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SECONDARY  
LEAD SMELTING 12.11  
AP-42 Section 7.11  
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10 a

# RESEARCH REPORT



Battelle

Environmental Research

RESEARCH REPORT

on

SECONDARY LEAD PLANT STACK EMISSION SAMPLING  
AT N. L. INDUSTRIES PLANT, BEECH GROVE,  
INDIANA

Contract No. 68-02-0230

Task Order No. 1

to

ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF AIR PROGRAMS

August 4, 1972

by

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INTRODUCTION

Gas and particulate emission measurements were made at the N. L. Industries secondary lead plant in Beech Grove, Indiana, on November 17 and 18, 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 effluent gas from a lead blast furnace was sampled in the stack from the process baghouse outlet after particulate removal by three cyclones and a baghouse in series. Particulate and SO<sub>2</sub> emissions were determined and Orsat analyses were made.

The blast furnace is used to melt down manufacturer's scrap and worn-out batteries, and to reduce various lead oxide drosses generated at several of the N. L. Industries Plants. The blast furnace is fed almost continuously with coke, iron, batteries, lead oxide, and a silaceous slag. A 7100-pound charge to the furnace yields about 4800 pounds of lead. About 10 charges are fed each 8-hour shift.

Figure 1 is a sketch showing the gas flow through the plant. Air is blown into the furnace to burn the coke in the feed. The heat of combustion melts the lead and the coke reduces the lead oxides. The off-gas from the furnace is heated in an afterburner to oxidize any odiferous compounds and to incinerate oily and sticky materials which may blind the

