle at various times during the day. Throughout the week of testing, this pattern of random and different operation on each of the furnaces continued. Therefore, it would be in error to interpret the following data as anything but as emissions occuring from three charging wells during various phases of operation.

Particulate Emissions

The total particualte results from the U.S. EPA Method 5 sampling are shown on Tables 5-2, 5-3, and 5-4.

The inlet emission rates varied from 5.56 lbs/hr to 17.06 lbs/hr while the outlet emission rates ranged from 0.19 lbs/hr to 26.85 lbs/hr. Several runs had higher emission rates at the outlet than at the inlet during the same time period. A review of the individual filter and probe-wash gravimetric results show that for the inlet samples most of the particulate matter collected came from the filter, while this was not true for the outlet samples. Outlet runs #1 and #8 had larger net weight gains in their washes while runs #2, #5, and #7 had almost all the catch found on the filters. A green coloration of the filters was noticed after outlet runs #5 and #7 (conducted in the morning) and the preceding overnight runs #1 and #2. The cause of this coloration was not identified but is suspected to be related to overnight plant operations. Outlet run #2 was immediately following a cycle of cleaning and recoating of the bags, and demonstrated a higher outlet than inlet emission rate.

Another factor to consider when comparing the emission rates is that stack gas flow (DSCFM) has a direct relationship to emission rates. In all but the first run, the outlet flow exceeded the inlet by from 9,000 to 16,000 DSCFM depending on the specific run. The fan system is rated at 39,000 SCFM at a pressure drop of 10 to 22 inches water column. This corresponds favorably with the average outlet flow of 43,202 DSCFM but not with the average inlet flow of 32,064 DSCFM. It is suspected that a in-leakage existed between the two sampling locations and probably was from the evaporating chamber's emergency damper or baghouse pre-coat inlet ducts.

Table 5-1. Schedule of Field Testing

Type	Location	Monday 10/18/76	Tuesday 10/19/76	Wednesday 10/20/76	Thursday 10/21/76	Friday 10/22/76
Particulates	Inlet	#1 14:55-16:40	#2 11:05-14:11	#3 10:34-13:06 #4 13:58-16:55	#5 10:27-12:56 #6 14:30-15:36	#7 08:39-10:54 #8 13:43-16:05
	Outlet	#1 15:22-16:06	#2 11:06-14:30	#3 10:45-13:22 #4 14:08-16:42	#5 10:28-13:02 #6 14:26-15:48	#7 08:38-11:12 #8 13:46-16:22
Fluorides (in particulate trains on Runs #1 and #5	Inlet	#1 14:55-16:40			#2 10:27-12:56	
	Outlet	#1 15:22-16:06			#2 10:28-13:02	
Chlorides	Inlet		#1 12:06-12:46	#2 11:35-12:00 #3 14:05-15:30		#4 13:40-15:30 #5 17:10-18:10
	Outlet		#1 13:50-14:30	#2 10:55-11:55 #3 14:05-15:30		#4 13:45-15:30 #5 17:10-18:10
Total Methane Hydrocarbons (Test Locations alternately sampled throughout the days indicated)	Inlet		193 minutes	264 minutes	200 minutes	200 minutes
	Outlet		171 minutes	184 minutes	190 minutes	260 minutes

Table 5-1. Schedule of Field Testing (CONTINUED)

Type	Location	Monday 10/18/76	Tuesday 10/19/76	Wednesday 10/20/76	Thursday 10/21/76	Friday 10/22/76
Sulfur Oxides	Inlet	#1 15:10-16:18	#2 13:44-14:50		#3 10:32-11:42 #4 12:35-13:05 #5 14:25-14:55	#6 09:00-09:35 #7 10:55-11:25
Sulfur Oxides	Outlet	#1 15:49-16:14	#2 11:55-13:05		#3 10:40-11:14 #4 11:45-12:15 #5 14:30-15:00	#6 08:55-09:30 #7 11:00-11:30
Particle Sizing	Inlet		#1 14:58-15:23	#2 11:23-12:13	#3 12:27-13:41	#4 14:35-17:05
	Outlet			#1 12:57-16:58	#2 12:10-17:00	#3 09:45-17:00
Cyclones	Inlet				#1 11:27-11:47 #2 14:20-15:20	#3 09:55-10:47 #4 13:35-14:10
	Outlet			#1 11:00-17:00 #2 18:05----------09:04		#3 17:40-------7:2 10/23/76
Overnight	Outlet			#1 17:20----------09:04	#2 16:40----------07:30	#3 17:40-------7:1 10/23/76 #4 17:45-------7:1 10/23/76

Table 5-2. Summary of U.S. EPA Method 5 Results - Combined Baghouse Inlet

Run	Date	Time	Stack Temp (°F)	Flow[1] (SCFMD)	Filter Weight (mg)	Wash Weight (mg)	Total Weight (mg)	Emissions (lbs/hr)
1	10/18/76	14:55-16:40	86.7	43,572	45.0	16.5	61.5	5.56
2	10/19/76	11:05-14:11	112.6	35,217	164.0	22.7	186.7	10.52
3	10/20/76	10:34-13:06	115.9	38,779	170.6	25.1	195.7	11.16
4	10/20/76	13:58-16:55	132.7	33,469	267.8	31.7	299.6	16.92
5	10/21/76	10:27-12:56	103.6	24,673	170.4	16.4	186.8	10.29
6	10/21/76	14:30-15:36	96.6	26,303	90.7	56.1	146.8	10.77
7	10/22/76	08:39-10:54	115.3	26,419	240.6	75.6	316.2	17.06
8	10/22/76	13:43-16:05	92.8	28,078	177.5	30.8	208.3	11.74
Average			107.0	32,064				11.75

1. Dry Standard conditions of 68°F and 29.92 inches Hg

Table 5-3. Summary of U.S. EPA Method 5 Results - Baghouse Outlet

Run	Date	Time	Stack Temp (°F)	Flow[1] (SCFMD)	Filter Weight (mg)	Wash Weight (mg)	Total Weight (mg)	Emissions (lbs/hr)
1	10/18/76	15:22-16:06	70.0[2]	45,689[2]	35.7	105.7	141.4	20.86
2	10/19/76	11:06-14:30	89.5	45,150	124.2	2.2	126.4	12.94
3	10/20/76	10:45-13:22	98.4	47,765	46.6	1.2	47.8	4.93
4	10/20/76	14:08-16:42	108.4	45,617	4.8	2.8	7.6	0.77
5	10/21/76	10:28-13:02	86.0	41,019	111.0	NEG[3]	111.0	11.21
6	10/21/76	14:26-15:48	91.4	42,002	NEG[3]	0.9	0.9	0.19
7	10/22/76	08:38-11:12	100.8	40,015	255.5	9.0	264.5	26.85
8	10/22/76	13:46-16:22	73.2	38,360	4.3	14.1	18.4	1.82
Average			89.7	43,202				9.95

1. Dry Standard conditions of 68 F and 29.92 inches Hg
2. Estimated from traversing only one outlet stack
3. Neg - Negative weight gain

Table 5-4. Summary of U.S. EPA Method 5 Results - Baghouse Collection Efficiencies

Run	Date	Time	Emission Rates Inlet (lb/hr)	Emission Rates Outlet (lb/hr)	Baghouse Efficiency (%)
1	10/18/76	14:55-16:40	5.56	20.86	Preliminary run - invalid for comparision.
2	10/19/76	11:05-14:30	10.52	12.94	Immediately after bags re-coated. Suspect coating bleed through.
3	10/20/76	10:34-13:22	11.16	4.93	55.8
4	10/20/76	13:58-16:55	16.92	0.77	95.4
5	10/21/76	10:27-13:02	10.29	11.21	Biased run due to green coloration of outlet filter; high weight gain.
6	10/21/76	14:26-15:48	10.77	0.19	98.2
7	10/22/76	08:38-11:12	17.06	26.85	Biased run due to green coloration of outlet filter; high weight gain.
8	10/22/76	13:43-16:22	11.74	1.82	84.5
					Average 83.5

The particulate control efficiencies ranged from 55.8 percent to 98.2 percent with an average of 83.5 percent. This is somewhat lower than expected for a baghouse but does agree with previous testing on this unit by Clayton Environmental Consultants, Inc.

Table 5-5 presents a summary of the overnight particulate sampling at the outlet and shows a lower average emission rate, 2.47 lbs/hr, than found during the daytime. This may relate to reduced furnace operations during the night, when usually two furnaces were idle and the other furnace was charged.

The emission rates from the one point sampling methods used for the Andersen Mark III Cascade Impactor and the three-stage cyclone sampler are presented in Tables 5-6 and 5-7, respectively. The Andersen impactor's results show an average of 8.78 lbs/hr at the inlet and 0.26 lbs/hr at the outlet. Likewise, the three-stage cyclone sampler had results of average emission rates of 8.19 lbs/hr and 0.09 lbs/hr for the inlet and outlet, respectively.

A summary of all the particulate sampling train results is presented in Table 5-8. The Andersen impactor and three-stage cyclone sampler gave lower results at both the inlet and outlet locations, but the degree of difference was much greater at the outlet. This may mean that the outlet emission levels are somewhat lower than reported by the U.S. EPA Method 5 test runs; outlet runs #5 and #7 mentioned before, show the greatest differences but this may be explained by the absence of the green coloration on the filters from the Andersen Impactor and three-stage cyclone sampler. It is important to note the difference in the methods of sampling (in-stack filters vs out-of-stack hot box, one point vs multiple points), sample recovery (no wash vs probe wash, dry brushing of stages vs brushing and acetone rinse) and analyses (on-site weighings vs off-site weighings) that exist between the Andersen Impactor and three stage cyclone sampler, and U.S. EPA Method 5. Therefore, direct numerical comparisons of the results may be erroneous. However, "ballpark" estimates and comparisons can be made to determine the possibility of gross errors or sample bias. For example, on October 21, 1976, the inlet runs show the same "ballpark" of emission rates even though two of the values (Impactor #3 and Cyclone #2) differ from the other three values by a multiple of 3. On the same day, the outlet runs differ significantly due to the green coloration on U.S. EPA Method 5 filters on runs #5 and overnight #2.

Total Fluorides

The results of the fluoride determination on the samples obtained from the U.S. EPA Method 5 runs #1 and #5 are presented in Table 5-9. The average emissions were found to be 1.23 lbs/hr at the inlet and 0.23 lbs/hr at the outlet with an average collection efficiency of 75.4 percent. Both of these test runs had higher outlet total particulate emission rates than found at the inlet, but showed a large removal of fluorides by passing through the coated baghouse system. These emission results are slightly

Table 5-5. Summary of Overnight U.S. EPA Method 5 Results

Run	Date	Time	Stack Temperature (°F)	Flow** (SCFM)	Emissions (lbs/hr)
Outlet 1	10/20/76	17:20-09:04	100	43,318	3.35
2	10/21/76	16:40-07:30	75	38,360	6.01
3	10/22/76	17:40-07:17	79	41,009	0.46
4*	10/22/76	17:45-07:10	100	38,360	0.04
Average					2.47

*No filter used

**Based upon an average of flows from the first full traverse particulate test runs before and after the overnight test.

Table 5-6. Summary of Andersen Impactor Results.

Run	Date	Time	Stack Temperature (°F)	D50 (μ)	Flow* (DSCFM)	Emissions (lbs/hr)
Inlet 1	10/19/76	14:58-15:23	117.2	0.3	35,217	13.43
2	10/20/76	11:23-12:13	117.7	0.4	38,779	15.03
3	10/21/76	12:27-13:41	96.7	3.6	24,673	3.65
4	10/22/76	14:35-17:05	95.4	0.4	28,078	3.02
					Average	8.78
Outlet 1	10/20/76	12:57-16:58	100.1	0.2	47,765	0.10
2	10/21/76	12:10-17:00	80.0	2.5	42,002	0.56
3	10/22/76	09:45-17:00	80.7	0.3	39,187	0.13
					Average	0.26

*Flows taken from particulate sampling.

Table 5-7. Summary of Three-Stage Cyclone Results

Run		Date	Time	Cyclone D50's 1st (μ)	2nd (μ)	3rd (μ)	Flow* (DSCFM)	Emissions (lbs/hr)
Inlet	1	10/21/76	11:27-11:47	2.05	0.75	0.40	38,779	11.45
	2	10/21/76	14:20-15:20	2.01	0.73	0.39	33,469	2.82
	3	10/22/76	09:55-10:47	2.43	0.89	0.47	24,673	10.64
	4	10/22/76	13:35-14:10	2.26	0.83	0.44	26,303	7.84
Average								8.19
Outlet	1	10/20/76	11:00-17:00	2.02	0.74	0.39	45,150	0.11
	2	10/20/76	18:05-09:04	2.03	0.74	0.39	46,458	0.08
	3	10/22-23/76	17:40-07:20	2.13	0.78	0.41	38,360	0.08
Average								0.09

* Flow taken from particulate sampling.

Table 5-8. Summary of Particulate Emission Results - All Sampling Trains

Date	Time	Train and Run			Inlet Emissions (lbs/hr)	Outlet Emissions (lbs/hr)
		Method 5	Impactor	Cyclone		
10/18/76	14:55-16:40	Inlet #1			5.56	
	15:22-16:06	Outlet #1				20.86
10/19/76	11:05-14:11	Inlet #2			10.52	
	11:06-14:30	Outlet #2				12.94
	14:58-15:23		Inlet #1		13.43	
10/20/76	10:34-13:06	Inlet #3			11.16	
	10:45-13:22	Outlet #3				4.93
	11:00-17:00			Outlet #1		0.11
	11:23-12:13		Inlet #2		15.03	
	12:57-16:58		Outlet #1			0.10
	13:58-16:55	Inlet #4			16.92	
	14:08-16:42	Outlet #4				0.77
	17:20-09:04	Overnight #1				3.35
	18:05-09:04			Outlet #2		0.08
10/21/76	10:27-12:56	Inlet #5			10.29	
	10:28-13:02	Outlet #5				11.21
	11:27-11:47			Inlet #1	11.45	
	12:10-17:00		Outlet #2			0.56
	12:27-13:41		Inlet #3		3.65	
	14:20-15:20			Inlet #2	2.82	
	14:26-15:48	Outlet #6				0.19
	14:30-15:36	Inlet #6			10.77	
	16:40-07:30	Overnight #2				6.01

Table 5-8. Summary of Particulate Emission Results - All Sampling Trains (CONTINUED)

Date	Time	Train and Run			Inlet Emissions (lbs/hr)	Outlet Emissions (lbs/hr)
		Method 5	Impactor	Cyclone		
10/22/76	08:38-11:12	Outlet #7				26.85
	08:39-10:54	Inlet #7			17.06	
	09:45-17:00		Outlet #3			0.13
	09:55-10:47			Inlet #3	10.64	
	13:35-14:10			Inlet #4	7.84	
	13:43-16:05	Inlet #8			11.74	
	13:46-16:22	Outlet #8				1.82
	14:35-17:05		Inlet #4		3.02	
	17:40-07:20			Outlet #3		0.08
	17:40-07:17	Overnight #3				0.46
	17:45-07:10	Overnight #4				0.04

Table 5-9. Summary of Total Fluoride Results

Location	Date	Time	FLUORIDE Concentration ($lbs/ft^3 \times 10^{-7}$)	FLUORIDE Emissions (lbs/hr)*	FLUORIDE Efficiency (%)
Inlet #1	10/18/76	14:55-16:40	6.23	1.63	92.3
Outlet #1	10/18/76	15:22-16:06	0.45	0.125	
Inlet #2	10/21/76	10:27-12:56	3.35	0.825	58.4
Outlet #2	10/21/76	10:28-13:02	1.39	0.343	
Average**					
	Inlet		4.79	1.23	75.4
	Outlet		0.92	0.234	

* Calculated based on the stack flow data from a concurrent full traverse particulate test run.
** Averages based on all test runs except that efficiencies are based on only those runs performed simultaneously.

higher than that reported by Clayton Enivornmental Consultants, Inc., but the average efficiency is similar. However, it should be noted that the previous test and analysis methodologies are unknown and the results may not be comparable.

Particle Sizing

The results of the Andersen Impactor test runs are summarized in Table 5-10. The D50 is the calculated particle diameter in microns at which 50 percent of particles will be larger than and 50 percent will be smaller than this size. The "percent less than" is the cumulative percentage of the total weight caught during each test run, for a particular stage and all the stages located downstream of it in the impactor, and includes the back-up filter.

The average D50 for each test run and for all test runs at the inlet and outlet are presented. The average D50 for all the inlet test runs was determined to be 0.4 microns. Interestingly, the average D50 for all the outlet tests was also 0.4 microns. These run averages were obtained by plotting the results on log-probability paper and finding the size for each test run at the 50 "percent less than" value as shown in Figures 5-1, 5-2, and 5-3. Based on a weight relationship, these D50 run averages and D50 inlet and outlet averages represent that particle size at 50 percent of the cumulative weight.

Inlet run #3 and outlet run #2 were performed during the same time period on October 21, 1976, and show much higher average D50's (3.6 and 2.5 microns respectively) than the other runs.

The major aspect of the particle sizing results is that the average particle found at the inlet is the same size as the average particle found after the coated baghouse. In view of this and the average particulate collection efficiency of 83.5 percent (Table 5-4) of the baghouse, the system appears to be operating in the expected range for a normal baghouse with the same D50 particle size. The higher efficiencies (99% +) usually associated with baghouses is for collecting particles larger than 5 microns

Three-Stage Cyclone Sampler

Elemental analysis of the particulate samples collected in the three cyclones was performed on outlet runs #1 and #3 (run #2 was retained for possible crystaline analysis). Table 5-11 presents a summary of 11 separate analyses performed with an atomic absorption unit on the three stages of both test runs. These 11 were chosen for further investigation following review of a qualitative analysis of a sample of the baghouse hopper dust by spark source spectroscopy. The metals of interest and their average percentage in the outlet dust samples is given on the bottom of the table. The iron results are apparently in error due to rust contamination from the sampler. The contamination was apparently less severe on run #3 which concurs with visual observations during sample recovery.

Table 5-10. Summary for Andersen Impactor Sizing Results

Stages	Run 1		Run 2		Run 3		Run 4		Average	
	D50 (μ)	Percent Less Than (%)	D50 (μ)	Percent Less Than (%)	D50 (μ)	Percent Less Than (%)	D50 (μ)	Percent Less Than (%)	D50 (μ)	Percent Less Than (%)
INLET										
1	8.64	89.3	7.97	94.9	8.33	55.8	8.75	85.8	8.42	81.4
2	7.28	89.1	6.72	93.3	7.02	54.3	7.37	85.4	7.10	80.5
3	5.02	88.2	4.63	91.5	4.84	52.4	5.09	84.0	4.89	79.0
4	3.71	86.7	3.41	88.8	3.57	49.5	3.75	82.0	3.61	76.7
5	1.76	85.8	1.62	85.4	1.69	46.4	1.78	79.6	1.71	74.3
6	1.12	77.3	1.03	78.8	1.08	41.8	1.14	73.9	1.09	67.9
7	0.59	67.6	0.54	63.0	0.57	36.0	0.60	60.0	0.57	56.6
8	0.29	51.9	0.26	42.6	0.28	29.4	0.29	44.9	0.28	42.2
D50 Average	>~~0.3~~ .29		0.4		3.6		0.4		0.4	
OUTLET										
1	7.79	88.2	7.97	84.4	8.04	97.7			7.93	90.1
2	6.56	79.3	6.72	74.2	6.78	95.1			6.67	82.9
3	4.53	79.3	4.63	62.4	4.68	87.8			4.61	76.5
4	3.34	79.3	3.42	54.4	3.45	79.5			3.40	71.1
5	1.58	77.5	1.62	45.7	1.63	78.4			1.61	67.2
6	1.01	65.7	1.03	41.2	1.04	76.2			1.03	61.0
7	0.53	58.0	0.55	44.2	0.55	65.0			0.54	54.7
8	0.26	53.3	0.26	40.7	0.27	49.0			0.26	47.7
D50 Average	>0.2		2.5		0.3				0.4	

