SECONDARY LEAD
SMELTING
AP-42 Section 7.11
Reference Number
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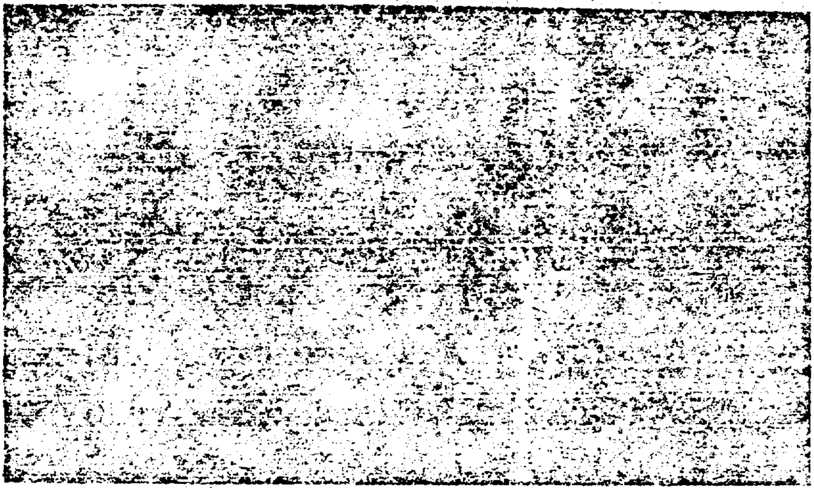
RESEARCH REPORT



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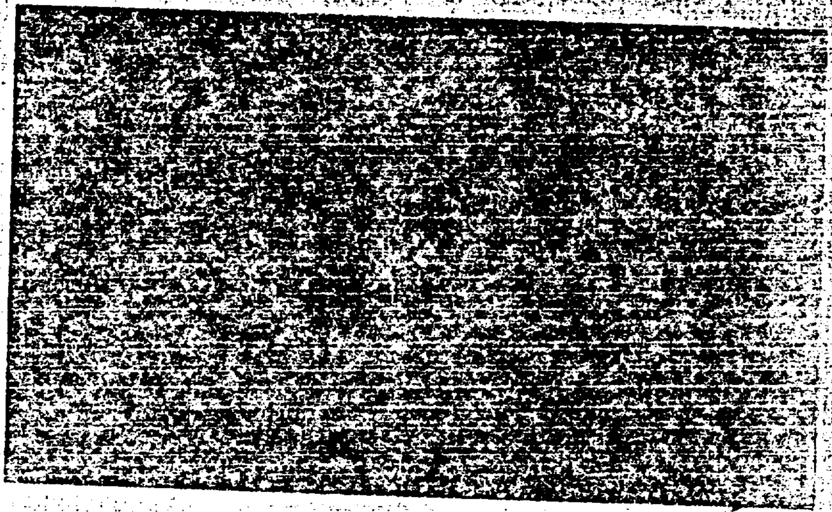

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RESEARCH REPORT

on

SECONDARY LEAD PLANT STACK EMISSION SAMPLING AT REVERE SMELTING AND REFINING PLANT, NEWARK, NEW JERSEY

Contract No. 68-02-0230

Task Order No. 1

to

ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF AIR PROGRAMS

August 11, 1972

by

P. R. Webb, H. E. Carlton,
and R. B. Engdahl

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INTRODUCTION AND BACKGROUND

Gas and particulate emission measurements were made at the Revere Smelting and Refining Company secondary lead plant in Newark, New Jersey, on December 8 and 9, 1971. The results of the measurements at this and other lead plants will be used in determining standards of performance for secondary lead plants.

The blast furnace, used to process lead plates from worn out batteries and lead cladding from electric cables, is fed continuously with battery plates, cladding, coke, iron, limestone, slag, dust, and sludge from the gas cleaning systems.

Figure 1 is a sketch showing gas flow through the plant. Air and oxygen are blown into the furnace to burn the coke in the feed. The heat of combustion melts the lead, while the coke reduces the lead oxides. The off-gases from the furnace are cleaned in two venturi scrubbers in parallel and then blown into the stack.

Effluent samples were withdrawn from the stack at a level of 33 feet above ground. The particulate samples were made using a 16-point

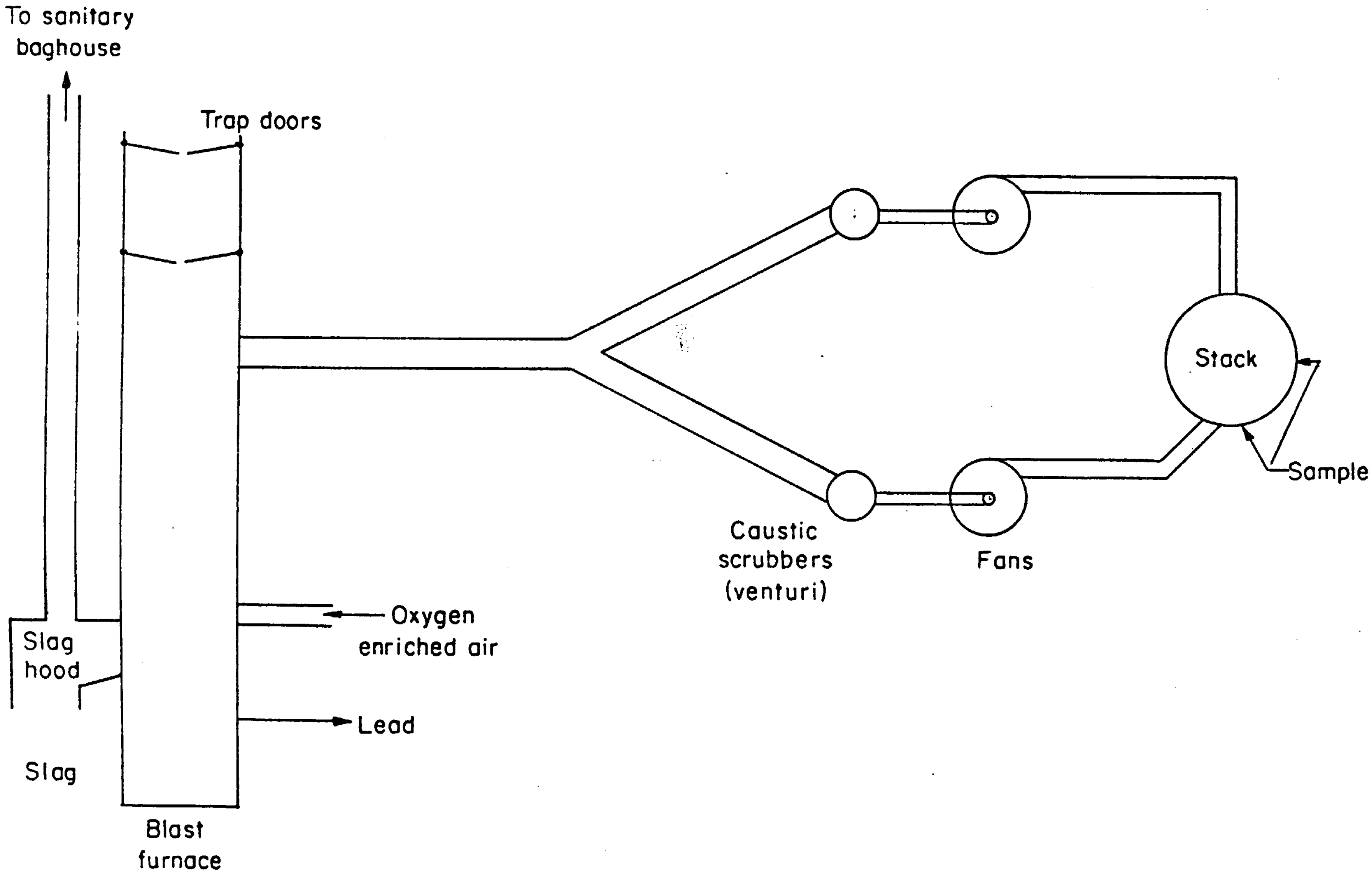


FIGURE 1. GAS FLOW DIAGRAM, REVERE SMELTING AND REF -, NEWARK, NEW JERSEY

traverse over a 2-hour period. The SO_2 samples were withdrawn from the center of the stack over a 1-1/2-hour period, and the Orsat samples were withdrawn from the center of the stack over a 1-hour period.

Samples were obtained in four runs; however, the first run was made while the blast furnace was shut down, and therefore, only three runs supplied complete data.

SUMMARY OF RESULTS

The process stack on the lead blast furnace of Revere Smelting and Refining Company in Newark, New Jersey, was sampled for particulates, SO_2, CO_2, O_2, and CO, in three runs on December 8 and 9, 1971.

A summary of the results is presented in Appendix G. The particulate emission caught in the filter and probe for the three runs averaged 0.0143 gr/scf. This is equivalent to an average emission rate from the plant of 1.511 lb/hr or 1.139 lb/ton of lead produced. The absorbables and condensables caught in the impingers increased the emission to 0.022 gr/scf which is equivalent to 2.341 lb/hr, or 1.785 lb/ton of lead. About one half of the impinger absorbables and condensables were extracted with ethyl either and chloroform and the other half remained in the water phase. The lead emission was low and averaged 0.001 gr/scf, which is equivalent to 0.111 lb/hr, or 0.085 lb/ton of lead. The SO_2 in the stack reached a detectable concentration of 0.1 ppm in only one of the three runs. The CO level in the stack averaged 0.4 percent. The flow rates and Orsat analysis indicated that only 5-7 percent of the sampled gas had passed through the blast furnace; the remainder being air that leaked into the system.

Runs 3 and 4 were typical of normal lead blast furnace operations. For Run 2, the furnace had been on stream for only about 1.5 hours before the sampling run was started. The oxygen enrichment during this run was about 10 percent rather than the normal three percent and the lead output was about 25 percent higher than the normal rate. The particulate emissions were increased in proportion to the greater lead output.

Analysis of the particulate by atomic absorption indicated only 5-10 percent lead; the major constituent measured by optical emission spectroscopy being sodium from the caustic scrubbing liquor. Lead and other fume constituents were present either because of incomplete scrubbing or trapped fumes in the mist from the scrubber were incompletely separated by the demister.

PROCESS DESCRIPTION

The Revere Plant in Newark processes various lead scraps into lead ingots. The plant consists of a receiving yard where the various lead scraps are stored; a blast furnace to melt or reduce the lead and lead oxides; refining kettles for lead; a continuous casting line for 60-pound ingots, and a shipping section.

Description of Feed Materials

Most scrap is received in the form of discarded batteries or scrap lead-sheathed electrical cable. The discarded batteries are broken apart; the acid and cases are discarded, and the lead plates are used as charge for the blast furnace. The lead sheath is usually removed from the electric cables and the copper wire sold, sometimes because of difficulty in removing the sheath, the entire cable is charged into the blast furnace. The sludge collected from the scrubber water is dried and recycled to the furnace as is the dust which settles in the flue. Crushed limestone and scrap cast iron were also fed to the furnace. About one-half of the slag from the furnace is crushed and fed back to the furnace and the other half is discarded in a landfill. A metallurgical grade coke is used for fuel. Oxygen enrichment is supplied from a large liquid oxygen tank.

Description of Blast Furnace

The blast furnace, rated at 45 tons/day, is a tube furnace approximately 20 feet high and about 4 feet in diameter as sketched in Figure 2. It contains a hearth for lead at the bottom. The slag floats on the lead and coke supports the charge in the tube. Oxygen enriched air is blown into the furnace at a level just above the slag at a constant water pressure of 1.5 psi and a rate of about 800 cfm. Gaseous oxygen is automatically supplied to the furnace by passing liquid oxygen through heat exchangers. Dusty gas is vented into a flue near the top of the furnace. Dusty gas from a hood over the slag tap is exhausted through the sanitary baghouse which also filters exhaust air from several other parts of the plant. Double trap doors are used to seal the top of the furnace and are forced open by the weight of a charge.

The lead from the blast furnace is tapped continuously into 1500-pound or 1650-pound water-cooled ingot molds which are then remelted in refining kettles and cast into 60-pound ingots in a continuous casting machine.

The slag is tapped intermittently into cast iron slag pots, each pot holding about 2000 pounds.

Description of Gas Cleaning Equipment

Separate gas cleaning systems are used for cleaning the various gas streams at Revere. The system which was sampled collected dust only from the blast furnace exhaust gas. The parts for the gas-cleaning system (manufactured by Chemico) were purchased second hand and assembled by Revere.

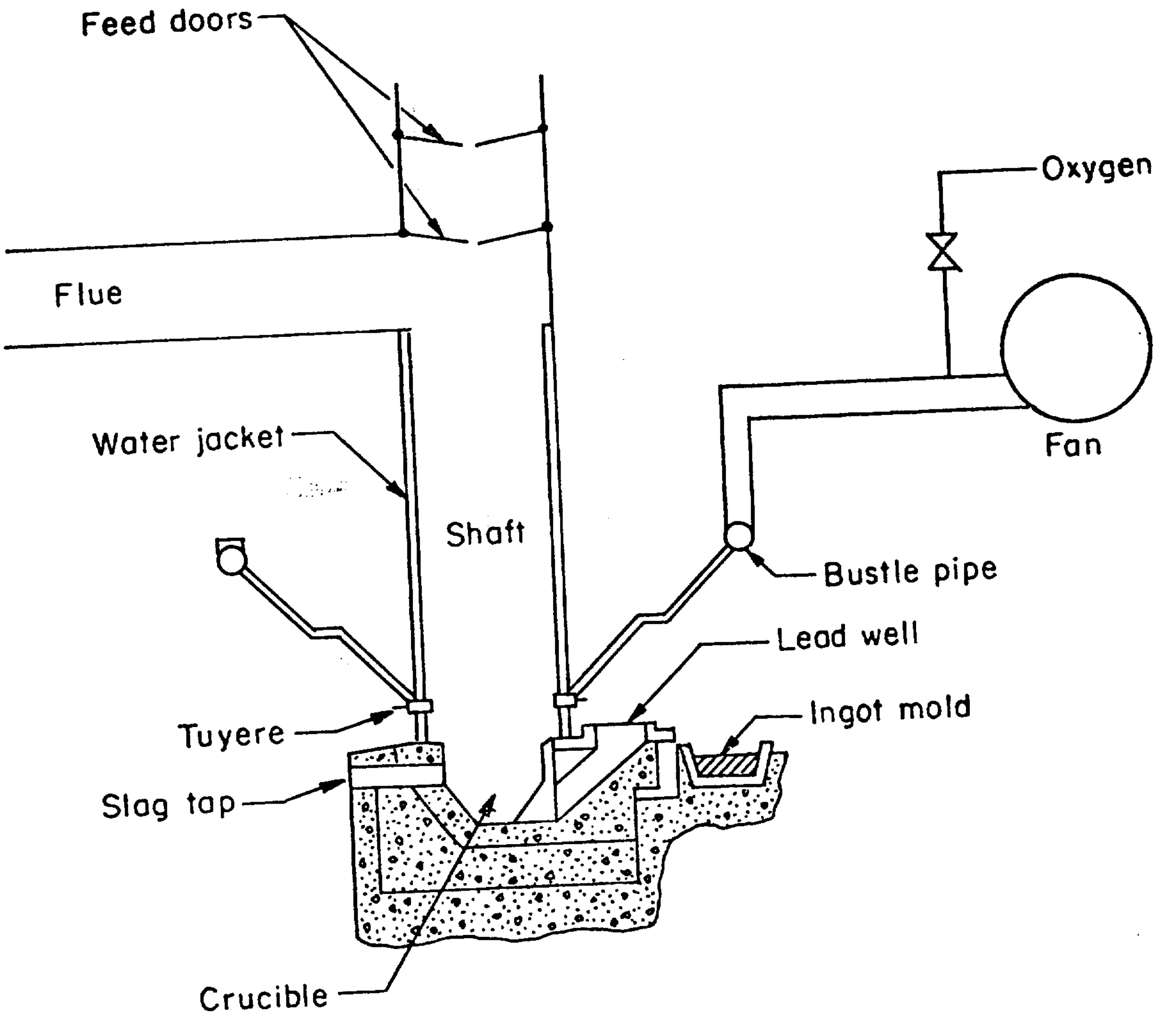


FIGURE 2. LEAD BLAST FURNACE

The exhaust gas from the blast furnace is vented through a long horizontal flue where the gas cools and causes a large portion of the particulates to settle. The duct was not air tight and allowed air to leak into the system.