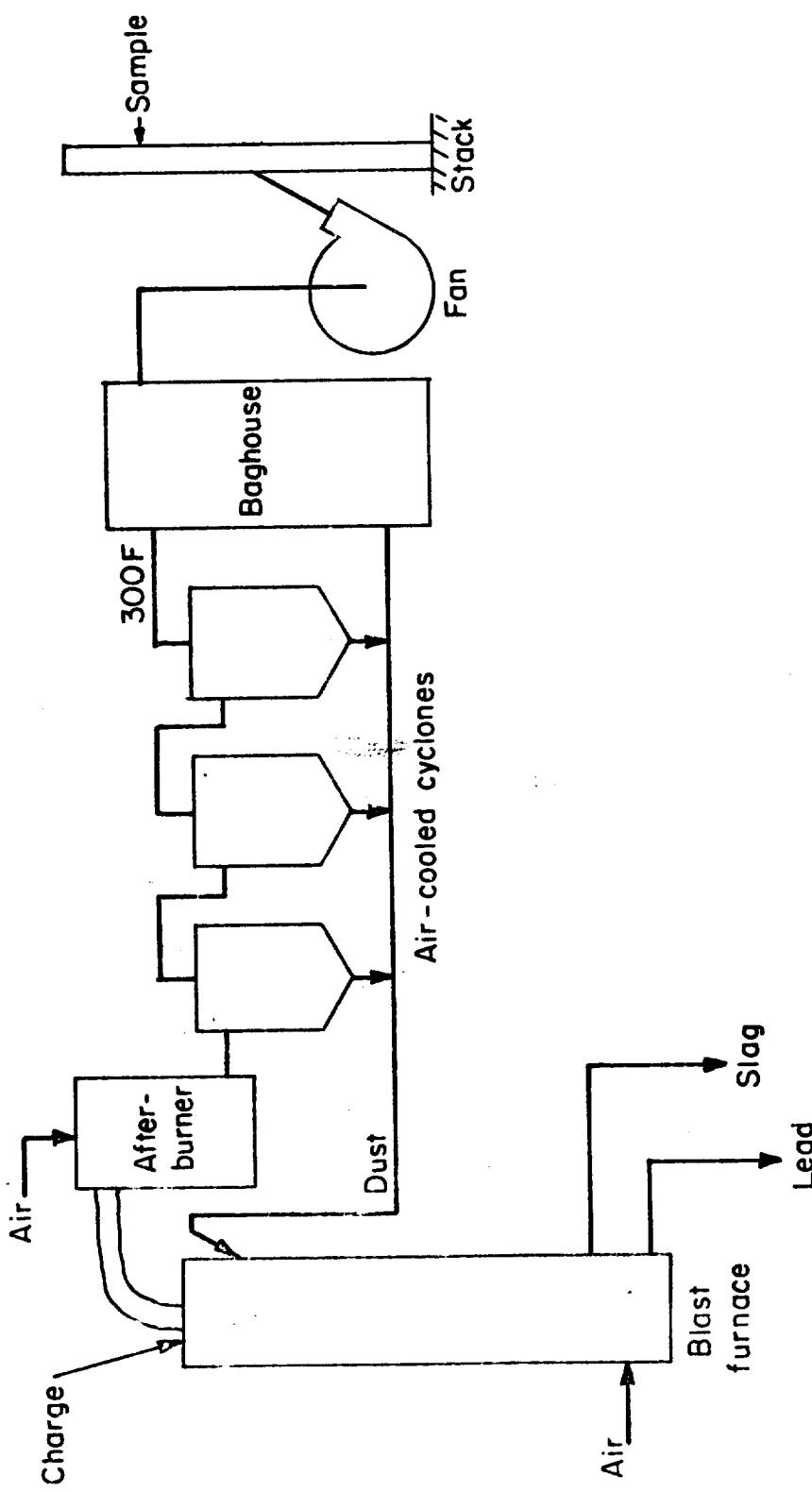


FIGURE 1. GAS FLOW DIAGRAM, N.L. INDUSTRIES,  
BEECH GROVE, INDIANA

fabric filters. The off-gas is cooled in three air-cooled cyclones in series which also remove most of the dust. The remaining dust is removed in a baghouse. The gas is then exhausted through a 154-foot-high stack. Particulate,  $\text{SO}_2$ , and Orsat samples were obtained by sampling from the stack just above the roof level.

#### SUMMARY OF RESULTS

The process stack on the lead blast furnace of N. L. Industries in Beech Grove, Indiana, was sampled for particulates,  $\text{SO}_2$ ,  $\text{CO}_2$ ,  $\text{O}_2$ , and CO in three runs on November 17 and 18, 1971. For the three tests, the particulate emission caught in the filter and probe averaged 0.0026 gr/scf which is equivalent to an emission from the plant of 0.51 lb/hr, or 0.17 lb/ton lead. The highest value was 0.0028 gr/scf.

The  $\text{SO}_2$  concentration in the stack varied from 204 to 443 ppm, which is equivalent to an emission of 46 to 101 lb/hr.

The operation of the blast furnace was essentially the same for all three runs. The afterburner was off a substantial part of Run 2, although monitored temperatures in the afterburner were never less than 1100 F. The bags in the baghouse were hand shaken during Run 2 which may account for the reduction in the dust load in Run 3.

The stack gas water vapor level varied from a high of 3.7 percent during Run 1 to a low of 2.3 percent during Run 2.

Each volume of gas from the blast furnace is diluted with about 11 volumes of air in the exhaust system. This large volume of air is necessary to keep dust from leaking into the plant. Consequently, the Orsat analysis was essentially equal to that of air. A small amount of

CO from the blast furnace was observed by Orsat analysis as well as CO<sub>2</sub> from the blast furnace and from the combustion of CO and natural gas in the afterburner.

A summary of the results is presented in Appendix J.

#### PROCESS DESCRIPTION

The N. L. Industries Plant in Beech Grove processes various lead scraps into lead ingots. The plant consists of a large receiving yard where the various lead scraps are stored (mainly under a roof); a blast furnace to melt or reduce the lead and lead oxide scraps, holding kettles, pot furnaces for molten lead, a continuous casting line for 60-pound ingots, an intermittent line for 1400-pound ingots, and a shipping section.

Most scrap is received in the form of battery plate groups from discarded batteries with the cases, but not the tops, removed. If batteries with cases are received they are sent to an outside company for case removal. Various other forms of lead scraps, such as toothpaste tubes and automobile weights, are included with the battery plates. Lead oxide scraps appeared to be battery plates that had been burned, perhaps to remove the cases. Various lead containing dross, obtained from skimming of lead pots, were also fed into the blast furnace.