FIGURE 5-1. INLET PARTICLE SIZE DISTRIBUTIONS AT ROCHESTER SMELTING AND REFINING

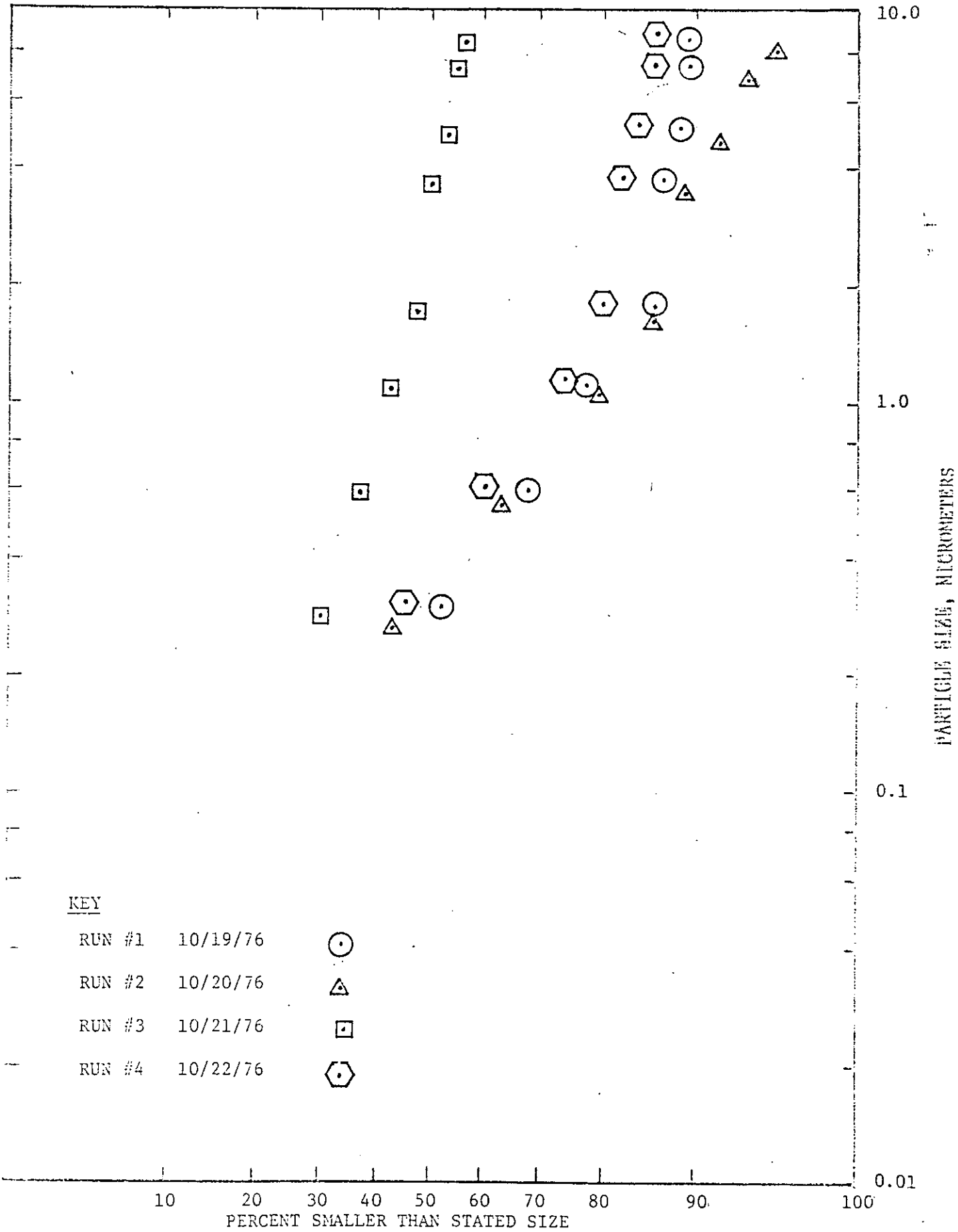


FIGURE 5-2. OUTLET PARTICLE SIZE DISTRIBUTIONS AT ROCHESTER SMELTING AND REFINING

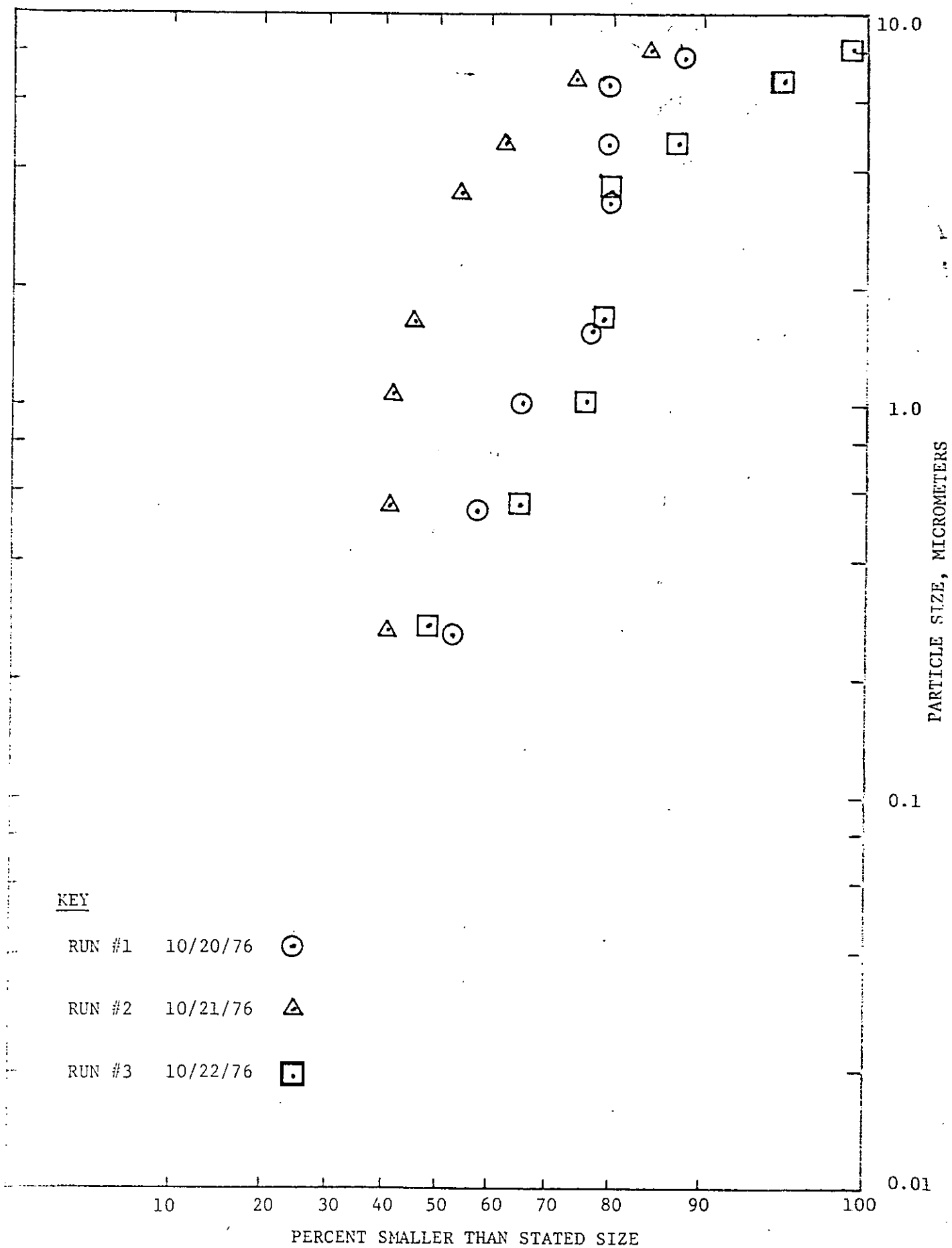


FIGURE 5-3. AVERAGE INLET AND AVERAGE OUTLET SIZE DISTRIBUTIONS AT ROCHESTER SMELTING AND REFINING

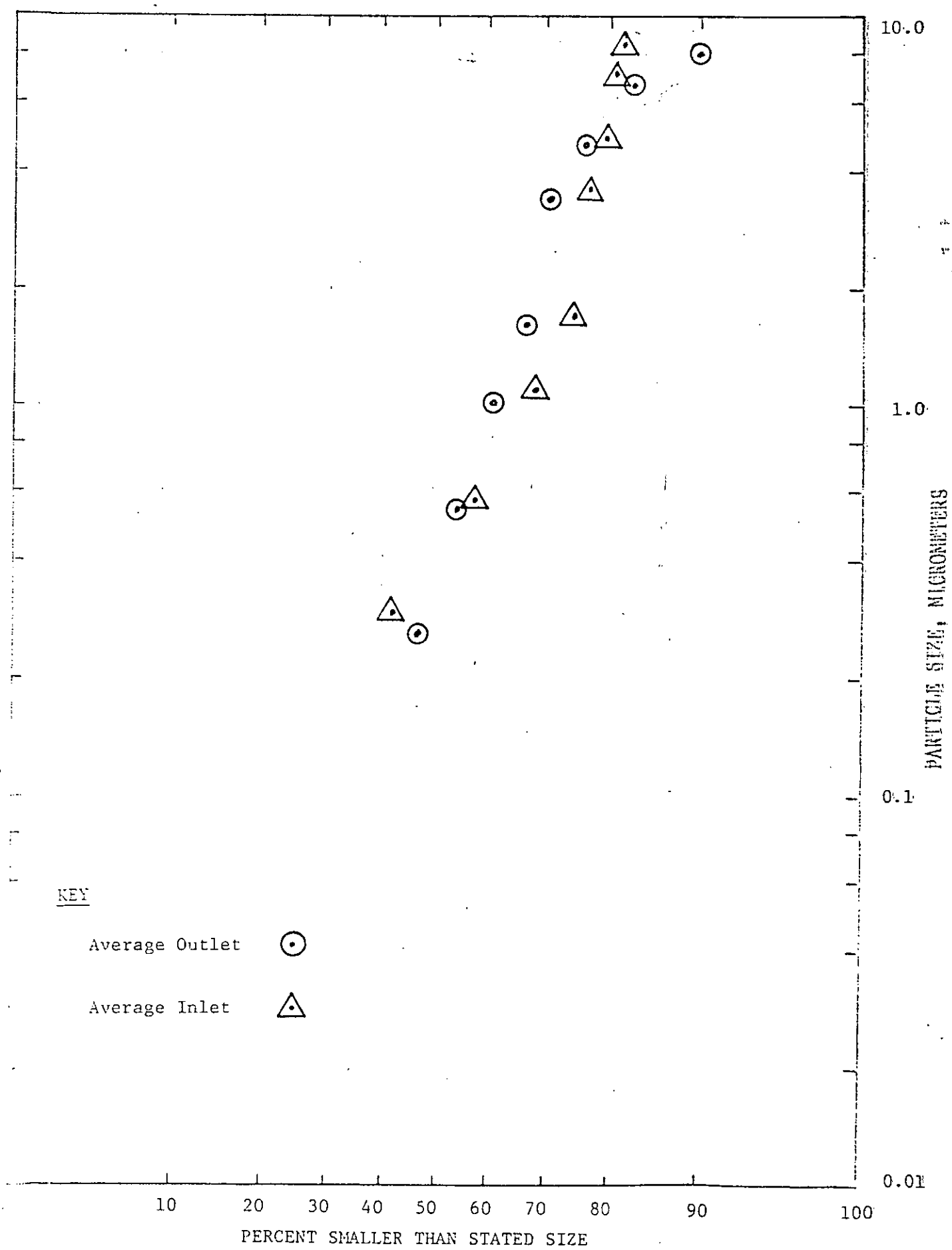


Table 5-11. Summary of Elemental Analysis of Particulate Catch From 3 Stage Cyclone Sampler

Run	Date	Time	Cyclone Run	Percentage of Total Weight										
				Na	K	Al	Sr	Fe*	Cu	Pb	Zn	Mg	Cd	Ca
Outlet #1	10/20/76	11:00-17:00	1	1.2	0.7	2.0	0	21.5	0.1	0.2	0.5	0.3	0.4	0.0
			2	0.4	0.2	0.8	0	34.5	0.1	0.1	0.2	0.1	0.1	0.2
			3	1.6	0.8	3.1	0	34.2	1.6	0.2	1.6	0.2	0.3	0.6
			Average	1.1	0.6	2.0	0	30.1	0.6	0.2	0.8	0.2	0.3	0.3
Outlet #3	10/22 + 10/23	17:40-07:20	1	0.7	1.0	1.7	0	19.0	0.3	0.1	0.5	0.2	0.1	0.3
			2	4.2	4.2	6.5	0	6.5	0.7	0.7	1.0	1.3	1.3	0.7
			3	3.4	5.1	6.4	0	4.2	1.7	0.4	2.1	1.1	0.4	0.0
			Average	2.8	3.4	4.9	0	9.9	0.9	0.4	1.2	0.9	0.6	0.3
TOTAL			AVERAGE	2.0	2.0	3.5	0	20.0	7.5	0.3	1.0	0.6	0.5	0.3

* High levels of Fe probably due to special connections between cyclones; appeared rusty but were not accessible for cleaning.

Sulfur Dioxide and Sulfur Trioxide

The results for sulfur dioxide and sulfur trioxide sampling are presented in Table 5-12. The average sulfur dioxide emission rates were 0.07 lbs/hr and 0.05 lbs/hr for the inlet and outlet, respectively. The average efficiency of the coated baghouse was found to be 54 percent for sulfur dioxide removal.

The sulfur trioxide emissions were determined to have an average inlet rate of 1.81 lbs/hr and an average outlet rate of 0.6 lbs/hr. The system had an average collection efficiency of 86 percent.

The results show some higher emission rates at the outlet than at the inlet. This may be due to a chromographic effect of the bag coating where inlet concentrations may be delayed or retained by the coating before being released sometime later. Also, the U.S. EPA Method 6 is reported to have a detectable limit of 2.1×10^{-7} lbs/DSCF for sulfur dioxide, and an equivalent detectable limit of 2.63×10^{-7} lbs/DSCF for sulfur trioxide. Since Table 5-12 shows that all the sulfur dioxide inlet and outlet results and most of the sulfur trioxide outlet results are considerably below these limits, the results may have a degree of lower limit "bounce". However, sample volumes were approximately four times the usual requirements of the method (~ 3.0 DSCF to 0.71 DSCF) which might have a lowering effect on the detection limits.

The important points of the results are that sulfur dioxide is emitted at both the inlet and outlet in very low concentrations and that sulfur trioxide concentrations are reduced effectively by the coated baghouse.

Hydrogen Chloride and Chlorine

The aspect of the possible reduction of chlorine gas and chlorides was the primary goal of this testing program. Table 5-13 presents the results of chlorine and hydrogen chloride testing and reports on the collection efficiencies. The average chlorine emission rates were 7.66 lbs/hr at the inlet and 1.29 lbs/hr at the outlet; the average chlorine collection efficiency was 84.4 percent. The average hydrogen chloride emission rates were 5.06 lbs/hr and 0.28 lbs/hr at the inlet and outlet, respectively; the average collection efficiency was 97.4 percent.

These results demonstrate that the coated baghouse was effectively reducing the chlorine/hydrogen chloride emissions.

In order to determine any residual effect of the chlorine demagging process, run #5 was conducted approximately two hours after chlorination was stopped and the "bell" removed from the charging well. While the emission rates of both gases decreased during this run, as compared to run #4, the outlet levels of chlorine increased and hydrogen chloride was not detectable. The increase in chlorine emissions may be related to a chromatographic effect of the bag coating or its saturation level. The significant aspects of the results are that chlorine and hydrogen emissions continued from both the charging well and the baghouse for at least several

Table 5-12. Summary of Sulfur Dioxide and Sulfur Trioxide Results

Run	Date	Time	Qs (SCFMD)	SULFUR DIOXIDE $\times 10^{-7}$ lbs/DSCF	SULFUR DIOXIDE lbs*/hr	SULFUR DIOXIDE Efficiency (%)	SULFUR TRIOXIDE $\times 10^{-7}$ lbs/DSCF	SULFUR TRIOXIDE lbs*/hr	SULFUR TRIOXIDE Efficiency (%)
Inlet #1	10/18/76	15:10-16:18	43,572	0.20	0.05	Neg	5.03	1.32	100
Outlet #1		15:49-16:14	45,707	0.45	0.12		0.00	0.00	
Inlet #2	10/19/76	13:44-14:50	35,217	0.14	0.03	---	0.09	0.19	---
Outlet #2		11:55-13:05	45,150	0.55	0.15		5.72	1.55	
Inlet #3	10/21/76	10:32-11:42	24,673	0.35	0.08	100	8.98	2.09	100
Outlet #3		10:40-11:14	41,019	0.00	0.00		0.00	0.00	
Inlet #4	10/21/76	12:35-13:05	24,673	0.39	0.06	---	1.38	0.20	---
Outlet #4		11:45-12:15	41,019	0.15	0.04		0.00	0.00	
Inlet #5	10/21/76	14:25-14:55	26,303	0.14	0.22	100	10.12	1.60	99
Outlet #5		14:30-15:00	42,002	0.00	0.00		0.07	0.02	
Inlet #6	10/22/76	09:00-09:35	26,419	0.05	0.01	0	20.27	3.21	72
Outlet #6		08:55-09:30	40,015	0.06	0.01		4.14	0.99	
Inlet #7	10/22/76	10:55-11:25	26,419	0.44	0.07	71	25.40	4.03	59
Outlet #7		11:00-11:30	40,015	0.06	0.02		6.83	1.64	
	Average:		Inlet		0.07	54		1.81	86
			Outlet		0.05			0.6	

NOTE: SO_2 detectable limit of 2.1×10^{-7} lbs/DSCF; SO_3 detectable limit of 2.63×10^{-7} lbs/DSCF.

* Calculated based on the stack flow data from a concurrent full traverse particulate test run.

** Averages based on all test runs except that efficiencies are based on only those runs performed simultaneously.

Table 5-13. Summary of Chlorine and Hydrogen Chloride Results

Run	Date	Time	CHLORINE			HYDROGEN CHLORIDE		
			Concentration (lbs/ft^3 $x10^{-7}$)	Emissions* (lbs/hr)	Efficiency (%)	Concentration (lbs/ft^3 $x10^{-7}$)	Emissions* (lbs/hr)	Efficiency (%)
Inlet #1	10/19/76	12:06-12:46	100.00	22.62	---	5.77	1.22	---
Outlet #1		13:50-14:30	2.37	0.64		0.66	0.18	
Inlet #2	10/20/76	11:35-12:00	25.10	5.85	86.5	21.76	5.06	99.4
Outlet #2		10:55-11:55	2.77	0.79		0.09	0.03	
Inlet #3	10/20/76	14:05-15:30	46.90	9.42	87.4	20.63	4.14	100
Outlet #3		14:05-15:30	4.35	1.19		0.00	0.00	
Inlet #4	10/22/76	13:40-15:30	86.10	13.64	89.5	77.80	12.33	90.2
Outlet #4		13:45-15:30	5.94	1.43		5.02	1.21	
Inlet #5	10/22/76	17:10-18:10	55.60	9.37	74.3	15.04	2.53	100
Outlet #5		17:10-18:10	10.49	2.41		0.00	0.00	
Average**								
Inlet			62.74	7.66	84.4	28.20	5.06	97.4
Outlet			5.18	1.29		2.16	0.28	

* Calculated based on the stack flow data from a concurrent full traverse particulate test run

** Averages based on all test runs except that efficiencies are based on only those runs performed simultaneously.

hours after chlorine demagging was completed and that the coated baghouse successfully reduced both of these emissions by 84 percent and 97 percent, respectively.

Total Methane Hydrocarbons

As summaried in Table 5-14, the emission levels of total methane hydrocarbons did not differ significantly by use of the coated baghouse. Simultaneous sampling was not conducted while making this determination, however, each location was adequately sampled for greater than 800 minutes during the course of the four days in order to characterize the emission rates. The inlet and outlet gases were sampled alternately for individual periods of up to 45 minutes throughout the testing period. This technique was designed to adequately describe the total methane hydrocarbon concentrations at both sampling locations.

The time-weighed average concentrations of total methane hydrocarbons were determined to be 46 ppm at the baghouse inlet and 40 ppm at its outlet. Due to the lack of a significant difference in concentrations, a control efficiency is not given.

Polynuclear Aromatics

The filters from the three-stage cyclone outlet run #2 and U.S. EPA Method 5 outlet runs #4 and #7 were analyzed on a gas chromatograph for polynuclear aromatic hydrocarbons (PNA). Based on comparison with a sample of α-Naphthyl Amine, a known PNA, none were detectable. Therefore, the scheduled gas chromatograph-mass spectroscopy analysis was cancelled.

Table 5-14. Summary of Total Methane Hydrocarbon Determinations

Location	Date	Total Sampling Time (min)		Emissions: Time Weighed Average (ppm)	Emissions: Non-Peak Maximum (ppm)	Emissions: Non-Peak Minimum (ppm)
Inlet	10/19/76	193		44	115	12
	10/20/76	264		38	111	20
	10/21/76	200		71	117	32
	10/22/76	200		35	78	119
	TOTAL	857	Average	46		
Outlet	10/19/76	171		34	110	12
	10/20/76	184		44	124	15
	10/21/76	190		51	115	16
	10/22/76	260		32	107	15
	TOTAL	811	Average	40		

APPENDIX A

TEST METHODS

Aug 11, 1977

ENVIRONMENTAL PROTECTION AGENCY REGULATIONS ON STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

(40 CFR 60: 36 FR 24876, December 23, 1971; Effective August 17, 1971; Amended as shown in Code of Federal Regulations, Volume 40, revised as of July 1, 1975; 40 FR 33152, August 6, 1975; 40 FR 42194, September 11, 1975; 40 FR 43850, September 23, 1975; 40 FR 45170, October 1, 1975; 40 FR 46250, October 6, 1975; 40 FR 48347, October 15, 1975; 40 FR 50718, October 31, 1975, 40 FR 53340 November 17, 1975; 40 FR 58415, December 16, 1975; 40 FR 59729, December 30, 1975; 41 FR 1913, January 13, 1976; 41 FR 2232, January 15, 1976; 41 FR 3826, January 26, 1976; 41 FR 4263, January 29, 1976; 41 FR 7749, February 20, 1976; 41 FR 8346, February 26, 1976; 41 FR 11820, March 22, 1976; 41 FR 17549, April 27, 1976; 41 FR 18498, May 4, 1976)

PART 60—STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

AUTHORITY: Secs. 111 and 114 of the Clean Air Act, as amended by sec. 4(a) of Pub. L. 91-604, 84 Stat. 1678 (42 U.S.C. 1857c-6, 1857c-9). Subpart B also issued under sec. 301(a) of the Clean Air Act, as amended by sec. 15(c)(2) of Pub. L. 91-604, 84 Stat. 1713 (42 U.S.C. 1857g).

[40 FR 53340, November 17, 1975]

Subpart A—General Provisions

§ 60.1 Applicability.

Except as provided in Subparts B and C, the provisions of this part apply to the owner or operator of any stationary source which contains an affected facility, the construction or modification of which is commenced after the date of publication in this part of any standard (or, if earlier, the date of publication of any proposed standard) applicable to that facility.

[39 FR 20790, June 14, 1974]

[40 FR 46250, October 6, 1975]

§ 60.2 Definitions.

As used in this part, all terms not defined herein shall have the meaning given them in the Act:

(a) "Act" means the Clean Air Act (42 U.S.C. 1857 et seq., as amended by Public Law 91-604, 84 Stat. 1676).

(b) "Administrator" means the Administrator of the Environmental Protection Agency or his authorized representative.

(c) "Standard" means a standard of performance proposed or promulgated under this part.

(d) "Stationary source" means any building, structure, facility, or installation which emits or may emit any air pollutant and which contains any one or combination of the following:

(1) Affected facilities.

(2) Existing facilities.

(3) Facilities of the type for which no standards have been promulgated in this part.

[40 FR 58415, December 16, 1975]

(e) "Affected facility" means, with reference to a stationary source, any apparatus to which a standard is applicable.

(f) "Owner or operator" means any person who owns, leases, operates, controls, or supervises an affected facility or a stationary source of which an affected facility is a part.

(g) "Construction" means fabrication, erection, or installation of an affected facility.

(h) "Modification" means any physical change in, or change in the method of operation of, an existing facility which increases the amount of any air pollutant (to which a standard applies) emitted into the atmosphere by that facility or which results in the emission of any air pollutant (to which a standard applies) into the atmosphere not previously emitted.

[40 FR 58415, December 16, 1975]

(i) "Commenced" means, with respect to the definition of "new source" in section 111(a)(2) of the Act, that an owner or operator has undertaken a continuous program of construction or modification or that an owner or operator has entered into a contractual obligation to undertake and complete, within a reasonable time, a continuous program of construction or modification.

(j) "Opacity" means the degree to which emissions reduce the transmission of light and obscure the view of an object in the background.

(k) "Nitrogen oxides" means all oxides of nitrogen except nitrous oxide, as measured by test methods set forth in this part.

(l) "Standard conditions" means a temperature of 20°C (68°F) and a pressure of 760 mm of Hg (29.92 in. of Hg).

(m) "Proportional sampling" means sampling at a rate that produces a constant ratio of sampling rate to stack gas flow rate.

(n) "Isokinetic sampling" means sampling in which the linear velocity of the gas entering the sampling nozzle is equal to that of the undisturbed gas stream at the sample point.

(o) "Startup" means the setting in operation of an affected facility for any purpose.

(p) "Shutdown" means the cessation of operation of an affected facility for any purpose.

(q) "Malfunction" means any sudden and unavoidable failure of air pollution control equipment or process equipment or of a process to operate in a normal or usual manner. Failures that are caused entirely or in part by poor maintenance, careless operation, or any other preventable upset condition or preventable

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(1) Method 5 for concentration of particulate matter and associated moisture content;

(2) Method 1 for sample and velocity traverses;

(3) Method 2 for velocity and volumetric flow rate; and

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling time for each run shall be at least four hours. When a single EAF is sampled, the sampling time for each run shall also include an integral number of heats. Shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator. The minimum sample volume shall be 4.5 dscm (160 dscf).

(c) For the purpose of this subpart, the owner or operator shall conduct the demonstration of compliance with 60.272(a)(3) and furnish the Administrator a written report of the results of the test.

(d) During any performance test required under § 60.8 of this part, no gaseous diluents may be added to the effluent gas stream after the fabric in any pressurized fabric filter collector, unless the amount of dilution is separately determined and considered in the determination of emissions.

(e) When more than one control device serves the EAF(s) being tested, the concentration of particulate matter shall be determined using the following equation:

$$C_s = \frac{\sum_{n=1}^{N} (C_s Q_s)_n}{\sum_{n=1}^{N} (Q_s)_n}$$

where:

C_s = concentration of particulate matter in mg/dscm (gr/dscf) as determined by method 5.

N = total number of control devices tested.

Q_s = volumetric flow rate of the effluent gas stream in dscm/hr (dscf/hr) as determined by method 2.

$(C_sQ_s)_n$ or $(Q_s)_n$ = value of the applicable parameter for each control device tested.

(f) Any control device subject to the provisions of this subpart shall be designed and constructed to allow measurement of emissions using applicable test methods and procedures.

(g) Where emissions from any EAF(s) are combined with emissions from facilities not subject to the provisions of this subpart but controlled by a common capture system and control device, the owner or operator may use any of the following procedures during a performance test:

(1) Base compliance on control of the combined emissions.

(2) Utilize a method acceptable to the Administrator which compensates for the emissions from the facilities not subject to the provisions of this subpart.

(3) Any combination of the criteria of paragraphs (g)(1) and (g)(2) of this section.

(h) Where emissions from any EAF(s) are combined with emissions from facilities not subject to the provisions of this subpart, the owner or operator may use any of the following procedures for demonstrating compliance with § 60.272(a)(3):

(1) Base compliance on control of the combined emissions.

(2) Shut down operation of facilities not subject to the provisions of this subpart.

(3) Any combination of the criteria of paragraphs (h)(1) and (h)(2) of this section.

* * * * *

(Secs. 111 and 114 of the Clean Air Act, as amended by sec. 4(a) of Pub. L. 91-604, 84 Stat. 1678 (42 U.S.C. 1857c-6, 1857c-9))

[40 FR 43850, September 23, 1975]

APPENDIX A – REFERENCE METHODS

[39 FR 20790, June 14, 1974]

METHOD 1—SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

1. *Principle and Applicability.*

1.1 Principle. A sampling site and the number of traverse points are selected to aid in the extraction of a representative sample.

1.2 Applicability. This method should be applied only when specified by the test procedures for determining compliance with the New Source Performance Standards. Unless otherwise specified, this method is not intended to apply to gas streams other than those emitted directly to the atmosphere without further processing.

2. *Procedure.*

2.1 Selection of a sampling site and minimum number of traverse points.

2.1.1 Select a sampling site that is at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, contraction, or visible flame. For rectangular cross section, determine an equivalent diameter from the following equation:

$$\text{Equivalent diameter} = 2\left(\frac{(\text{length})(\text{width})}{\text{length}+\text{width}}\right)$$

equation 1-1

2.1.2 When the above sampling site criteria can be met, the minimum number of traverse points is twelve (12).

2.1.3 Some sampling situations render the above sampling site criteria impractical. When this is the case, choose a convenient sampling location and use Figure 1-1 to determine the minimum number of traverse points. Under no conditions should a sampling point be selected within 1 inch of the stack wall. To obtain the number of traverse points for stacks or ducts with a diameter less than 2 feet, multiply the number of points obtained from Figure 1-1 by 0.67.

2.1.4 To use Figure 1-1 first measure the distance from the chosen sampling location to the nearest upstream and downstream disturbances. Determine the corresponding number of traverse points for each distance from Figure 1-1. Select the higher of the two numbers of traverse points, or a greater value, such that for circular stacks the number is a multiple of 4, and for rectangular stacks the number follows the criteria of section 2.2.2.

2.2 Cross-sectional layout and location of traverse points.

2.2.1 For circular stacks locate the traverse points on at least two diameters according to Figure 1-2 and Table 1-1. The traverse axes shall divide the stack cross section into equal parts.

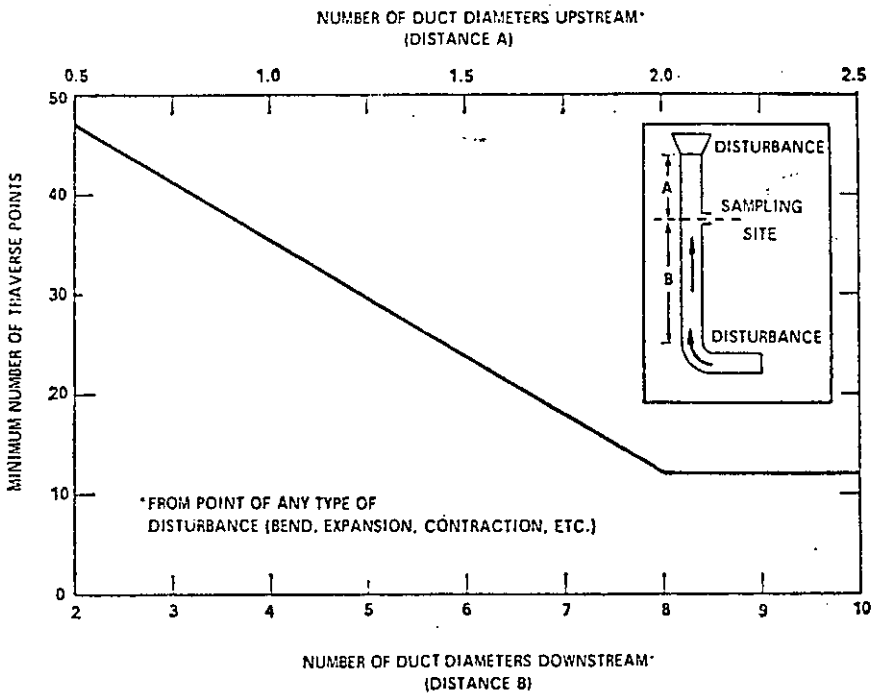

Figure 1-1. Minimum number of traverse points.