The gas stream is divided into two streams flowing in parallel to venturi scrubbers with approximately 60 percent of the flow in one duct and 40 percent in the other. The venturi scrubbers, as illustrated in Figure 3, are of the dry inlet type. Water is injected at the venturi throat.

The larger scrubber had a throat diameter of 12 inches and the smaller scrubber had a throat diameter of 10 inches. A name plate on the smaller scrubber indicated it had been made by the Chemical Construction Company.

The discharge from each scrubber passes through demisters to separate the gas and liquid. The liquid is then directed into cylindrical sludge tanks that are 10 feet in diameter and 30 feet long. A 150 gpm pump recirculates the liquid to the smaller scrubber and a 300 gpm pump supplies the larger scrubber. Fresh caustic soda solution is prepared in a small tank and added to either sludge tank as needed.

The gases from the demister are pumped through centrifugal blowers (rated at 50 inches of water pressure) and exhausted into a 36-inch stack. The blower on the larger scrubber was manufactured by the Robbins Company.

LOCATION OF SAMPLING POINTS

The stack gas was sampled for emissions from sample ports located 7.5 stack diameters from the last flow disturbance, a distance requiring a minimum traverse of 16 sampling points. (See Figure 4). The SO_2 and Orsat samples were obtained from a single point at the center of the stack.

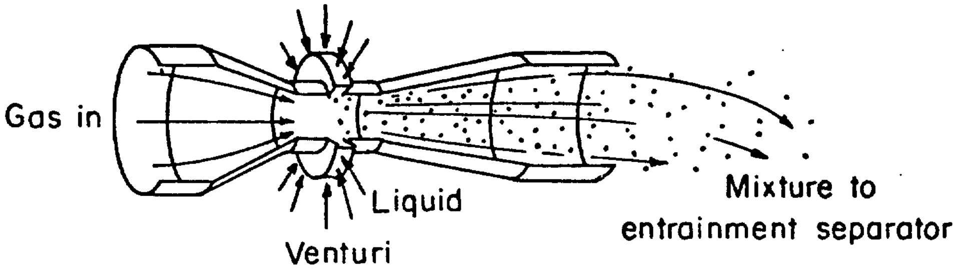
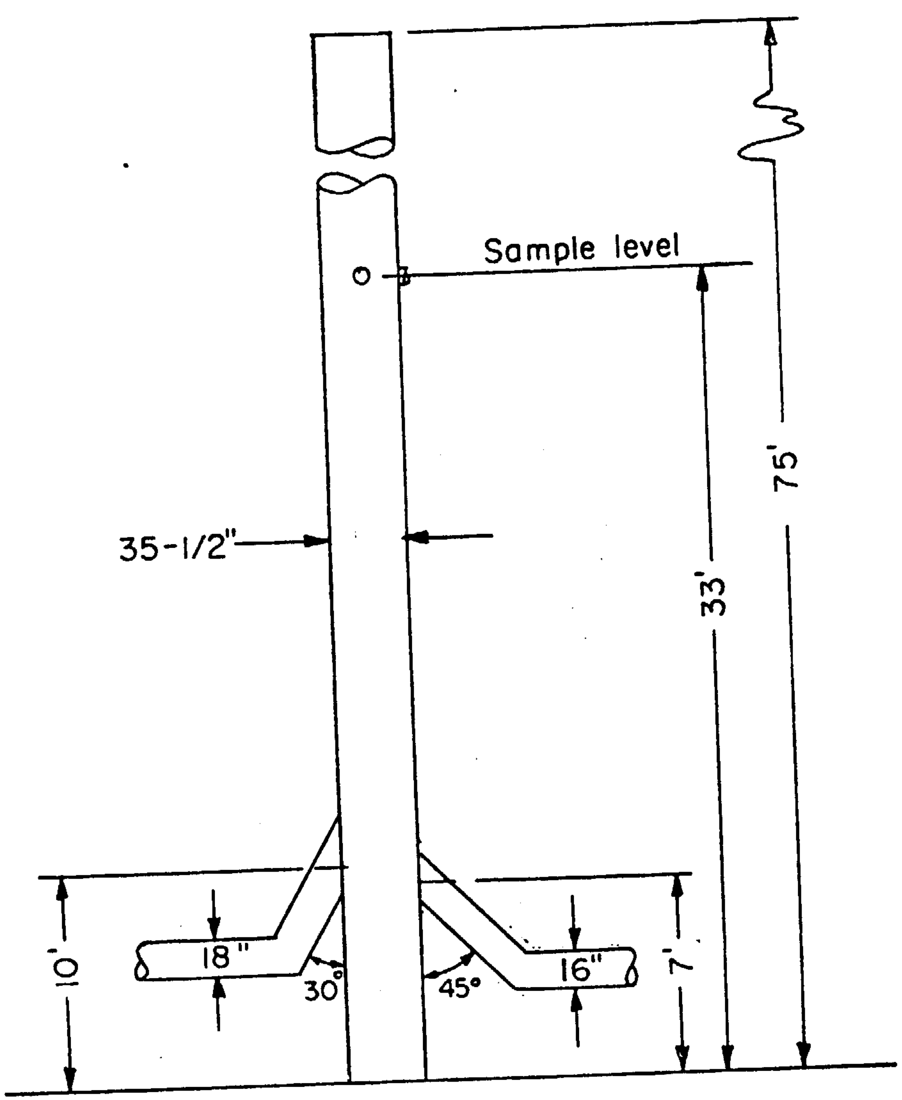
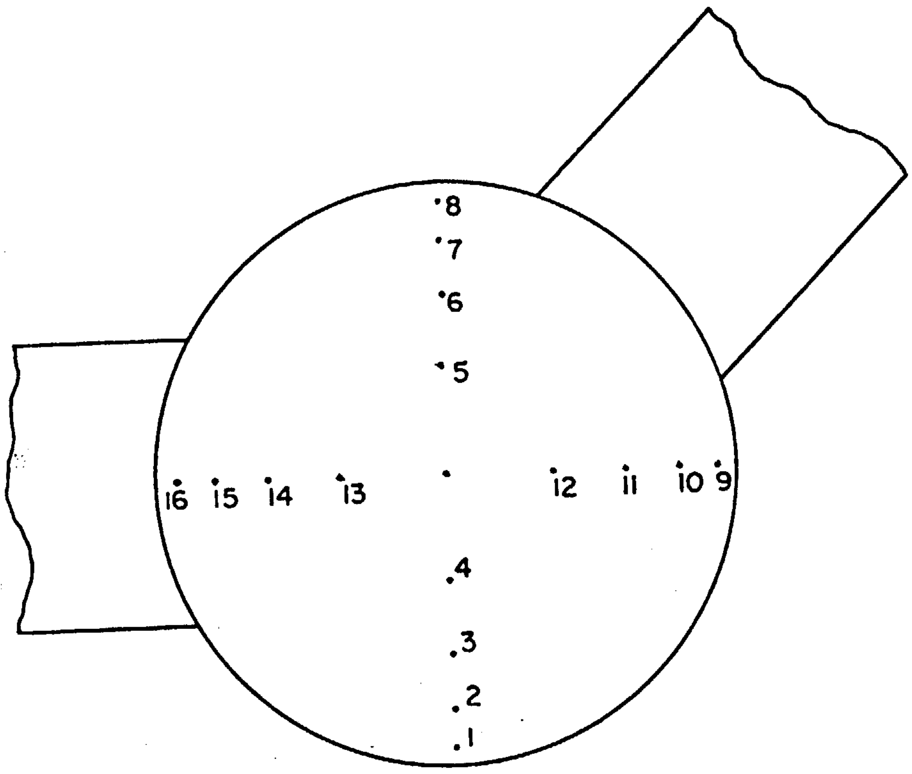


FIGURE 3. VENTURI SCRUBBER





Distance of Sampling Point From Wall

Point	1 9	2 10	3 11	4 12	5 13	6 14	7 15	8 16
Distance, inches	1-$\frac{1}{4}$	3-$\frac{3}{4}$	7	11-$\frac{1}{2}$	24	28-$\frac{3}{4}$	32	34-$\frac{1}{4}$
% of Diam	3.3	10.5	19.4	32.3	67.7	80.6	89.5	96.7

FIGURE 4. LOCATION OF SAMPLING POINTS

PROCESS OPERATION

Oxygen enriched air is blown through the blast furnace at a rate of about 800 cfm and at a water pressure of 1.5 psi. The materials charged to the furnace are burned, reduced, or melted into a lead output, a slag output, and a gas output. Lead is the main product of the plant. Part of the slag is recycled to the blast furnace and the remainder is dumped in a landfill. The exhaust is cleaned in scrubbers and exhausted through a steel stack. While only about 800 cfm of gas passed through the blast furnace, 12,000 cfm was exhausted through the stack because of air inleakage at several points.

Operation of the Blast Furnace

Process air to the blast furnace is furnished by a centrifugal blower at a constant water pressure of 1.5 psi. The volume of air which flows through the furnace is governed by the porosity of the charge in the furnace and plugging of the tuyeres. To increase furnace output and maintain an improved heat balance, the process air is enriched with about three percent oxygen. The oxygen enrichment is manually controlled to maintain the proper furnace temperature as estimated by the operator from the color of burning coke. The furnace was down during Run 1 because of a burned out tuyere although the exhaust fans were operational. The furnace had been placed in operation 1-1/2 hours before the start of Run 2, and during this run combustion air was enriched initially with an estimated 10 percent oxygen and reduced to an estimated 5 percent by the end of the run. No method was available to determine the exact percent of oxygen being added to the process air. Runs 3 and 4 were considered conducted under normal operating conditions.

About 400 pounds of charge was mixed in the skip hoist used for loading the furnace. The proper amounts of limestone, coke, battery plates, cast iron, slag, and sludge were loaded into the hoist as determined by the furnace operator. While no measurements were made on the skip hoist loads, management estimated the weight to be 400 pounds. The charging rate varied and was dependent on the level of material in the furnace. A time log of furnace feed is presented in Appendix C.

Although lead tapping was generally continuous, the rate varied as a function of furnace slag level. Lead flow essentially stops during slag tapping. The lead tapping rate was determined by noting the number of 1500 and 1650 pound ingots filled during the time the stack emission measurements were taken.

Slag was tapped intermittantly at intervals of slightly more than 1 hour, the exact time being determined by the rise of the slag level to the center of the tuyeres. About 2,000 pounds, as estimated by management, can be tapped each time and requires approximately 15 to 20 minutes. The reported slag rate was determined by the number of slag taps during the particulate run. The tuyeres are rodded immediately following the slag tap and the blast air and oxygen are turned off during this operation to prevent dust from being blown into the plant from the open tuyeres. A time log of slag and lead tapping is presented in Appendix C.

Operation of the Air Cleaning System

The gas from the blast furnace passes through a long horizontal duct which is used to cool the gases to about 100 F by natural convection and dilution. About one-half of the dust from the blast furnace settles in this duct and is removed manually during a brief shutdown each Monday and Friday morning.

The gas from the exhaust duct is cleaned further by two venturi scrubbing systems operating in parallel. A flow diagram for the venturi systems is shown in Figure 5. The liquid and gas are intimately mixed in the venturi. The particulates in the gas are trapped in liquid droplets and vapors in the gas condense or are absorbed by the caustic scrubbing liquid.

The liquid-gas mixture passes through a demister where the liquid droplets are separated from the gas that goes to the stack. The liquid containing the dust in suspension flows into a sludge tank. The solid particles settle in the tank and the liquid is decanted off and recycled to the scrubber. Makeup caustic must be added to the settling tank as the caustic is neutralized by absorbing SO_2. The sludge is removed from the settling tank every few weeks and placed in a pit on the plant site to drain. When reasonably dry, it is fed back to the furnace. The plant manager estimated that about 1500 tons of dru sludge, which contains 50-70 percent lead, is produced in ten months of operation. Slightly less than 2 lb/hr of particulates escape the air cleaning system, therefore, the collection efficiency of the air cleaning system is estimated at 99.96 percent for lead since about 0.1 lb/hr is emitted.

SAMPLING AND ANALYTICAL PROCEDURES

The off gases from the lead blast furnace were sampled for particulates, SO_2, CO_2, O_2, and CO using methods described in Appendix D of the Federal Register dated August 17, 1971. The gas was sampled for seven minutes at each point using points recommended for an eight point traverse. About 60 cubic feet of stack gas was sampled during each run using a 0.235-inch-diameter probe tip.