The blast furnace, rated at 77 tons/day, is a tube furnace about 30 feet high and 5 or 6 feet in diameter as is sketched in Figure 2. It contains a hearth for lead at the bottom. Slag normally floats on the lead. Coke supports the charge in the tube. Air is blown into the furnace at a level just above the slag at a pressure of 30 to 60 inches of water. A Roots blower supplies the air at a constant rate of about 1600

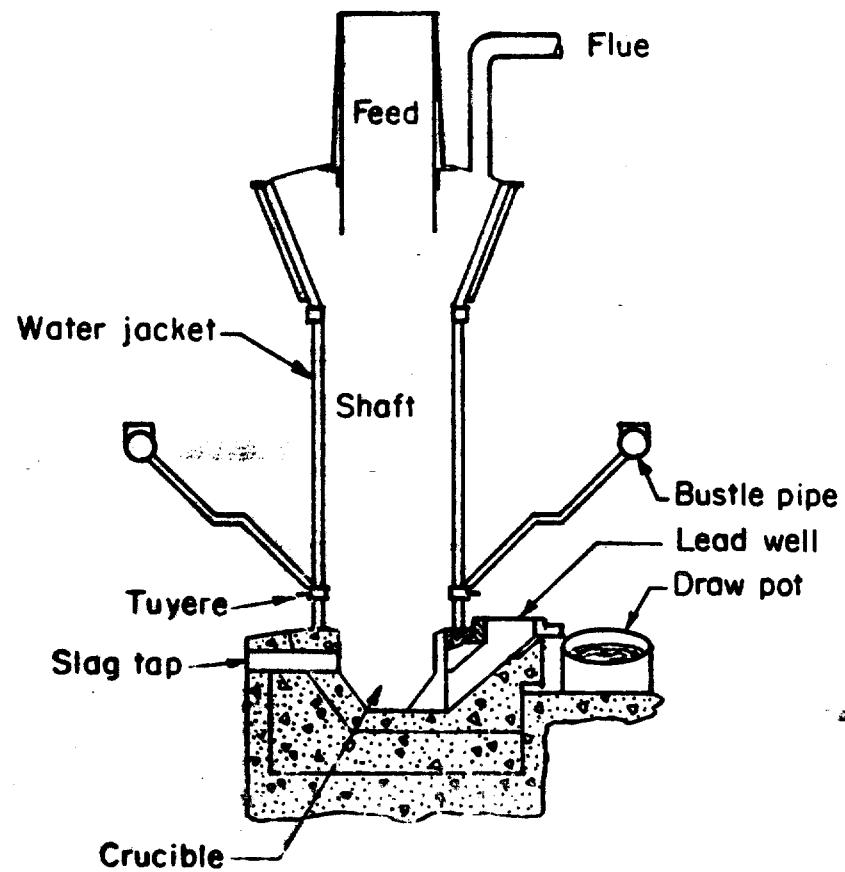


FIGURE 2. LEAD BLAST FURNACE

to 1800 cfm. The furnace is loaded from the top and tapped for lead and slag from separate taps at the bottom. Dusty gas is removed from the furnace at the main exhaust stream near the top of the furnace, from the skip hoist hood, and from a hood over the slag tap as shown in Figure 3. Only the main exhaust stream from the furnace was exhausted through the main stack. The other streams were exhausted through the sanitary bag-house which also filters exhaust air from several other parts of the plant.

The lead from the blast furnace is drained into a series of kettles and then is pumped into one of two kettles used to feed the casting machines. The lead is cast into 60-pound ingots in a continuous casting machine or into 1400-pound ingots intermittently.

#### Description of Gas Cleaning Equipment

Three separate gas cleaning systems are used for cleaning the several gas streams at N. L. Industries. One system, the only one sampled, collects dust from the blast furnace exhaust gas. A second system is used to collect dust from the top of the blast furnace, from the blast furnace slag tap and from the lead kettles. The third system is used to collect dust from small kettles used for making arsenic - lead alloys.

The main gas stream leaving the blast furnace is first heated to about 1200 F in a refractory afterburner with a natural gas flame. Most of the hydrocarbons and odoriferous compounds, and some CO, in the exhaust gas are burned here. The gas then passes through three forced air-and-water-cooled cyclones which remove some particulates. The gas stream is cooled from about 1200 F to about 300 F in the cyclones by heat transfer and by dilution with leakage air.