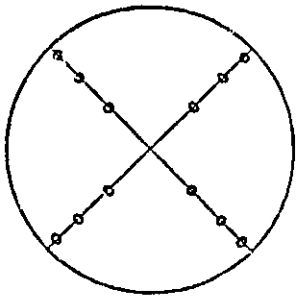

Figure 1-2. Cross section of circular stack divided into 12 equal areas, showing location of traverse points at centroid of each area.

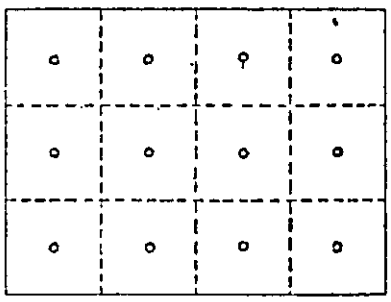

Figure 1-3. Cross section of rectangular stack divided into 12 equal areas, with traverse points at centroid of each area.

Table 1-1. Location of traverse points in circular stacks (Percent of stack diameter from inside wall to traverse point)

Traverse point number on a diameter	Number of traverse points on a diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.3	2.5	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.7	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.5	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.5	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.3	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5
6			95.6	80.6	65.8	35.5	28.9	22.0	18.8	16.5	14.6	13.2
7				89.5	77.4	64.5	38.6	28.3	23.6	20.4	18.0	15.8
8				96.7	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4
9					91.8	82.3	73.1	62.5	39.2	30.6	26.1	23.0
10					97.5	89.2	79.9	71.7	61.8	38.8	31.5	28.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2
14							98.2	91.5	85.4	79.6	73.9	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.5	92.1
22											98.9	94.5
23												96.8
24												98.9

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2.2.2 For rectangular stacks divide the cross section into as many equal rectangular areas as traverse points, such that the ratio of the length to the width of the elemental areas is between one and two. Locate the traverse points at the centroid of each equal area according to Figure 1-3.

3. *References.*

Determining Dust Concentration in a Gas Stream. ASME Performance Test Code #27, New York, N.Y., 1957.

Devorkin, Howard, et al., Air Pollution Source Testing Manual. Air Pollution Control District, Los Angeles, Calif. November 1963.

Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases, Western Precipitation Division of Joy Manufacturing Co., Los Angeles, Calif. Bulletin WP-50, 1968.

Standard Method for Sampling Stacks for Particulate Matter. In: 1971 Book of ASTM Standards, Part 23, Philadelphia, Pa. 1971, ASTM Designation D-2928-71.

METHOD 2—DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE (TYPE S PITOT TUBE)

1. *Principle and applicability.*

1.1 Principle. Stack gas velocity is determined from the gas density and from measurement of the velocity head using a Type S (Stauscheibe or reverse type) pitot tube.

1.2 Applicability. This method should be applied only when specified by the test procedures for determining compliance with the New Source Performance Standards.

2. *Apparatus.*

2.1 Pitot tube—Type S (Figure 2-1), or equivalent, with a coefficient within ±5% over the working range.

2.2 Differential pressure gauge—Inclined manometer, or equivalent, to measure velocity head to within 10% of the minimum value.

2.3 Temperature gauge—Thermocouple or equivalent attached to the pitot tube to measure stack temperature to within 1.5% of the minimum absolute stack temperature.

2.4 Pressure gauge—Mercury-filled U-tube manometer, or equivalent, to measure stack pressure to within 0.1 in. Hg.

2.5 Barometer—To measure atmospheric pressure to within 0.1 in. Hg.

2.6 Gas analyzer—To analyze gas composition for determining molecular weight.

2.7 Pitot tube—Standard type, to calibrate Type S pitot tube.

3. *Procedure.*

3.1 Set up the apparatus as shown in Figure 2-1. Make sure all connections are tight and leak free. Measure the velocity head and temperature at the traverse points specified by Method 1.

3.2 Measure the static pressure in the stack.

3.3 Determine the stack gas molecular weight by gas analysis and appropriate calculations as indicated in Method 3.

4. *Calibration.*

4.1 To calibrate the pitot tube, measure the velocity head at some point in a flowing gas stream with both a Type S pitot tube and a standard type pitot tube with known coefficient. Calibration should be done in the laboratory and the velocity of the flowing gas stream should be varied over the normal working range. It is recommended that the calibration be repeated after use at each field site.

4.2 Calculate the pitot tube coefficient using equation 2-1.

$$C_{p_{test}} = C_{p_{std}} \sqrt{\frac{\Delta p_{std}}{\Delta p_{test}}} \quad \text{equation 2-1}$$

where:

$C_{p_{test}}$ = Pitot tube coefficient of Type S pitot tube.

$C_{p_{std}}$ = Pitot tube coefficient of standard type pitot tube (if unknown, use 0.99).

Δp_{std} = Velocity head measured by standard type pitot tube.

Δp_{test} = Velocity head measured by Type S pitot tube.

4.3 Compare the coefficients of the Type S pitot tube determined first with one leg and then the other pointed downstream. Use the pitot tube only if the two coefficients differ by no more than 0.01.

5. *Calculations.*

Use equation 2-2 to calculate the stack gas velocity.

$$(V_s)_{ave.} = K_p C_p (\sqrt{\Delta p})_{ave.} \sqrt{\frac{(T_s)_{ave.}}{P_s M_s}}$$

Equation 2-2

where:

$(V_s)_{ave.}$ = Stack gas velocity, feet per second (f.p.s.);

$K_p = 85.48 \frac{\text{ft.}}{\text{sec.}} \left(\frac{\text{lb.}}{\text{lb. mole-°R}}\right)^{1/2}$ when these units are used.

C_p = Pitot tube coefficient, dimensionless.

$(T_s)_{ave.}$ = Average absolute stack gas temperature, °R.

$(\sqrt{\Delta p})_{ave.}$ = Average velocity head of stack gas, inches H_2O (see Fig. 2-2).

P_s = Absolute stack gas pressure, inches Hg.

M_s = Molecular weight of stack gas (wet basis), lb./lb.-mole.

$M_s(1-B_{wo}) + 18B_{wo}$

M_d = Dry molecular weight of stack gas (from Method 3).

B_{wo} = Proportion by volume of water vapor in the gas stream (from Method 4).

Figure 2-2 shows a sample recording sheet for velocity traverse data. Use the averages in the last two columns of Figure 2-2 to determine the average stack gas velocity from Equation 2-2.

Use Equation 2-3 to calculate the stack gas volumetric flow rate.

$$Q_s = 3600\,(1-B_{wo})\,V_s A \left(\frac{T_{std}}{(T_s)_{ave.}}\right)\left(\frac{P_s}{P_{std}}\right)$$

Equation 2-3

where:

Q_s = Volumetric flow rate, dry basis, standard conditions, ft.³/hr.

A = Cross-sectional area of stack, ft.²

T_{std} = Absolute temperature at standard conditions, 530° R.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

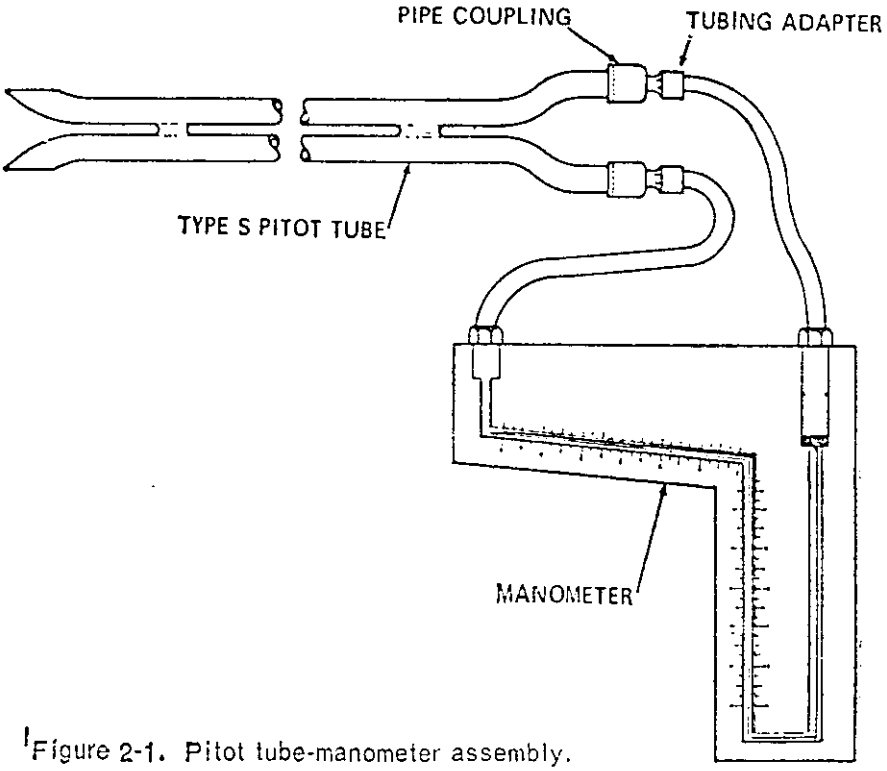

Figure 2-1. Pitot tube-manometer assembly.

6. *References.*

Mark, L. S., Mechanical Engineers' Handbook, McGraw-Hill Book Co., Inc., New York, N.Y., 1951.

Perry, J. H., Chemical Engineers' Handbook, McGraw-Hill Book Co., Inc., New York, N.Y., 1960.

Shigehara, R. T., W. F. Todd, and W. S. Smith, Significance of Errors in Stack Sampling Measurements. Paper presented at the Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14–19, 1970.

Standard Method for Sampling Stacks for Particulate Matter, In: 1971 Book of ASTM Standards, Part 23, Philadelphia, Pa., 1971, ASTM Designation D-2928-71.

Vennard, J. K., Elementary Fluid Mechanics. John Wiley & Sons, Inc., New York, N.Y., 1947.

PLANT____________________

DATE____________________

RUN NO.____________________

STACK DIAMETER, in.____________________

BAROMETRIC PRESSURE, in. Hg.____________________

STATIC PRESSURE IN STACK (P_g), in. Hg.____________________

OPERATORS____________________

SCHEMATIC OF STACK CROSS SECTION

Traverse point number	Velocity head, in. H_2O	$\sqrt{\Delta_p}$	Stack Temperature (T_S), °F
	AVERAGE:		

Figure 2-2. Velocity traverse data.

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METHOD 3—GAS ANALYSIS FOR CARBON DIOXIDE, EXCESS AIR, AND DRY MOLECULAR WEIGHT

1. *Principle and applicability.*

1.1 Principle. An integrated or grab gas sample is extracted from a sampling point and analyzed for its components using an Orsat analyzer.

1.2 Applicability. This method should be applied only when specified by the test procedures for determining compliance with the New Source Performance Standards. The test procedure will indicate whether a grab sample or an integrated sample is to be used.

2. *Apparatus.*

2.1 Grab sample (Figure 3-1).

2.1.1 Probe—Stainless steel or Pyrex[1] glass, equipped with a filter to remove particulate matter.

2.1.2 Pump—One-way squeeze bulb, or equivalent, to transport gas sample to analyzer.

[1] Trade name.

2.2 *Integrated sample (Figure 3-2).*

2.2.1 Probe—Stainless steel or Pyrex[1] glass, equipped with a filter to remove particulate matter.

2.2.2 Air-cooled condenser or equivalent—To remove any excess moisture.

2.2.3 Needle valve—To adjust flow rate.

2.2.4 Pump—Leak-free, diaphragm type, or equivalent, to pull gas.

2.2.5 Rate meter—To measure a flow range from 0 to 0.035 cfm.

2.2.6 Flexible bag—Tedlar,[1] or equivalent, with a capacity of 2 to 3 cu. ft. Leak test the bag in the laboratory before using.

2.2.7 Pitot tube—Type S, or equivalent, attached to the probe so that the sampling flow rate can be regulated proportional to the stack gas velocity when velocity is varying with time or a sample traverse is conducted.

2.3 *Analysis.*

2.3.1 Orsat analyzer, or equivalent.

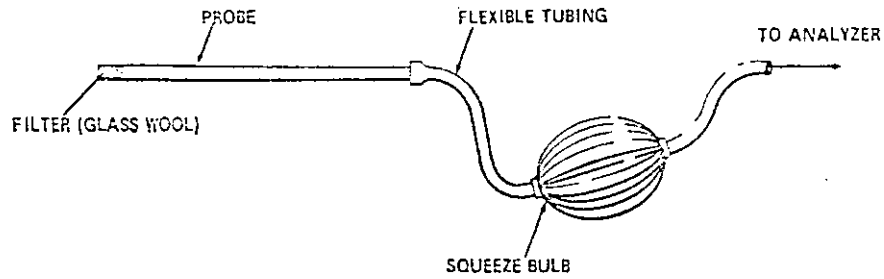

Figure 3-1. Grab-sampling train.

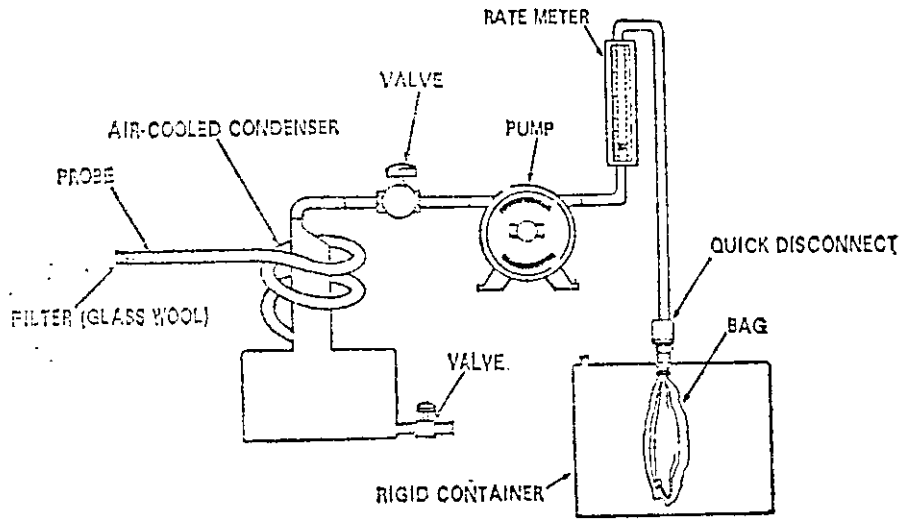

Figure 3-2. Integrated gas - sampling train.

3. *Procedure.*

3.1 Grab sampling.

3.1.1 Set up the equipment as shown in Figure 3-1, making sure all connections are leak-free. Place the probe in the stack at a sampling point and purge the sampling line.

3.1.2 Draw sample into the analyzer.

3.2 Integrated sampling.

3.2.1 Evacuate the flexible bag. Set up the equipment as shown in Figure 3-2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are tight and that there are no leaks.

3.2.2 Sample at a rate proportional to the stack velocity.

3.3 Analysis.

3.3.1 Determine the CO_2, O_2, and CO concentrations as soon as possible. Make as many passes as are necessary to give constant readings. If more than ten passes are necessary, replace the absorbing solution.

3.3.2 For grab sampling, repeat the sampling and analysis until three consecutive samples vary no more than 0.5 percent by volume for each component being analyzed.

3.3.3 For integrated sampling, repeat the analysis of the sample until three consecutive analyses vary no more than 0.2 percent by volume for each component being analyzed.

4. *Calculations.*

4.1 Carbon dioxide. Average the three consecutive runs and report the result to the nearest 0.1% CO_2.

4.2 Excess air. Use Equation 3-1 to calculate excess air, and average the runs. Report the result to the nearest 0.1% excess air.

$$\%\,EA = \frac{(\%\,O_2) - 0.5(\%\,CO)}{0.264(\%\,N_2) - (\%\,O_2) + 0.5(\%\,CO)} \times 100 \qquad \text{equation 3-1}$$

where:

$\%EA$ = Percent excess air.

$\%O_2$ = Percent oxygen by volume, dry basis.

$\%N_2$ = Percent nitrogen by volume, dry basis.

$\%CO$ = Percent carbon monoxide by volume, dry basis.

0.264 = Ratio of oxygen to nitrogen in air by volume.

4.3 Dry molecular weight. Use Equation 3-2 to calculate dry molecular weight and average the runs. Report the result to the nearest tenth.

$$M_d = 0.44(\%\,CO_2) + 0.32(\%\,O_2) + 0.28(\%\,N_2 + \%\,CO) \qquad \text{equation 3-2}$$

where:

M_d = Dry molecular weight, lb./lb-mole.

$\%CO_2$ = Percent carbon dioxide by volume, dry basis.

$\%O_2$ = Percent oxygen by volume, dry basis.

$\%N_2$ = Percent nitrogen by volume, dry basis.

0.44 = Molecular weight of carbon dioxide divided by 100.

0.32 = Molecular weight of oxygen divided by 100.

0.28 = Molecular weight of nitrogen and CO divided by 100.

5. *References.*

Altshuller, A. P., et al., Storage of Gases and Vapors in Plastic Bags, Int. J. Air & Water Pollution, 6:75–81, 1963.

Conner, William D., and J. S. Nader, Air Sampling with Plastic Bags, Journal of the American Industrial Hygiene Association, 25:291–297, May–June 1964.

Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif., November 1963.

METHOD 4—DETERMINATION OF MOISTURE IN STACK GASES

1. *Principle and applicability.*

1.1 Principle. Moisture is removed from the gas stream, condensed, and determined volumetrically.

1.2 Applicability. This method is applicable for the determination of moisture in stack gas only when specified by test procedures for determining compliance with New Source Performance Standards. This method does not apply when liquid droplets are present in the gas stream[1] and the moisture is subsequently used in the determination of stack gas molecular weight.

Other methods such as drying tubes, wet bulb-dry bulb techniques, and volumetric condensation techniques may be used.

2. *Apparatus.*

2.1 Probe—Stainless steel or Pyrex[2] glass sufficiently heated to prevent condensation and equipped with a filter to remove particulate matter.

2.2 Impingers—Two midget impingers, each with 30 ml. capacity, or equivalent.

2.3 Ice bath container—To condense moisture in impingers.

2.4 Silica gel tube (optional)—To protect pump and dry gas meter.

2.5 Needle valve—To regulate gas flow rate.

2.6 Pump—Leak-free, diaphragm type, or equivalent, to pull gas through train.

2.7 Dry gas meter—To measure to within 1% of the total sample volume.

2.8 Rotameter—To measure a flow range from 0 to 0.1 c.f.m.

2.9 Graduated cylinder—25 ml.

2.10 Barometer—Sufficient to read to within 0.1 inch Hg.

2.11 Pitot tube—Type S, or equivalent, attached to probe so that the sampling flow rate can be regulated proportional to the stack gas velocity when velocity is varying with time or a sample traverse is conducted.

3. *Procedure.*

3.1 Place exactly 5 ml. distilled water in each impinger. Assemble the apparatus without the probe as shown in Figure 4-1. Leak check by plugging the inlet to the first impinger and drawing a vacuum. Insure that flow through the dry gas meter is less than 1% of the sampling rate.

3.2 Connect the probe and sample at a constant rate of 0.075 c.f.m. or at a rate proportional to the stack gas velocity. Continue sampling until the dry gas meter registers 1 cubic foot or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4-2.

3.3 After collecting the sample, measure the volume increase to the nearest 0.5 ml.

4. *Calculations.*

4.1 Volume of water vapor collected.

$$V_{wc} = \frac{(V_f - V_i)\rho_{H_2O} R T_{std}}{P_{std} M_{H_2O}} = 0.0474 \frac{\text{ft.}^3}{\text{ml.}} (V_f - V_i) \qquad \text{equation 4-1}$$

where:

V_{wc} = Volume of water vapor collected (standard conditions), cu. ft.

V_f = Final volume of impinger contents, ml.

V_i = Initial volume of impinger contents, ml.

R = Ideal gas constant, 21.83 inches Hg—cu. ft./lb. mole-°R.

ρ_{H_2O} = Density of water, 1 g./ml.

T_{std} = Absolute temperature at standard conditions, 530° R.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

M_{H_2O} = Molecular weight of water, 18 lb./lb.-mole.

[1] If liquid droplets are present in the gas stream, assume the stream to be saturated, determine the average stack gas temperature by traversing according to Method 1, and use a psychrometric chart to obtain an approximation of the moisture percentage.

[2] Trade name.

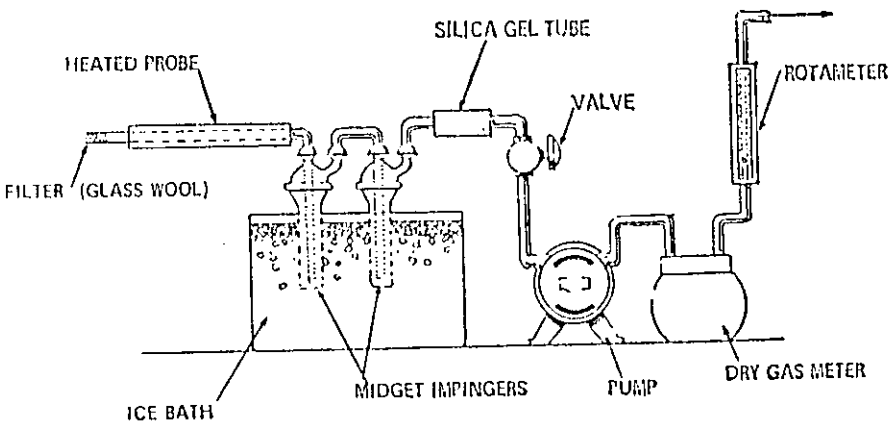

Figure 4-1. Moisture-sampling train.

LOCATION _____ COMMENTS

TEST _____

DATE _____

OPERATOR _____

BAROMETRIC PRESSURE _____

CLOCK TIME	GAS VOLUME THROUGH METER, (Vm), ft^3	ROTAMETER SETTING ft^3/min	METER TEMPERATURE, °F

Figure 4-2. Field moisture determination.

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4.2 Gas volume.

$$V_{mc} = V_m \left(\frac{P_m}{P_{std}}\right)\left(\frac{T_{std}}{T_m}\right) = 17.71 \frac{°R}{in.\ Hg}\left(\frac{V_m P_m}{T_m}\right) \quad \text{equation 4-2}$$

where:

V_{mc} = Dry gas volume through meter at standard conditions, cu. ft.
V_m = Dry gas volume measured by meter, cu. ft.
P_m = Barometric pressure at the dry gas meter, inches Hg.
P_{std} = Pressure at standard conditions, 29.92 inches Hg.
T_{std} = Absolute temperature at standard conditions, 530° R.
T_m = Absolute temperature at meter (°F + 460), °R.

4.3 Moisture content.

$$B_{wo} = \frac{V_{wc}}{V_{wc} + V_{mc}} + B_{wm} = \frac{V_{wc}}{V_{wc} + V_{mc}} + (0.025) \quad \text{equation 4-3}$$

where:

B_{wo} = Proportion by volume of water vapor in the gas stream, dimensionless.
V_{wc} = Volume of water vapor collected (standard conditions), cu. ft.
V_{mc} = Dry gas volume through meter (standard conditions), cu. ft.
B_{wm} = Approximate volumetric proportion of water vapor in the gas stream leaving the impingers, 0.025.

5. *References.*

Air Pollution Engineering Manual, Danielson, J. A. (ed.), U.S. DHEW, PHS, National Center for Air Pollution Control, Cincinnati, Ohio, PHS Publication No. 999-AP-40, 1967.

Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif., November 1963.

Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases, Western Precipitation Division of Joy Manufacturing Co., Los Angeles, Calif., Bulletin WP-50, 1968.

METHOD 5—DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and its weight is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources only when specified by the test procedures for determining compliance with New Source Performance Standards.

2. *Apparatus.*

2.1 Sampling train. The design specifications of the particulate sampling train used by EPA (Figure 5-1) are described in APTD-0581. Commercial models of this train are available

2.1.1 Nozzle—Stainless steel (316) with sharp, tapered leading edge.

2.1.2 Probe—Pyrex[1] glass with a heating system capable of maintaining a minimum gas temperature of 250° F. at the exit end during sampling to prevent condensation from occurring. When length limitations (greater than about 8 ft.) are encountered at temperatures less than 600° F., Incoloy 825[1], or equivalent, may be used. Probes for sampling gas streams at temperatures in excess of 600° F. must have been approved by the Administrator.

2.1.3 Pitot tube—Type S, or equivalent, attached to probe to monitor stack gas velocity.

[1] Trade name.

2.1.4 Filter Holder—Pyrex[1] glass with heating system capable of maintaining minimum temperature of 225° F.

2.1.5 Impingers / Condenser—Four impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a ½-inch ID glass tube extending to one-half inch from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip. A condenser may be used in place of the impingers provided that the moisture content of the stack gas can still be determined.

2.1.6 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5° F., dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume.

2.1.7 Barometer—To measure atmospheric pressure to ±0.1 inches Hg.

2.2 Sample recovery.

2.2.1 Probe brush—At least as long as probe.

2.2.2 Glass wash bottles—Two.

2.2.3 Glass sample storage containers.

2.2.4 Graduated cylinder—250 ml.

2.3 Analysis.

2.3.1 Glass weighing dishes.

2.3.2 Desiccator.

2.3.3 Analytical balance—To measure to ±0.1 mg.

2.3.4 Trip balance—300 g. capacity, to measure to ±0.05 g.

3. *Reagents.*

3.1 Sampling.

3.1.1 Filters—Glass fiber, MSA 1106 BH[1], or equivalent, numbered for identification and preweighed.

3.1.2 Silica gel—Indicating type, 6–16 mesh, dried at 175° C. (350° F.) for 2 hours.

3.1.3 Water.

3.1.4 Crushed ice.

3.2 Sample recovery.

3.2.1 Acetone—Reagent grade.

3.3 Analysis.

3.3.1 Water.

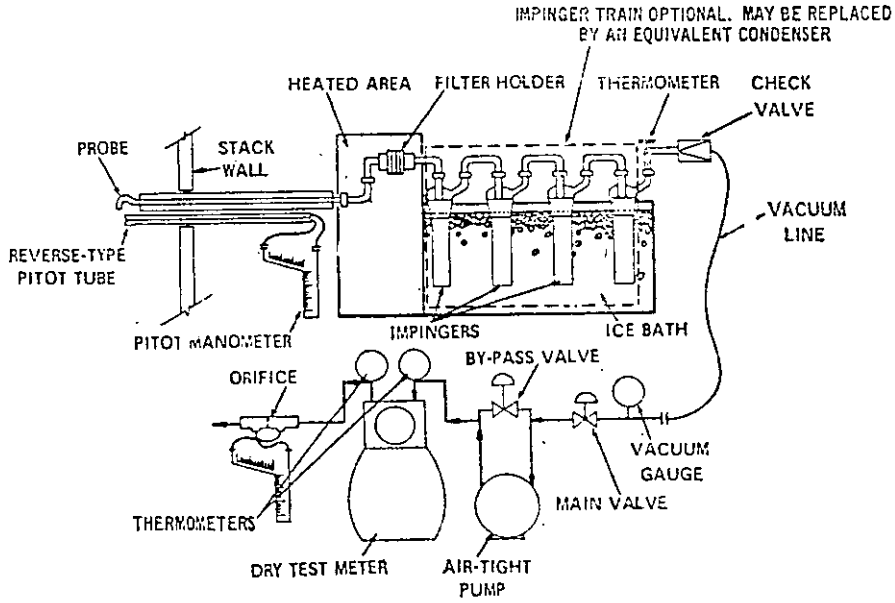

Figure 5-1. Particulate-sampling train.