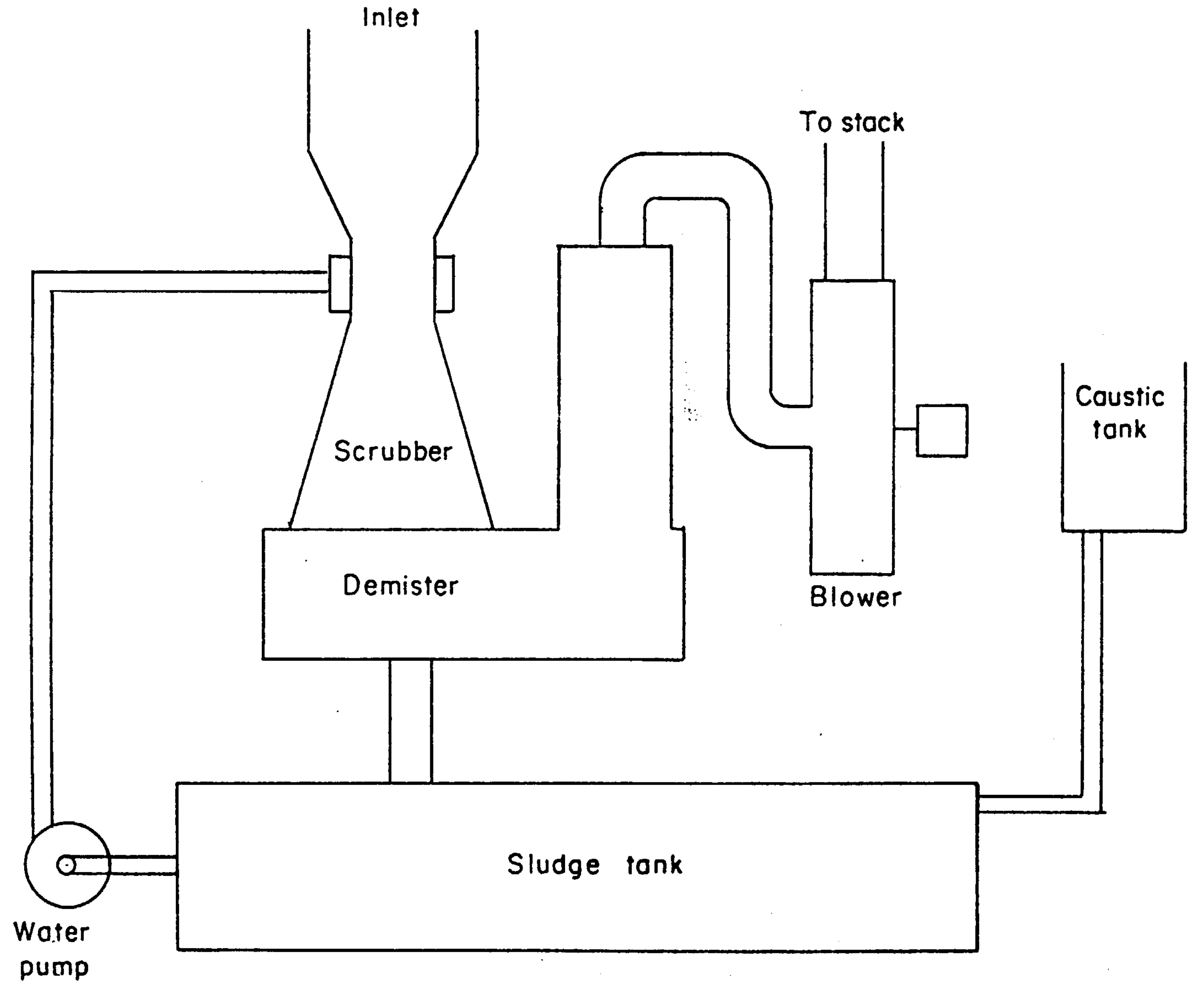


FIGURE 5. VENTURI SCRUBBING SYSTEM

Particulate sample weight was obtained by desiccating the filter paper before and after sampling and measuring the increase in weight from the tare. The probe was washed with acetone and the solution residue evaporated to a constant weight.

The SO_2 and particulate were sampled simultaneously for a period of 1-1/4 hours, during which time a sample volume of about 7.5 cubic feet was collected. The impinger liquid and washings were more than 50 ml and, therefore, the sample was diluted to 100 ml for a convenient value for analysis. Ten ml of the sample was pippeted into an Erlenmeyer flask for analysis and was titrated against 0.01 normal barium perchlorate using a thorin indicator.

Two Orsat samples were taken from the center of the stack simultaneously with the particulate sample. These samples were drawn into a 2-cubic-foot plastic bag over a period of one hour and, after flushing the Orsat with the collected sample, a 100-ml volume was taken and analyzed as follows: the volume was adjusted to 100 ml over acidulated water in the measuring burette; the CO_2 was absorbed with potassium hydroxide solution, and the volume decrease measured; the oxygen was absorbed with alkaline pyrogallol, and the volume decrease measured, and CO was absorbed with acid cuprous chloride solution and after absorbing any acid vapors from the cuprous chloride solution with potassium hydroxide solution, the volume decrease was measured. The gas analysis was repeated using another 100-ml sample from the 2-cubic-foot plastic bag.

APPENDIX A

COMPLETE PARTICULATE RESULTS WITH EXAMPLE CALCULATIONS

APPENDIX A

COMPLETE PARTICULATE RESULTS WITH SAMPLE CALCULATIONS

SOURCE TESTING CLACULATION FORMS

Test No. 2, 3, 4 No. Runs 3

Name of Firm Revere Smelting and Refining Company

Location of Plant Newark, New Jersey

Type of Plant Secondary Lead Blast Furnace

Control Equipment Wet Scrubbers

Sampling Point Locations Stack

Pollutants Sampled Particulates - SO_2

Time of Particulate Test:

Run No. 2 Date 12-8-71 Begin 3:10 pm End 5:39 pm

Run No. 3 Date 12-9-71 Begin 9:50 am End 11:55 am

Run No. 4 Date 12-9-71 Begin 12:58 pm End 3:08 pm

PARTICULATE EMISSION DATA

Run No.	2	3	4
P_b-barometric pressure, in. Hg Absolute	30.18	30.23	30.13
P_m-orifice pressure drop, in. H_2O	0.71	0.75	0.80
V_m-volume of dry gas sampled @ meter conditions, ft^3	70.2	57.3	59.3
T_m-average gas meter temperature, F	66	61	64
$V_{m_{std.}}$ - volume of dry gas sampled @ standard conditions, ft^3, dry	71.4	59.0	60.5
V_w- total H_2O collected, impingers and silical gel., ml.	37.3	21.4	30.0
$V_{w_{gas}}$ - volume of water vapor collected ft^3 @ standard conditions$^{(a)}$	1.8	1.0	1.4
V_{total}- total gas volume, standard conditions, ft^3	73.2	60.0	61.9
Moisture in the stack gas volume, percent	2.4	1.7	2.3
M_d-mole fraction of dry gas	0.98	0.98	0.98

(a) 70 F, 29.92 in. Hg

PARTICULATE EMISSION DATA (Cont'd)

Run No.	2	3	4
CO_2, dry, percent	0.5	0.8	0.8
O_2, dry, percent	20.3	20.2	18.8
CO, dry, percent	0.8	0.3	0.2
N_2, dry, percent	78.4	78.7	80.2
$M\,W_d$ - molecular weight of dry stack gas	28.9	28.9	28.9
M W - molecular weight of stack gas	28.6	28.8	28.6
T_s - stack temperature, F	97	95	93
$\sqrt{\Delta P_s \times (T_s + 460)}$	12.938	13.049	13.300
P_s - stack pressure, in. Hg absolute	30.18	30.23	30.13
V_s - stack velocity @ stack conditions, fpm	1914	1925	1970
A_s - stack area, in^2	972	972	972
Q_s - stack gas volume @ standard conditions,(a) ft^3 dry	12100	13330	12450
T_t - net time of test, min.	120	112	112
D_n - sampling nozzle diameter, in.	0.235	0.235	0.235
Percent I - percent isokinetic	109	95	96
m_f - particulate-probe and filter, mg	91.0	32.3	58.7
m_t - particulate-total, mg	127.3	60.0	92.1
C_{an} - particulate, probe, and filter, gr/scf, dry	0.0196	0.0084	0.0149
C_{ao} - particulate, total, gr/scf	0.0275	0.0157	0.0235

(a) 70 F, 29.92 in. Hg

PARTICULATE EMISSION DATA (Cont'd)

Run	2	3	4
C_{at} - particulate; probe and filter, gr/ft^3 @ stack conditions	0.0184	0.0080	0.0141
C_{au} - particulate; total, gr/ft^3 @ stack conditions	0.0257	0.0149	0.0221
C_{aw} - particulate; probe and filter, lb/hr	2.0354	0.8913	1.6061
C_{ax} - particulate; total, lb/hr	2.8474	1.6556	2.5200
C_p - particulate; probe and filter, lb/ton lead	1.3570	0.7750	1.2849
C_{pt} - particulate; total lb/ton lead	1.8983	1.4397	2.0160
C_{1s} - lead emission; probe and filter, gr/scf	0.00127	0.00061	0.00127
C_{1a} - lead emission; probe and filter, gr/ft^3	0.00119	0.00058	0.00120
C_{1h} - lead emission; probe and filter, lb/hr	0.1320	0.0646	0.1368
C_{1t} - lead emission; probe and filter, lb/ton	0.0880	0.0562	0.1095

EXAMPLE PARTICULATE CALCULATIONS

SAMPLE NUMBER 3

1. Volume of dry gas sampled at standard conditions - 70 F, 29.92 in. Hg, ft^3

$$V_{m_{std}} = \frac{17.7 \times V_m \left(P_b + \frac{P_m}{13.6}\right)}{(T_m + 460)}$$

$$= \frac{17.7 \times 70.2 \left(30.18 + \frac{0.71}{13.6}\right)}{66 + 460} \quad \text{dry}$$

$$= 71.4 \text{ ft}^3$$

2. Volume of water vapor at 70 F and 29.92 in. Hg, ft^3

$$V_{w_{gas}} = 0.0474 \times V_w$$

$$V_{w_{gas}} = 0.0474 \times 37.3$$

$$= 1.8 \text{ ft}^3$$

3. Percent moisture in stack gas

$$\% M = \frac{100 \times V_{w_{gas}}}{V_{m_{std}} + V_{w_{gas}}}$$

$$= \frac{100 \times 1.8}{71.4 + 1.8}$$

$$= 2.4 \text{ percent}$$

4. Mole fraction of dry gas

$$M_d = \frac{100 - \%M}{100}$$

$$= \frac{100 - 2.4}{100}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

5. Average molecular weight of dry stack gas

$$M\ W_d = (CO_2 \times \frac{44}{100}) + (O_2 \times \frac{32}{100}) + (N_2 + CO) \times \frac{28}{100}$$

$$= (0.5 \times 0.44) + (20.3 \times 0.32) + 79.2 \times 0.28)$$

$$= 28.9$$

6. Molecular weight of stack gas

$$M\ W = M\ W_d \times M_d + 18\ (1 - M_d)$$

$$= 28.9 \times 0.98 + 18\ (1 - 0.98)$$

$$= 28.6$$

7. Stack velocity @ stack conditions, fpm

$$V_s = 4350 \times \sqrt{\triangle P_s \times (T_s + 460)} \quad \left(\frac{1}{P_s \times M\ W}\right)^{1/2} \text{ fpm}$$

$$= 4350 \times 12.938 \quad \left[\frac{1}{30.18 \times 28.6}\right]^{1/2}$$

$$= 1914 \text{ fpm}$$

8. Stack gas volume at standard conditions, scfm

$$Q_s = \frac{0.123 \times V_s \times A_s \times P_s}{(T_s + 460)} M_d \text{ scfm}$$

$$= \frac{0.123 \times 1914 \times 972 \times 30.18 \times 0.98}{97 + 460}$$

$$= 12{,}100 \text{ scfm}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

9. Sampling velocity, percent of isokinetic

$$\%I = \frac{1032 \times (Ts + 460) \times V_{m_{std}}}{V_s \times T_t \times P_s \times M_d \times (D_n)^2}$$

$$= \frac{1032 \times (97 + 460) \times 71.4}{1914 \times 120 \times 30.18 \times 0.98 \times (0.235)^2}$$

$$= 109 \text{ percent}$$

10. Percent excess air at sampling point

$$\%\ EA = \frac{100 \times \%\ O_2}{(0.266 \times \%\ N_2) - \%\ O_2}$$

$$= \frac{100 \times 20.3}{(0.266 \times 78.4) - 20.3}$$

$$= 3690 \text{ percent}$$

11. Particulate - probe and filter, gr/scf

$$C_{an} = 0.0154 \times \frac{m_f}{V_{m_{std}}}$$

$$= 0.0154 \times \frac{91.0}{71.4}$$

$$= 0.0196 \text{ gr/scf}$$

12. Particulate - total, gr/scf

$$C_{ao} = 1.54 \times 10^{-2} \left(\frac{m_t}{V_{m_{std}}}\right)$$

$$= 1.54 \times 10^{-2}\ \frac{127.3}{71.4}$$

$$= 0.0274 \text{ gr/scf}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

13. Particulate - probe and filter, gr/ft^3 at stack conditions

$$C_{at} = \frac{17.7 \times C_{an} \times P_s M_d}{(T_s + 460)}$$

$$= \frac{17.7 \times 0.0196 \times 30.18 \times 0.98}{(97 + 460)}$$

$$= 0.0184 \text{ gr/ft}^3$$

14. Particulate, total, gr/ft^3 at stack conditions

$$C_{au} = \frac{17.7\ C_{ao}\ P_s\ M_d}{(T_s + 460)}$$

$$= \frac{17.7 \times 0.0275 \times 30.18 \times 0.98}{(97 + 460)}$$

$$= 0.0257 \text{ gr/ft}^3$$

15. Particulate - probe and filter

$$C_{aw} = 0.00857 \times C_{an} \times Q_s \text{ lb/hr}$$

$$= 0.00857 \times 0.0196 \times 12,100$$

$$= 2.0354 \text{ lb/hr}$$

16. Particulate - total, lb/hr

$$C_{ax} = 8.57 \times 10^{-3}\ C_{ao}\ Q_s$$

$$= 8.57 \times 10^{-3} \times 0.0275 \times 12,100$$

$$= 2.8474 \text{ lb/hr}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

17. Particulate; probe and filter, lb/ton lead

$$C_p = C_{aw}/R$$
$$= 2.0354/1.5$$
$$= 1.357 \text{ lb/ton lead}$$

18. Particulate; total lb/ton lead

$$C_{pt} = C_{ax}/R$$
$$= 2.8474/1.5$$
$$= 1.8983 \text{ lb/ton lead}$$

19. Lead emission; probe and filter, gr/scf

$$C_{ls} = 0.0154 \times \frac{M_l}{V_{m\ std}}$$
$$= 0.0154 \times \frac{5.9}{71.4}$$
$$= 0.00127 \text{ gr/scf}$$

20. Lead emission; probe and filter, gr/scf

$$C_{la} = \frac{17.7 \times C_{ls} \times P_s \times M_d}{(T_s + 960)}$$
$$= \frac{17.7 \times 0.00127 \times 30.18 \times 0.98}{(97 + 460)}$$
$$= 0.00119 \text{ gr/scf}$$

21. Lead emission; probe and filter, lb/hr

$$C_{lh} = 8.57 \times 10^{-3} \times C_{ls} \times Q_s$$
$$= 8.57 \times 10^{-3} \times 0.00127 \times 12{,}100$$
$$= 0.1320 \text{ lb/hr}$$

22. Lead emission; probe and filter, lb/ton lead

$$C_{lt} = C_{lh}/R$$
$$= 0.1320/1.5$$
$$= 0.0880 \text{ lb/ton lead}$$

APPENDIX B

COMPLETE GASEOUS RESULTS WITH EXAMPLE CALCULATION

APPENDIX B

COMPLETE GASEOUS RESULTS WITH EXAMPLE CALCULATIONS

SO_2 EMISSION DATA

Run Number	1	2	3	4
Date (1971)	12-8	12-8	12-9	12-9
T_m - average gas meter temperature, F	56	62	61	63
P_b - barometer pressure, inches Hg absolute	30.2	30.2	30.2	30.1
V_m - volume of dry gas sampled at meter conditions, ft^3	4.9	6.7	9.9	10.6
V_m standard, ft^3	5.1	6.9	10.3	10.8
V_t - volume Ba $(ClO_4)_2$, ml	0.01	<0.01	<0.01	0.02
V_{tb}- volume Ba $(ClO_4)_2$ blank ml	0	0	0	0
N - normality of Ba $(ClO_4)_2$	0.01	0.01	0.01	0.01
V_{soln} - volume of SO_2 soln, ml	50	100	100	100
V_a - volume aliquot, ml	10	10	10	10
C_{SO_2} - lb/feet^3 x 10^{-5}	0.001	0	0	0.001
C_{SO_2} - ppm (dry)	0.1	0	0	0.1
W_{SO_2} - lb/hour	0	0	0	0

SO_2 EXAMPLE CALCULATIONS

Run 4

1. $$V_{m_{std}} = V_m \frac{(T_{std})\ (P_{Bar})}{(T_m)\ \ (P_{std})}$$

$$= 10.6 \times \frac{530}{523} \times \frac{30.1}{29.9}$$

$$= 10.8\ ft^3$$

2. $$C_{SO_2} = \frac{7.05 \times 10^{-5}\ (V_t - V_{tb})\ (N)\ (V_{soln})}{(V_{m_{std}})\ (V_a)} \quad lb/ft^3$$

$$= \frac{7.05 \times 10^{-5} \times 0.02 \times 0.01 \times 100}{(10.8)\ (10)}$$

$$= 0.001 \times 10^{-5}\ \ lb/ft^3$$

3. $$C_{SO_2}\ ppm = 6.05 \times 10^6 \times C_{SO_2}$$

$$= 6.05 \times 10^6 \times 0.001 \times 10^{-5}$$

$$= 0.1\ ppm$$

4. $$W_{SO_2} = C_{SO_2} \times Q_s \times 60$$

$$= 0.001 \times 10^{-5} \times 12{,}540$$

$$= 0\ lb/hr$$

APPENDIX C

FIELD DATA

Note: Original Handwritten Data on file at Battelle's Columbus Laboratories.