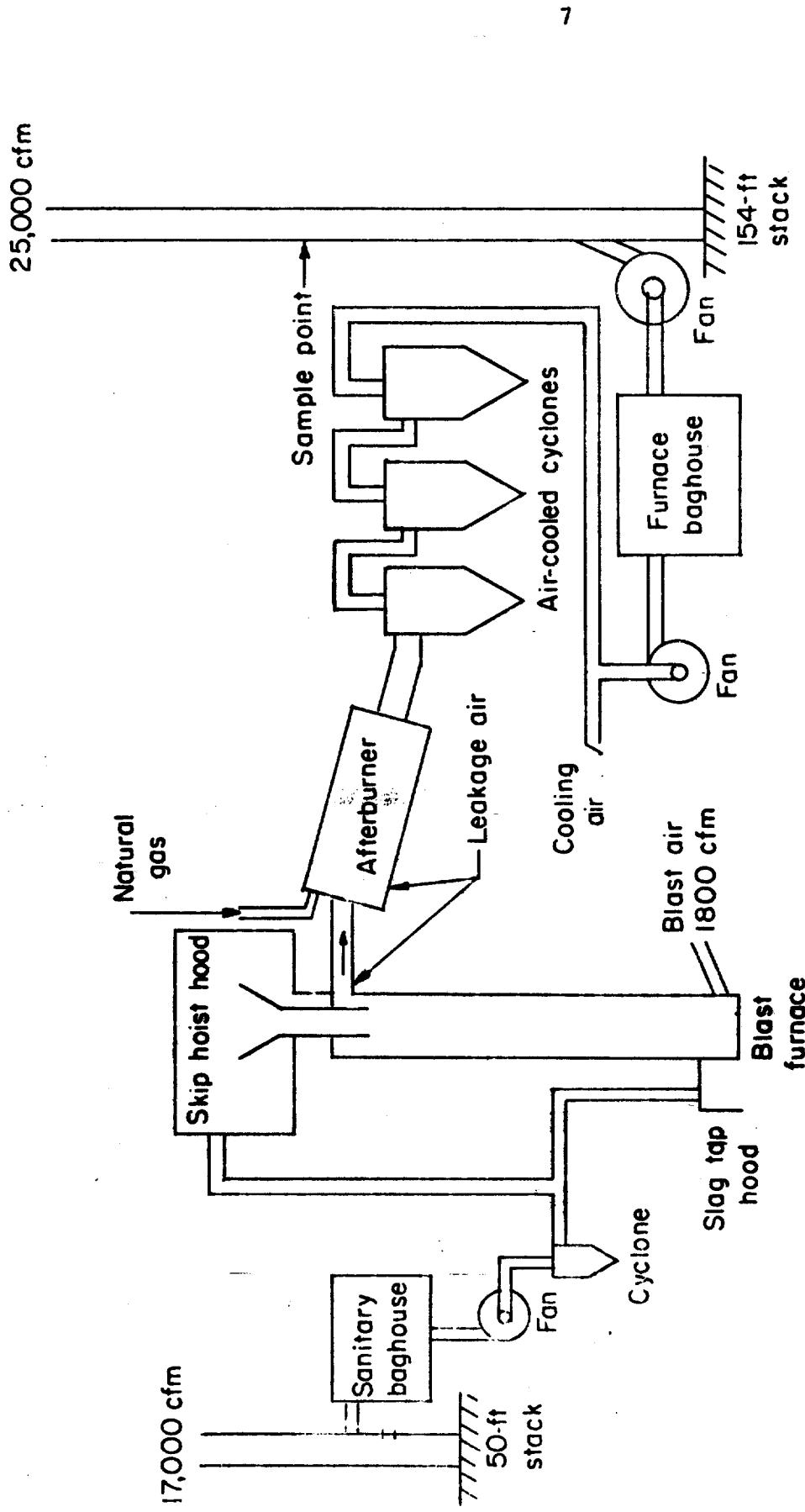


FIGURE 3. GAS CLEANING SYSTEM FLOW DIAGRAM, SECONDARY LEAD BLAST FURNACE, BEECH GROVE, INDIANA

The baghouse was purchased from Buell-Norblo and is constructed in eight equal sections. The baghouse has a total of 320 dacron bags, 25 feet, 7 inches long, and 11-1/2 inches in diameter, with a total cloth area of 24,000 square feet. Since the gas flow was about 22,000 cfm, the air/cloth ratio is about 1. Every 15 minutes, each of the eight sections is shaken separately for 25 seconds while the air flow is diverted with dampers. Equal time intervals are maintained between shaking of the eight sections.