3.3.2 Desiccant—Drierite,[1] indicating.

4. *Procedure.*

4.1 Sampling

4.1.1 After selecting the sampling site and the minimum number of sampling points, determine the stack pressure, temperature, moisture, and range of velocity head.

4.1.2 Preparation of collection train. Weigh to the nearest gram approximately 200 g. of silica gel. Label a filter of proper diameter, desiccate[2] for at least 24 hours and weigh to the nearest 0.5 mg. in a room where the relative humidity is less than 50%. Place 100 ml. of water in each of the first two impingers, leave the third impinger empty, and place approximately 200 g. of preweighed silica gel in the fourth impinger. Set up the train without the probe as in Figure 5-1. Leak check the sampling train at the sampling site by plugging up the inlet to the filter holder and pulling a 15 in. Hg vacuum. A leakage rate not in excess of 0.02 c.f.m. at a vacuum of 15 in. Hg is acceptable. Attach the probe and adjust the heater to provide a gas temperature of about 250° F. at the probe outlet. Turn on the filter heating system. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger as low as possible and preferably at 70° F., or less. Temperatures above 70° F. may result in damage to the dry gas meter from either moisture condensation or excessive heat.

[1] Trade name.
[2] Dry using Drierite[1] at 70° F. ± 10° F.

4.1.3 Particulate train operation. For each run, record the data required on the example sheet shown in Figure 5-2. Take readings at each sampling point, at least every 5 minutes, and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Sample for at least 5 minutes at each traverse point; sampling time must be the same for each point. Maintain isokinetic sampling throughout the sampling period. Nomographs are available which aid in the rapid adjustment of the sampling rate without other computations. APTD-0576 details the procedure for using these nomographs. Turn off the pump at the conclusion of each run and record the final readings. Remove the probe and nozzle from the stack and handle in accordance with the sample recovery process described in section 4.2.

PLANT _____
LOCATION _____
OPERATOR _____
DATE _____
RUN NO. _____
SAMPLE BOX NO. _____
METER BOX NO. _____
METER $\Delta H_@$ _____
C FACTOR _____

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE _____
BAROMETRIC PRESSURE _____
ASSUMED MOISTURE, % _____
HEATER BOX SETTING _____
PROBE LENGTH, in. _____
NOZZLE DIAMETER, in. _____
PROBE HEATER SETTING _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE (P_g), in. Hg.	STACK TEMPERATURE (T_s), °F	VELOCITY HEAD (ΔP_s),	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER (ΔH), in. H_2O	GAS SAMPLE VOLUME (V_m), ft^3	GAS SAMPLE TEMPERATURE AT DRY GAS METER		SAMPLE BOX TEMPERATURE, °F	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °F
							INLET ($T_{m_{in}}$), °F	OUTLET ($T_{m_{out}}$), °F		
TOTAL							Avg.	Avg.		
AVERAGE							Avg.			

Figure 5-2. Particulate field data.

4.2 Sample recovery. Exercise care in moving the collection train from the test site to the sample recovery area to minimize the loss of collected sample or the gain of extraneous particulate matter. Set aside a portion of the acetone used in the sample recovery as a blank for analysis. Measure the volume of water from the first three impingers, then discard. Place the samples in containers as follows:

Container No. 1. Remove the filter from its holder, place in this container, and seal.

Container No. 2. Place loose particulate matter and acetone washings from all sample-exposed surfaces prior to the filter in this container and seal. Use a razor blade, brush, or rubber policeman to lose adhering particles.

Container No. 3. Transfer the silica gel from the fourth impinger to the original container and seal. Use a rubber policeman as an aid in removing silica gel from the impinger.

4.3 Analysis. Record the data required on the example sheet shown in Figure 5-3. Handle each sample container as follows:

Container No. 1. Transfer the filter and any loose particulate matter from the sample container to a tared glass weighing dish, desiccate, and dry to a constant weight. Report results to the nearest 0.5 mg.

Container No. 2. Transfer the acetone washings to a tared beaker and evaporate to dryness at ambient temperature and pressure. Desiccate and dry to a constant weight. Report results to the nearest 0.5 mg.

Container No. 3. Weigh the spent silica gel and report to the nearest gram.

5. *Calibration.*

Use methods and equipment which have been approved by the Administrator to calibrate the orifice meter, pitot tube, dry gas meter, and probe heater. Recalibrate after each test series.

6. *Calculations.*

6.1 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5-2).

6.2 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (70° F., 29.92 inches Hg) by using Equation 5-1.

$$V_{m_{std}} = V_m\left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{P_{std}}\right) = \left(17.71\ \frac{°R}{in.\ Hg}\right)V_m\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{T_m}\right)$$

equation 5-1

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_{std} = Absolute temperature at standard conditions, 530° R.

$$V_{w_{std}} = V_{l_c}\frac{\rho H_2O}{M_{H_2O}}\ \frac{RT_{std}}{P_{std}}\ \frac{lb.}{454\ gm.} = 0.0474\frac{cu.\ ft.}{ml.}V_{l_c}$$

equation 5-2

where:

$V_{w_{std}}$ = Volume of water vapor in the gas sample (standard conditions), cu. ft.

V_{l_c} = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.

ρH_2O = Density of water, 1 g./ml.

M_{H_2O} = Molecular weight of water, 18 lb./lb.-mole.

R = Ideal gas constant, 21.83 inches Hg—cu. ft./lb.-mole-°R.

T_{std} = Absolute temperature at standard conditions, 530° R.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.4 Moisture content.

$$B_{wo} = \frac{V_{w_{std}}}{V_{m_{std}} + V_{w_{std}}}$$

equation 5-3

where:

B_{wo} = Proportion by volume of water vapor in the gas stream, dimensionless.

$V_{w_{std}}$ = Volume of water in the gas sample (standard conditions), cu. ft.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

6.5 Total particulate weight. Determine the total particulate catch from the sum of the weights on the analysis data sheet (Figure 5-3).

6.6 Concentration.

6.6.1 Concentration in gr./s.c.f.

$$c'_s = \left(0.0154\frac{gr.}{mg.}\right)\left(\frac{M_n}{V_{m_{std}}}\right)$$

equation 5-4

where:

c'_s = Concentration of particulate matter in stack gas, gr./s.c.f., dry basis.

M_n = Total amount of particulate matter collected, mg.

$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

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PLANT _____

DATE _____

RUN NO. _____

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			

	VOLUME OF LIQUID WATER COLLECTED		
	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g	
FINAL			
INITIAL			
LIQUID COLLECTED			
TOTAL VOLUME COLLECTED		g*	ml

CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER. (1 g/ml):

$$\frac{\text{INCREASE, g}}{(1\ \text{g/ml})} = \text{VOLUME WATER, ml}$$

Figure 5-3. Analytical data.

6.6.2 Concentration in lb./cu. ft.

$$c_s = \frac{\left(\frac{1}{453{,}600}\ \frac{\text{lb.}}{\text{mg.}}\right) M_n}{V_{m_{std}}} = 2.205 \times 10^{-6} \frac{M_n}{V_{m_{std}}} \qquad \text{equation 5-5}$$

where:

c_s = Concentration of particulate matter in stack gas, lb./s.c.f., dry basis.

453,600 = Mg/lb.

M_n = Total amount of particulate matter collected, mg.

$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

6.7 Isokinetic variation.

$$I = \frac{T_s\left[\left(\frac{0.00267\ \text{in. Hg-cu. ft.}}{\text{ml}-°\text{R}}\right) V_{l_c} + \frac{V_m}{T_m}\left(P_{bar} + \frac{H}{13.6}\right)\right]\left(1.667\ \frac{\text{min.}}{\text{sec.}}\right)}{\theta V_s P_s A_n} \qquad \text{equation 5-6}$$

where:

I = Percent of isokinetic sampling.

V_{l_c} = Total volume of liquid collected in impingers and silica gel (See Fig. 5-3), ml.

ρ_{H_2O} = Density of water, 1 g./ml.

R = Ideal gas constant, 21.83 inches Hg-cu. ft./lb. mole-°R.

M_{H_2O} = Molecular weight of water, 18 lb./lb.-mole.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_m = Absolute average dry gas meter temperature (see Figure 5-2), °R.

P_{bar} = Barometric pressure at sampling site, inches Hg.

ΔH = Average pressure drop across the orifice (see Fig. 5-2), inches H_2O.

T_s = Absolute average stack gas temperature (see Fig. 5-2), °R.

θ = Total sampling time, min.

V_s = Stack gas velocity calculated by Method 2, Equation 2-2, ft./sec.

P_s = Absolute stack gas pressure, inches Hg.

A_n = Cross-sectional area of nozzle, sq. ft.

6.8 Acceptable results. The following range sets the limit on acceptable isokinetic sampling results:

If $90\% \leq I \leq 110\%$, the results are acceptable, otherwise, reject the results and repeat the test.

7. *Reference.*

Addendum to Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, Dec. 6, 1967.

Martin, Robert M., Construction Details of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0581.

Rom, Jerome J., Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0576.

Smith, W. S., R. T. Shigehara, and W. F. Todd, A Method of Interpreting Stack Sampling Data, Paper presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14-19, 1970.

Smith, W. S., et al., Stack Gas Sampling Improved and Simplified with New Equipment, APCA paper No. 67-119, 1967.

Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, 1967.

METHOD 6—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

1.1 Principle. A gas sample is extracted from the sampling point in the stack. The acid mist, including sulfur trioxide, is separated from the sulfur dioxide. The sulfur dioxide fraction is measured by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfur dioxide emissions from stationary sources only when specified by the test procedures for determining compliance with New Source Performance Standards.

2. *Apparatus.*

2.1 Sampling. See Figure 6-1.

2.1.1 Probe—Pyrex[1] glass, approximately 5 to 6 mm. ID, with a heating system to prevent condensation and a filtering medium to remove particulate matter including sulfuric acid mist.

2.1.2 Midget bubbler—One, with glass wool packed in top to prevent sulfuric acid mist carryover.

2.1.3 Glass wool.

2.1.4 Midget impingers—Three.

2.1.5 Drying tube—Packed with 6 to 16 mesh indicating-type silica gel, or equivalent, to dry the sample.

2.1.6 Valve—Needle valve, or equivalent, to adjust flow rate.

2.1.7 Pump—Leak-free, vacuum type.

2.1.8 Rate meter—Rotameter or equivalent, to measure a 0-10 s.c.f.h. flow range.

2.1.9 Dry gas meter—Sufficiently accurate to measure the sample volume within 1%.

2.1.10 Pitot tube—Type S, or equivalent,

[1] Trade names.

necessary only if a sample traverse is required, or if stack gas velocity varies with time.

2.2 Sample recovery.

2.2.1 Glass wash bottles—Two.

2.2.2 Polyethylene storage bottles—To store impinger samples.

2.3 Analysis.

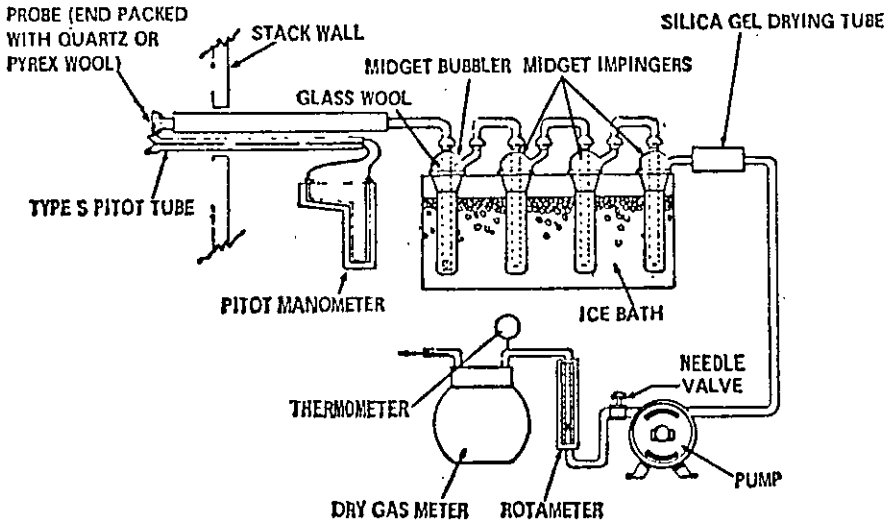

Figure 6-1. SO_2 sampling train.

2.3.1 Pipettes—Transfer type, 5 ml. and 10 ml. sizes (0.1 ml. divisions) and 25 ml. size (0.2 ml. divisions).

2.3.2 Volumetric flasks—50 ml., 100 ml., and 1,000 ml.

2.3.3 Burettes—5 ml. and 50 ml.

2.3.4 Erlenmeyer flask—125 ml.

3. *Reagents.*

3.1 Sampling.

3.1.1 Water—Deionized, distilled.

3.1.2 Isopropanol, 80%—Mix 80 ml. of isopropanol with 20 ml. of distilled water.

3.1.3 Hydrogen peroxide, 3%—dilute 100 ml. of 30% hydrogen peroxide to 1 liter with distilled water. Prepare fresh daily.

3.2 Sample recovery.

3.2.1 Water—Deionized, distilled.

3.2.2 Isopropanol, 80%.

3.3 Analysis.

3.3.1 Water—Deionized, distilled.

3.3.2 Isopropanol.

3.3.3 Thorin indicator—1-(o-arsonophenylazo)-2-naphthol-3,6-disulfonic acid, disodium salt (or equivalent). Dissolve 0.20 g. in 100 ml. distilled water.

3.3.4 Barium perchlorate (0.01 *N*)—Dissolve 1.95 g. of barium perchlorate [$Ba(ClO_4)_2 \cdot 3H_2O$] in 200 ml. distilled water and dilute to 1 liter with isopropanol. Standardize with sulfuric acid. Barium chloride may be used.

3.3.5 Sulfuric acid standard (0.01 *N*)—Purchase or standardize to ±0.0002 *N* against 0.01N NaOH which has previously been standardized against potassium acid phthalate (primary standard grade).

4. *Procedure.*

4.1 Sampling.

4.1.1 Preparation of collection train. Pour 15 ml. of 80% isopropanol into the midget bubbler and 15 ml. of 3% hydrogen peroxide into each of the first two midget impingers. Leave the final midget impinger dry. Assemble the train as shown in Figure 6-1. Leak check the sampling train at the sampling site by plugging the probe inlet and pulling a 10 inches Hg vacuum. A leakage rate not in excess of 1% of the sampling rate is acceptable. Carefully release the probe inlet plug and turn off the pump. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 70° F. or less.

4.1.2 Sample collection. Adjust the sample flow rate proportional to the stack gas velocity. Take readings at least every five minutes and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the tip of the probe at the first sampling point and start the pump. Sample proportionally throughout the run. At the conclusion of each run, turn off the pump and record the final readings. Remove the probe from the stack and disconnect it from the train. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes.

4.2 Sample recovery. Disconnect the impingers after purging. Discard the contents of the midget bubbler. Pour the contents of the midget impingers into a polyethylene shipment bottle. Rinse the three midget impingers and the connecting tubes with distilled water and add these washings to the same storage container.

4.3 Sample analysis. Transfer the contents of the storage container to a 50 ml. volumetric flask. Dilute to the mark with deionized, distilled water. Pipette a 10 ml. aliquot of this solution into a 125 ml. Erlenmeyer flask. Add 40 ml. of isopropanol and two to four drops of thorin indicator. Titrate to a pink endpoint using 0.01 *N* barium perchlorate. Run a blank with each series of samples.

5. *Calibration.*

5.1 Use standard methods and equipment which have been approved by the Administrator to calibrate the rotameter, pitot tube, dry gas meter, and probe heater.

5.2 Standardize the barium perchlorate against 25 ml. of standard sulfuric acid containing 100 ml. of isopropanol.

6. *Calculations.*

6.1 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (70° F. and 29.92 inches Hg) by using equation 6-1.

$$V_{m_{std}} = V_m \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}}{P_{std}}\right) = 17.71 \frac{°R}{in.\ Hg}\left(\frac{V_m P_{bar}}{T_m}\right) \quad \text{equation 6-1}$$

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_{std} = Absolute temperature at standard conditions, 530° R.

T_m = Average dry gas meter temperature, °R.

P_{bar} = Barometric pressure at the orifice meter, inches Hg.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.2 Sulfur dioxide concentration.

$$C_{SO_2} = \left(7.05 \times 10^{-5} \frac{lb.\text{-}l.}{g.\text{-}ml.}\right) \frac{(V_t - V_{tb}) N \left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}} \quad \text{equation 6-2}$$

where:

C_{SO_2} = Concentration of sulfur dioxide at standard conditions, dry basis, lb./cu. ft.

7.05×10^{-5} = Conversion factor, including the number of grams per gram equivalent of sulfur dioxide (32 g./g.-eq.), 453.6 g./lb., and 1,000 ml./l., lb.-l./g.-ml.

V_t = Volume of barium perchlorate titrant used for the sample, ml.

V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.

N = Normality of barium perchlorate titrant, g.-eq./l.

V_{soln} = Total solution volume of sulfur dioxide, 50 ml.

V_a = Volume of sample aliquot titrated, ml.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft., see Equation 6-1.

7. *References.*

Atmospheric Emissions from Sulfuric Acid Manufacturing Processes, U.S. DHEW, PHS, Division of Air Pollution, Public Health Service Publication No. 999-AP-13, Cincinnati, Ohio, 1965.

Corbett, P. F., The Determination of SO_2 and SO_3 in Flue Gases, Journal of the Institute of Fuel, 24:237-243, 1961.

Matty, R. E. and E. K. Diehl, Measuring Flue-Gas SO_2 and SO_3, Power *101*:94-97, November, 1957.

Patton, W. F. and J. A. Brink, Jr., New Equipment and Techniques for Sampling Chemical Process Gases, J. Air Pollution Control Association, *13*, 162 (1963).

METHOD 7—DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except

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METHOD 13B

DETERMINATION OF TOTAL FLUORIDE EMISSIONS

PLANT _____
LOCATION _____
OPERATOR _____
DATE _____
RUN NO. _____
SAMPLE BOX NO. _____
METER BOX NO. _____
METER ΔH@ _____
C FACTOR _____
PITOT TUBE COEFFICIENT, C_p _____

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE _____
BAROMETRIC PRESSURE _____
ASSUMED MOISTURE, % _____
PROBE LENGTH, m (ft) _____
NOZZLE IDENTIFICATION NO. _____
AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) _____
PROBE HEATER SETTING _____
LEAK RATE, m³/min (cfm) _____
PROBE LINER MATERIAL _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE mm Hg (in. Hg)	STACK TEMPERATURE T_s °C (°F)	VELOCITY HEAD (Δp_s), mm H₂O (in. H₂O)	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER mm H₂O (in. H₂O)	GAS SAMPLE VOLUME m³ (ft³)	GAS SAMPLE TEMPERATURE AT DRY GAS METER INLET °C (°F)	GAS SAMPLE TEMPERATURE AT DRY GAS METER OUTLET °C (°F)	FILTER HOLDER TEMPERATURE, °C (°F)	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
TOTAL										
AVERAGE										

Figure 13A-2. Field data.

METHOD 13A—DETERMINATION OF TOTAL FLUORIDE EMISSIONS FROM STATIONARY SOURCES—SPECIFIC ION ELECTRODE METHOD.

1. *Principle and Applicability.*

1.1 Principle. Gaseous and particulate fluorides are withdrawn isokinetically from the source using a sampling train. The fluorides are collected in the impinger water and on the filter of the sampling train. The weight of total fluorides in the train is determined by the specific ion electrode method.

1.2 Applicability. This method is applicable for the determination of fluoride emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. Fluorocarbons such as Freons, are not quantitatively collected or measured by this procedure.

2. *Range and Sensitivity.*

The fluoride specific ion electrode analytical method covers the range of 0.02–2,000 μg F/ml; however, measurements of less than 0.1 μg F/ml require extra care. Sensitivity has not been determined.

3. *Interferences.*

During the laboratory analysis, aluminum in excess of 300 mg/liter and silicon dioxide in excess of 300 μg/liter will prevent complete recovery of fluoride.

4. *Precision, Accuracy and Stability.*

The accuracy of fluoride electrode measurements has been reported by various researchers to be in the range of 1–5 percent in a concentration range of 0.04 to 80 mg/l. A change in the temperature of the sample will change the electrode response; a change of 1°C will produce a 1.5 percent relative error in the measurement. Lack of stability in the electrometer used to measure EMF can introduce error. An error of 1 millivolt in the EMF measurement produces a relative error of 4 percent regardless of the absolute concentration being measured.

5. *Apparatus.*

5.1 Sample train. See Figure 13A-1 (Method 13A); it is similar to the Method 5 train except for the interchangeability of the position of the filter. Commercial models of this train are available. However, if one desires to build his own, complete construction details are described in APTD-0581; for clarifications, the APTD-0581 document and for allowable modifications to Figure 13A-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein.

5.1.1 Probe nozzle—Stainless steel (316) with sharp, tapered leading edge. The angle of taper shall be ≦30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. The wall thickness of the nozzle shall be less than or equal to that of 20 gauge tubing, i.e., 0.165 cm (0.065 in.) and the distance from the tip of the nozzle to the first bend or point of disturbance shall be at least two times the outside nozzle diameter. The nozzle shall be constructed from seamless stainless steel tubing. Other configurations and construction material may be used with approval from the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 cm (⅛ in.) up to 1.27 cm (½ in.) (or larger if higher volume sampling trains are used) inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in the calibration section.

5.1.2 Probe liner—Borosilicate glass or stainless steel (316). When the filter is located immediately after the probe, a probe heating system may be used to prevent filter plugging resulting from moisture condensation. The temperature in the probe shall not exceed 120±14°C (248±25°F).

5.1.3 Pitot tube—Type S, or other device approved by the Administrator, attached to probe to allow constant monitoring of the stack gas velocity. The face openings of the pitot tube and the probe nozzle shall be adjacent and parallel to each other, not necessarily on the same plane, during sampling. The free space between the nozzle and pitot tube shall be at least 1.9 cm (0.75 in.). The free space shall be set based on a 1.3 cm (0.5 in.) ID nozzle, which is the largest size nozzle used.

The pitot tube must also meet the criteria specified in Method 2 and be calibrated according to the procedure in the calibration section of that method.

5.1.4 Differential pressure gauge—Inclined manometer capable of measuring velocity head to within 10 percent of the minimum measured value. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers are not easily adaptable to field conditions and are not easy to use with pulsating flow. Thus, other methods or devices acceptable to the Administrator may be used when conditions warrant.

5.1.5 Filter holder—Borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g. if probe liner is stainless steel, then filter holder may be stainless steel. The holder design shall provide a positive seal against leakage from the outside or around the filter.

5.1.6 Filter heating system—When moisture condensation is a problem, any heating system capable of maintaining a temperature around the filter holder during sampling of no greater than 120±14°C (248±25°F). A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that when the filter heater is used, the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

5.1.7 *Impingers—Four* impingers connected as shown in Figure 13A-1 with ground glass (or equivalent), vacuum tight fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a 1¼ cm (½ in.) inside diameter glass tube extending to 1¼ cm (½ in.) from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip.

5.1.8 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry gas meter with 2 percent accuracy at the required sampling rate, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

5.1.9 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value shall be requested and an adjustment for elevation differences shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase.

5.2 Sample recovery.

5.2.1 Probe liner and probe nozzle brushes—Nylon bristles with stainless steel wire handles. The probe brush shall have extensions, at least as long as the probe, of stainless steel, teflon, or similarly inert material. Both brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

5.2.2 Glass wash bottles—Two.

5.2.3 Sample storage containers—Wide mouth, high density polyethylene bottles, 1 liter.

5.2.4 Plastic storage containers—Air tight containers of sufficient volume to store silica gel.

5.2.5 Graduated cylinder—250 ml.

5.2.6 Funnel and rubber policeman—To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

5.3 Analysis.

5.3.1 Distillation apparatus—Glass distillation apparatus assembled as shown in Figure 13A-2 (Method 13A).

5.3.2 Hot plate—Capable of heating to 500°C.

5.3.3 Electric muffle furnace—Capable of heating to 600°C.

5.3.4 Crucibles—Nickel, 75 to 100 ml capacity.

5.3.5 Beaker—1000 ml.

5.3.6 Volumetric flask—50 ml.

5.3.7 Erlenmeyer flask or plastic bottle—500 ml.

5.3.8 Constant temperature bath—Capable of maintaining a constant temperature of ±1.0°C in the range of room temperature.

5.3.9 Trip balance—300 g capacity to measure to ±0.5 g.

5.3.10 Fluoride ion activity sensing electrode.

5.3.11 Reference electrode—Single junction; sleeve type. (A combination-type electrode having the references electrode and the fluoride-ion sensing electrode built into one unit may also be used).

5.3.12 Electrometer—A pH meter with millivolt scale capable of ±0.1 mv resolution, or a specific ion meter made specifically for specific ion use.

5.3.13 Magnetic stirrer and TFE fluorocarbon coated stripping bars.

6. *Reagents.*

6.1 Sampling.

6.1.1 Filters—Whatman No. 1 filters, or equivalent, sized to fit filter holder.

6.1.2 Silica gel—Indicating type, 6–16 mesh. If previously used, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received.