APPENDIX C

PRELIMINARY FIELD DATA

STACK GEOMETRY

Plant - Revere Smelting and Refining Company
Newark, New Jersey

Location - Stack - Wall thickness, inches, 1/4

Date - December 7, 1971 - Inside diameter of stack, inches, 35.5
Stack area, 989 inches^2, 6.87 feet^2

Point	Percent Diameter for Circular Stack	Distance from Outside of Sample Port, inches,
1	3.3	1-1/4 + 3-3/4 = 5
2	10.5	3-3/4 + 3-3/4 = 7-1/2
3	19.4	7 + 3-3/4 = 10-3/4
4	32.3	11-1/2 + 3-3/4 = 15-1/4
5	67.7	24 + 3-3/4 = 27-3/4
6	80.6	28-3/4 + 3-3/4 = 32-1/2
7	89.5	32 + 3-3/4 = 34-1/4
8	96.7	34-1/4 + 3-3/4 = 38

PARTICULATE CLEANUP SHEET

Plant Revere Smelting and Refining Company

Operators P.R. Webb, B.E. Campbell

Run No.	2	3	4
Impinger water volume, ml			
Final	224	212	216
Initial	200	200	200
Collected	24	12	16
Drierite, weight, gm			
Final	514.6	523.9	530.5
Initial	501.3	514.5	516.5
Gain	13.3	9.4	14.0
Total moisture, gm plus ml	37.3	21.4	30.0
Probe, acetone wash residue, mg(a)	4.0	0	0
Filter particulate weight, mg			
Final	362.3	326.6	356.5
Tare	275.3	294.3	297.8
Gain	87.0	32.3	58.7
Total front half, mg	91.0	32.3	58.7
Weight residue from chloroform-ether extract, mg			
Impinger water	19.3	13.5	14.4
Impinger water wash	2.9	3.0	3.5
Total	21.2	16.5	17.9
Weight residue from aqueous phase, mg(a)			
Impinger water	10.4	8.4	12.4
Impinger water wash	0	0	0
Total	10.4	8.4	12.4
Weight residue from acetone wash, mg(a)	4.7	2.8	3.1
Total, back half residue, mg	36.3	27.7	33.4
Total front half plus back half, mg	127.3	60.0	92.1

(a) Acetone and water blank subtracted; acetone blank = 0.035 mg/ml,
water blank = 0.022 mg/ml

PARTICULATE FIELD DATA

Plant Revere Smelting and Refining Company
Date 12/8/71
Run No. 2
Operator P.R. Webb, B.E. Campbell
Sample Box No. ______
Meter Box No. ______
Meter ΔH @ 1.84
C Factor 1.0

Ambient Temperature, F 51
Barometric Pressure, in. Hg 30.18
Assumed Moisture, Percent 10
Heater Box Setting, F ______
Probe Tip Diameter, Inches 0.235
Probe Length 5 feet
Probe Heater Setting ______
Average ΔP ______ Average ΔH ______

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H_2O ΔP	Orifice ΔH, In. H_2O		Dry Gas Temp., F		Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press., In. Hg	Stack Temp., F
				Desired	Actual	Inlet	Outlet					
16	3:10 pm	386.816	0.30	--	0.75	58	55	5	255	60	(a)	95
15	3:18 pm	392.0	0.33	--	0.79	59	55	5	260	58	(a)	95
14	3:26 pm	395.0	0.32	--	0.80	61	56	5	260	57	(a)	95
13	3:34 pm	399.2	0.32	--	0.80	63	59	5	249	58	(a)	95
12	3:42 pm	403.6	0.35	--	0.85	64	62	5	242	58	(a)	95
11	3:50 pm	407.7	0.34	--	0.83	69	63	5	240	59	(a)	95
10	3:58 pm	413.5	0.30	--	0.75	70	65	5	238	55	(a)	95
9(b)	4:06 pm	418.8	0.22	--	0.46	70	65	5	238	52	(a)	95
8	4:35 pm	422.9	0.40	--	0.90	64	64	5	235	46	(a)	97
7	4:43 pm	429.2	0.40	--	0.90	70	66	5	242	49	(a)	97
6	4:51 pm	433.5	0.40	--	0.90(c)	72	66	5	242	50	(a)	97
5	4:59 pm	438.8	---	--	---	74	68	6	248	52	(a)	105
4	5:07 pm	443.5	0.28	--	0.62	75	68	5	250	50	(a)	99
3	5:15 pm	447.3	0.24	--	0.52	74	68	5	250	50	(a)	104
2	5:23 pm	450.8	0.23	--	0.54	72	68	5	250	50	(a)	95
1	5:31 pm	454.0	0.15	--	0.32	72	69	5	255	50	(a)	96
Off	5:39 pm	457.										
Average			0.32		0.64	68	63					97
Total	120 min	70.2										

(a) Atmospheric (b) Test stopped (c) Cleaned pitot tube

PARTICULATE FIELD DATA

Plant Revere Smelting and Refining Company	Ambient Temperature, F 52
Date 12-9-71	Barometric Pressure, in. Hg 30.23
Run No. 3	Assumed Moisture, Percent 10
Operator P.R. Webb, B.E. Campbell	Heater Box Setting, F 255
Sample Box No. 1	Probe Tip Diameter, Inches 0.235
Meter Box No. 1	Probe Length 5 feet
Meter ΔH @ 1.84	Probe Heater Setting
C Factor 10	Average ΔP ______ Average ΔH ______

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H_2O ΔP	Orifice ΔH, In. H_2O		Dry Gas Temp., F		Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press., In. Hg	Stack Temp., F
				Desired	Actual	Inlet	Outlet					
8	9:50 am	457.1083	0.28	--	0.71	58	46	4	220	51	(a)	95
7	9:57 am	460.5	0.40	--	1.0	54	48	5	233	51	(a)	96
6	10:04 am	464.1	0.40	--	1.0	61	51	5	240	53	(a)	96
5	10:11 am	468.8	0.38	--	0.96	65	53	5	240	55	(a)	96
4	10:18 am	473.1	0.33	--	0.77	68	56	5	250	56	(a)	96
3	10:25 am	476.5	0.30	--	0.72	69	59	4	255	56	(a)	96
2	10:32 am	480.0	0.28	--	0.65	71	62	3	260	56	(a)	96
1(b)	10:39 am	483.2	0.18	--	0.41	70	64	2	260	57	(a)	93
16	10:59 am	486.2	0.32	--	0.76	62	62	4.5	252	52	(a)	95
15	11:06 am	489.7	0.32	--	0.76	64	60	4.5	262	53	(a)	95
14	11:13 am	493.4	0.32	--	0.76	64	58	4.5	255	54	(a)	93
13	11:20 am	497.0	0.32	--	0.76	64	58	4.5	260	54	(a)	93
12	11:27 am	500.9	0.36	--	0.86	63	58	5.0	264	55	(a)	93
11	11:34 am	505.0	0.34	--	0.80	65	59	4.5	266	55	(a)	93
10	11:41 am	508.0	0.26	--	0.60	79	62	4.	266	57	(a)	93
	11:48 am	512.0	0.18	--	0.45	68	60	4.	260	59	(a)	93
Off	11:55 am	514.										
Average			0.31			65	57					95
Total	112 min.	57.5										

(a) Atmospheric (b) Stopped at 10:46 am. Comments: 7 minutes/point. Gas b [illegible] arted 10:18 am, ended 10:46 am.

PARTICULATE FIELD DATA

lant Revere Smelting and Refining Company
ate 12-9-71
un No. 4
perator P.R. Webb, B.E. Campbell
ample Box No.
eter Box No.
eter ΔH @ 1.84
Factor 1.0

Ambient Temperature, F 51
Barometric Pressure, in. Hg 30.13
Assumed Moisture, Percent 10
Heater Box Setting, F
Probe Tip Diameter, Inches 0.235
Probe Length 5 feet
Probe Heater Setting
Average ΔP Average ΔH

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H_2O ΔP	Orifice ΔH, In. H_2O		Dry Gas Temp., F		Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press, In. Hg	Stack Temp., F
				Desired	Actual	Inlet	Outlet					
16	12:58 pm	515.0	0.32	--	0.77	52	51	5	175	60	(a)	90
15	1:05 pm	518.6	0.34	--	0.81	56	54	5.5	250	55	(a)	93
14	1:12 pm	522.2	0.34	--	0.81	63	55	5.5	242	60	(a)	93
13	1:19 pm	526.0	0.34	--	0.81	63	57	5.5	250	63	(a)	93
12	1:26 pm	529.7	0.36	--	0.88	64	57	5.5	255	62	(a)	93
11	1:33 pm	533.6	0.36	--	0.88	66	58	5.5	255	62	(a)	93
10	1:40 pm	537.9	0.32	--	0.76	68	60	5.5	261	62	(a)	93
9(b)	1:47 pm	541.0	0.18	--	0.40	67	61	--	260	60	(a)	93
8	2:12 pm	544.8	0.39	--	0.98	59	59	--	244	48	(a)	93
7	2:19 pm	549.1	0.39	--	0.98	68	62	7.0	254	54	(a)	95
6	2:26 pm	552.6	0.40	--	1.0	72	64	7.0	254	57	(a)	95
5	2:33 pm	556.7	0.36	--	0.97(c)	74	66	7.4	254	57	(a)	95
4	2:40 pm	560.8	0.32	--	0.80	75	68	6.2	250	58	(a)	95
3	2:47 pm	564.7	0.30	--	0.76	75	69	6.0	250	57	(a)	94
2	2:54 pm	568.7	0.27	--	0.65	75	69	6.0	249	54	(a)	93
1	3:04 pm	571.7	0.20	--	0.50	74	68	4.9	248	52	(a)	92
Off	3:01 pm	574.3										
Average			0.32			67	61					93
Total	112 min.	59.3										

(a) Atmospheric. (b) Stopped at 1:54 pm. (c) Cleaned pitot tube.

GAS SAMPLING FIELD DATA

Date 12-8-71

Material Sampled for SO_2

Barometric Pressure 30.2 in. Hg

Ambient Temperature 51 F

Run Number 1

Power Stat Setting Heated

Filter Used: Yes X No ___

Operator W.C. Baytos, H. Hess

Plant Revere Smelting and Refining Co.

Comments: Blast furnace not operating..

Probe temperature, 100 F.
Recovered 40 ml - diluted to 50 ml.

Titration		
Aliquot, ml	Titer, ml	0.01 N $Ba(ClO_4)_2$
10.0	0.01	
blank	0	

Clock Time	Meter (ft^3)	Flow Meter Setting (CFH)	Meter Temperature, F
2:00 pm	450.9	6.0	50
2:05 pm	451.6	6.0	--
2:15 pm	452.3	6.0	--
2:25 pm	453.3	6.0	--
2:30 pm	453.7	6.0	51
2:50 pm	454.2	6.0	--
3:10 pm	455.0	6.0	--
3:15 pm	455.2	6.0	--
Total	4.3		

GAS SAMPLING FIELD DATA

Date 12-8-71
Material Sampled for SO_2
Barometric Pressure 30.2 in. Hg
Ambient Temperature 52 F
Run Number 2
Power Stat Setting Heated
Filter Used: Yes X No ___
Operator W.C. Baytos, H. Hess

Plant Revere Smelting and Refining Co.

Comments:

Titration		
Aliquot, ml	Titer, ml	
10.0	0.01	0.01 N $Ba(ClO_4)_2$
10.0	<0.01	
blank	0	

Probe temperature, 104 F.
Recovered 84 ml - diluted to 100 ml.

Clock Time	Meter (ft^3)	Flow Meter Setting (CFH)	Meter Temperature, F
4:15 pm	77.7	6.0	60
4:20 pm	78.3	6.0	60
4:25 pm	78.7	6.0	60
4:30 pm	--	0	--
4:40 pm	--	0	60
4:45 pm	79.7	0	59
4:50 pm	80.2	6.0	59
5:00 pm	81.1	6.0	61
5:10 pm	82.0	6.0	63
5:15 pm	82.5	6.0	64
5:20 pm	83.1	6.0	65
5:25 pm	83.7	6.0	65
5:30 pm	84.1	6.0	65
5:35 pm	84.4	6.0	65
Total	6.7		

GAS SAMPLING FIELD DATA

Date 12-9-71

Material Sampled for SO_2

Barometric Pressure 30.1 in. Hg

Ambient Temperature 53 F

Run Number 4

Power Stat Setting Heated

Filter Used: Yes X No ___

Operator W.C. Baytos, H. Hess

Plant Revere Smelting and Refining CO.

Comments: Probe temperature, 180 F.
Recovered ml - diluted to 100 ml.

Titration

Aliquot, ml	Titer, ml	
10.0	0.02	0.01 N $Ba(ClO_4)_2$
0	4.58	
blank	0	

Clock Time	Meter (ft^3)	Flow Meter Setting (CFH)	Meter Temperature, F
12:50 pm	95.4	8.0	59
1:00 pm	96.5	7.5	59
1:10 pm	97.2	8.0	60
1:20 pm	98.2	7.5	60
1:30 pm	99.3	8.5	60
1:40 pm	100.2	8.5	64
1:50 pm	101.3	8.0	63
2:00 pm	102.0	--	63
2:10 pm	102.7	0	65
2:20 pm	102.8	--	65
2:30 pm	103.7	--	65
2:40 pm	104.5	7.0	65
2:50 pm	105.3	--	65
3:00 pm	106.0	7.0	65
Total	10.6		

GAS SAMPLING FIELD DATA

Date 12-9-71

Material Sampled for SO_2

Barometric Pressure 30.2 in. Hg

Ambient Temperature 54 F

Run Number 3

Power Stat Setting Heated

Filter Used: Yes X No ___

Operator W.C. Baytos, H. Hess

Plant Revere Smelting and Refining Co.