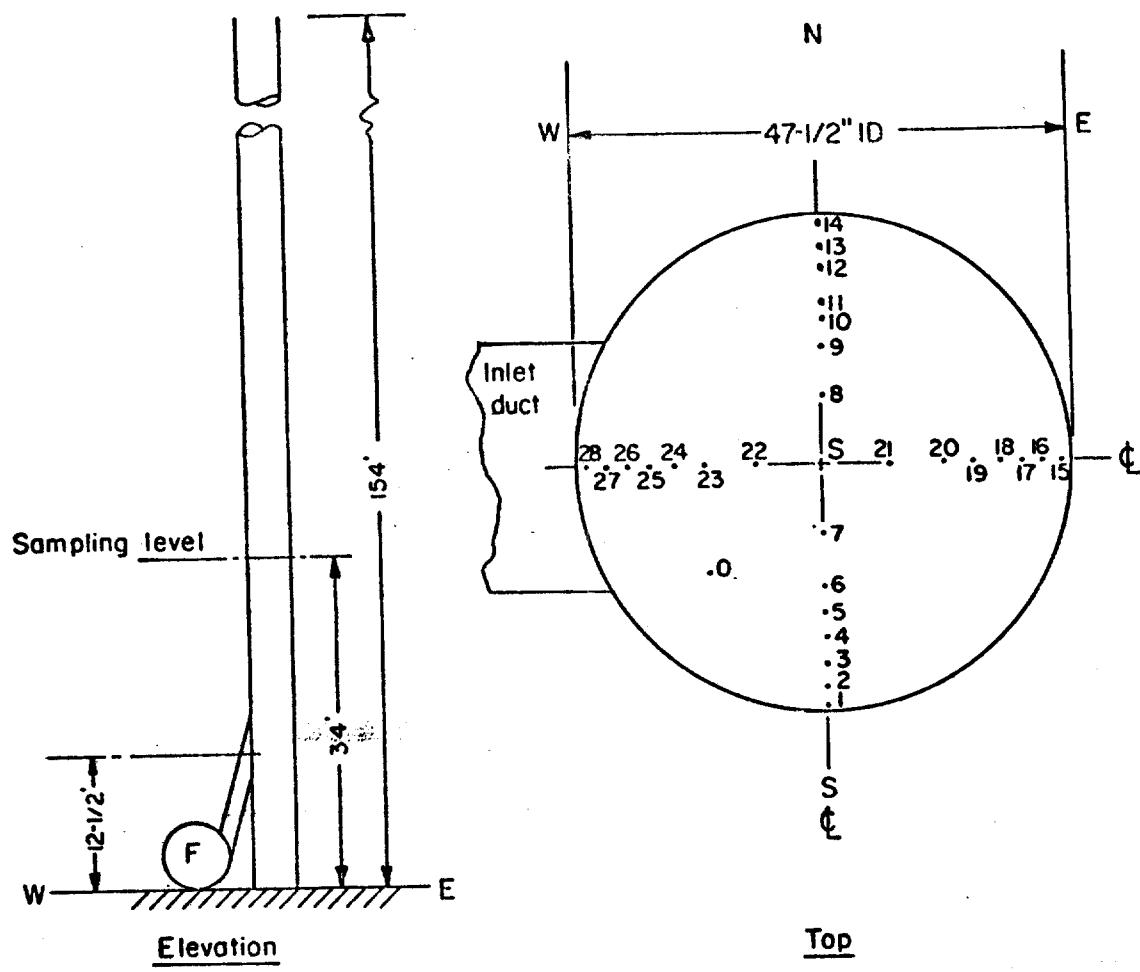
During the three runs, baghouse operation was as follows. Gas passing through the baghouse is cooled by heat losses and air inleakage to about 150 F. The bags were partially blinded during Run 1 and the inlet pressure to the baghouse was slightly positive, causing dust-containing gas to be blown out of the baghouse conveyor. During Run 1, one section of the baghouse was inoperative and during parts of Run 2, an additional section was inoperative. The bags had become blinded before Run 2, and each section in turn was bypassed while the bags were shaken by hand.

#### LOCATION OF SAMPLING POINTS

All of the samples were obtained from the 154-foot stack at roof level, 34 feet above ground, after the gas from the blast furnace left the baghouse. (See Figure 4). The SO<sub>2</sub> sample was obtained from the center of the stack, the Orsat sample from a point 18 inches into the stack indicated by "0" in Figure 4, and the particulate sample by means of a 28-point stack traverse. Points 1 and 15 were not sampled because of possible contamination from dust on the stack wall. The sampling level is 5.5 stack diameters downstream from the inlet duct, a distance which requires a minimum of 28 sampling points.