6.1.3 Water—Distilled.

6.1.4 Crushed ice.

6.1.5 Stopcock grease—Acetone insoluble, heat stable silicone grease. This is not necessary if screw-on connectors with teflon sleeves, or similar, are used.

6.2 Sample recovery.

6.2.1 Water—Distilled from same container as 6.1.3.

6.3 Analysis.

6.3.1 Calcium oxide (CaO)—Certified grade containing 0.005 percent fluoride or less.

6.3.2 Phenolphthalein Indicator—0.1 percent in 1:1 ethanol water mixture.

6.3.3 Sodium hydroxide (NaOH)—Pellets, ACS reagent grade or equivalent.

6.3.4 Sulfuric acid (H_2SO_4)—Concentrated, ACS reagent grade or equivalent.

6.3.5 Filters—Whatman No. 541, or equivalent.

6.3.6 Water—Distilled, from same container as 6.1.3.

6.3.7 Total Ionic Strength Adjustment Buffer (TISAB)—Place approximately 500 ml of distilled water in a 1-liter beaker. Add 57 ml glacial acetic acid, 58 g sodium chloride, and 4 g CDTA (Cyclohexylene dinitrilo tetraacetic acid). Stir to dissolve. Place the beaker in a water bath to cool it. Slowly add 5 M NaOH to the solution, measuring the pH continuously with a calibrated pH/reference electrode pair, until the pH is 5.3. Cool to room temperature. Pour into a 1-liter flask and dilute to volume with distilled water. Commercially prepared TISAB buffer may be substituted for the above.

6.3.8 Fluoride Standard Solution—0.1 M fluoride reference solution. Add 4.20 grams of reagent grade sodium fluoride (NaF) to a 1-liter volumetric flask and add enough distilled water to dissolve. Dilute to volume with distilled water.

7. *Procedure.*

Note: The fusion and distillation steps of this procedure will not be required, if it can be shown to the satisfaction of the Administrator that the samples contain only water-soluble fluorides.

7.1 Sampling. The sampling shall be conducted by competent personnel experienced with this test procedure.

7.1.1 Pretest preparation. All train components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh approximately 200-300 g of silica gel in air tight containers to the nearest 0.5 g. Record the total weight, both silica gel and container, on the container. More silica gel may be used but care should be taken during sampling that it is not entrained and carried out from the impinger. As an alternative, the silica gel may be weighed directly in the impinger or its sampling holder just prior to the train assembly.

7.1.2 Preliminary determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2 and moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate calculations. Estimates may be used. However, final results will be based on actual measurements made during the test.

Select a nozzle size based on the range of velocity heads such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the differential pressure gauge is capable of measuring the minimum velocity head value to within 10 percent, or as specified by the Administrator.

Select a suitable probe liner and probe length such that all traverse points can be sampled. Consider sampling from opposite sides for large stacks to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that the sampling time per point is not less than 2 min. or select some greater time interval as specified by the Administrator, and such that the sample volume that will be taken will exceed the required minimum total gas sample volume specified in the test procedures for the specific industry. The latter is based on an approximate average sampling rate. Note also that the minimum total sample volume is corrected to standard conditions.

It is recommended that a half-integral or integral number of minutes be sampled at each point in order to avoid timekeeping errors.

In some circumstances, e.g. batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

7.1.3 Preparation of collection train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and place approximately 200-300 g or more, if necessary, of preweighed silica gel in the fourth impinger. Record the weight of the silica gel and container on the data sheet. Place the empty container in a clean place for later use in the sample recovery.

Place a filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to not allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed.

When glass liners are used, install selected nozzle using a Viton A O-ring. The Viton A O-ring is installed as a seal where the nozzle is connected to a glass liner. See APTD-0576 for details. When metal liners are used, install the nozzle as above or by a leak free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Unless otherwise specified by the Administrator, attach a temperature probe to the metal sheath of the sampling probe so that the sensor extends beyond the probe tip and does not touch any metal. Its position should be about 1.9 to 2.54 cm (0.75 to 1 in.) from the pitot tube and probe nozzle to avoid interference with the gas flow.

Assemble the train as shown in Figure 13A-1 (Method 13A) with the filter between the third and fourth impingers. Alternatively, the filter may be placed between the probe and first impinger. A filter heating system may be used to prevent moisture condensation, but the temperature around the filter holder shall not exceed 120°±14°C (248±25°F). [(Note: Whatman No. 1 filter decomposes at 150°C (300°F)).] Record filter location on the data sheet.

Place crushed ice around the impingers.

7.1.4 Leak check procedure—After the sampling train has been assembled, turn on and set (if applicable) the probe and filter heating system(s) to reach a temperature sufficient to avoid condensation in the probe. Allow time for the temperature to stabilize. Leak check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum. A leakage rate in excess of 4% of the average sampling rate or 0.0057 m³/min. (0.02 cfm), whichever is less, is unacceptable.

The following leak check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until 380 mm Hg (15 in. Hg) vacuum is reached. *Do Not* reverse direction of by-pass valve. This will cause water to back up into the filter holder. If 380 mm Hg (15 in. Hg) is exceeded, either leak check at this higher vacuum or end the leak check as described below and start over.

When the leak check is completed, first slowly remove the plug from the inlet to the probe or filter holder and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder (if placed before the impingers) and silica gel from being entrained backward into the third impinger.

Leak checks shall be conducted as described whenever the train is disengaged, e.g. for silica gel or filter changes during the test, prior to each test run, and at the completion of each test run. If leaks are found to be in excess of the acceptable rate, the test will be considered invalid. To reduce lost time due to leakage occurrences, it is recommended that leak checks be conducted between port changes.

7.1.5 Particulate train operation—During the sampling run, an isokinetic sampling rate within 10%, or as specified by the Administrator, of true isokinetic shall be maintained.

For each run, record the data required on the example data sheet shown in Figure 13A-3 (Method 13A). Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment when changes in flow rates are made, and when sampling is halted. Take other data point readings at least once at each sampling point during each time increment and additional readings when significant changes (20% variation in velocity head readings) occur.

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make additional adjustments in flow rate. Be sure to level and zero the manometer.

Clean the portholes prior to the test run to minimize chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify (if applicable) that the probe heater is working and filter heater is up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available for sampling trains using type S pitot tubes with 0.85±0.02 (coefficients (C_s), and when sampling in air or a stack gas with equivalent density (molecular weight, M_d, equal to 29±4), which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. APTD-0576 details the procedure for using these nomographs. If C_s and M_d are outside the above stated ranges, do not use the nomograph unless appropriate steps are taken to compensate for the deviations.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to avoid water backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and portholo to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes to minimize chance of extracting deposited material.

During the test run, make periodic adjustments to keep the probe and (if applicable) filter temperatures at their proper values. Add more ice and, if necessary, salt to the ice bath, to maintain a temperature of less than 20°C (68°F) at the impinger/silica gel outlet, to avoid excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes high enough to make isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. After the new filter or filter assembly is installed, conduct a leak check. The final emission results shall be based on the summation of all filter catches.

A single train shall be used for the entire sample run, except for filter and silica gel changes. However, if approved by the Administrator, two or more trains may be used for a single test run when there are two or more ducts or sampling ports. The final emission results shall be based on the total of all sampling train catches.

At the end of the sample run, turn off the pump, remove the probe and nozzle from the stack, and record the final dry gas meter reading. Perform a leak check.[1] Calculate percent isokinetic (see calculation section) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

[1] With acceptability of the test run to be based on the same criterion as in 7.1.4.

7.2 Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to keep from losing part of the sample. Do not cap off the probe tip tightly while the sampling train is cooling down, as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate, if present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Ground glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Inspect the train prior to and during disassembly and note any abnormal conditions. Using a graduated cylinder, measure and record the volume of the water in the first three impingers, to the nearest ml; any condensate in the probe should be included in this determination. Treat the samples as follows:

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7.2.1 Container No. 1. Transfer the impinger water from the graduated cylinder to this container. Add the filter to this container. Wash all sample exposed surfaces, including the probe tip, probe, first three impingers, impinger connectors, filter holder, and graduated cylinder thoroughly with distilled water. Wash each component three separate times with water and clean the probe and nozzle with brushes. A maximum wash of 500 ml is used, and the washings are added to the sample container which must be made of polyethylene.

7.2.2 Container No. 2. Transfer the silica gel from the fourth impinger to this container and seal.

7.3 Analysis. Treat the contents of each sample container as described below.

7.3.1 Container No. 1.

7.3.1.1 Filter this container's contents, including the Whatman No 1 filter, through Whatman No. 541 filter paper, or equivalent into a 1500 ml beaker. Note: If filtrate volume exceeds 900 ml make filtrate basic with NaOH to phenolphthalein and evaporate to less than 900 ml.

7.3.1.2 Place the Whatman No. 541 filter containing the insoluble matter (including the Whatman No. 1 filter) in a nickel crucible, add a few ml of water and macerate the filter with a glass rod.

Add 100 mg CaO to the crucible and mix the contents thoroughly to form a slurry. Add a couple of drops of phenolphthalein indicator. The indicator will turn red in a basic medium. The slurry should remain basic during the evaporation of the water or fluoride ion will be lost. If the indicator turns colorless during the evaporation, an acidic condition is indicated. If this happens add CaO until the color turns red again.

Place the crucible in a hood under infrared lamps or on a hot plate at low heat. Evaporate the water completely.

After evaporation of the water, place the crucible on a hot plate under a hood and slowly increase the temperature until the paper chars. It may take several hours for complete charring of the filter to occur.

Place the crucible in a cold muffle furnace and gradually (to prevent smoking) increase the temperature to 600°C, and maintain until the contents are reduced to an ash. Remove the crucible from the furnace and allow it to cool.

7.3.1.3 Add approximately 4 g of crushed NaOH to the crucible and mix. Return the crucible to the muffle furnace, and fuse the sample for 10 minutes at 600°C.

Remove the sample from the furnace and cool to ambient temperature. Using several rinsings of warm distilled water transfer the contents of the crucible to the beaker containing the filtrate from container No. 1 (7.3.1). To assure complete sample removal, rinse finally with two 20 ml portions of 25 percent (v/v) sulfuric acid and carefully add to the beaker. Mix well and transfer to a one-liter volumetric flask. Dilute to volume with distilled water and mix thoroughly. Allow any undissolved solids to settle.

7.3.2 Container No. 2. Weigh the spent silica gel and report to the nearest 0.5 g.

7.3.3 Adjustment of acid/water ratio in distillation flask—(Utilize a protective shield when carrying out this procedure). Place 400 ml of distilled water in the distilling flask and add 200 ml of concentrated H_2SO_4. Caution: Observe standard precautions when mixing the H_2SO_4 by slowly adding the acid to the flask with constant swirling. Add some soft glass beads and several small pieces of broken glass tubing and assemble the apparatus as shown in Figure 13A-2. Heat the flask until it reaches a temperature of 175°C to adjust the acid/water ratio for subsequent distillations. Discard the distillate.

7.3.4 Distillation—Cool the contents of the distillation flask to below 50°C. Pipette an aliquot of sample containing less than 0.6 mg F directly into the distilling flask and add distilled water to make a total volume of 220 ml added to the distilling flask. [For an estimate of what size aliquot does not exceed 0.6 mg F, select an aliquot of the solution and treat as described in Section 7.3.6. This will give an approximation of the fluoride content, but only an approximation since interfering ions have not been removed by the distillation step.]

Place a 250 ml volumetric flask at the condenser exist. Now begin distillation and gradually increase the heat and collect all the distillate up to 175°C. Caution: Heating the solution above 175°C will cause sulfuric acid to distill over.

The acid in the distilling flask can be used until there is carryover of interferences or poor fluoride recovery. An occasional check of fluoride recovery with standard solutions is advised. The acid should be changed whenever there is less than 90 percent recovery or blank values are higher than 0.1 µg/ml.

7.3.5 Determination of concentration—Bring the distillate in the 250 ml volumetric flask to the mark with distilled water and mix thoroughly. Pipette a 25 ml aliquot from the distillate. Add an equal volume of TISAB and mix. The sample should be at the same temperature as the calibration standards when measurements are made. If ambient temperature fluctuates more than ±2°C from the temperature at which the calibration standards were measured, condition samples and standards in a constant temperature bath. Measurement with the sample with a magnetic stirrer during measurement to minimize electrode response

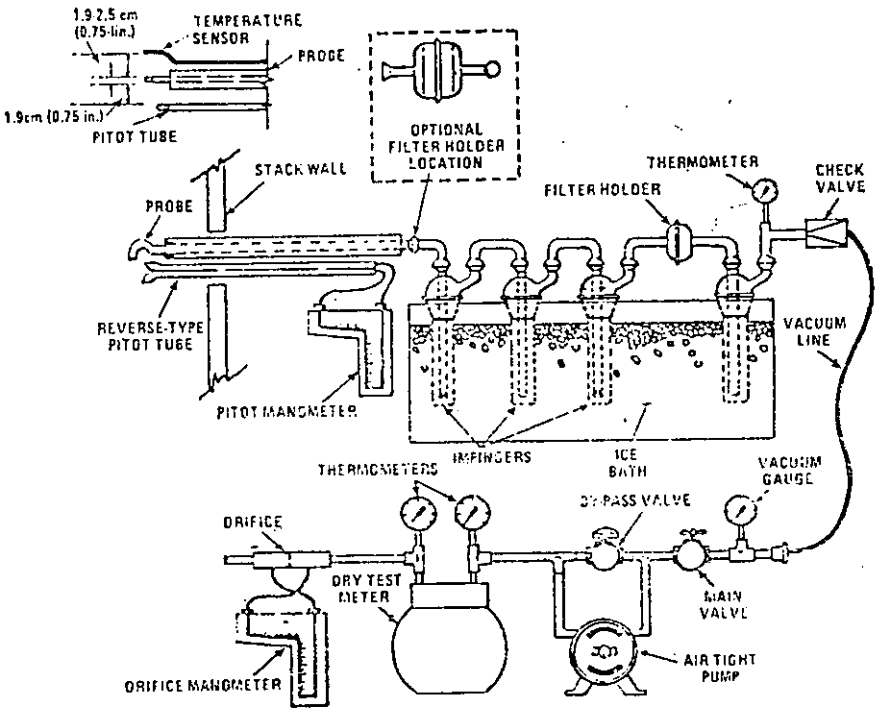

Figure 13A-1. Fluoride sampling train.

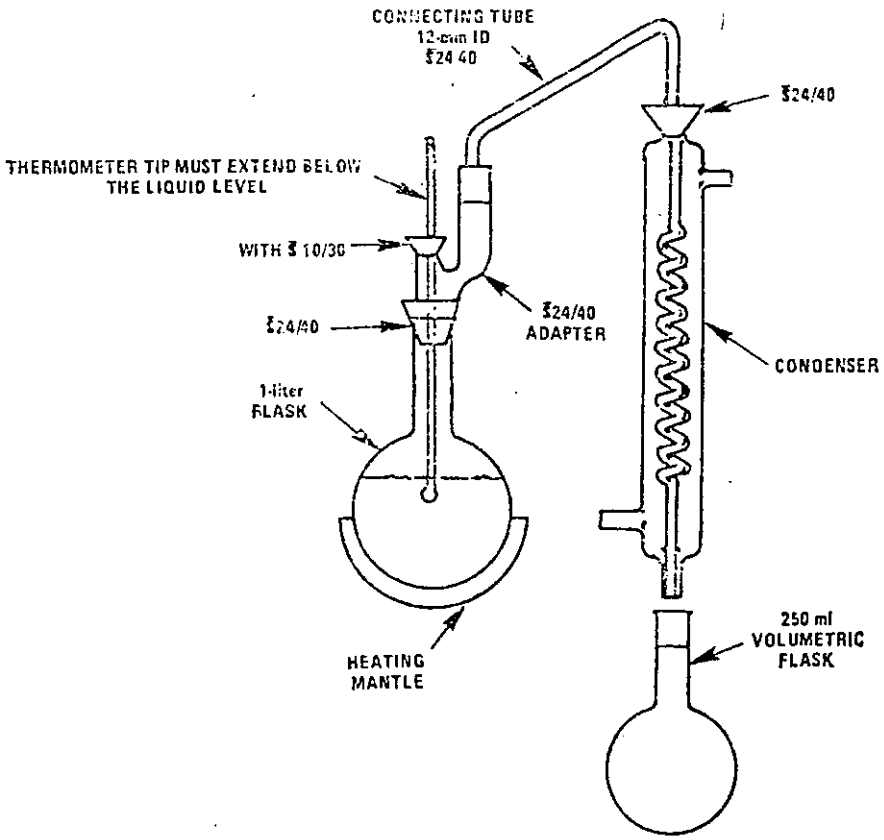

Figure 13A-2. Fluoride Distillation Apparatus

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time. If the stirrer generates enough heat to change solution temperature, place a piece of insulating material such as cork between the stirrer and the beaker. Dilute samples (below 10^{-4} M fluoride ion content) should be held in polyethylene or polypropylene beakers during measurement.

Insert the fluoride and reference electrodes into the solution. When a steady millivolt reading is obtained, record it. This may take several minutes. Determine concentration from the calibration curve. Between electrode measurements, soak the fluoride sensing electrode in distilled water for 30 seconds and then remove and blot dry.

8. *Calibration.*

Maintain a laboratory log of all calibrations.

8.1 Sampling Train.

8.1.1 Probe nozzle—Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make 3 separate measurements using different diameters each time and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.).

When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use.

Each nozzle shall be permanently and uniquely identified.

8.1.2 Pitot tube—The pitot tube shall be calibrated according to the procedure outlined in Method 2.

8.1.3 Dry gas meter and orifice meter. Both meters shall be calibrated according to the procedure outlined in APTD-0576. When diaphragm pumps with by-pass valves are used, check for proper metering system design by calibrating the dry gas meter at an additional flow rate of 0.0057 m³/min. (0.2 cfm) with the by-pass valve fully opened and then with it fully closed. If there is more than ±2 percent difference in flow rates when compared to the fully closed position of the by-pass valve, the system is not designed properly and must be corrected.

8.1.4 Probe heater calibration—The probe heating system shall be calibrated according to the procedure contained in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

8.1.5 Temperature gauges—Calibrate dial and liquid filled bulb thermometers against mercury-in-glass thermometers. Thermocouples need not be calibrated. For other devices, check with the Administrator.

8.2 Analytical Apparatus.

8.2.1 Fluoride Electrode—Prepare fluoride standardizing solutions by serial dilution of the 0.1 M fluoride standard solution. Pipet 10 ml of 0.1 M NaF into a 100 ml volumetric flask and make up to the mark with distilled water for a 10^{-2} M standard solution. Use 10 ml of 10^{-2} M solution to make a 10^{-3} M solution in the same manner. Repeat 10^{-4} and 10^{-5} M solutions.

Pipet 50 ml of each standard into a separate beaker. Add 50 ml of TISAB to each beaker. Place the electrode in the most dilute standard solution. When a steady millivolt reading is obtained, plot the value on the linear axis of semi-log graph paper versus concentration on the log axis. Plot the nominal value for concentration of the standard on the log axis, e.g., when 50 ml of 10^{-3} M standard is diluted with 50 ml TISAB, the concentration is still designated "10^{-3} M".

Between measurements soak the fluoride sensing electrode in distilled water for 30 seconds, and then remove and blot dry. Analyze the standards going from dilute to concentrated standards. A straight-line calibration curve will be obtained, with nominal concentrations of 10^{-2}, 10^{-3}, 10^{-4}, 10^{-5} [illegible] of 10^{-2}, 10^{-3}, 10^{-4}, 10^{-5}, 10^{-6} concentrations of 10^{-3}, 10^{-4}, 10^{-5}, 10^{-6}, 10^{-7} fluoride molarity on the log axis plotted versus electrode potential (in millivolts) on the linear scale.

Calibrate the fluoride electrode daily, and check it hourly. Prepare fresh fluoride standardizing solutions daily of 10^{-2} M or less. Store fluoride standardizing solutions in polyethylene or polypropylene containers. (Note: Certain specific ion meters have been designed specifically for fluoride electrode use and give a direct readout of fluoride ion concentration. These meters may be used in lieu of calibration curves for fluoride measurements over narrow concentration ranges. Calibrate the meter according to manufacturer's instructions.)

9. *Calculations.*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

9.1 Nomenclature.

A_n = Cross sectional area of nozzle, m² (ft²).

A_t = Aliquot of total sample added to still, ml.

B_{ws} = Water vapor in the gas stream, proportion by volume.

C_s = Concentration of fluoride in stack gas, mg/m³, corrected to standard conditions of 20° C, 760 mm Hg (68° F, 29.92 in. Hg) on dry basis.

F_t = Total weight of fluoride in sample, mg.

I = Percent of isokinetic sampling.

M = Concentration of fluoride from calibration curve, molarity.

m_n = Total amount of particulate matter collected, mg.

M_w = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole).

m_a = Mass of residue of acetone after evaporation, mg.

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 mm Hg-m³/°K-g-mole (21.83 in. Hg-ft³/°R-lb-mole).

T_m = Absolute average dry gas meter temperature (see fig. 13A-3), °K (°R).

T_s = Absolute average stack gas temperature (see fig. 13A-3), °K (°R).

T_{std} = Standard absolute temperature, 293° K (528° R).

V_a = Volume of acetone blank, ml.

V_{aw} = Volume of acetone used in wash, ml.

V_d = Volume of distillate collected, ml.

V_{if} = Total volume of liquid collected in impingers and silica gel, ml. Volume of water in silica gel equals silica gel weight increase in grams times 1 ml/gram. Volume of liquid collected in impinger equals final volume minus initial volume.

V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf).

$V_{w(std)}$ = Volume of water vapor in the gas sample corrected to standard conditions, scm (scf).

V_t = Total volume of sample, ml.

v_s = Stack gas velocity, calculated by Method 2, Equation 2-7 using data obtained from Method 5, m/sec (ft/sec).

W_a = Weight of residue in acetone wash, mg.

ΔH = Average pressure differential across the orifice (see fig. 13A-3), meter, mm H₂O (in. H₂O).

ρ_a = Density of acetone, mg/ml (see label on bottle).

ρ_w = Density of water, 1 g/ml (0.00220 lb/ml).

θ = Total sampling time, min.

13.6 = Specific gravity of mercury.

60 = Sec/min.

100 = Conversion to percent.

9.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 13A-2 of Method 13A).

9.3 Dry gas volume. Use Section 9.2 of Method 13A.

9.4 Volume of Water Vapor. Use Section 9.4 of Method 13A.

9.5 Moisture Content. Use Section 9.5 of Method 13A.

9.6 Concentration.

9.6.1 Calculate the amount of fluoride in the sample according to equation 13B-1.

$$F_t = K - (V_a) \frac{V_t}{A_t} (M)$$

where:

K = 19 mg/ml.

9.6.2 Concentration of fluoride in stack gas. Use Section 9.6.2 of Method 13A.

9.7 Isokinetic variation. Use Section 9.7 of Method 13A.

9.8 Acceptable results. Use Section 9.8 of Method 13A.

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(Sections 111 and 114 of the Clean Air Act, as amended by section 4(a) of Pub. L. 91-604, 84 Stat. 1678 (42 U.S.C. 1857 c-6, c-9))

[40 FR 33152, August 6, 1975]

METHOD 14—DETERMINATION OF FLUORIDE EMISSIONS FROM POTROOM ROOF MONITORS OF PRIMARY ALUMINUM PLANTS

1. *Principle and applicability.*

1.1 *Principle.* Gaseous and particulate fluoride roof monitor emissions are drawn into a permanent sampling manifold through several large nozzles. The sample is transported from the sampling manifold to ground level through a duct. The gas in the duct is sampled using Method 13A or 13B—DETERMINATION OF TOTAL FLUORIDE EMISSIONS FROM STATIONARY SOURCES. Effluent velocity and volumetric flow rate are determined with anemometers permanently located in the roof monitor.

1.2 *Applicability.* This method is applicable for the determination of fluoride emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards.

2. *Apparatus.*

2.1 *Anemometers.* Vane or propeller anemometers with a velocity measuring threshold as low as 15 meters per minute and a range up to at least 600 meters per minute. Each anemometer shall generate an electrical signal which can be calibrated to the velocity measured by the anemometer. Anemometer shall be rated by manufacturer for corrosive atmospheres.

DETERMINATION OF HYDROGEN CHLORIDE AND CHLORINE

DETERMINATION OF HYDROGEN CHLORIDE AND CHLORINE IN STACK GAS

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April, 1968

DETERMINATION OF HYDROGEN CHLORIDE AND CHLORINE IN STACK GAS

INTRODUCTION

This method is intended for the determination of hydrogen chloride and chlorine in stack gases, as would be encountered in emissions from hydrochloric acid manufacture. Hydrogen chloride is collected from the gas stream in midget impingers or a grab sampling flask containing sodium hydroxide and determined by the Volhard Titration.[1] If chlorine is suspected to be present in the stack effluent, both gases are collected in the same manner, except that a known quantity of alkaline arsenite absorbing reagent is substituted for the sodium hydroxide. Chlorine is reduced to chloride by arsenite, and is measured by titration of the unconsumed arsenite with standard iodine solution. Total chloride content of the sample is determined by the Volhard Titration. Hydrogen chloride concentration is calculated by subtraction of the chlorine concentration from total chloride concentration. This method is applicable to the determination of hydrogen chloride and chlorine in the concentration range from 10 ppm to percentage quantities.

REAGENTS

All chemicals must be ACS analytical reagent grade.

Water

Deionized-distilled water.

Absorbing Reagent

A. Sodium hydroxide (1 N) - Dissolve 40 g of sodium hydroxide in water and dilute to 1 liter. Use this reagent when the stack gas concentration of HCl is less than 1000 ppm.

B. Sodium hydroxide (2.5 N) - Dissolve 100 g of sodium hydroxide in water and dilute to 1 liter. Use this reagent when the stack gas concentration of HCl is greater than 1000 ppm.