Comments: Probe temperature, 104 F.
Recovered 61 ml - diluted to 100 ml

Titration

Aliquot, ml	Titer, ml	
10.0	<0.01	0.01 N $Ba(ClO_4)_2$
blank	0	

Clock Time	Meter (ft^3)	Flow Meter Setting (CFH)	Meter Temperature, F
9:40 am	85.4	8.0	51
9:50 am	86.9	8.0	52
10:00 am	87.8	8.0	56
10:10 am	88.8	8.0	59
10:20 am	89.3	8.0	61
10:30 am	89.8	8.0	62
10:40 am	90.6	8.0	63
10:50 am	91.1	0	63
11:00 am	91.2	8.0	63
11:10 am	92.5	8.0	63
11:20 am	93.3	8.0	63
11:30 am	94.0	8.0	63
11:40 am	94.7	8.0	63
11:50 am	95.3	8.0	63
Total	9.9	8.0	63

ORSAT GAS COMPOSITION

Plant Revere Smelting and Refining Company

Run	Date	Time	CO_2, Percent	O_2, Percent	CO, Percent	N_2, Percent
2	12/8/71	4:30-5:30 pm	0.5	20.0	< 0.1	79.5
			0.5	20.4	1.1	78.0
			0.5	20.3	1.2	78.0
		Average -	0.5	20.2	0.8	78.5
3	12/9/71	10:18-10:46 am	0.8	20.0	< 0.1	79.2
			0.8	20.4	0.6	78.2
		Average -	0.8	20.2	0.3	78.7
4	12/9/71	1:00-1:40 pm	0.8	18.8	0.2	80.2

APPENDIX D

STANDARD SAMPLING PROCEDURES

APPENDIX D

STANDARD SAMPLING PROCEDURES

The sampling procedures, as described in the August 17, 1971 issue of the Federal Register, reproduced as a part of this Appendix, have been used as a reference for the source emission test conducted at Revere Smelting and Refining Company, Newark, New Jersey.

After selecting the appropriate stack sampling location, number and position of sampling points as described in Method 1, a preliminary traverse of the stack was completed to determine the average Δp and average gas temperature. These data were then used, with the aid of a nomograph, to determine the appropriate nozzle diameter which would allow isokinetic sampling at an air flow rate consistent with proper sampling train operation.

The sample train was prepared for operation as outlined in Method 5. The glass filter used for particulate collection was desiccated for 24 hours and the tare weight determined. Two hundred grams of indicating drierite, which had been stored in a sealed container, was placed in one of the three modified impingers.

One hundred ml of distilled water was placed in each of two impingers. The sample train was then assembled and leak checked to within the specified tolerances.

During the sampling period, isokinetic flow was maintained by adjusting the sampling flow rate to compensate for stack Δp and temperature variations. Appendix D is a record of the field data for the three tests completed at the Revere Smelting & Refinery.

After the completion of each test and the recording of the appropriate field data, the sampling train was removed from the sampling platform and cleaned. The probe assembly was removed from the sampling train; the ends were plugged and the probe allowed to cool. The Pyrex tube and stainless steel nozzle were then thoroughly cleaned with a nylon brush and rinsed with reagent-grade acetone into a precleaned screw-cap glass container. The glass filter was removed from its holder and placed into an appropriate container. The side of the glass filter holder facing the probe was then rinsed with reagent-grade acetone and emptied into the probe-acetone-wash container. The weight of acetone wash residue and the net filter weight comprise the "front half" of the collected sample.

The water from the first three impingers was measured to determine the volume increase and then poured into precleaned screw-cap glass containers. The weight gain from the water volume increase plus the weight increase of the drierite was used to determine the stack gas moisture content. All of the glassware from the back half of the filter holder, up to, but not including the drierite container was rinsed with distilled water and the rinse water poured into a precleaned screw-cap glass container. This same glassware was then rinsed with reagent-grade acetone and the acetone rinse poured into a precleaned screw-cap glass container. These three solutions were then extracted and evaporated as outlined in the Federal Register and the weight residue determined. These weights comprise the "back half" of the collected sample.

Samples of the water and acetone used to clean the glassware were taken from each container that was used in the field to determine the amount of residue from blank water and blank acetone. These values were subtracted from the sample data to obtain net values.

The sampling procedures are those specified in the Federal Register, Volume 36, No. 159, dated August 17, 1971.

PROPOSED RULE MAKING

15708

APPENDIX—TEST METHODS

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

New Source Performance Standards. 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 a 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.

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 four, 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 two perpendicular diameters according to Figure 1-2 and Table 1-1.

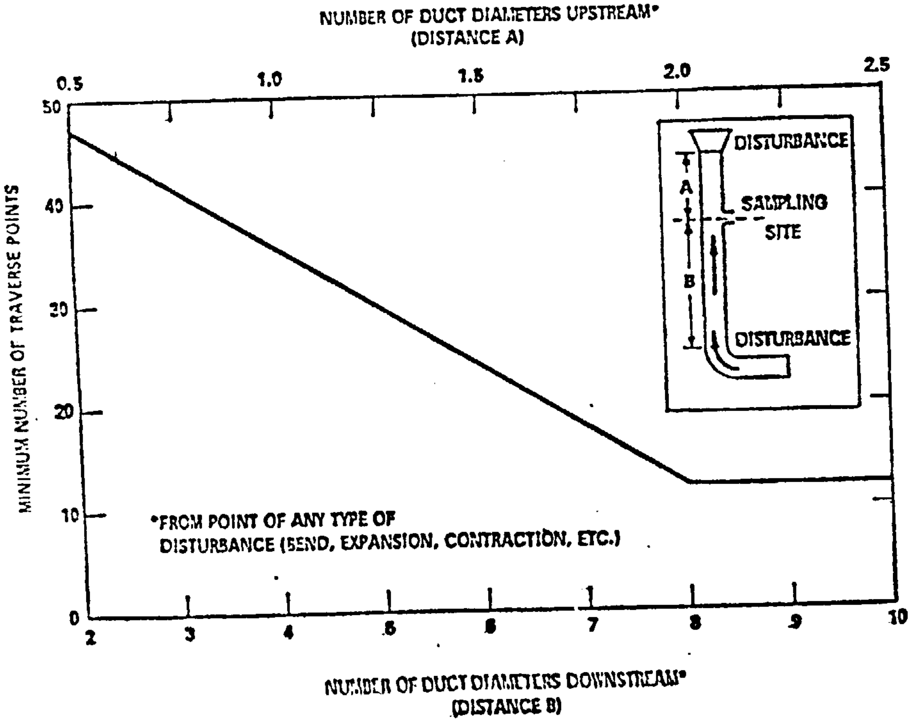


Figure 1-1. Minimum number of traverse points.

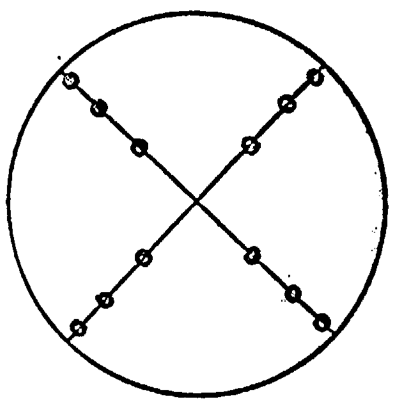


Figure 1-2. Cross section of circular stack showing location of traverse points on perpendicular diameters.

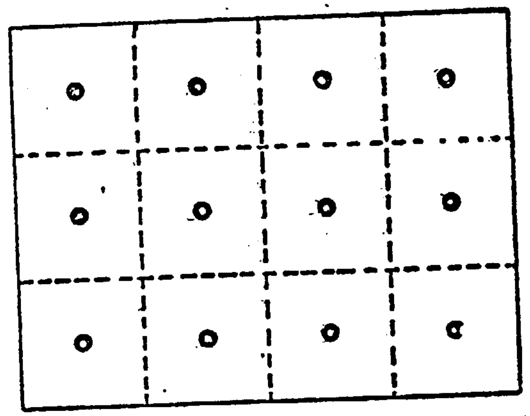


Figure 1-3. Cross section of rectangular stack divided into 12 equal areas, with traverse points at centroid of each area.

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									
	6	8	10	12	14	16	18	20	22	24
1	4.4	3.3	2.5	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	14.7	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3	29.5	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4	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	26.9	22.0	18.8	16.5	14.6	13.2
7		89.5	77.4	64.5	36.6	28.3	23.6	20.4	18.0	16.1
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	38.2	30.6	26.1	23.0
10			97.5	88.2	79.9	71.7	61.8	38.8	31.5	27.2
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

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, 1957.

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

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

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

METHOD 2—DETERMINATION OF STACK GAS VELOCITY (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 New Source Performance Standards. Being a directional instrument, a pitot tube should not be used in the case of nondirectional flow.

2. *Apparatus.*

2.1 Pitot tube—Type S (Figure 2-1), or equivalent.

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

2.3. Temperature gauge—Thermocouples, bimetallic thermometers, liquid filled systems, or equivalent, to measure stack temperature to within 1.5 percent 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 at the traverse points specified by Method 1.

3.2 Measure the temperature of the stack gas. If the total temperature variation with time is less than 50° F., a point measurement will suffice. Otherwise, conduct a temperature traverse.

3.3 Measure the static pressure in the stack.

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

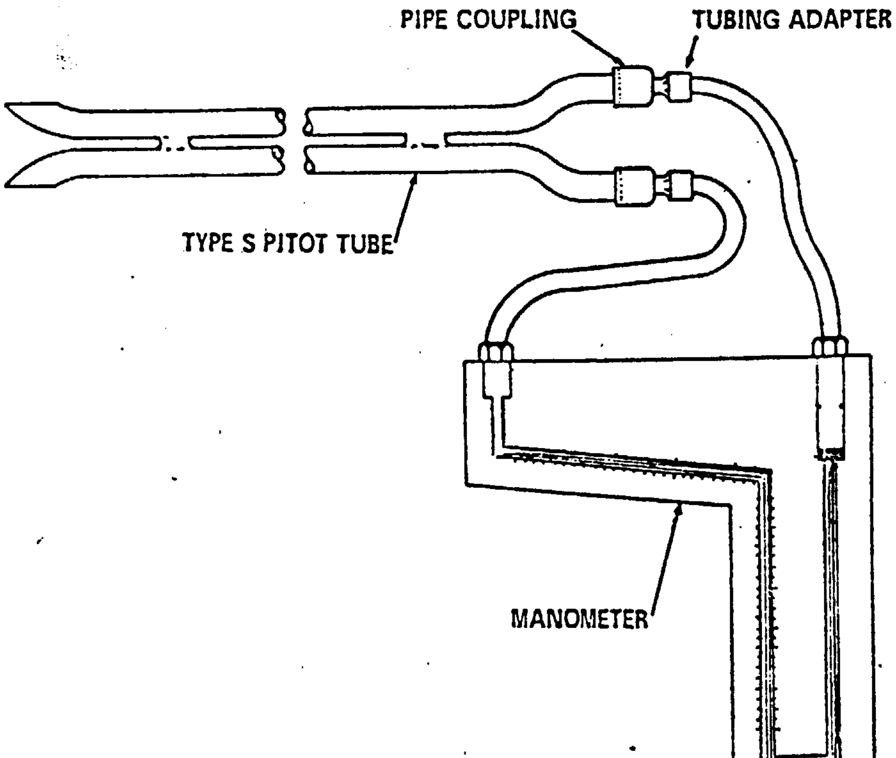


Figure 2-1. Pitot tube - manometer assembly.

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. The velocity of the flowing gas stream should be within the normal working range.

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.

$$V_s=K_pC_p\sqrt{\frac{T_s\Delta_p}{P_sM_s}} \quad \text{equation 2-2}$$

where:

V_s=Stack gas velocity, feet per second (f.p.s.).

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

C_p=Pitot tube coefficient, dimensionless.

T_s=Absolute stack gas temperature, °R.

Δ_p=Velocity head of stack gas, in H_2O (see fig. 2-2).

P_s=Absolute stack gas pressure, in Hg.

M_s=Molecular weight of stack gas, lb./lb.-mole.

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.

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.

6. *References.*

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

Perry, J. H. Chemical Engineers' Handbook. McGraw-Hill Book Co., Inc., New York, 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, 1971. ASTM Designation D-2928-71.

Vennard, J. K. Elementary Fluid Mechanics. John Wiley and Sons, Inc., New York, 1947.

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

1. *Principle and applicability.*

1.1 Principle. An integrated or gra sample is extracted from a sampling and analyzed for its components usi Orsat analyzer.

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

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.

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—To remove any excess moisture.

2.2.3 Needle valve—To adjust flow rate.

3.2.4 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 c.f.m.

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.

3. *Procedure.*

3.1 Grab sampling.

3.1.1 Set up the equipment as shown in Figure 3-1. Place the probe in the stack at a sampling point and purge the sampling line.

---

[1] Trade name.

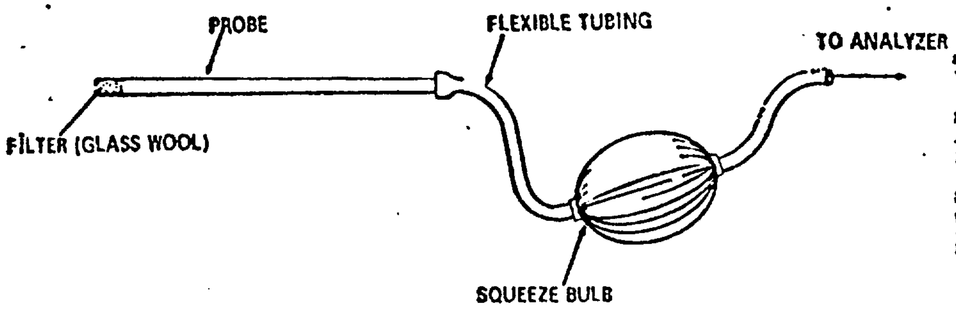


Figure 3-1. Grab-sampling train.

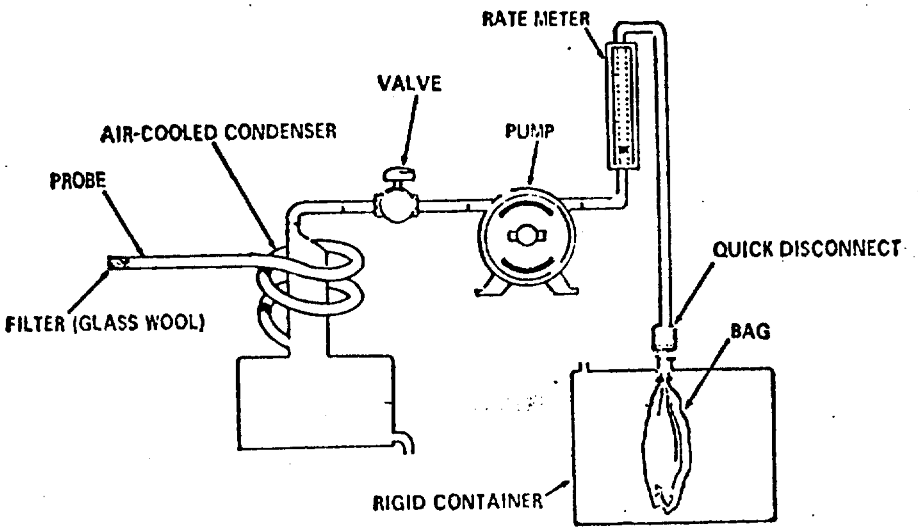


Figure 3-2. Integrated gas - sampling train.

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 gas 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 10 passes are necessary, replace the absorbing solution.

3.3.2 For integrated sampling, repeat the analysis until three consecutive runs 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 result to the nearest 0.1 percent 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 percent excess air.

$$\%EA = \frac{(\%O_2) - 0.5(\%CO)}{0.264(\%N_2) - (\%O_2) + 0.5(\%CO)} \times 100$$

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.

$$M_d = 0.44(\%CO_2) + 0.32(\%O_2) + 0.28(\%N_2 + \%CO)$$

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 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. 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 gravimetrically.