#### PROCESS OPERATION

The process operation is as follows. Air is blown through the blast furnace at a rate of 1600-1800 cfm and at a pressure of 30-100 inches of water. The materials charged to the furnace are burned, re-



Sampling Points	1 15	2 16	3 17	4 18	5 19	6 20	7 21	8 22	9 23	10 24	11 25	12 26	13 27	14 28
Distance From Wall, inches	.85	2.7	4.7	6.9	9.5	12.8	17.4	30.1	34.7	38.	40.6	42.8	44.8	46.6
Percent of Diameter	1.8	5.7	9.9	14.6	20.1	26.9	36.6	63.4	73.1	79.9	85.4	90.1	94.3	98.2

FIGURE 4. SAMPLING POINTS

duced, 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 gas is treated in an afterburner, cleaned in cyclones and a baghouse, and exhausted through a steel stack.

While only about 1800 cfm of gas is passed through the blast furnace, it is diluted at several points so that about 25,000 cfm is exhausted through the stack. The first point of dilution is air leakage into the furnace through the feed system. Additional air is added in the afterburner. A regulator bleeds air into the system just before the baghouse to maintain the baghouse below a maximum temperature of 300 F. Air also enters the system through leaks in the baghouse structure.

#### Operation of Blast Furnace

Process air to the blast furnace is furnished by a Roots blower at a constant volume of 1600-1800 cfm. The air pressure at the bottom is determined by the porosity of the charge in the furnace and plugging of the tuyeres. The pressure is usually reduced by about 10 inches of water each time the slag is tapped and the tuyeres are rodded. In the first run the pressure range was 40-50 inches; in the second, 30-40 inches, and in the third, 50-65 inches.

The lead was tapped continuously at the rate indicated by the table below.

<u>Run</u>	<u>Rate, tons/hr</u>
1	3.7
2	2.5
3	5.1

The furnace is rated at 77 tons per day or 3.2 tons per hour. The high rate observed on Run 3 may have been due to measuring the rate during a short period of time (the first half of the run) because the lead rate

for the entire run was inadvertently lost. The operator estimated that the rate was about equal to that in Run 1. On a short-term basis, the lead output stops or is greatly reduced for 5 to 15 minutes after tapping slag.

The slag was tapped intermittently and although the slag was not weighed, about 1500 lb was tapped at a time. The slag was tapped once during Run 1, and immediately before and after the start of the run. During Run 2, the slag was tapped 6 times; the first time at the start of the run and the last time at the end of the run. The slag was tapped four times during Run 3, and had been tapped just before and immediately after the run. Tapping slag is an extremely dusty operation and an effective hood was used to exhaust the dust into the sanitary baghouse. Air flow to the furnace was not stopped while tapping slag and rodding tuyeres, a departure from practice at other plants.

The gas outlet of the blast furnace is kept under a slight negative pressure. Air sucked through the feed charge and into the gas outlet dilutes the gases coming from the blast furnace. The air above the feed thimble is sucked into the sanitary baghouse as shown in Figure 2, and is quite dusty because of the loading operations.

#### Operation of the Afterburner

The exhaust from the blast furnace is sucked into the afterburner where a natural gas flame burns the carbon monoxide and hydrocarbons. The natural gas flow to the afterburner was designed to be controlled by the afterburner temperature but the control system was inoperative. The gas flow was adjusted manually to control the temperature to approximately 1200 F. In the first sampling run, the afterburner was on and the temperature was in the 1100-1130 F range. In the second run, the afterburner was off the first 15 minutes and the last hour of the run. The temperature was at a minimum of 1100 F when it was turned on and the gas was turned off when the temperature reached 1550 F. The temperature continued to rise and peaked at 1700 F. It had cooled to about 1200 F

at the end of the run. During the third run the afterburner was on and the temperature was  $1210\text{ F} \pm 10$  for the entire run. Large volumes of air leak into the system through the afterburner.