C. Alkaline arsenite (1 N NaOH - 0.1 N $NaAsO_2$) - Dissolve 40 g of NaOH and 6.5 g of sodium arsenite ($NaAsO_2$) in water and dilute to 1 liter in a volumetric flask. Use this reagent when the stack gas concentration of HCl and Cl_2 is less than 1000 ppm.

D. Alkaline arsenite (2.5 N NaOH - 0.5 N $NaAsO_2$) - Dissolve 100 g of NaOH and 32.5 g of $NaAsO_2$ in water and dilute to 1 liter in a volumetric flask. Use this reagent when the stack gas concentration of HCl and Cl_2 is greater than 1000 ppm.

Ferric Indicator

Dissolve 28.0 g of ferric ammonium sulfate $\left[FeNH_4(SO_4)_2 \cdot 12\ H_2O\right]$ in 70 ml of hot water. Cool, filter, add 10 ml of concentrated nitric acid (HNO_3), and dilute to 100 ml in a volumetric flask.

Nitric Acid (8 N)

Prepare NO_X free nitric acid by adding 100 ml of HNO_3 to 100 ml

of water and boiling in a conical flask until the solution is colorless. Store in a glass reagent bottle.

Nitrobenzene

Reagent grade.

Sodium Chloride (Primary Standards)

A. NaCl (0.1 N) - Dissolve 5.846 g of dried sodium chloride (NaCl) in water and dilute to 1 liter in a volumetric flask.

B. NaCl (0.01 N) - Dissolve 0.5846 g of dried NaCl in water and dilute to 1 liter in a volumetric flask, or dilute 100 ml of 0.1 N NaCl to 1 liter.

Ammonium Thiocyanate

A. NH_4CNS (0.1 N) - Dissolve 8 g of NH_4CNS in water and dilute to 1 liter in a volumetric flask.

B. NH_4CNS (0.01 N) - Dilute 100 ml of 0.1 N NH_4CNS to 1 liter in a volumetric flask, or dissolve 0.8 g NH_4CNS in 1 liter of distilled water.

Silver Nitrate

A. $AgNO_3$ (0.1 N) - Dissolve 17.0 g of silver nitrate ($AgNO_3$) in water and dilute to 1 liter in a volumetric flask. Transfer to an amber reagent bottle. Standardize this solution against standard 0.1 N NaCl solution, according to the Volhard Titration.[1]

B. $AgNO_3$ (0.01 N) - Dissolve 1.7 g of $AgNO_3$ in water and dilute to 1 liter in a volumetric flask. Transfer to an amber reagent bottle. Standardize this solution against standard 0.01 N NaCl solution, according to the Volhard Titration.

Starch solution (iodine indicator), 1.0%

Make a thin paste of 1 g of soluble starch in cold water and pour into 100 ml of boiling water while stirring. Boil for a few minutes. Store in a glass stoppered bottle.

Standard iodine solution (0.1 N)

Dissolve 12.69 g of resublimed iodine (I_2) in 25 ml of a solution containing 15 g of iodate-free potassium iodide (KI); dilute to 1 liter in a volumetric flask. Standardize this solution against a standard thiosulfate solution using starch as an indicator. This solution should be stored in an amber reagent bottle and refrigerated when not in use.

APPARATUS

Absorbers

A. Midget impingers - An all glass midget impinger capable of removing HCl and Cl_2 from an air sample by using 15 ml of absorbing reagent. This device should be used when sampling stack gas containing less than 0.1 percent HCl or Cl_2.

B. Grab sampling flask - A 2-liter glass bottle equipped with Teflon stopcocks on either end. This device should be used when sampling percentage quantities of HCl or Cl_2. Absorbing

reagent is added to the grab sample flask after the sample has been collected.

Dispenser (absorbing reagent) - One-hundred milliliter round-bottom flask, modified with a Teflon stopcock and ball joint extension (see Figure 2). Use to add absorbing reagent to grab sampling bottle after the sample has been collected.

Buret (50 ml)

Erlenmeyer flasks (250 ml)

ANALYTICAL PROCEDURE

Collection of samples

A. Midget impingers: The apparatus for sampling of HCl and Cl_2 consists of a probe (1), impingers (5, 6, 7, 8), pump (12), rotameter (14), and dry gas test meter (16). (Figure 1.)

The probe (1) is 18" long and made from medium wall pyrex glass. The first 6 inches is 5/8" OD tube and the last 12 inches is 1/4" OD tube. Nubs are impressed into the larger tube just before the contraction to act as a stop for glass wool which is placed in the end of the probe to act as a filter. The 1/4" end has a male 12/5 ball joint on the end. The whole glass tube is wrapped with 7 feet of 26 gauge nichrome heating wire (2). A variable transformer (3) connected to the wire controls the voltage and hence the temperature of probe. The wire is covered with fiberglass insulation tape and placed in a stainless steel probe sheath.

A three-way valve (4) with ball joint connections to the probe and first impinger allows the ice bath (9) (a plastic bucket) and impinger system to be sealed off and removed after a run is completed. This valve is also used to purge the probe prior to sampling. Due to the sensitivity of the analytical method, only a small sample is required; therefore, four midget impingers (5, 6, 7) contain 15 ml of the absorbing reagent. The fourth impinger (8) is left dry to collect any entrained liquid. This impinger is attached to the umbilical cord.

The umbilical cord (10) leading from the collection device to the pump and metering system consists of a 1/4" OD - 1/8" ID polyethylene sample line, and an electrical circuit for the probe. The sample line is first connected to a drying tube (11) containing silica gel, which removes moisture from the sample gas. From the drying tube, the gas passes through a diaphragm pump (12) to a gross flow control rotameter (14), where the sampling rate is maintained at 0.1 CFM with a needle valve (13). The sampled gas then passes through a 0.1 CFM per revolution dry gas test meter (16), where the sample volume is read with an accuracy of ± 1 percent. A thermometer (15) placed on the inlet side of the meter measures the temperature of the gas in the meter. This temperature reading is later used to correct the sample volume to standard conditions.

The equipment described above is leak tested prior to sampling by plugging the end of the probe and then letting the pump

create a vacuum on the system. If the dry test meter continues to revolve, the system has a leak; if the meter does not revolve, then the system is tight at operating vacuum pressures.

When the leak test has been completed, the probe should be heated to the desired operating temperature (200 to 250°F) before sampling begins. To sample, simply turn on the pump and adjust the rotameter via the needle valve to the desired setting. This setting should be checked at 10 to 15 minute intervals during the test. Generally, a sampling interval of 30 to 60 minutes is used; however, this may vary with the concentration of HCl present in the source effluent. At the end of the test turn off pump and disconnect the sample collection unit.

Cleanup procedure for the midget impinger train consists of emptying the four impingers into a sample bottle and then rinsing each one twice with distilled water into the sample bottle.

B. Grab sampling: The apparatus used for purged flask gas sampling consists of an accurately calibrated 2-liter glass bottle with Teflon stopcocks on either end. The flask, with both stopcocks in the open position, is placed in the sampling line and stack gas (under positive pressure) is allowed to flow through the flask. (A purge volume 10 times

the volume of the flask should be used.) After the flask has been purged, both valves are shut off. The flask is removed from the sampling line and one valve opened to the atmosphere to allow the flask to come to atmospheric pressure.

Pipet 25 ml of absorbing reagent into the modified buret and connect it to the ball joint on one side of the flask. Absorbing reagent is allowed to flow into the flask by opening the stopcocks. The reaction of HCl and/or Cl_2 with the appropriate absorbing reagent creates a negative pressure in the flask and pulls more reagent into the flask. After completion of the reaction, wash all the absorbing reagent into the flask with a small quantity of water. This step is important only when alkaline arsenite reagent is used. Remove the buret and shake the flask for several minutes to insure complete absorption of the gases.

Quantitatively transfer the contents of the grab sampling bottle to a sample container, such as a polyethylene bottle, with deionized-distilled water.

Sample Preparation

Measure the actual volume of the liquid sample or adjust to a known volume using a graduated cylinder or volumetric flask.

PROCEDURE A. Analysis of Hydrogen Chloride

Pipet an aliquot of the sample into a 250 ml Erlenmeyer flask. Add 25 ml of water, 5 ml of nitric acid and swirl to mix. Add 0.1 N

or 0.01 N silver nitrate, depending on chloride content, from a buret until it is in excess. Near the equivalence point, the silver chloride precipitate will coagulate. When coagulation occurs, add an additional 5 ml of silver nitrate. Add 3 ml of nitrobenzene, 2 ml of ferric indicator, and back-titrate with 0.1 N or 0.01 N ammonium thiocyanate until the first appearance of the reddish-brown $Fe(CNS)_6^{-3}$ complex. A blank determination for chloride in the absorbing reagent should be run simultaneously and subtracted from the sample results. From the titer of NH_4CNS solution, as determined previously by titration against standard $AgNO_3$ solution using ferric alum as an indicator, calculate the net volume of $AgNO_3$ required for precipitation of the chloride.

CALCULATIONS

Compute the number of milligrams of HCl present in the sample by the following equation:

$$HCl, mg = \text{net ml } AgNO_3 \times T \times F$$

T = hydrogen chloride equivalent of standard $AgNO_3$

(T = 3.65 mg HCl/ml for 0.1 N $AgNO_3$)

(T = 0.365 mg HCl/ml for 0.01 N $AgNO_3$)

$$F = \frac{\text{sample volume, ml}}{\text{aliquot volume, ml}}$$

Convert the volume of air sampled to the volume at standard conditions of 70°F and 29.92 in. Hg.

$$V_S = V \times \frac{P}{29.92} \times \frac{530^\circ R}{(t + 460^\circ R)}$$

V = volume of gas sampled, cu. ft.

P = barometric pressure, in. Hg

t = temperature of gas sampled, ^{o}R

Determine the concentration of HCl in the air sample by the following formula:

$$\text{ppm HCl} = \frac{(662)\ (C)}{V_S\ (28.32)}$$

662 = ul/mg of HCl at $70^{o}F$ and 29.92 in. Hg

C = concentration of HCl, mg

V_S = volume of gas sampled in cu. ft. at $70^{o}F$ and 29.92 in. Hg

28.32 = liters per cu. ft.

PROCEDURE B. Analysis of Hydrogen Chloride in the Presence of Chlorine

Pipet an aliquot of the sample into a 250-ml Erlenmeyer flask and proceed with the Volhard Titration for total chlorides as described under Procedure A. A blank determination for chloride in the absorbing reagent (alkaline arsenite reagent) should be run simultaneously and subtracted from the sample results.

Pipet another aliquot of the sample into a 250-ml Erlenmeyer flask. Add a few drops of phenolphthalein indicator, neutralize carefully with concentrated hydrochloric acid, and cool. Add sufficient solid sodium bicarbonate ($NaHCO_3$) to neutralize any excess hydrochloric acid, then add 2-3 g in excess. Add 2 ml of starch indicator and titrate with 0.1 N iodine solution to the blue endpoint. For the reagent blank, determine the number of ml of 0.1 N I_2 required to titrate 25 ml of alkaline-arsenite absorbing reagent, as described above.

CALCULATIONS

Determine the number of milliliters of 0.1 N I_2 required to titrate the entire sample by the following formula:

Sample (ml 0.1 N I_2) = (ml of 0.1 N I_2 for aliquot) x F

$$F = \frac{\text{volume of sample, ml}}{\text{volume of aliquot, ml}}$$

Cl_2, mg = Blank (ml 0.1 N I_2) - Sample (ml 0.1 N I_2) x 3.546

3.546 = chlorine equivalent of 0.1 N I_2, mg

Convert the volume of gas sampled at standard conditions of 70°F, 29.92 in. Hg, using the formula in Procedure A. Calculate the concentration of Cl_2 in the sample using the following formula:

$$\text{ppm } Cl_2 \text{ by volume} = \frac{(340)\ (C)}{V_S}$$

340 = µl/mg of Cl_2 at 70°F and 29.92 in. Hg

C = concentration of Cl_2, mg

V_S = volume of gas sampled at standard conditions, liters

Determine the number of milligrams of HCl present in the sample by subtracting the number of milligrams of Cl_2 present, as determined by the iodine titration, from the total number of milligrams of chloride present as determined by the Volhard Titration. Calculate the concentration of HCl in parts per million using the formula in Procedure A.

DISCUSSION OF PROCEDURE

The estimated error for the combined sampling and analytical procedure is $\pm$ 10 percent. The precision of the analytical methods is $\pm$ 2 percent on standard samples containing NaCl and $NaAsO_2$.

The usual volumetric errors are encountered with the Volhard Titration. Premature endpoints may occur, if the NH_4CNS is not added dropwise near the equivalence point and the solution shaken before the next addition. Nitrobenzene is used to effectively remove AgCl by forming an oily coating on the precipitate and preventing reaction with the thiocyanate.[2] Substances which form insoluble silver salts and bivalent mercury, which forms a stable complex ion with the thiocyanate, interfere in the analysis and must be absent from the sample. Titrations should be made at temperatures below 25°C, as is customary in other titrations with thiocyanate.[3]

The chief source of error in the iodine titration of arsenite is failure to use sufficient bicarbonate to neutralize all the excess acid. If insufficient bicarbonate is added, serious errors are incurred due to a fading endpoint. Reducing agents, such as sulfur dioxide, and oxidizing agents, such as iodine, nitrogen dioxide, and ozone, if present in the stack gas sample, would interfere with the iodine titration and yield high results.

REFERENCES

1. Standard Methods of Chemical Analysis, D. Van Nostrand Company, Inc., 6th Ed., Vol. 1, 329-330 (1962).

2. Caldwell, J. R., and Mayer, H. V., Ind. Eng. Chem., Anal. Ed., 7, 38 (1935).

3. Jacobs, M. B., The Chemical Analysis of Air Pollutants, Interscience Publishers, Inc., 10, 199 (1960).

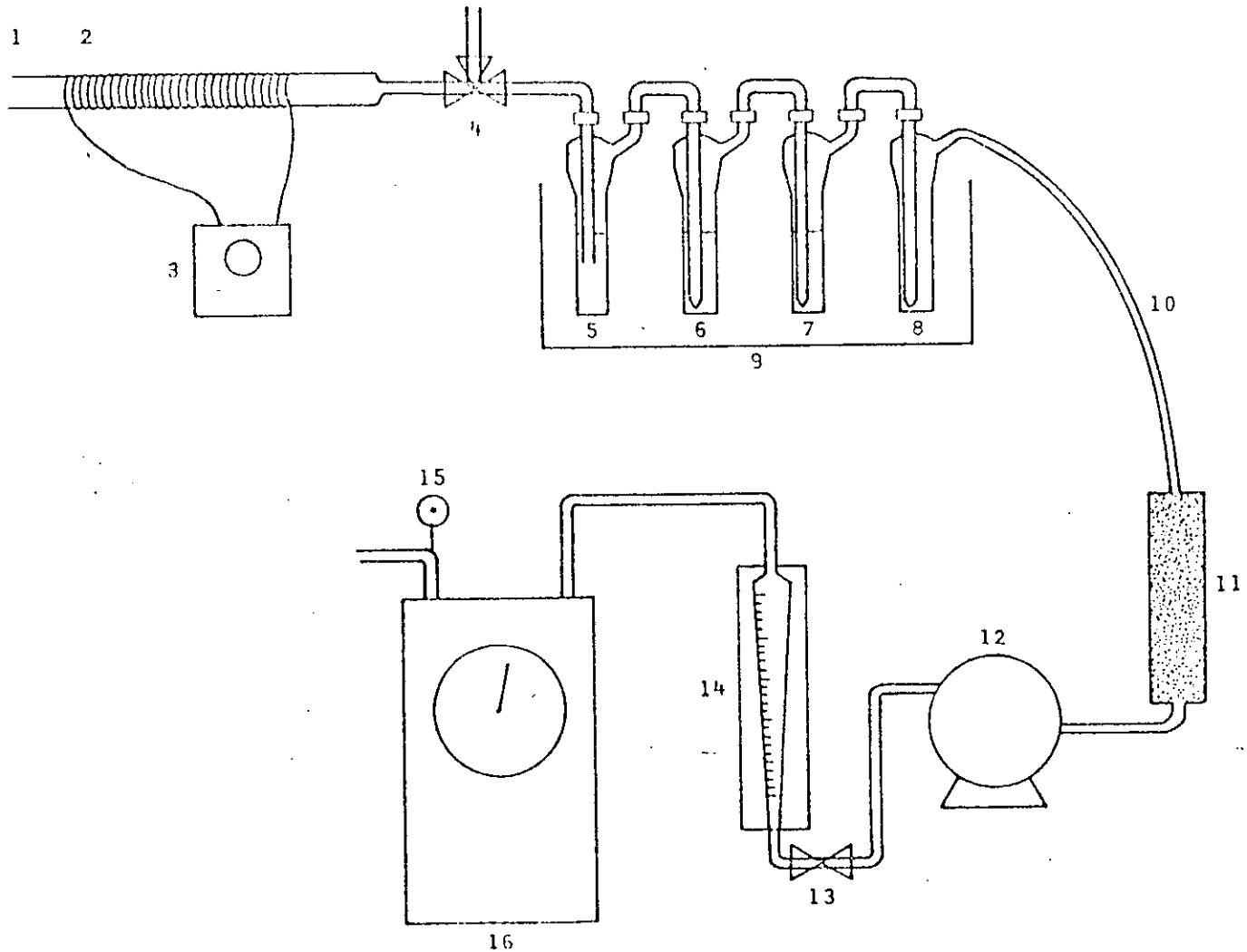

Figure 1. Schematic of sampling apparatus.

APPENDIX B

PROCESS DATA

ROCHESTER SMELTING
AND REFINING CO., INC.

26 SHERER STREET · ROCHESTER, N. Y. 14611

MAILING ADDRESS: P. O. BOX 547 · ROCHESTER, N.Y. 14602

SINCE 1895

TELEPHONE 716/328-4740

February 3, 1977

MR. ROBERT A. BAKER, P. E.
ENVIRONMENTAL ENGINEER
ENVIRONMENTAL SCIENCE AND ENGINEERING, INC.
P. O. Box 13454
Gainesville, Florida 32604

Dear Mr. Baker:

Enclosed herewith is the information that you requested.

If you have any more questions or need any further information, please do not hesitate to call.

Cordially yours,

STEVEN R. SHENCUP

SRS:rd
Enc.

ALL AGREEMENTS AND SALES ARE CONTINGENT UPON STRIKES, ACCIDENTS, DELAYS OF CARRIERS AND OTHER DELAYS UNAVOIDABLE OR BEYOND OUR CONTROL. ALL PRICES SUBJECT TO ACCEPTANCE BY RETURN MAIL. ALL STENOGRAPHICAL AND CLERICAL ERRORS SUBJECT TO CORRECTION. ALL AGREEMENTS MADE BY AGENTS ARE SUBJECT TO OUR APPROVAL

FURNACE # 4

DATE	ALUMINUM SCRAP INPUT	ALSO ADDED	SKIMMED OFF	FINISHED PRODUC
10/18/76	7775# 380 ALUMINUM INGOT	6000# FLUX	7341#	46,780#
	5435# CAST ALUMINUM (DIRTY)	3550# SILICON		
	3750# ALUMINUM BUTTS	15 BAGS ALUMINUM FLUORIDE		
	3111# PAINTED ALUMINUM SIDING (DIRTY)			
	5121# #3-S ALUMINUM SPINNINGS			
	31496# ALUMINUM CHIPS (CLEAN)			
	600# ALUMINUM COPPER RAD. FINS			
10/19/76	4256# ALUMINUM BUTTS	4100# SILICON	8842#	54,084#
	988# CASTINGS	15 BAGS ALUMINUM FLUORIDE		
	11254# CAST ALUMINUM (DIRTY)			
	9946# #3-S ALUMINUM SPINNINGS	6000# FLUX		
	25859# ALUMINUM CHIPS (CLEAN)			
	1480# ALUMINUM COPPER RAD. FINS			
10/20/76	12606# CLEAN CASTINGS	2750# SILICON		44,268#
	16106# #3-S ALUMINUM SPINNINGS			
	5842# PAINTED ALUMINUM SIDING (DIRTY)			
	15160# ALUMINUM CHIPS (CLEAN)			
	1000# COPPER			
10/21/76	8086# ALUMINUM CAST (DIRTY)	6000# FLUX	9718#	51,784#
	9996# ALUMINUM BUTTS	2800# SILICON		
	6334# PAINTED ALUMINUM SIDING (DIRTY)			
	25465# ALUMINUM CHIPS (CLEAN	20 BAGS FLUORIDE		
	700# ALUMINUM COPPER RAD. FINS			
	450# ZAMAK DROSS			
10/22/76	8199# ALUMINUM CAST (DIRTY)	6000# FLUX	5531#	56,182#
	12230# ALUMINUM BUTTS	3250# SILICON		
	5136# 3-S ALUMINUM SPINNINGS	19 BAGS FLUORIDE		
	3703# 3-S ALUMINUM			
	7254# PAINTED ALUMINUM SIDING (DIRTY)			
	17774# ALUMINUM CHIPS			
	676# Z-5 HARDENER			
	550# COPPER			

ROCHESTER SMELTING
AND REFINING CO., INC.
26 SHERER STREET · ROCHESTER, N. Y. 14611
MAILING ADDRESS: P. O. BOX 547 · ROCHESTER, N.Y. 14602

SINCE 1895
TELEPHONE 716/328-4740

October 25, 1976

Mr. Robert A. Baker
ENVIRONMENTAL SCIENCE AND ENGINEERING, INC.
P. O. Box 13454
Gainesville, Florida 32604

Dear Mr. Baker:

Enclosed is the FURNACE information that you requested.

If you have any further questions regarding this, please do not hesitate to call me.

Yours very truly,

STEVEN R. SHENCUP

SRS:rd
Enc.

ALL AGREEMENTS AND SALES ARE CONTINGENT UPON STRIKES, ACCIDENTS, DELAYS OF CARRIERS AND OTHER DELAYS UNAVOIDABLE OR BEYOND OUR CONTROL. ALL PRICES SUBJECT TO ACCEPTANCE BY RETURN MAIL. ALL STENOGRAPHICAL AND CLERICAL ERRORS SUBJECT TO CORRECTION. ALL AGREEMENTS MADE BY AGENTS ARE SUBJECT TO OUR APPROVAL.

NO. 1 FURNACE

DATE	ALUMINUM SCRAP INPUT	ALSO ADDED	SKIMMED OFF	FINISHED PRODUCT
10/18/76	3427# Sheet Aluminum (Dirty)	6000# Flux		
	6166# Loose Aluminum Sheets & (Sheet was Dirty) Clips	10 Bags Alum Fluoride (for testing purpose)		
	7555# Big Alum Bricquettes			
	14818# Loose Sheet & Brick Alum (Sheet was Dirty)			
	5463# Aluminum Rolls (Clean		10,300#	38,554#
10/19/76	3476# Sheet Aluminum (Dirty)	265# Iron		
	5402# Big Aluminum Bricquettes	6000# Flux		
	5488# Loose Alum Sheet & Clips (Sheet was Dirty)	10 Bags Alum Fluoride (for testing purpose)		
	2158# Aluminum Sheet (Dirty)			
	13118# Sheet Alum in Bales (Dirty)			
	7611# Clean bricks & Sheet Aluminum (Sheet was Dirty)			
	3535# Clean Alum Spinnings in Bales			
	6184# Large Aluminum Bricks		5,405#	39,374#
10/20/76	5564# Contaminated Clips	90# Iron		
	720# Clips	6000# Flux		
	11074# Loose Aluminum Sheets (Dirty)			
	6247# Bales of Sheet Aluminum (Dirty)			
	8602# Large Aluminum Bricks & 8718# Aluminum Rolls (Clean)			
	1427# Aluminum Wire		3,274#	39,948#
10/21/76	15352# Loose Sheet Aluminum (Dirty)	6000# Flux		
	7820# Large Aluminum Bricks			
	4694# Aluminum Chips			
	11236# Aluminum Rolls			
	3528# Aluminum Spinnings		9,090#	40,024#
10/22/76	4846# Aluminum Clippings	156# Iron		
	13650# Loose Aluminum Sheets (Dirty)	6000# Flux		
	18044# Bricks			
	2277# Aluminum Sheet in Bales (Dirty)			
	3855# Aluminum Can Stock			
	921# Aluminum Litho Sheet		7,274#	29,716#

NO. 3 FURNACE

DATE	ALUMINUM SCRAP INPUT	ALSO ADDED	SKIMMED OFF	FINISHED PRODUCT
10/18/76	6018# Cast Aluminum (Dirty)	800# Silicon		
	4312# 3S Clippings (Clean)	8 Bags Alum Fluoride		
	5084# Aluminum Chips (Clean)	4000# Flux	1736#	16,650#
	300# Alum/Copper Rad.Fins			
10/19/76	6110# Cast Aluminum (Dirty)	10 Bags Fluoride		
	5872# Painted Aluminum Siding (Dirty)	50# Magnesium		
	3043# Mxd.Aluminum Clippings(Clean)	4000# Flux		
	500# Alum/Copper Rad. Fins	1700# Silicon		
		100# Copper	2330#	13,440#
NOTE:	On our day shift the only thing we added was the Magnesium. Everything else was already in the furnace from the midnight shift.			
10/20/76	4086# Cast Aluminum (Dirty)	4000# Flux		
	3890# Painted Alum Siding (Dirty)	1420# Silicon		
	4902# Mxd. Alum Clippings (Clean)	9 Bags Alum Fluoride		
	300# Alum/Copper Rad. Fins(Clean)	40# Magnesium	5394#	15,740#
10/21/76	2612# Aluminum Butts	4000# Flux		
	6516# Cast Aluminum (Dirty)	800# Silicon		
	6106# Aluminum Chips (Clean	10 Bags Alum Fluoride		
	1956# Mxd. Aluminum Clippings(Clean)		5599#	14,280#
10/22/76	5010# Cast Aluminum (Dirty)	3870# #384 Alum Ingot		
	1840# Aluminum Butts	4000# Flux		
	3140# Mxd. Aluminum Clippings (Clean)			
		770# Silicon		
		100# Copper		
		14 Bags Alum Fluoride		
		25# Magnesium	2294#	19,970#

Certificate of Analysis

Rochester Smelting & Refining Co., Inc.

Order No. .....

Lab No. 977 Heat No. 1160F #2

Sample Of 384alloy Dominos

Date 10-22-76

Ship To .....