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.[2]

Other methods such as drying tubes, wet bulb-dry bulb techniques, and volumetric condensation techniques may be used subject to the approval of the Administrator.

2. *Apparatus.*

2.1 Probe—Stainless steel or Pyrex[1] 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—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 percent of the total sample volume.

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

2.9 Balance—Capable of measuring to the nearest 0.1 g.

2.10 Barometer—Sufficient to read to within 0.1 in. 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 about 5 ml. distilled water in each impinger and weigh the impinger and contents to the nearest 0.1 g. 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 percent 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 not to exceed 0.075 c.f.m. Continue sampling until the dry gas meter registers 1 cu. ft. or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter reading as required by Figure 4-2.

3.3 After collecting the sample, weigh the impingers and their contents again to the nearest 0.1 g.

---

[1] Trade name.

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

4. *Calculations.*

4.1 Volume of water collected.

$$V_{wc}=\frac{(W_f-W_i)RT_{std}}{P_{std}M_w}=\left(0.0474\frac{\text{ft.}^3}{\text{g.}}\right)(W_f-W_i)$$

equation 4-1

where:

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

W_f=Final weight of impingers and contents, g.

W_i=Initial weight of impingers and contents, g.

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

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

P_{std}=Pressure at standard conditions, 29.92 in. Hg.

M_w=Molecular weight of water, 18 lb./lb. mole.

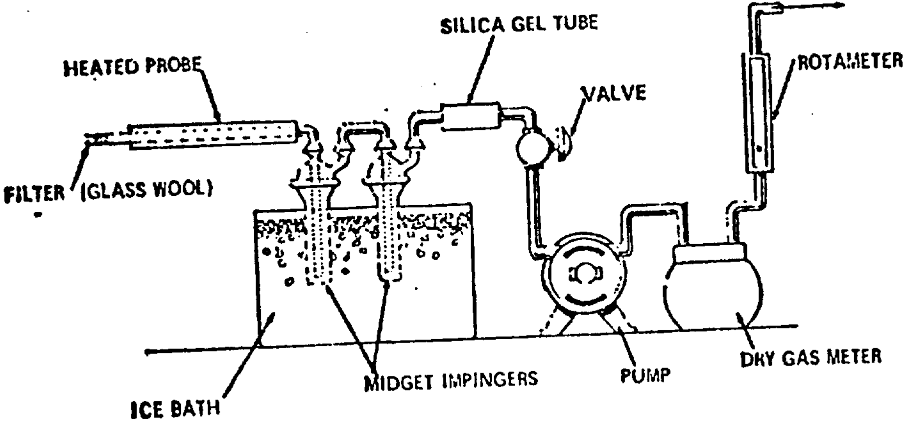


Figure 4-1. Moisture-sampling train.

LOCATION________________________ COMMENTS

TEST________________________

DATE________________________

OPERATOR________________________

BAROMETRIC PRESSURE________________________

CLOCK TIME	GAS VOLUME THROUGH METER, (Vm), ft³	ROTAMETER SETTING, ft³/min	METER TEMPERATURE, °F

Figure 4-2. Field moisture determination.

4.2 Gas volume.

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

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, in. Hg.

P_{std}=Pressure at standard conditions, 29.92-in. Hg.

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

T_m=Absolute temperature at meter (°F.+460), °R.

4.3 Moisture content.

$$B_{ws}=\frac{V_{wc}}{V_{wc}+V_{mc}}+B_{wm}=\frac{V_{wc}}{V_{wc}+V_{mc}}+(0.025)$$

equation 4-3

where:

B_{ws}=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 gas temperature of 250° F. at the exit end during sampling. When temperature or length limitations are encountered, 316 stainless steel, or equivalent, may be used, as approved by the Administrator.

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

2.1.4 Filter holder—Pyrex [1] glass with heating system capable of maintaining any temperature to a maximum of 225° F.

2.1.5 Impingers—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 ½-inch from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip.

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

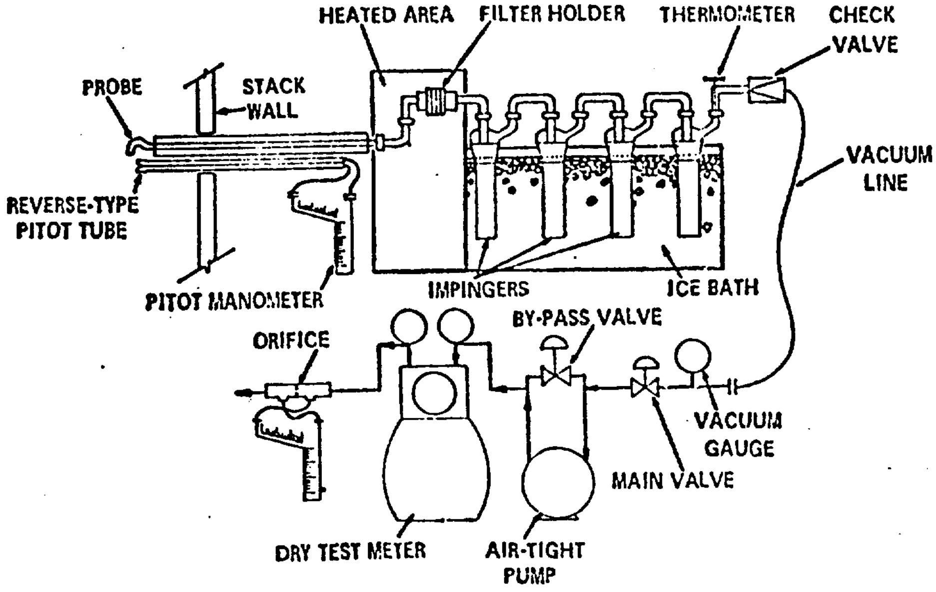


Figure 5-1. Particulate-sampling train.

2.1.7 Barometer—To measure atmospheric pressure to ±0.1 in. 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 Beakers—250 ml.

2.3.5 Separatory funnels—500 ml. and 1,000 ml.

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

2.3.7 Graduated cylinder—25 ml.

3. *Reagents.*

3.1 Sampling

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

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

3.1.3 Water—Deionized, distilled.

3.1.4 Crushed ice.

3.2 Sample recovery

3.2.1 Water—Deionized, distilled.

3.2.2 Acetone—Reagent grade.

3.3 Analysis

3.3.1 Water—Deionized, distilled.

3.3.2 Chloroform—Reagent grade.

3.3.3 Ethyl ether—Reagent grade.

3.3.4 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 percent. 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. Save a portion of the water for use as a blank in the sample analysis. Set up the train without the probe as in Figure 5-1. Leak check the sampling train at the sampling site by plugging 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 at 70° F. or less.

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. 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.

---

[1] Trade name.

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

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_S), 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	IMPINGER TEMPERATURE, °F
							INLET ($T_{m\ in}$), °F	OUTLET ($T_{m\ out}$), °F		
							Avg.	Avg.		
TOTAL							Avg.			
AVERAGE										

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 portions of the water and acetone used in the sample recovery as blanks for analysis. 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 loosen adhering particles.

Container No. 3. Measure the volume of water from the first three impingers and place the water in this container. Place water rinsings of all sample-exposed surfaces between the filter and fourth impinger in this container prior to sealing.

Container No. 4. 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.

Container No. 5. Thoroughly rinse all sample-exposed surfaces between the filter and fourth impinger with acetone, place the washings in this container, and seal.

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, dessicate, 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. Dessicate and dry to a constant weight. Report results to the nearest 0.5 mg.

Container No. 3. Extract organic particulate from the impinger solution with three 25 ml. portions of chloroform. Complete the extraction with three 25 ml. portions of ethyl ether. Combine the ether and chloroform extracts, transfer to a tared beaker and evaporate at 70° F. until no solvent remains. Dessicate, dry to a constant weight, and report the results to the nearest 0.5 mg.

Container No. 4. Weigh the spent silica gel and report to the nearest gram.

PLANT____________________

DATE____________________

RUN NO.____________________

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
3a*			
3b**			
5			
TOTAL	╳	╳	

*3a - ORGANIC EXTRACT FRACTION.
**3b - RESIDUAL WATER FRACTION.

	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.

Container No. 5. Transfer the acetone washings to a tared beaker and evaporate to dryness at ambient temperature and pressure. Desiccate, dry to a constant weight, and report the results to the nearest 0.5 mg.

5. *Calibration.*

Use standard methods and equipment approved by the Administrator to calibrate the orifice meter, pitot tube, dry gas meter, and probe heater.

6. *Calculations.*

6.1 Sample concentration method.

6.1.1 Average dry gas meter temperature. See data sheet (Figure 5-2).

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

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

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.
T_m=Average dry gas meter temperature, °R.
P_{bar}=Barometric pressure at the orifice meter, in. Hg.
ΔH=Pressure drop across the orifice meter, in H_2O.
13.6=Specific gravity of mercury.
P_{std}=Absolute pressure at standard conditions, 29.92 in. Hg.

6.1.3 Volume of Water vapor.

$$V_{w_{std}} = V_{l_c}\left(\frac{\rho_{H_2O}}{M_{H_2O}}\right)\left(\frac{RT_{std}}{P_{std}}\right) = \left(0.0474\,\frac{\text{cu. ft.}}{\text{ml.}}\right)V_{l_c}$$

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.
ρ_{H_2O}=Density of water, 1 g./ml.
M_{H_2O}=Molecular weight of water, 18 lb./lb. mole.
R=Ideal gas constant, 21.83 in Hg-cu. ft./lb. mole-°R.
T_{std}=Absolute temperature at standard conditions, 530° R.
P_{std}=Absolute pressure at standard conditions, 29.92 in. Hg.

6.1.4 Total gas volume.

$$V_{total} = V_{m_{std}} + V_{w_{std}}$$

equation 5-3

where:

V_{total}=Total volume of gas sample (standard conditions), cu. ft.
$V_{m_{std}}$=Volume of gas through dry gas meter (standard conditions), cu. ft.
$V_{w_{std}}$=Volume of water vapor in the gas sample (standard conditions), cu. ft.

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

6.1.6 Concentration.

$$c_s' = \left(0.0154\,\frac{\text{gr.}}{\text{mg.}}\right)\left(\frac{M_n}{V_{total}}\right)$$

equation 5-4

where:

c'_s=Concentration of particulate matter in stack gas (Sample Concentration Method), gr./s.c.f.
M_n=Total amount of particulate matter collected, mg.
V_{total}=Total volume of gas sample (standard conditions), cu. ft.

6.2 Ratio of area method.

6.2.1 Stack gas velocity. Collect the necessary data as detailed in Method 2. Correct the stack gas velocity to standard conditions (29.92 in. Hg, 530° R.) as follows:

$$V_{s,std}=V_s\left(\frac{P_s}{P_{std}}\right)\left(\frac{T_{std}}{T_s}\right)=\left(17.71\,\frac{°R}{\text{in. Hg}}\right)\left(\frac{V_sP_s}{T_s}\right)\quad \text{equation 5-5}$$

where:

$V_{s,std}$=Stack gas velocity at standard conditions ft./sec.

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

P_s=Absolute stack gas pressure, in. Hg.

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

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

T_s=Absolute stack gas temperature (average), °R.

6.2.2 Concentration.

$$c_s=\frac{M_s}{Q_s}=\frac{\frac{M_n}{\theta}\,\frac{A_s}{A_n}}{A_sV_{s,std}}=\left(2.57\times10^{-4}\,\frac{\text{gr.-min.}}{\text{mg.-sec.}}\right)\left(\frac{M_n}{\theta V_{s,std}A_n}\right)\quad \text{equation 5-6}$$

where:

c_s=Concentration of particulate matter in the stack gas (Ratio of Area Method), gr./s.c.f.

M_s=Particulate mass flow rate through the stack (standard conditions), mass time.

Q_s=Volumetric flow rate of gas stream through the stack (standard conditions), volume time.

M_n=Total amount of particulate matter collected by train, mg.

θ=Total sampling time, min.

A_s=Cross-sectional area of stack, sq. ft.

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

$V_{s,std}$=Stack gas velocity at standard conditions, ft./sec.

6.3 Isokinetic variation.

$$I=\frac{c_s}{c_s'}\times100=\frac{T_s\left[\frac{V_{l_c}\rho_{H_2O}R}{M_{H_2O}}+\frac{V_m}{T_m}\left(P_{bar}+\frac{\Delta H}{13.6}\right)\right]}{\theta V_sP_sA_n}\times100=$$

$$\frac{\left(1.667\,\frac{\text{min.}}{\text{sec.}}\right)\left[\left(0.00267\,\frac{\text{in. Hg-cu. ft.}}{\text{ml.-°R}}\right)V_{l_c}+\frac{V_m}{T_m}\left(P_{bar}+\frac{\Delta H}{13.6}\right)\right]T_s}{\theta V_sP_sA_n}\quad \text{equation 5-7}$$

where:

I=Percent of isokinetic sampling.

C_s=Concentration of particulate matter in the stack gas (Ratio of Area Method), gr./s.c.f.

C_s'=Concentration of particulate matter in the stack gas (Sample Concentration Method), gr./s.c.f.

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

ρ_{H_2O}=Density of water, 1 g./ml.

R=Ideal gas constant, 21.83 in. 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, in Hg.

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

T_s=Absolute average stack gas temperature (see Figure 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, in. Hg.

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

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

If 82 percent ≤I≤120 percent, the results are acceptable; otherwise, reject the results and repeat the test.

6.5 Average particulate concentration. If the criteria for acceptability are met, calculate the average concentration of particulate in the stack from the following equation:

$$c_s=\frac{\overline{c}_s+c_s'}{2}\quad \text{Equation 5-8}$$

where:

$\overline{c}_s$=Average particulate concentration in the stack gas, gr./s.c.f.

c_s=Concentration of particulate matter in the stack gas (Ratio of Area Method), gr./s.c.f.

c_s'=Concentration of particulate matter in the stack gas (Sample Concentration Method), gr./s.c.f.

7. *References.*

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

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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, 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, and 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 5-6 mm. ID, with a heating system to prevent condensation and a filter 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 Pump—Leak-free, vacuum type.

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

2.1.8 Dry gas meter—Sufficiently accurate to measure the sample volume within 1 percent.

2.1.9 Pitot tube—Type S, or equivalent, 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.

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[1] Trade name.

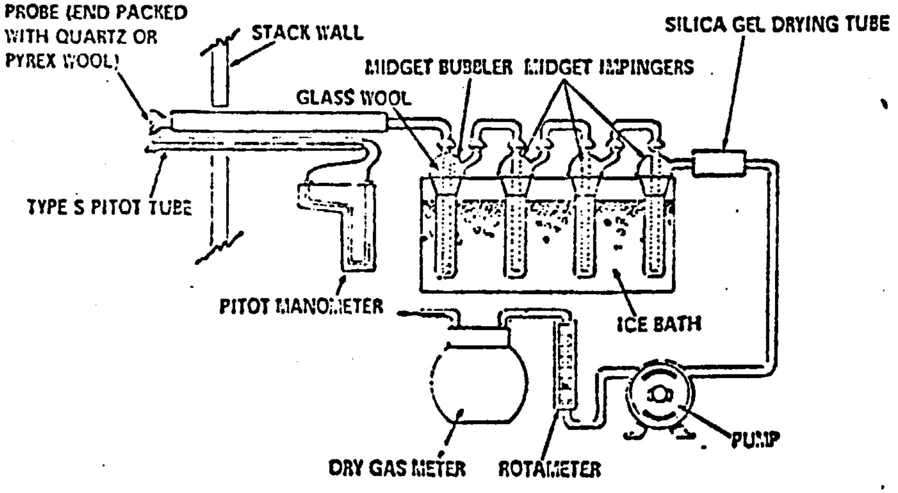

Figure 6-1. SO_2 sampling train.