Copper	3.40	%
Tin	.07	%
Lead	.11	%
Antimony		%
Iron	1.10	%
Nickel	.07	%
Zinc	.71	%
Silicon	11.75	%
Manganese	.28	%
Magnesium	.12	%
Aluminum	Bal	%
Titanium	.04	%
Cadmium		%
Sulphur		%
Phosphorous		%
Chromium	.03	%

Metallurgist

Certificate of Analysis

Rochester Smelting & Refining Co., Inc.

Order No. .....

Lab No. 977 Heat No. 1160F #1

Sample Of 384alloy Dominos

Date 10-22-76

Ship To .....

Copper	3.40	%
Tin	.07	%
Lead	.11	%
Antimony		%
Iron	1.11	%
Nickel	.06	%
Zinc	.90	%
Silicon	11.95	%
Manganese	.28	%
Magnesium	.19	%
Aluminum	Bal	%
Titanium	.04	%
Cadmium		%
Sulphur		%
Phosphorous		%
Chromium	.03	%

Metallurgist

Certificate of Analysis

Rochester Smelting & Refining Co., Inc.

Order No. .....

Lab No. 962 Heat No. 234P2

Sample Of Grade 4

Date 10-18-76

Ship To .....

Copper	.126	%
Tin	<.01	%
Lead	.105	%
Antimony		%
Iron	1.47	%
Nickel	.102	%
Zinc	.104	%
Silicon	.144	%
Manganese	.141	%
Magnesium	.154	%
Aluminum	96.66	%
Titanium	.102	%
Cadmium		%
Sulphur		%
Phosphorous		%
Chromium	.101	%

Metallurgist

#1 10/18/76 Pre Flaoride demagging

Certificate of Analysis

Rochester Smelting & Refining Co., Inc.

Order No. .....

Lab No. 962 Heat No. 234F

Sample Of Grade 4

Date 10-18-76

Ship To .....

Copper	.138	%
Tin	<.01	%
Lead	.105	%
Antimony		%
Iron	1.69	%
Nickel	.102	%
Zinc	.104	%
Silicon	.147	%
Manganese	.142	%
Magnesium	.118	%
Aluminum	96.64	%
Titanium	.102	%
Cadmium		%
Sulphur		%
Phosphorous		%
Chromium	.108	%

Metallurgist

#2 Fluoride demagging

Certificate of Analysis

Rochester Smelting & Refining Co., Inc.

Order No. .....

Lab No. 970 Heat No. 1165F #1

Sample Of Daily Demag

Date 10-20-76

Ship To .....

Copper	3.25	%
Tin	.05	%
Lead	.09	%
Antimony		%
Iron	1.14	%
Nickel	.05	%
Zinc	.58	%
Silicon	11.80	%
Manganese	.07	%
Magnesium	.17	%
Aluminum	Bal	%
Titanium	.05	%
Cadmium		%
Sulphur		%
Phosphorous		%
Chromium	.01	%

Metallurgist

Certificate of Analysis

Rochester Smelting & Refining Co., Inc.

Order No. .....

Lab No. 970 Heat No. 1165F #2

Sample Of Daily Demag

Date 10-20-76

Ship To .....

Copper	3.25	%
Tin	.05	%
Lead	.09	%
Antimony		%
Iron	1.14	%
Nickel	.05	%
Zinc	.58	%
Silicon	11.80	%
Manganese	.07	%
Magnesium	.14	%
Aluminum	Bal	%
Titanium	.05	%
Cadmium		%
Sulphur		%
Phosphorous		%
Chromium	.01	%

Metallurgist

Certificate of Analysis

Rochester Smelting & Refining Co., Inc.

Order No. .....

Lab No. 970 Heat No. 1165 F #3

Sample Of 374 alloy Dec 10 mg

Date 10-20-76

Ship To .....

Copper	3.25	%
Tin	.05	%
Lead	.09	%
Antimony		%
Iron	1.14	%
Nickel	.05	%
Zinc	.58	%
Silicon	11.70	%
Manganese	.27	%
Magnesium	.09	%
Aluminum	Bal	%
Titanium	.05	%
Cadmium		%
Sulphur		%
Phosphorous		%
Chromium	.01	%

Metallurgist

Certificate of Analysis

Rochester Smelting & Refining Co., Inc.

Order No. #2

Lab No. 966 Heat No. 1164F Drums

Sample Of 387 alloy

Date 10-19-76

Ship To

Copper	3.35	%
Tin	.05	%
Lead	.07	%
Antimony		%
Iron	.97	%
Nickel	.05	%
Zinc	.72	%
Silicon	11.50	%
Manganese	.39	%
Magnesium	.15	%
Aluminum	Rest	%
Titanium	.01	%
Cadmium		%
Sulphur		%
Phosphorous		%
Chromium	.01	%

Metallurgist

Certificate of Analysis

Rochester Smelting & Refining Co., Inc.

Order No.

Lab No. 966 Heat No. 1164F Drums

Sample Of 387 alloy

Date 10-19-76

Ship To

Copper	3.70	%
Tin	.05	%
Lead	.07	%
Antimony		%
Iron	.96	%
Nickel	.05	%
Zinc	.70	%
Silicon	11.90	%
Manganese	.38	%
Magnesium	.23	%
Aluminum	Rest	%
Titanium	.05	%
Cadmium		%
Sulphur		%
Phosphorous		%
Chromium	.01	%

Metallurgist

Sample 3
Chemical Analysis

#3 Sample

Certificate of Analysis

Rochester Smelting & Refining Co., Inc.

Order No. #3

Lab No. 966 Heat No. 1164F

Sample Of 77Y alloy Demag EPA

Date 10-19-76

Ship To

Copper	2.35	%
Tin	.05	%
Lead	.08	%
Antimony		%
Iron	.96	%
Nickel	.05	%
Zinc	.70	%
Silicon	11.65	%
Manganese	.38	%
Magnesium	.09	%
Aluminum	Bal	%
Titanium	.04	%
Cadmium		%
Sulphur		%
Phosphorous		%
Chromium	.01	%

Metallurgist

APPENDIX C

ADDITIONAL INFORMATION

U.S. EPA METHOD 5

COMPUTER PRINT OUT

AND

LABORATORY ANALYSIS

PLANT NAME - IBM LOCATION - ROCHESTER

STACK ID - COMBINED INLET SAMPLING TRAIN - PARTICULATES

		RUN 1	RUN 2	RUN 3	AVG
DATE		10/18/76	10/19/76	10/20/76	
STARTING TIME		1455	1105	1034	
ENDING TIME		1640	1411	1306	
MINUTES		88.	132.	132.	
NUMBER OF POINTS		44.	44.	44.	
PB	(IN HG)	29.93	29.76	29.61	
PS	(IN HG)	29.86	29.69	29.54	
DP		0.84	0.84	0.84	
AS	(SF)	19.63	19.63	19.63	
ASI	(SF)	19.63	19.63	19.63	
DN	(IN)	0.255	0.255	0.255	
AN	(SF)	0.000355	0.000355	0.000355	
TM	(DEG F)	510.5	528.3	524.4	
TS	(DEG F)	546.7	572.6	575.9	565.07
VM	(ACF)	61.812	82.835	89.855	
VMSTD	(SCFD)	63.734	82.658	89.933	
CONDENSATE	(ML)	17.5	25.5	21.5	
CALC % H2O		1.28	1.43	1.11	1.28
% H2O @ SATURATION		4.29	9.43	10.40	8.04
% CO2		0.0	0.0	0.0	0.00
% O2		21.0	21.0	21.0	21.00
% CO		0.0	0.0	0.0	0.00
MWD	(LB/LB-MOLE)	28.84	28.84	28.84	28.84
MWW	(LB/LB-MOLE)	28.70	28.68	28.72	28.70
DELTA H AVG	(IN H2O)	1.816	1.542	1.791	
SQRT DELTA H AVG	(IN H2O)	0.678	0.563	0.622	
VS	(F/S)	38.87	33.15	36.78	36.27
QSI	(ACFM)	45785.	39039.	43317.	42714.
QSII	(ACFMD)	45200.	38479.	42835.	42171.
QS	(SCFMD)	43572.	35217.	38779.	39189.
% ISO		92.04	98.45	97.28	95.92
FILTER NO -		00375	00433	00445	
		0.	0.	0.	
		0.	0.	0.	
		0.	0.	0.	
		0.	0.	0.	
TOTAL FILTER CATCH	(MG)	45.00	164.00	170.60	
TOTAL ASH CATCH	(MG)	16.50	22.70	15.30	
TOTAL CATCH	(MG)	61.50	186.70	185.90	
CONCENTRATION	(GR/ACF)	0.0141	0.0314	0.0285	0.0247
CONCENTRATION	(GR/SCF)	0.0149	0.0348	0.0318	0.0272
EMISSION RATE	(LB/HR)	5.5625	10.5239	10.6052	8.8972

PLANT NAME - IBM LOCATION - ROCHESTER

STACK ID - COMBINED INLET SAMPLING TRAIN - PARTICULATES

		RUN 4	RUN 5	RUN 6	AVG
DATE		10/20/76	10/21/76	10/21/76	
STARTING TIME		1358	1027	1430	
ENDING TIME		1655	1256	1536	
MINUTES		132.	132.	66.	
NUMBER OF POINTS		40.	40.	40.	
PB	(IN HG)	29.61	29.18	29.18	
PS	(IN HG)	29.54	29.11	29.11	
CP		0.84	0.84	0.84	
AS	(SF)	19.63	19.63	19.63	
AS1	(SF)	19.63	19.63	19.63	
PI	(")	0.255	0.255	0.255	
AN	(SF)	0.000355	0.000355	0.000355	
TM	(DEG F)	530.0	531.5	519.6	
TS	(DEG F)	592.7	563.6	553.0	569.90
VM	(ACF)	79.277	61.062	47.618	
VMSTD	(SCFD)	78.427	59.251	47.434	
CONDENSATE	(ML)	25.5	18.0	7.5	
CALC % H2O		1.51	1.01	0.74	1.22
% H2O @ SATURATION		16.47	7.00	5.43	9.74
% CO2		0.0	0.0	0.0	0.00
% O2		21.0	21.0	20.5	20.83
% CO		0.0	0.0	0.0	0.00
MWD	(LB/LB-MOLE)	28.80	28.80	28.82	28.53
MW	(LB/LB-MOLE)	28.68	28.69	28.70	28.70
DELTA H AVG	(IN H2O)	1.380	0.788	2.028	
SQRT DELTA P AVG	(IN H2O)	0.546	0.395	0.415	
VS	(F/S)	32.80	23.31	24.23	26.78
QS	(ACFM)	38629.	27451.	28563.	31541.
QSD	(ACFMD)	35046.	27058.	28332.	31107.
QS	(SCFMD)	33069.	24673.	26303.	28146.
% IKP		98.29	100.78	151.30	116.79
FILTER NO -		00413	00389	00421	
		0.	0.	0.	
		0.	0.	0.	
		0.	0.	0.	
		0.	0.	0.	
TOTAL FILTER CATCH	(MG)	267.80	170.00	90.70	
TOTAL WASH CATCH	(MG)	18.60	16.00	19.70	
TOTAL CATCH	(MG)	286.40	186.00	110.40	
CONCENTRATION	(GR/ACF)	0.0087	0.0036	0.0330	0.0018
CONCENTRATION	(GR/SCF)	0.0562	0.0085	0.0358	0.0465
EMISSION RATE	(LB/HR)	18.1698	10.2858	8.0968	11.5161

[illegible]

LOCATION - ROCHESTER

STACK ID - COMBINED INLET

SAMPLING TRAIN - PARTICULATES

		RUN 7	RUN 8	RUN	AVG
[illegible]		1?/22/76	1?/22/76		
[illegible]		870	13?3		
[illegible]		1058	14?5		
[illegible]		?32.	?32.		
[illegible]		?8.	?8.		
[illegible]	[illegible]	29.20	29.2?		
[illegible]	[illegible]	2?.22	??.??		
[illegible]		[illegible]	[illegible]		
[illegible]	[illegible]	??.43	??.43		
[illegible]	[illegible]	??.43	??.43		
	[illegible]	?.255	?.255		
	[illegible]	?.??0755	[illegible]		
[illegible]	[illegible]	522.5	[illegible]		
[illegible]	[illegible]	578.3	[illegible]		[illegible]
[illegible]	[illegible]	46,?0?	[illegible]		
[illegible]	[illegible]	48,?0?	[illegible]		
[illegible]	[illegible]	[illegible]	[illegible]		
[illegible]		?.52	[illegible]		[illegible]
[illegible]		6.07	[illegible]		[illegible]
[illegible]		[illegible]	[illegible]		[illegible]
[illegible]		2?.5	[illegible]		[illegible]
[illegible]		[illegible]	[illegible]		[illegible]
	[illegible]	26.??	[illegible]		[illegible]
	[illegible]	2?.??	[illegible]		[illegible]
[illegible]	[illegible]	[illegible]	[illegible]		
[illegible]	[illegible]	?.?27	[illegible]		
[illegible]	[illegible]	25.2?	[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]	[illegible]		
[illegible]		[illegible]	[illegible]		
[illegible]	[illegible]	[illegible]	[illegible]		[illegible]
[illegible]	[illegible]	?.?752	[illegible]		[illegible]
[illegible]	[illegible]	??.??5?	[illegible]		[illegible]

PLANT NAME - [illegible]

LOCATION - [illegible]

STACK ID - [illegible]

SAMPLING TRAIN - PARTICULATES

		RUN 1	RUN 2	RUN 3	AVG
DATE		10/18/76	10/19/76	10/20/76	
TESTING TIME		[illegible]	1104	[illegible]	
STARTUP TIME		[illegible]	1030	[illegible]	
MINUTES		[illegible]	[illegible]	144.	
NUMBER OF POINTS		44.	72.	72.	
[illegible]	[illegible]	29.03	29.71	29.61	
[illegible]	[illegible]	29.66	29.77	29.57	
[illegible]		[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	21.21	21.21	21.21	
[illegible]	[illegible]	21.21	21.21	21.21	
[illegible]	[illegible]	21.21	21.21	21.21	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	521.3	521.0	527.4	
[illegible]	[illegible]	530.0	540.8	555.5	[illegible]
[illegible]	[illegible]	38.828	47.273	42.143	
[illegible]	[illegible]	41.027	44.736	41.235	
[illegible]	[illegible]	[illegible]	30.4	10.	
[illegible]		[illegible]	[illegible]	[illegible]	[illegible]
[illegible]		[illegible]	[illegible]	[illegible]	[illegible]
[illegible]		[illegible]	[illegible]	[illegible]	[illegible]
[illegible]		21.4	[illegible]	21.4	[illegible]
[illegible]		[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	20.00	29.[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	21.[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]	[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]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

PLANT NAME - IBM

LOCATION - ROCHESTER

STACK ID - OUTLET

SAMPLING TRAIN - PARTICULATES

		RUN 4	RUN 5	RUN 6	AVG
DATE		10/20/76	10/21/76	[illegible]	
[illegible]		[illegible]	1028	[illegible]	
[illegible]		[illegible]	1302	[illegible]	
[illegible]		144.	[illegible]	72.	
[illegible]		72.	72.	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	29.57	[illegible]	[illegible]	
[illegible]		[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	21.21	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]		[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	527.7	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	15.3	[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]	[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]		47.3	[illegible]	[illegible]	
[illegible]		52.1	[illegible]	[illegible]	
[illegible]	[illegible]	.0123	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	.0136	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	5.3111	[illegible]	[illegible]	[illegible]

PLANT NAME - [illegible]　　　　LOCATION - ROCHESTER

STACK ID - OUTLET　　　　SAMPLING TRAIN - PARTICULATES

		RUN 7	RUN 8	RUN	AVG
DATE		10/22/76	10/22/76		
STARTING TIME		838	[illegible]		
[illegible] TIME		1112	1622		
[illegible]		144.	144.		
[illegible] POINTS		72.	72.		
[illegible]	(IN HG)	29.29	[illegible]		
[illegible]	(IN HG)	29.24	29.25		
[illegible]		0.84	[illegible]		
[illegible]	(SF)	21.21	21.21		
[illegible]	(SF)	21.21	21.21		
[illegible]	[illegible]	0.187	0.187		
[illegible]	(SF)	0.000191	0.000191		
[illegible]	(DEG R)	517.0	515.3		
[illegible]	(DEG R)	560.2	533.2		546.7
[illegible]	(ISF)	52.120	51.510		
[illegible]	[illegible]	52.107	51.037		
[illegible]	[illegible]	15.0	7.6		
[illegible]		1.01	[illegible]		[illegible]
[illegible]		6.77	[illegible]		[illegible]
[illegible]		0.0	[illegible]		[illegible]
[illegible]		20.5	[illegible]		[illegible]
[illegible]		0.0	[illegible]		[illegible]
[illegible]	[illegible]	25.52	[illegible]		[illegible]
[illegible]	[illegible]	25.47	[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]	[illegible]	[illegible]		[illegible]
[illegible]	[illegible]	[illegible]	[illegible]		[illegible]
[illegible]	[illegible]	[illegible]	[illegible]		[illegible]

LABORATORY ANALYSIS - PARTICULATES

1.0 Filter

Sample	Run No.	Filter No.	Tare Weight (gm)	Color	WEIGHINGS 1st (gm)	2nd (gm)	3rd (gm)	4th (gm)	5th (gm)	6th (gm)	7th (gm)	8th (gm)	Final (gm)	Net (gm)
Inlet:														
9437	1	375	.4682	green-gray	.6203	.5740	.5741	.5698	.5460	.5207	.5165	.5132	.5132	.0450
9440	2	433	.4274	dk black w/gray	.5970	.5939	.5951	.5968	.5917	.5968	.5934	.5911	.5914	.1640
9454	3	445	.3279	dark black	.5027	.4998	.4996	.5027	.4984	.5013	.4989	.4980	.4985	.1706
9448	4	413	.4299	black	.7073	.7040	.7062	.7052	.7000	.7008	.6992	.6963	.6978	.2679
9455	5	389	.4656	black	.6529	.6525	----	.6529	.6399	.6457	.6360	.6359	.6360	.1704
9463	6	421	.4288	black	.5330	.5290	.5264	.5250	.5225	.5253	.5202	.5188	.5195	.0907
9471	7	448	.3302	black w/glass	.5854	.5858	----	.5749	.5713	.5730	.5718	.5702	.5708	.2406
9473	8	402	.4265	black w/green	.6158	.6106	.6119	.6100	.6046	.6112	.6080	.6033	.6040	.1775
Outlet:														
9432	1A	378	.4902	white	.5244	.5239	----	.5227	.5187	.5259	.5208	.5187	.5187	.0285
9374	1B	379	.4541	white	.4655	.4651	----	.4649	.4612	.4711	.4618	.4613	.4613	.0072
9442	2	425	.4315	lt gray in ctr	.5585	.5562	.5568	.5571	.5552	.5574	.5563	.5570	.5557	.1242
9446	3	436	.4290	lt gray in ctr	.4765	.4760	----	.4767	.4751	.4768	.4791	.4799	.4756	.0466
9449	4	417	.3921	black gray	.3972	.3975	----	.3974	.3966	.3980	.3975	.3991	.3969	.0048
9458	5	370	.4869	green	.6042	.6001	.6025	.6024	.6006	.6140	.5979	.5991	.5979	.1110
9461	6	434	.4309	off white	.4316	.4312	----	.4313	.4304	.4318	.4315	.4312	.4308	-.0001
9468	7	418	.3896	dk gray green	.6567	.6542	.6526	.6549	.6482	.6568	.6465	.6451	.6451	.2555
9379	8	444	.3266	light gray	.3340	.3316	.3316	.3325	.3307	.3319	.3310	.3320	.3309	.0043
Overnight Runs:														
9452	1	446	.3318	green black-- corroded state	.5608	.5607	----	.5610	.5528	.5631	.5523	.5490	.5490	.2172
9465	2	435	.4268	green	.8798	.8796	.8796	.8910	.8724	.8847	.8698	.8640	.8640	.4372
9381	3	441	.3984	black gray	.4105	.4100	----	.4105	.4089	.4088	.4075	.4082	.4079	.0095

2.0 Beakers

Sample	Run	Beaker	Volume (ml)	Tare (gm)	WEIGHINGS 1st (gm)	2nd (gm)	3rd (gm)	4th (gm)	Final Weight (gm)	Net Gain (gm)	Adjustment for Blank (gm)	Adjusted Net Gain (gm)
Inlet:												
9441	1	13	85	81.6445	81.6543	81.6544			81.6544	.0099	.0014	.0085
		14	135	80.8667	80.8767	80.8771			80.8769	.0102	.0022	.0080
9444	2	11	118	79.8626	79.8747	79.8749			79.8748	.0122	.0019	.0103
		12	146	81.7659	81.7804	81.7807			81.7806	.0147	.0023	.0124
9445	3	3	115	82.2915	82.3087	82.3083			82.3085	.0170	.0018	.0152
		5	130	81.3906	81.4024	81.4028			81.4026	.0120	.0021	.0099
9451	4	2	110	81.4655	81.4856	81.4862	81.4858		81.4859	.0204	.0018	.0186
		1	145	82.3022	82.3175	82.3177			82.3176	.0154	.0023	.0131
9456	5	8	130	81.0174	81.0292	81.0295			81.0294	.0120	.0021	.0099
		9	115	81.4521	81.4603	81.4605			81.4604	.0083	.0018	.0065
9464	6	6	50	81.5212	81.5416	81.5418			81.5417	.0205	.0008	.0197
		7	140	80.8630	80.9011	80.9017	80.9019		80.9016	.0386	.0022	.0364
9472	7	16	130	83.2780	83.3556	83.3558			83.3557	.0777	.0021	.0756
9474	8	15	150	80.1897	80.2226	80.2231			80.2229	.0332	.0024	.0308
Blank		10	50	82.0571	82.0577	82.0580			82.0579	.0008	.0008	----

2.0 Beakers

Sample	Run	Beaker	Volume (ml)	Tare (gm)	WEIGHINGS 1st (gm)	2nd (gm)	3rd (gm)	4th (gm)	Final Weight (gm)	Net Gain (gm)	Adjustment for Blank (gm)	Adjusted Net Gain (gm)
Outlet:												
9434	1A	3	145	82.2915	82.3447	82.3430	82.3407	82.3407	82.3407	.0492	.0023	.0469
		15	140	80.1897	80.2507	80.2507			80.2507	.0610	.0022	.0588
9376	1B	15	145	80.2229	80.2240	80.2240			80.2240	.0011	.0023	-.0012
		14	25	80.8769	80.8713	80.8710			80.8712	-.0057	.0004	-.0061
9443	2	16	150	83.3557	83.3532	83.3532			83.3532	-.0025	.0024	-.0049
		1	140	82.3176	82.3219	82.3221			82.3220	.0044	.0022	.0022
9447	3	3	145	82.3085	82.3120	82.3120			82.3120	.0035	.0023	.0012
		12	140	81.7806	81.7820	81.7816			81.7818	.0012	.0022	-.0010
9450	4	7	90	80.8630	80.8672	80.8672			80.8672	.0042	.0014	.0028
		16	110	83.2780	83.2830	83.2809	83.2788	83.2789	83.2789	.0009	.0018	-.0009
9459	5	6	145	81.5417	81.5436	81.5436			81.5436	.0019	.0023	-.0004
		9	30	81.4604	81.4567	81.4560	81.4565		81.4564	.0040	.0005	-.0045
9462	6	11	140	79.8749	79.8782	79.8777			79.8780	.0031	.0022	.0009
		10	55	82.0579	82.0590	82.0581	82.0586		82.0586	.0007	.0009	-.0002
9469	7	4	140	84.2189	84.2302	84.2300			84.2301	.0112	.0022	.0090
9380	8	8	140	81.0294	81.0464	81.0451	81.0455		81.0457	.0163	.0022	.0141
		5	50	81.4026	81.3993	81.4002	81.3988		81.3994	-.0032	.0008	-.0040

2.0 Beakers

Sample	Run	Beaker	Volume (ml)	Tare (gm)	WEIGHINGS 1st (gm)	2nd (gm)	3rd (gm)	4th (gm)	Final Weight (gm)	Net Gain (gm)	Adjustment for Blank (gm)	Adjusted Net Gain (gm)
Overnight:												
9453	1	54	225	96.7278	96.7495	96.7489	96.7488		96.7489	.0211	.0036	.0175
9467-catch	2	35	110	106.5266	106.5518	106.5517	106.5517		106.5517	.0251	.0000	.0251
9382-catch	3	Ace 30	200	107.4211	107.4300	107.4304	107.4324		107.4302	.0091	.0032	.0059
9383		Water 52	250	87.4415	87.4675	87.4677	87.4676		87.4676	.0261	----	.0261
9470	3*	51	250	105.7463	105.7455	105.7450	105.7451		105.7451	-.0012	.0000	-.0012
		53	225	104.7784	104.7835	104.7833	104.7834		104.7833	.0049	.0000	.0049

* No filter.