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 percent—Mix 80 ml. of isopropanol with 20 ml. of distilled water.

3.1.3 Hydrogen peroxide, 3 percent—dilute 100 ml. of 30 percent hydrogen peroxide with 900 ml. of distilled water. Prepare fresh daily.

3.2 Sample recovery.

3.2.1 Water—Deionized, distilled.

3.2.2 Isopropanol, 80 percent.

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.

3.3.5 Sulfuric acid standard (0.01*N*)—Purchase or standardize against a primary standard to ±0.0002*N*.

4. *Procedure.*

4.1 Sampling.

4.1.1 Preparation of collection train. Pour 15 ml. of 80 percent isopropanol into the midget bubbler and 15 ml. of 3 percent hydrogen peroxide to 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-in. Hg vacuum. A leakage rate not in excess of 1 percent 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 5 minutes and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle with the tip pointing directly into the gas stream 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 the purging period. 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 to a 125-ml. erlenmyer flask. Add 40 ml. of isopropanol and 2 to 4 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 approved by the Administrator to calibrate the orifice meter, pitot tube, dry gas meter, and probe heater.

5.2 Standardize the sulfuric acid with potassium acid phthalate as a primary standard. Standardize the barium perchlorate with 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 in. Hg) by using Equation 6-1.

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

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, in. Hg.

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

6.2 Sulfur dioxide concentration.

$$c_{SO_2}=\left(7.05\times10^{-5}\,\frac{\text{lb.-l.}}{\text{g.-ml.}}\right)\frac{(V_t-V_{tb})\,(N)\left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}} \qquad \text{equation 6-2}$$

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, ml.

V_a=Volume of sample aliquot titrated, ml.

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

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. Paper presented at the 55th Annual Meeting of APCA. Chicago, Ill. May 20-24, 1962.

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 nitrous oxide, are measured colorimetrically using the phenoldisulfonic acid (PDS) procedure.

1.2 Applicability. This method is applicable for the measurement of nitrogen oxides 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 7-1.

2.1.1 Probe—Pyrex[1] glass, heated, with filter to remove particulate matter. Heating is unnecessary if the probe remains dry during the purging period.

2.1.2 Collection flask—Two liter, Pyrex[1] round bottom with short neck and 24/40 standard taper opening, protected against implosion or breakage.

2.1.3 Flask valve—T-bore stopcock connected to a 24/40 standard taper joint.

2.1.4 Temperature gauge—Dial-type thermometer, or equivalent, capable of measuring 2° F. intervals from 25° to 125° F.

2.1.5 Vacuum line—Tubing capable of withstanding a vacuum of 3-in. Hg absolute pressure, with "T" connection and T-bore stopcock, or equivalent.

2.1.6 Pressure gauge—U-tube manometer, 36 inches, with 0.1 inch divisions, or equivalent.

2.1.7 Pump—Capable of producing a vacuum of 3-in. Hg absolute pressure.

2.1.8 Squeeze bulb—One way.

2.2 Sample recovery.

2.2.1 Pipette or dropper.

2.2.2 Glass storage containers—Cushioned for shipping.

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[1] Trade name.

APPENDIX E

LABORATORY REPORT

APPENDIX E

LABORATORY REPORT

BACK HALF IMPINGER WATER RESIDUE PHASE ANALYSIS BY WET CHEMISTRY

Plant Revere Smelting and Refining Company, Newark, New Jersey

Date December 8, 1971

Run 1

	Organic, mg	Inorganic, mg	Total, mg
Split Residue Mass Before Analysis	6.7	12.5[(a)]	19.2
Constituent Mass per Split Residue			
SO_3	< 0.3	< 0.3	< 0.3
SO_4	0.33	8.8	9.13
Total Sample Residue Mass	13.4	25.0[(a)]	38.4
Constituent Mass per Total Residue			
SO_3	< 0.6	< 0.6	< 0.6
SO_4	0.66	17.6	18.26

(a) Water blank subtracted; water blank = 0.022 mg/ml.

Plant Revere Smelting and Refining Company, Newark, New Jersey

Date December 8 and 9, 1972

FRONT HALF LABORATORY REPORT
ANALYSIS BY ATOMIC ABSORPTION

Run	Probe Acetone Wash Residue, μg(a)	Pb, μg per Residue, μg	Pb, μg per Total Residue	Filter Catch, μg	Pb, μg per Split Filter Sample, μg	Pb, μg per Total Filter Catch
1	13,500	0.077	1,035	(b)	(b)	(b)
2	9,300	0.097	900	87,000	0.056	5,000
3	3,100	0.048	150	32,000	0.068	2,190
4	3,000	0.067	200	58,700	0.082	4,800

(a) Acetone blank values were relatively high, and for Run 3 and 4, were in excess of the reported values; therefore, analysis data for all runs are being reported for the uncorrected mass values. Blank value = 0.035 mg/ml.

(b) Not available.

BACK HALF IMPINGER WATER LIQUID PHASE ANALYSIS BY WET CHEMISTRY

Plant Revere Smelting and Refining Company, Newark, New Jersey

Date December 8, 1972

Run 1

Total Sample Volume - 260 ml

Split Sample Volume Analyzed - 130 ml

Constituent	μg/ml	mg per Split Sample Volume	mg per Total Sample Volume
Total Acid	2	0.26	0.52
$SO_3^=$	160	20.8	41.6
$SO_4^=$	24	3.12	6.24
Cl^-	2	0.26	0.52
NH_4^+	43	5.60	11.20
pH(a)	3.6		

(a) Value is pH units.

LABORATORY REPORT

OPTICAL EMISSION SPECTROSCOPY TRACE METAL
(μg element per total sample)

Total Sample Volume, Run 1 - 260 ml

Split Sample Volume Analyzed - 130 ml

Element	Impinger Water Run 1
Hg[(a)]	< 0.3
Be	< 0.3
Cd	< 13.
As	< 13.
V	< 3.
Mn	< 1.
Ni	< 10.
Sb	< 13.
Cr	< 10.
Zn	< 26.
Cu	2.
Pb	26.
Se[(b)]	(c)
B	2.
F[(b)]	(c)
Li	26.
Ag	< 0.3
Sn	< 13.
Fe	10.
Sr	< 10.
Na	< 26.
K	< 90.
Ca	10.
Si	26.
Mg	2.
Co	< 10.
Ba	< 3.
Al	< 10.
Total Mass, μg[(c)]	38,500

(a) Sample dried and ignited at 450 C prior to analysis; Hg values are not considered significant.

(b) Not detectable by OES.

(c) Water blank subtracted; water blank = 0.022 mg/ml

LABORATORY REPORT

OPTICAL EMISSION SPECTROSCOPY TRACE METAL
(μg of element for total filter catch)(a)

Element	Filter Analysis			
	Run 2	Run 3	Run 4	Blank
Be	< 1.	< 1.	< 1.	< 1.
Cd	< 20.	< 20.	< 20.	< 20.
As	< 40.	< 40.	< 40.	< 20.
V	< 1.	< 1.	< 1.	< 1.
Mn	40.	10.	20.	2.
Ni	< 20.	< 20.	< 20.	< 20.
Sb	< 20.	< 20.	< 20.	< 20.
Cr	< 20.	< 20.	< 20.	< 20.
Zn	2000.	200.	600.	< 200.
Cu	20.	4.	8.	2.
Pb(b)	---	---	---	50.
B	0	0	0	4000.
Ag	< 2.	< 2.	< 2.	< 2.
Sn	400.	300.	200.	< 10.
Fe	1000.	300.	500.	< 100.
Sr	5.	1.	1.	5.
Na	14000.	10000.	14000.	2000.
K	2000.	1000.	1000.	1000.
Ca	0	0	2000.	10000.
Si	---	---	---	---
Mg	0	0	0	4000.
Ba	100.	100.	100.	100.
Al	0	0	0	4000.
Total Catch, μg	87,000	32,300	58,700	

(a) Filter blank subtracted.
(b) Pb determined by Atomic Absorption.
Note: Symbol < indicates minimal detection limits.

LABORATORY REPORT

OPTICAL EMISSION SPECTROSCOPY TRACE METAL
(μg of element per total sample)

Element	Probe Residue Run 1	Run 2	Run 3	Run 4
Be	< 1.	< 1.	< 1.	< 1.
Cd	< 10.	< 10.	< 10.	< 10.
As	< 20.	< 20.	< 20.	< 20.
V	< 1.	< 1.	< 1.	< 1.
Mn	1.	1.	< 1.	< 1.
Ni	< 10.	< 10.	< 10.	< 10.
Sb	< 10.	< 10.	< 10.	< 10.
Cr	< 10.	< 10.	< 10.	< 10.
Zn	100.	100.	< 100.	< 100.
Cu	3.	3.	1.	1.
Pb(a)	---	---	---	---
B	1.	< 1.	3.	10.
Ag	< 1.	< 1.	< 1.	< 1.
Sn	50.	50.	10.	10.
Fe	1000.	500.	30.	30.
Sr	< 1.	< 1.	< 1.	< 1.
Na	200.	300.	200.	200.
K	< 500.	< 500.	< 500.	< 500
Ca	100.	100.	100.	100
Si	50.	50.	50.	50
Mg	200.	300.	100.	50.
Ba	< 10.	< 10.	< 10.	< 1
Al	10.	10.	10.	10.
Total Weight, μg(b)	13,500	9,300	3,100	3,000

(a) Pb determined by atomic absorption.

(b) Acetone blank values were relatively high, and for Run 3 and 4, were in excess of the reported values; therefore, analysis data for all runs are being reported for the uncorrected mass values. Blank value = 0.035 mg/ml.

Plant Revere Smelting and Refining Company, Newark, New Jersey

EXAMPLE DETERMINATION
FILTER, RUN 2

A - Initial Extract Volume per Split Filter Sample, ml	4.5
B - Aliquot, ml	1.0
C - Dilution Factor	50
D - Absorption, Percent	32.5
E - Absorbance from Atomic Absorbance Tables, Based on Percent Absorption	0.1707
F - Lead, μg/ml of Diluted Aliquot(a)	11.1
G - Lead, μg/Split Filter Sample	2500
H - Lead, μg/Total Filter	5000