TOTALS

3.0 Total

Sample	Run	Sample No.	Filter No.	Filter (gm)	Sample No.	Wash (gm)	Totals (gm)
Inlet	1	9437	375	0.0450	9441	0.0165	0.0615
	2	9440	433	0.1640	9444	0.0227	0.1867
	3	9454	445	0.1706	9445	0.0251	0.1957
	4	9448	413	0.2679	9451	0.0317	0.2996
	5	9455	389	0.1704	9456	0.0164	0.1868
	6	9463	421	0.0907	9464	0.0561	0.1468
	7	9471	448	0.2406	9472	0.0756	0.3162
	8	9473	402	0.1775	9474	0.0308	0.2083
Outlet	1A	9432	378	0.0285	9434	0.1057	0.1342
	1B	9374	379	0.0072	9376	Neg	0.0072
	2	9442	425	0.1242	9443	0.0022	0.1264
	3	9446	436	0.0466	9447	0.0012	0.0478
	4	9449	417	0.0048	9450	0.0028	0.0076
	5	9458	370	0.1110	9459	Neg	0.1110
	6	9461	434	-0.0001	9462	0.0009	0.0009
	7	9468	418	0.2555	9469	0.0090	0.2645
	8	9379	444	0.0043	9380	0.0141	0.0184
Overnight	1	9452	446	0.2172	9453	0.0175	0.2347
	2	9465	435	0.4372	9467	0.0251	0.4623
	3	9381	441	0.0095	9383	0.0320	0.0415
					9382		
	3		No filter		9470	0.0049	0.0049

PARTICLE SIZING

COMPUTER PRINT OUT

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LABORATORY ANALYSIS

ROCHESTER SMELTING AND REFINING OUTLET RUN NO.1

FLOW RATE = 0.068 ACFM TEMPERATURE = 100.0 F

SAMPLING DURATION = 221.0 MIN

ASSUMED PARTICLE DENSITY = 2.00 GM/CC BAR OMETRIC PRESSURE = 29.82 HG

CALC. GRAIN LOADING = 0.00023 GR/ACF 0.00025 GR/SDCF

DELTA P = 0.0 HG

LOWER SIZE

	LIMIT	MICROGRAMS	CUM MASS	PER CENT CUM	GRAINS/SDCF
S1	7.70	150.0	1895.0	88.17	0.0000220
S2	4.54	0.0	1345.0	70.29	0.0
S3	2.43	0.0	1345.0	70.29	0.0
S4	2.34	30.0	1345.0	70.29	0.0000008
S5	1.58	200.0	1315.0	77.52	0.0000294
S6	1.01	130.0	1115.0	65.69	0.0000191
S7	0.53	80.0	985.0	57.99	0.0000117
S8	0.24	0.0	905.0	53.26	0.0

ROCHESTER SMELTING AND REFINING OUTLET RUN NO. 2

FLOW RATE = 0.039 ACFM TEMPERATURE = 81.0 F

SAMPLING DURATION = 200.0 MIN

ASSUMED PARTICLE DENSITY = 2.00 GM/CC BAR OMETRIC PRESSURE = 29.20 HG

CALC. GRAIN LOADING = 0.00164 GR/ACF 0.00154 GR/SDCF

DELTA P = 0.0 HG

LOWER SIZE

	LIMIT	MICROGRAMS	CUM MASS	PER CENT CUM	GRAINS/SDCF
S1	7.87	1930.0	10195.0	84.02	0.0001594
S2	4.72	1030.0	8965.0	73.92	0.0001401
S3	2.73	800.0	7935.0	65.34	0.0001236
S4	2.42	1100.0	6565.0	54.10	0.0001352
S5	1.62	540.0	5515.0	45.46	0.0000845
S6	1.03	0.0	4975.0	41.02	0.0
S7	0.55	60.0	4975.0	41.02	0.0000077
S8	0.28	350.0	4915.0	40.52	0.0000451

ROCHESTER SMELTING AND REFINING OUTLET RUN NO.3

FLOW RATE = 0.068 ACFM TEMPERATURE = 81.0 F

SAMPLING DURATION = 220.0 MIN

ASSUMED PARTICLE DENSITY = 2.00 GM/CC BAR OMETRIC PRESSURE = 29.32 HG

CALC. GRAIN LOADING = 0.00038 GR/ACF 0.00038 GR/SDCF

DELTA P = 0.0 HG

LOWER SIZE

	LIMIT	MICROGRAMS	CUM MASS	PER CENT CUM	GRAINS/SDCF
S1	8.04	110.0	8155.0	97.65	0.0000098
S2	4.75	310.0	8045.0	95.04	0.0000276
S3	2.48	340.0	7735.0	87.77	0.0000312
S4	3.45	60.0	7395.0	79.53	0.0000055
S5	1.43	80.0	7335.0	78.34	0.0000073
S6	1.04	460.0	7245.0	76.24	0.0000428
S7	0.56	460.0	6765.0	68.99	0.0000428
S8	0.27	250.0	6285.0	63.95	0.0000383

ROCHESTER SPELTING AT OPERATING INLET RUN NO.1

FLOW RATE = 0.357 ACFM TEMPERATURE = 117.0 F

SAMPLING DURATION = 25.0 MIN

ASSUMED PARTICLE DENSITY = 2.00 GM/CC BAR CHARTRIC PRESSURE = 29.40 HG

CALC. GRAIN LOADING = 0.03090 GR/ACF 0.00393 GR/SDCF

DELTA P = 0.0 HG

LOWER SIZE

	LIMIT	VELOCITY	FLOW RATE	PER CENT CUM	GRAINS/SDCF
S1	8.60	43.0	22394.0	89.32	0.0001104
S2	7.28	813.0	22333.0	89.04	0.0003733
S3	5.02	300.0	22120.0	88.21	0.0004830
S4	3.71	225.0	21730.0	86.66	0.0003993
S5	1.74	2125.0	21505.0	85.74	0.0037234
S6	1.13	2437.0	19390.0	77.29	0.0042705
S7	0.59	3913.0	14503.0	57.87	0.0068570
S8	0.29	3850.0	13050.0	51.96	0.0067064

ROCHESTER SPELTING AT OPERATING INLET RUN 2

FLOW RATE = 0.255 ACFM TEMPERATURE = 118.0 F

SAMPLING DURATION = 50.0 MIN

ASSUMED PARTICLE DENSITY = 2.00 GM/CC BAR CHARTRIC PRESSURE = 29.50 HG

CALC. GRAIN LOADING = 0.06034 GR/ACF 0.00000 GR/SDCF

DELTA P = 0.0 HG

LOWER SIZE

	LIMIT	VELOCITY	FLOW RATE	PER CENT CUM	GR/DS/SDCF
S1	9.07	925.0	56505.0	94.80	0.0005873
S2	4.72	1000.0	55580.0	93.33	0.0008219
S3	2.43	1025.0	54580.0	91.58	0.0012124
S4	1.41	1075.0	53555.0	89.78	0.0010736
S5	1.42	3575.0	50880.0	85.46	0.0029663
S6	1.13	3775.0	44905.0	75.38	0.0049540
S7	0.51	5150.0	37530.0	63.02	0.0090488
S8	0.26	11255.0	25380.0	42.60	0.0065258

ROCHESTER SPELTING AT OPERATING INLET RUN 3

FLOW RATE = 0.487 ACFM TEMPERATURE = 94.0 F

SAMPLING DURATION = 43.0 MIN

ASSUMED PARTICLE DENSITY = 2.00 GM/CC BAR CHARTRIC PRESSURE = 29.11 HG

CALC. GRAIN LOADING = 0.01847 GR/ACF 0.01700 GR/SDCF

DELTA P = 0.0 HG

LOWER SIZE

	LIMIT	VELOCITY	FLOW RATE	PER CENT CUM	GRAINS/SDCF
S1	6.33	475.0	16540.0	55.79	0.0002402
S2	7.02	510.0	16180.0	54.24	0.0003253
S3	4.44	740.0	15680.0	52.35	0.0004879
S4	3.47	805.0	15030.0	49.68	0.0005204
S5	1.49	1250.0	12130.0	38.42	0.0007817
S6	1.08	1545.0	10930.0	41.87	0.0009921
S7	0.57	1725.0	9205.0	35.99	0.0011222
S8	0.28	1525.0	7680.0	29.39	0.0009921

1 MECHANICAL SCRUBBER AND FABRIC JULET RUN 4

FLOW RATE = 0.346 ACF TEMPERATURE = 95.0 F

SCRUBBER OPERATION = 150.0 MIN

ASSUMED PARTICLE DENSITY = 2.00 GM/CC BAR OMETRIC PRESSURE = 29.21 HG

CALC. GRAIN LOADING = 0.01156 GR/ACF 0.01236 GR/SDCF

DELTA P = 0.0 IN

LOWER SIZE

	LIMIT	MICROGRAMS	CUM PASS	PER CENT CUM	GRAINS/SDCF
01	8.75	171.0	35526.0	85.76	0.0000908
02	7.37	854.0	35354.0	85.35	0.0001643
03	6.00	928.0	34806.0	84.02	0.0002465
04	3.75	1417.0	33951.0	82.03	0.0002988
05	1.75	2388.0	32951.0	79.62	0.0007021
06	1.10	5763.0	30631.0	73.94	0.0017299
07	0.60	8250.0	24568.0	60.03	0.0018670
08	0.20	6043.0	16318.0	44.94	0.0018195

LABORATORY ANALYSIS OF ANDERSEN FILTERS

Run ID	Filter No.	Stage	Tare (gm)	WEIGHINGS 1st (gm)	WEIGHINGS 2nd (gm)	Final Weight (gm)	Net Gain (gm)
Inlet #1	416	Pre-catch	0.257350	0.260050	0.260000	0.260030	0.002680
	H	1	0.179775	0.179900	0.179775	0.179838	0.000063
	B	2	0.166850	0.167275	0.166850	0.167063	0.000213
	G	3	0.172800	0.173200	0.173180	0.173190	0.000390
	A	4	0.170225	0.170475	0.170425	0.170450	0.000225
	F	5	0.172750	0.174925	0.174825	0.174875	0.002125
	E	6	0.168200	0.170650	0.170625	0.170637	0.002437
	I	7	0.178075	0.182050	0.181925	0.181988	0.003913
	C	8	0.168900	0.172825	0.172675	0.172750	0.003850
	14	Final	0.240525	0.249750	0.249650	0.249700	0.009175
						TOTAL	0.025071
Inlet #2	415	Pre-catch	0.258700		0.261750	0.261750	0.003050
	T	1	0.173875		0.174800	0.174800	0.000925
	Y	2	0.170325		0.171425	0.171425	0.001100
	S	3	0.179750		0.181375	0.181375	0.001625
	W	4	0.169850		0.171825	0.171825	0.001975
	R	5	0.178400		0.182375	0.182375	0.003975
	X	6	0.170025		0.179400	0.179400	0.009375
	U	7	0.175675		0.187825	0.187825	0.012150
	V	8	0.167825		0.179250	0.179250	0.011425
	18	Final	0.246100		0.260050	0.260050	0.013950
						TOTAL	0.059550

LABORATORY ANALYSIS OF ANDERSEN FILTERS (continued)

Run ID	Filter No.	Stage	Tare (gm)	WEIGHINGS 1st (gm)	WEIGHINGS 2nd (gm)	Final Weight (gm)	Net Gain (gm)
Inlet #3	407	Pre-catch	0.230550		0.242100	0.242100	0.011550
	25	1	0.174425		0.174825	0.174825	0.000400
	67	2	0.171775		0.172275	0.172275	0.000500
	26	3	0.179475		0.180225	0.180225	0.000750
	66	4	0.168400		0.169200	0.169200	0.000800
	27	5	0.176150		0.177350	0.177350	0.001200
	65	6	0.171100		0.172625	0.172625	0.001525
	28	7	0.173775		0.175500	0.175500	0.001725
	68	8	0.163850		0.165375	0.165375	0.001525
	424	Final	0.255050		0.261200	0.261200	0.006150
						TOTAL	0.026125
Inlet #4	628	Pre-catch	0.299800	0.305750	0.305650	0.305700	0.005900
	34	1	0.175950	0.176188	0.176050	0.176119	0.000170
	76	2	0.163400	0.163950	0.163950	0.163950	0.000550
	33	3	0.181950	0.182800	0.182750	0.182775	0.000825
	25	4	0.172350	0.173400	0.173300	0.173350	0.001000
	35	5	0.179800	0.182350	0.181950	0.182150	0.002350
	74	6	0.164200	0.170000	0.169925	0.169963	0.005763
	36	7	0.181200	0.187300	0.187600	0.187450	0.006250
	73	8	0.170375	0.176525	0.176350	0.176438	0.006063
	17	Final	0.244450	0.256800	0.256650	0.256725	0.012275
	16	Final	0.244000	0.244250	0.244300	0.244275	0.000275
						TOTAL	0.041421

LABORATORY ANALYSIS OF ANDERSEN FILTERS (continued)

Run ID	Filter No.	Stage	Tare (gm)	WEIGHINGS 1st (gm)	WEIGHINGS 2nd (gm)	Final Weight (gm)	Net Gain (gm)
Outlet #1	409	Pre-catch	0.230350	0.230550		0.230550	0.00020
	38	1	0.172825	0.172975		0.172975	0.00015
	78	2	0.169450	0.169450		0.169450	0.00000
	37	3	0.173050	0.173050		0.173050	0.00000
	79	4	0.172000	0.172025		0.172025	0.00003
	39	5	0.178950	0.179150		0.179150	0.00020
	80	6	0.162325	0.162450		0.162450	0.00013
	40	7	0.171300	0.171375		0.171375	0.00008
	77	8	0.169225	0.169150		0.169150	-0.00008
	427	Final	0.253700	0.254600		0.254600	0.00090
						TOTAL	0.00169
Outlet #2	408	Pre-catch	0.231850	0.233750	0.233700	0.233725	0.00188
	24	1	0.174550	0.175650	0.175900	0.175775	0.00123
	61	2	0.172175	0.173475	0.173725	0.173600	0.00143
	23	3	0.174550	0.175375	0.175650	0.175513	0.00096
	62	4	0.162175	0.163175	0.163275	0.163225	0.00105
	21	5	0.173225	0.173675	0.173850	0.173763	0.00054
	63	6	0.160275	0.162050	0.162050	0.162050	-0.00003
	22	7	0.179775	0.179775	0.179900	0.179838	0.00006
	64	8	0.167925	0.168175	0.168375	0.168275	0.00035
	413	Final	0.254600	0.256550	0.256600	0.256575	0.00198
	414	Final	0.253550	0.256050	0.256200	0.256125	0.00258
						TOTAL	0.01206

LABORATORY ANALYSIS OF ANDERSEN FILTERS (continued)

Run ID	Filter No.	Stage	Tare (gm)	WEIGHINGS 1st (gm)	WEIGHINGS 2nd (gm)	Final Weight (gm)	Net Gain (gm)
Outlet #3	614	Pre-catch	0.287600	0.287600	0.287800	0.287700	0.00010
	J	1	0.180300	0.180600	0.180525	0.180414	0.00011
	N	2	0.172200	0.172475	0.172550	0.172513	0.00031
	K	3	0.180150	0.180575	0.180425	0.180500	0.00035
	O	4	0.170025	0.170025	0.170125	0.170075	0.00005
	L	5	0.181725	0.181775	0.181850	0.181813	0.00009
	P	6	0.169275	0.169700	0.169800	0.169750	0.00048
	M	7	0.176200	0.176850	0.176900	0.176875	0.00068
	Q	8	0.170400	0.170800	0.170850	0.170825	0.00043
	406	Final	0.233400	0.234900	0.235200	0.235050	0.00165
						TOTAL	0.00425

THREE-STAGE CYCLONE SAMPLER

LABORATORY PARTICULATE ANALYSIS

LABORATORY ANALYSIS ON CYCLONE CATCH

Run	Stage	Tare Weight (mg)	Final Weight (mg)	Net Gain (mg)	Percentage of Total (%)
Inlet 1	1st	0.000	12.380	12.380	27.9
	2nd	0.080	4.130	4.050	9.1
	3rd	0.080	6.280	6.200	14.0
	Filter 625	137.425	157.600	20.175	49.0
	Filter 631	134.200	135.750	1.550	
	TOTAL			44.355	
Inlet 2	1st	0.080	22.510	22.430	55.6
	2nd	0.210	0.610	0.400	1.0
	3rd	0.180	2.160	1.980	4.9
	Filter 664	134.075	147.900	13.825	38.5
	Filter 656	140.045	142.150	1.700	
	TOTAL			40.335	
Inlet 3	1st	0.160	58.290	58.130	46.9
	2nd	0.250	2.160	1.910	1.5
	3rd	0.230	5.590	5.360	4.3
	Filter 693	138.600	139.425	0.825	47.2
	Filter 694	138.275	195.900	57.625	
	TOTAL			123.850	
Inlet 4	1st	0.150	14.800	14.650	22.1
	2nd	0.140	1.970	1.830	2.8
	3rd	0.160	7.890	7.730	11.7
	Filter 616	143.700	143.450	-0.250	63.4
	Filter 612	144.325	186.275	41.950	
	TOTAL			66.410	

LABORATORY ANALYSIS ON CYCLONE CATCH (continued)

Run	Stage	Tare Weight (mg)	Final Weight (mg)	Net Gain (mg)	Percentage of Total (%)
Outlet 1	1st	0.000	1.024	1.024	14.0
	2nd	0.027	2.706	2.679	36.7
	3rd	0.058	1.346	1.288	17.7
	Filter 612	141.975	144.125	2.150	31.5
	Filter 613	138.300	138.450	0.150	
	TOTAL			7.291	
Outlet 2	1st	0.058	2.403	2.345	18.6
	2nd	0.107	0.710	0.603	4.8
	3rd	0.082	0.441	0.359	2.8
	Filter 692	141.950	149.925	7.975	73.8
	Filter 619	138.500	139.825	1.325	
	TOTAL			12.607	
Outlet 3	1st	0.102	6.167	6.065	52.1
	2nd	0.147	0.455	0.308	2.6
	3rd	0.120	0.592	0.472	4.1
	Filter 615	140.450	140.475	0.025	41.2
	Filter 609	143.325	148.100	4.775	
	TOTAL			11.645	

POLYNUCLEAR AROMATIC HYDROCARBONS

LABORATORY ANALYSIS

POLYNUCLEAR AROMATIC HYDROCARBON ANALYSIS

The outlet filters (#417, Method 5, Run 4; #692, Cyclone Run 2; #418, Method 5, Run 7) were extracted with methylene chloride and concentrated to one ml. A gas chromatograph with a flame ionization detector with a Tenex-GC column was used for the analysis. The sample contained less than 1 μg (10^{-7} g) PNA as α-naphthyl amine, and as such PNA was not detectable on the gas chromatograph nor would it have been on the GC/MS.

CHLORINE/HYDROGEN CHLORIDE

LABORATORY ANALYSIS

CHLORINE ANALYSIS

Run	Date	Source	Aliquot	Titrate ml Iodine	Total ml Iodine	Blank ml Iodine	Total mg Chlorine
1	10/19/76	Inlet	1/4 sample	11.55, 11.65	11.60	12.64	13.130
1		Outlet	"	12.60, 12.61	12.61	12.64	0.379
2	10/20/76	Inlet	"	12.11, 12.30	12.21	12.64	5.430
2		Outlet	"	12.50, 12.68	12.59	12.64	0.631
3	10/20/76	Inlet	"	11.80, 11.89	11.85	12.64	9.970
3		Outlet	"	12.48, 12.55	12.52	12.64	1.510
4	10/22/76	Inlet	"	11.66, 11.60	11.63	12.64	12.750
4		Outlet	"	12.38, 12.46	12.42	12.64	2.780
5*	10/22/76	Inlet	"	11.60, 11.64	11.62	12.64	12.880
5*		Outlet	"	12.50, 12.54	12.52	12.64	1.510

["Chlorine Equivalent" = 3.1559]

* Sampled 1 hour after "chlorine demagging"
** This value has already been multiplied by the F factor

HYDROGEN CHLORIDE ANALYSIS

Run	Date	Source	Aliquot	ml $AgNO^3$ Added	ml Am. Thio back	Equivalent Amount $AgNO^3$	Net ml** $AgNO^3$	Blank ml $AgNO^3$	Total mg HCl
1	10/19/76	Inlet	1/4 sample	9.00	8.01, 8.05	8.45	2.20	0.00	0.765
1		Outlet	"	9.00	8.41, 8.55	8.92	0.32	0.00	0.111
2	10/20/76	Inlet	"	21.00	16.20, 16.85	17.38	14.48	0.00	5.040
2		Outlet	"	15.00	14.30, 14.20	14.99	0.04	0.00	0.014
3	10/20/76	Inlet	"	25.00	20.52, 20.56	21.61	13.56	0.00	4.720
3		Outlet	"	15.00	14.21, 14.31	15.00	0.00	0.00	0.000
4	10/22/76	Inlet	"	25.00	15.25, 15.39	16.12	35.32	0.00	12.360
4		Outlet	"	20.00	17.20, 17.32	18.16	7.36	0.00	2.560
5*	10/22/76	Inlet	"	25.00	21.19, 21.22	22.31	10.76	0.00	3.740
5*		Outlet	"	10.00	9.50, 9.52	10.00	0.00	0.00	0.000

["T" = 0.3478]

* Sampled 1 hour after "chlorine demagging"
** This value has already been multiplied by the F factor

SULFUR DIOXIDE/SULFUR TRIOXIDE

LABORATORY ANALYSIS

Sulfur Dioxide and Sulfur Trioxide Analysis

Run	Date	Source	SULFUR DIOXIDE			SULFUR TRIOXIDE		
			Aliquot	Blank (ml)	Sample (ml)	Aliquot	Blank (ml)	Sample (ml)
1	10/18/76	Inlet	Entire Sample	0.00	1.60	Entire Sample	0.00	0.08
1		Outlet	"	0.00	0.00	"	0.00	0.13
2	10/19/76	Inlet	"	0.00	0.02	"	0.00	0.04
2		Outlet	"	0.00	1.76	"	0.00	0.21
3	10/21/76	Inlet	"	0.00	2.86	"	0.00	0.14
3		Outlet	"	0.00	0.00	"	0.00	0.00
4	10/21/76	Inlet	"	0.00	0.28	"	0.00	0.10
4		Outlet	"	0.00	0.00	"	0.00	0.04
5	10/21/76	Inlet	"	0.00	2.96	"	0.00	0.51
5		Outlet	"	0.00	0.02	"	0.00	0.00
6	10/22/76	Inlet	"	0.00	6.26	"	0.00	0.02
6		Outlet	"	0.00	1.70	"	0.00	0.03
7	10/22/76	Inlet	"	0.00	5.10	"	0.00	0.11
7		Outlet	"	0.00	1.78	"	0.00	0.02

Normality = 0.0106

ELEMENTAL ANALYSIS OF

BAGHOUSE HOPPER DUST

Coors / SPECTRO-CHEMICAL LABORATORY
DIVISION OF COORS PORCELAIN COMPANY
GOLDEN, COLORADO, U.S.A.
303-279-6565 Ext: 3202

Mailing Address:
P.O. Box 500
Golden, Colorado 80401

Analytical Report

TO: Environmental Science and Engineering, Inc.
P.O. Box 13454
Gainesville, FL 32604

Attention: Stu Whitlock

LABORATORY NUMBER	27316-00
DATE	12-7-76
CUSTOMER ORDER NO.	2132
MATERIAL	
SAMPLE NUMBER	

ELEMENT	%	ELEMENT	%	ELEMENT	%	ELEMENT	%
[illegible] (Al)	> 10	Gallium (Ga)	0.005	Silicon (Si)	10	Rubidium (Rb)	0.005
[illegible]	0.05	Germanium (Ge)	< 0.005	Silver (Ag)	< 0.001	Hafnium (Hf)	< 0.01
[illegible]	< 0.01	Indium (In)	< 0.005	Strontium (Sr)	0.005	Niobium (Nb)	< 0.1
[illegible] (Fe)	0.005	Iron (Fe)	0.2	Tin (Sn)	0.007	Tellurium (Te)	< 0.05
[illegible] (Be)	< 0.001	Lead (Pb)	0.1	Titanium (Ti)	0.05	Thorium (Th)	< 0.1
[illegible]	0.01	Magnesium (Mg)	0.1	Vanadium (V)	< 0.005	Thallium (Tl)	< [illegible]
[illegible]	[illegible]	Manganese (Mn)	0.01	Zinc (Zn)	< 0.00[illegible]	Tungsten (W)	< 0.1
[illegible] (Co)	< [illegible]	Mercury (Hg)		Zirconium (Zr)	0.01	Gold (Au)	< 0.001
[illegible]	[illegible]	Molybdenum (Mo)	< 0.01	Sodium (Na)	4.0	Iridium (Ir)	< 0.11
[illegible]	[illegible]	Nickel (Ni)	< 0.01	Cesium (Cs)	< 0.001	Osmium (Os)	< 0.01
[illegible]	< 0.01	Niobium (Nb)		Lithium (Li)	0.002	Palladium (Pd)	< 0.005
[illegible]	[illegible]	Phosphorus (P)	[illegible]	Potassium (K)	[illegible]	[illegible]	< [illegible]

[illegible] on sample as received.

[illegible] basis on [illegible]

[illegible]

[illegible] (semi-quantitative to 50%)

[illegible] (as indicated)

< = Less than

> = Greater than

☐ Atomic Absorption

☒ Optical Emission

☐ Wet Chemistry

☐ X-Ray

Coors / SPECTRO-CHEMICAL LABORATORY

BY [signature]
Frank B. Schweitzer, Manager

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CUSTOMER

Coors / SPECTRO-CHEMICAL LABORATORY
DIVISION OF COORS PORCELAIN COMPANY
GOLDEN, COLORADO, U.S.A.
303-279-6565 Ext. 3202

Mailing Address:
P.O. Box 500
Golden, Colorado 80401

Analytical Report

TO: Environmental Science and Engineering, Inc.

LABORATORY NUMBER	27516-00
DATE	12-7-76
CUSTOMER ORDER NO.	2132

Element	Result
Rhenium(Re)	< 0.005
Rhodium(Rh)	< 0.005
Ruthenium(Ru)	< 0.005
Cerium(Ce)	< 0.05
Dysprosium(Dy)	< 0.01
Erbium(Er)	< 0.01
Europium(Eu)	< 0.01
Gadolinium(Gd)	< 0.005
Holmium(Ho)	< 0.005
Lutetium(Lu)	< 0.005
Lutetium(Tm)	< 0.001
Neodymium(Nd)	< 0.1
Praseodymium(Pr)	< 0.1
Samarium(Sm)	< 0.01
Terbium(Tb)	< 0.01
Thulium(Tm)	< 0.005
Yttrium(Y)	< 0.001
Ytterbium(Yb)	< 0.001

< = Less than.

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Coors / SPECTRO-CHEMICAL LABORATORY

BY Frank E. Schweitzer, Manager

[illegible] GREEN — Numerical Files CANARY — Customer Files

COMPOSITION OF TESISORB

TESISORB as supplied by Teller Environmental Systems, Inc.

Silicon as SiO_2 - 61%

Aluminum as Al_2O_3 - 23.3%

Calcium as CaO - 0.7%

Sodium as Na_2O - 9.8%

Potassium as K_2O - 4.6%

Magnesium as MgO - Trace

Iron as Fe_2O_3 - Trace

Titanium as TiO_2 - Trace