Example Computation

C x A x F = G

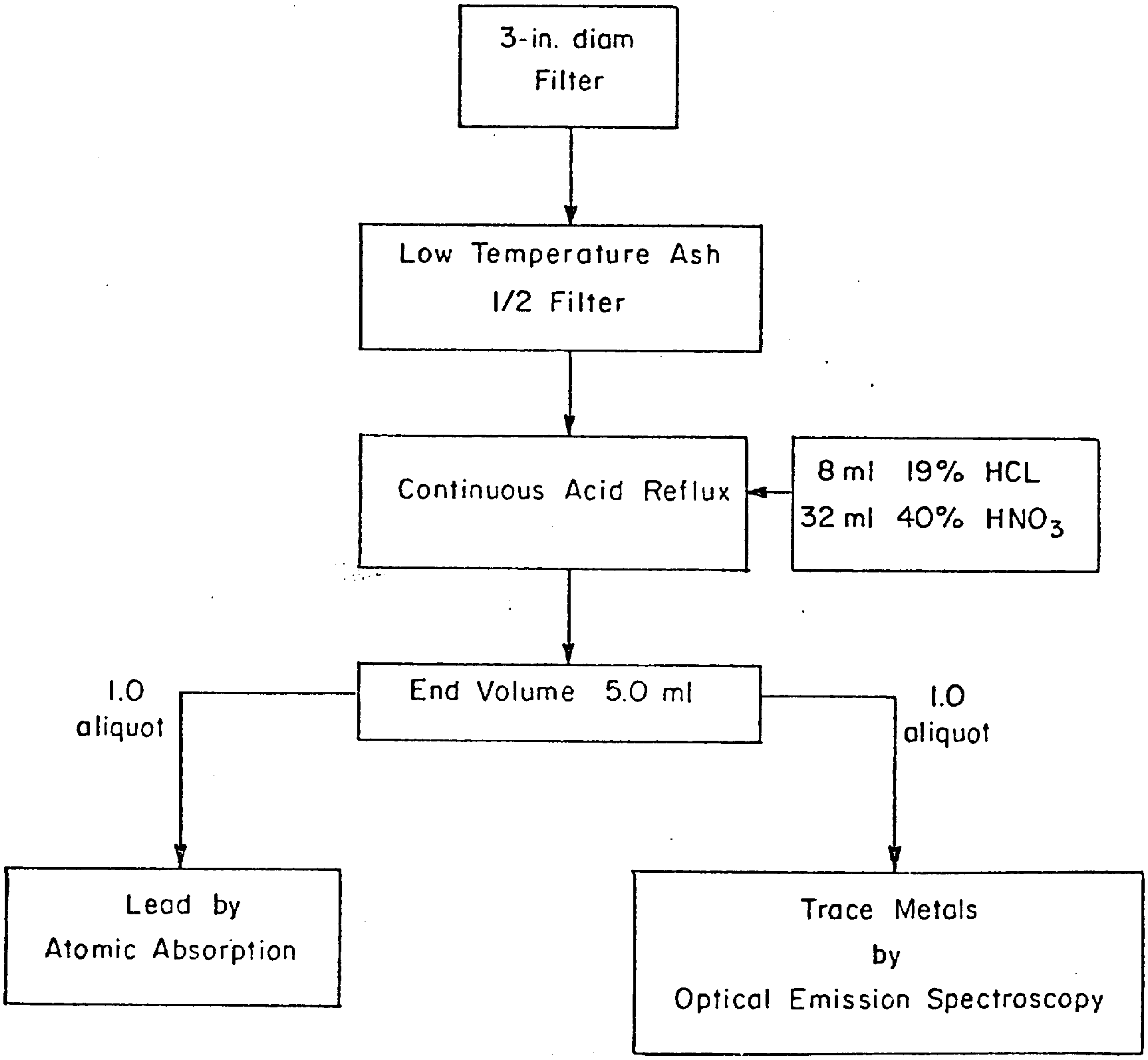
50 x 4.5 x 11.1 = 2500

2 x G = H

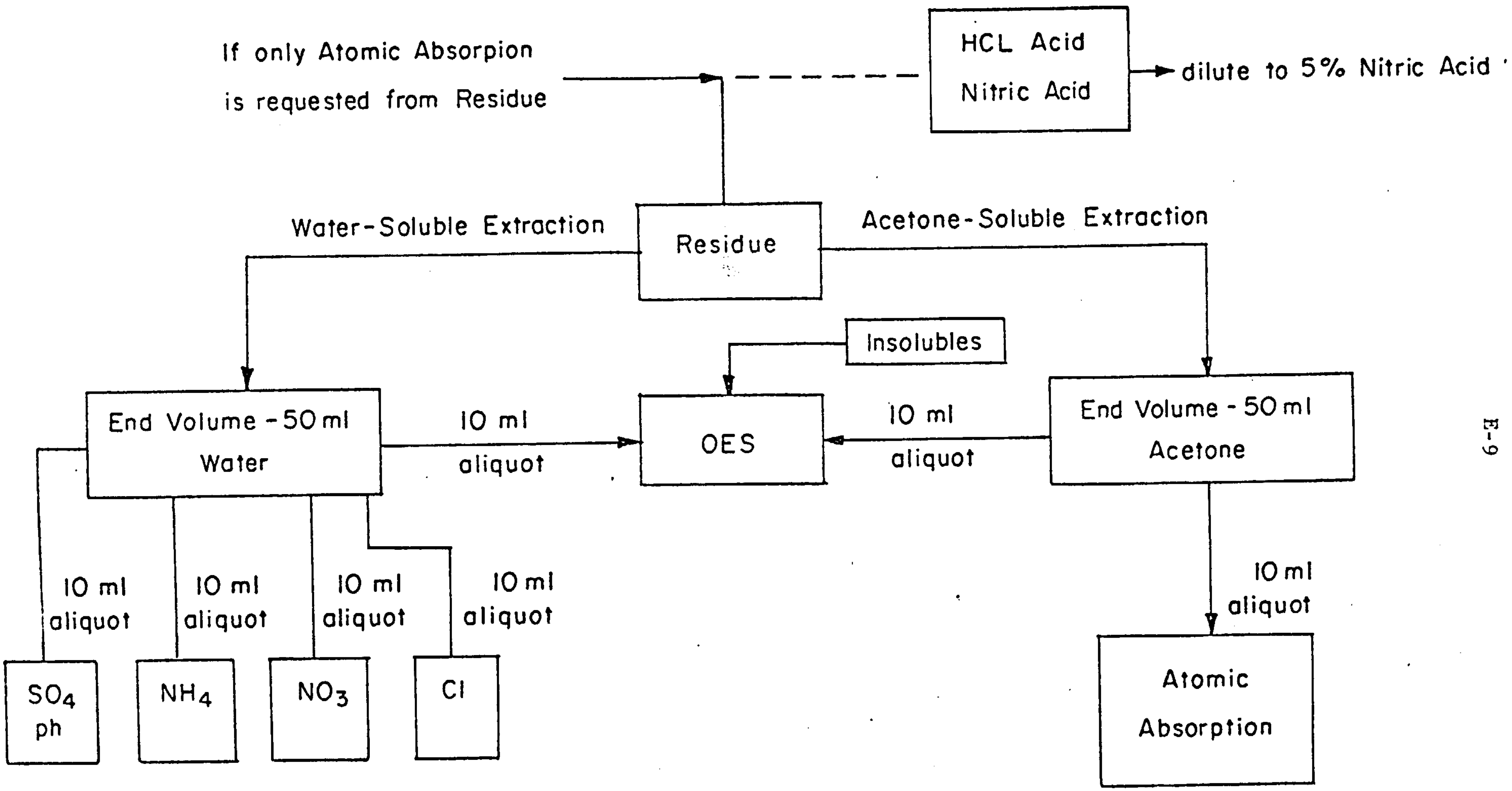
2 x 2500 = 5000

(a) Determined from calibration curve; based on 0.1707 absorbance.

FILTER ANALYSIS FLOW DIAGRAM



RESIDUE ANALYSIS FLOW DIAGRAM



CHEMICAL METHODS USED TO ANALYZE EPA RESIDUES

(The water-soluble portion of the residue has been transferred to a 50-ml volumetric flask, diluted to the mark and mixed).

$SO_4^=$

1. Add 4 ml of 1:1 HCl to the solution, heat to a boil, and continue boiling for about 5 minutes.
2. Cool slightly and transfer to a 250-ml beaker, rinsing out the 50-ml beaker well with deionized water. Dilute to 125 to 150-ml volume with deionized water.
3. Heat to near boiling temperature, add 10 ml of 10 percent $BaCl_2$ solution, stir well, and digest at near boiling temperature for an hour.
4. Remove from the hot-plate and cool to room temperature. Allow the solution to stand at room temperature at least 2 hours - preferably overnight.
5. Filter off the precipitated $BaSO_4$ through a tight filter paper (Whatman No. 42 or equivalent). Wash the paper and precipitate with hot deionized water until the washings are free of chloride.
6. Place the paper and precipitate in a crucible, dry under a gas hot-plate, and then heat in a muffle furnace for at least an hour at a temperature of 900 C.
7. Remove the crucibles from the muffle furnace, cool in a desiccator, and weigh as $BaSO_4$. Calculate SO_4 by multiplying by the factor 0.4116. Multiply this result by 5 to get total amount of $SO_4^=$ in the residue.

DISCUSSION OF METHODS

The methods used for the chemical analysis of residues were chosen because they represent reliable procedures used almost routinely in our laboratory. Because of this no preliminary testing nor experimentation were required before the analytical work was begun and results could be obtained in the short time allowed. No claim is made that these methods represent the best analytical scheme for this work.

The gravimetric method for sulfate lacks the sensitivity one might desire for this type of work if measurement of less than 1 milligram is necessary. However, it is capable of determining the occasional high sulfate found in some samples without repeating the determination as would be necessary when using some methods.

PREPARATION OF EPA RESIDUE SAMPLES

(1) Extract residues in beakers with warm water. Combine where indicated and adjust volume to 50 ml in a volumetric flask.

(2) Extract remaining residues in beakers with acetone. Police beakers thoroughly, making sure that all material is removed, even though insoluble. Combine where indicated and adjust volume to 50 ml in a volumetric flask.

(3) Transfer 10 ml of the water extracts to crucibles for OES. Add 10 ml of the corresponding acetone extracts to the water extracts, being sure to shake the flasks well before sampling to assure a representive portion of suspended matter is included. Dry combined extracts and proceed with OES.

(4) Remove 25 ml of the remainder of the acetone extracts, filter on Millipore filter if necessary, and transfer to tared aluminum dishes. Dry down at ~100 C. Reweigh vessel and record weight as "Acetone Soluble". Multiply results by 2 to convert to original sample.

(5) Proceed with chemical analysis of the water extracts in this manner.

40 ml water extract

- 10 ml for pH and $SO_4^=$
- 10 ml for Cl^-
- 10 ml for NH_4^+
- 10 ml for total nitrogen (Devarda's method) ⊗

⊗ NO_3^- will be calculated from the difference between total nitrogen and NH_4^+

Note: "Dry pipetting" (no rinsing of pipette) must be practiced to conserve sample.

If the weight of residue removed by the acetone treatment is useful in interpreting results these data may be obtained in the following manner.

Water Insoluble Residue

Note: If filtering is necessary in step 4, weight the filter pad, air dry after filtration is complete, and reweigh to obtain weight of retained residue.

(6) Wash the acetone solution and suspended matter remaining in the 50 ml volumetric flasks into the same dishes used in step 4. Dry down at ~100 C. Reweigh and record weight as "Water Insoluble". Multiply by 5/4 to convert to original sample. If a filtration step was included the weight of residue retained by the filter is added before multiplying by 5/4.

Preparation of Filters for Chemical Analysis

(1) Remove 1/4 of the filter circle and carefully cut it up into small pieces. Place the pieces in a 50 ml beaker, add 10 ml of deionized water, and stir with a clean glass stirring rod. Allow to stand an hour, stirring occasionally.

(2) Measure the pH of the solution using the small electrode assembly.

(3) Filter the slurry into a 50 ml volumetric flask. Wash the retained filter material with small increments of deionized water until the 50 ml volume is reached.

(4) Stopper the volumetric flask, mix well, and proceed with the chemical determinations as described for the residue samples.

(5) Multiply all results by 4 to obtain the amounts of SO_4, Cl, NH_4, and NO_3 on the entire filter circle.

APPENDIX F

TEST LOG

APPENDIX F

TEST LOG

December 6, 1971	Arrive on site Start erection of scaffold
December 7, 1971	Complete erection of scaffold Set up equipment
December 8, 1971	Make Run 1 while furnace is under repair Make Run 2
December 9, 1971	Make Runs 3 and 4
December 10, 1971	Pack equipment Leave test site.

APPENDIX G

RELATED REPORTS

APPENDIX G

RELATED REPORTS

(1) RESEARCH REPORT ON SECONDARY LEAD PLANT STACK EMISSION SAMPLING AT GENERAL BATTERY CORPORATION, READING, PENNSYLVANIA

(2) RESEARCH REPORT ON SECONDARY LEAD PLANT STACK EMISSION SAMPLING AT N.L. INDUSTRIES PLANT, McCOOK, ILLINOIS

(3) RESEARCH REPORT ON SECONDARY LEAD PLANT STACK EMISSION SAMPLING AT QUEMETCO LEAD COMPANY, INDUSTRY, CALIFORNIA

(4) RESEARCH REPORT ON SECONDARY LEAD PLANT STACK EMISSION SAMPLING AT N. L. INDUSTRIES PLANT, BEECH GROVE, INDIANA

APPENDIX H

PROJECT PARTICIPANTS AND TITLES

APPENDIX H

PROJECT PARTICIPANTS AND TITLES

Technical Supervision

Richard B. Engdahl, Senior Project Leader

Field Team

Paul R. Webb, Senior Technologist (Team Leader)

Herbert E. Carlton, P.E., Research Chemical Engineer

Bernard E. Campbell. Physicist

William C. Baytos, Scientist

Harold Hess, Technician

Administrative Support

Richard E. Barrett, Senior Mechanical Engineer

John M. Allen, Division Chief

Irene Whitener, Secretary

APPENDIX I

COMPLETE OPERATIONAL RESULTS

APPENDIX I

COMPLETE OPERATIONAL RESULTS

Run Number	2	3	4
Particulate sampling start time	3:10	9:50	12:58
Particulate sampling start time	5:39	11:55	3:08
Slag tap	3:51 5:10	10:55 12:26	12:26 1:45
Lead ingot mold change	3:00 (b) 3:22 (b) 3:47 (b) 4:45 (a) 5:07 (a) 5:40	9:41 (a) 10:10 (a) 10:45 (b) 11:30 (b) 12:10	12:10 (a) 1:12 (a) 1:50 (a) 2:35 (b) 3:05
Furnace feed times (c)	3:05 3:18 3:27 3:36 3:47 4:05 4:18 4:21 4:31 4:40 4:51 5:08 5:22 5:31	10:11 10:25 10:46 11:10 11:23 11:42 11:53 12:11	12:53 1:05 1:13 1:23 1:32 1:40 1:50 2:00 2:20 2:30 2:38 2:45 2:50 3:08
Lead rate, lb/hr	3000	2300	2500
Slag rate, lb/hr	1500	1000	1500

(a) Small mold (1500 pounds) filled

(b) Large mold (1650 pounds) filled

(c) 400 pounds of mixed feed

OPERATIONAL NOTES

Plant - Revere Smelting and Refining Company
Newark, New Jersey

12/9/71

1:30 p.m.	Furnace started after replacement of tuyere
2:10	Started filling lead mold Operating 10 percent oxygen enrichment
3:00	Change lead molds
3:05	Load furnace
3:18	Load furnace
3:22	Change lead mold - 1650 pounds
3:27	Load furnace
3:36	Load furnace 10 percent oxygen enrichment (estimated)
3:47	Load furnace
3:51	Start tapping slag
4:05	Rod tuyeres Finish tapping slag - 2000 pounds
4:09	Finish rodding tuyeres Lead flow stops while tapping slag
4:18	Load furnace
4:21	Load furnace
4:31	Load furnace
4:40	Load furnace
4:45	Change lead mold - 1650 pounds
4:51	Load furnace
4:59	Load furnace
5:07	Change lead mold - 1500 pounds
5:08	Load furnace
5:10	Tap slag - 2000 pounds
5:22	Load furnace
5:31	Load furnace
5:34	Road tuyeres
5:36	Finish tapping slag
5:40	Change mold - 1500 pounds
5:48	Finish rodding tuyeres Finish observations.

OPERATIONAL NOTES (Continued)

12/9/71

09:10 a.m.	Change lead mold
09:30	Velocity pressure in small duct 0.5-0.7 inches H_2O, duct diameter 15.5 inches. This is duct from smaller scrubber system to stack
09:41	Change lead mold - 1500 pounds
09:50	Started observations
10:10	Change lead mold - 1500 pounds
10:11	Load furnace
10:25	Load furnace
10:35	Load furnace
10:45	Change lead mold - 1500 pounds
10:46	Load furnace
10:55	Tap slag - 2000 pounds
11:05	Rod tuyeres
11:10	Load furnace
11:10	Finish rodding tuyeres
11:23	Load furnace
11:30	Change lead mold - 1650 pounds
11:42	Load furnace
11:53	Load furnace
12:10 p.m.	Change lead mold - 1650 pounds
12:11	Load furnace
12:26	Start slag tap - 2000 pounds
12:29	Load furnace Pressure; 47 inches H_2O at inlet to fan on large scrubber Temperature gas at scrubber inlet 95 F
12:45	Finish slag tap
12:53	Load furnace
1:05	Load furnace
1:12	Change lead molds - 1500 pounds
1:13	Load furnace
1:23	Load furnace
1:32	Load furnace
1:40	Load furnace

OPERATIONAL NOTES (Continued)

12/9/71

1:45	Tap slag
1:50	Change lead molds - 1500 pounds
2:00	Load furnace
2:03	Clean tuyeres
2:15	Stop slag tap
2:20	Load furnace
2:35	Change lead molds - 1500 pounds
2:38	Load furnace
2:45	Load furnace
2:50	Load furnace
3:05	Change molds - 1650 pounds
3:08	Load furnace
	End observation notes

APPENDIX J

SUMMARY OF RESULTS

APPENDIX J

SUMMARY OF RESULTS

Run Number	2	3	4	Average
Date	12-8-72	12-9-72	12-9-72	
Test Time - Minutes	120	112	112	115
Lead Production - TPH	1.5	1.2	1.2	1.3
Stack Effluent				
Flow rate - DSCFM	12,100	13,330	12,540	12,657
Flow rate - DSCF/ton	8,067	11,108	10,450	9,875
Temperature - ^{o}F	97	95	93	95
Water vapor - Vol. %	2.4	1.7	2.3	2.13
CO_2 - Vol. % dry	0.5	0.8	0.8	0.7
O_2 - Vol. % dry	20.3	20.2	18.8	19.7
CO - Vol. % dry	0.8	0.3	0.2	0.4
CO - lb/hr	420	160	109	330
SO_2 - ppm dry	0.06	0.04	0.08	0.06
SO_2 - lb/hr	0.01	0.01	0.01	0.01
Visible emissions - % opacity	10-15	10-20	5-15	12
Particulate Emissions				
Probe and filter catch				
gr/DSCF	0.0196	0.0084	0.0149	0.0143
gr/ACF	0.0184	0.0080	0.0140	0.0135
lb/hr	2.0354	0.8913	1.6061	1.5109
lb/ton lead	1.3570	0.7750	1.2849	1.1389
Total Catch				
gr/DSCF	0.0275	0.0157	0.0235	0.0222
gr/ACF	0.0257	0.0149	0.0221	0.0208
lb/hr	2.8474	1.6556	2.5200	2.3410
lb/ton lead	1.8983	1.4397	2.0160	1.7847
Lead Emissions				
Probe and filter catch				
gr/DSCF	0.00127	0.00061	0.00127	0.00105
gr/ACF	0.00119	0.00058	0.00120	0.00099
lb/hr	0.1320	0.0646	0.1368	0.1111
lb/ton lead	0.0880	0.0562	0.1095	0.0846
Total Catch	Essentially the same as probe and filter.			