#### Operation of Air Cleaning System

Gases from the afterburner pass through three cyclones in series which remove larger particulates and cool the gas. The cyclones are cooled by forced air blown over a cooling section and by evaporative cooling of water on the tops of the cyclones. The outside of the cyclones are cooled by natural convection. If the temperature of the gas leaving the cyclones is over 300 F, air is bled into the system to reduce the temperature to 300 F. The air bleed damper for this cooling air was closed during Run 1. During Run 2 it was open allowing several thousand cfm to enter the system, and was closed, or opened slightly for Run 3.

The dust from the cyclones is mixed with the dust from the bag-houses in a closed system so that it was not possible to estimate the relative amounts of dust collected in each system. The amount of dust collected in the dust collection systems was not measured. However, all of it was fed back into the furnace at a rate of about 650 lb/hr. If it is assumed that 90 percent of the dust collected is from the smelting process, uncontrolled emissions amount to 585 lb/hr, or 160 to 235 pounds of dust per ton of reclaimed lead.

#### SAMPLING AND ANALYTICAL PROCEDURES

The off-gases from the lead blast furnace were sampled for particulates,  $\text{SO}_2$ ,  $\text{CO}_2$ ,  $\text{O}_2$ , and  $\text{CO}$ , by the methods described in Appendix D and the Federal Register of August 17, 1971. A single thirteen-point traverse was made for the first particulate sample and the gas was sampled for seven minutes at each point. The points were those recommended for a fourteen-point traverse with the points nearest the stack wall omitted because of possible wall dust contamination. The Project Officer authorized use of this run although only a single traverse was made. Each of

the other two runs was made using two thirteen-point traverses at right angles to each other. Again, the points were those recommended for a fourteen-point traverse with the points nearest to the stack wall omitted. Each point was sampled for seven minutes. A 0.235-inch-diameter probe tip was used for all samples. About 50 cu ft of stack gas was sampled during Run 1 and 100 cu ft was sampled during each of Runs 2 and 3.

Particulate weight was obtained by dessicating the filter paper to constant weight and measuring the increase in weight from the tare. The probe was washed with acetone and the acetone evaporated to a constant weight.

The  $\text{SO}_2$  samples were taken simultaneously with the particulate sample for a period of 1-1/4 hours during which time a sample volume of about 7.5 cu ft 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 when diluting for analysis. One ml of the sample was pippetted into an Erlenmeyer flask for analysis and was titrated against 0.01 normal barium perchlorate using a thorin indicator.

Two Orsat grab samples were taken simultaneously with the particulate sample from a point about 18 inches into the stack. The sample was drawn directly into the analyzer through stainless steel and rubber tubing. About 500 ml of stack gas was drawn through the analyzer to flush the sampling tube and then a 100-ml sample was taken. It was analyzed as follows: the volume was adjusted to 100 ml over acidulated water in the measuring burette; the  $\text{CO}_2$  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 removing any acid vapors with potassium hydroxide solution and the volume decrease measured.

APPENDIX A

COMPLETE PARTICULATE RESULTS  
WITH SAMPLE CALCULATIONS

APPENDIX A  
COMPLETE PARTICULATE RESULTS WITH SAMPLE CALCULATIONS  
SOURCE TESTING CALCULATION FORMS

Test No. 1, 2, 3

No. Runs 3

Name of Firm N.L. Industries

Location of Plant Beech Grove, Indiana

Type of Plant Secondary Lead

Control Equipment Afterburner, cyclones, and baghouse, in series

Sampling Point Locations Stack

Pollutants Sampled Particulates, SO<sub>2</sub>, Orsat

Time of Particulate Test:

Run No. <u>1</u>	Date <u>11/17/71</u>	Begin <u>3:40 pm</u>	End <u>5:11 pm</u>
Run No. <u>2</u>	Date <u>11/18/71</u>	Begin <u>9:15 am</u>	End <u>12:23 pm</u>
Run No. <u>3</u>	Date <u>11/18/71</u>	Begin <u>1:55 pm</u>	End <u>5:01 pm</u>

PARTICULATE EMISSION DATA

Run No.	1	2	3
P <sub>b</sub> - barometric pressure, in. Hg Absolute	29.17	28.99	29.03
P <sub>m</sub> - orifice pressure drop, in. H <sub>2</sub> O	0.92	1.13	1.09
V <sub>m</sub> - volume of dry gas sampled @ meter conditions, ft <sup>3</sup>	56.5	117.7	112.4
T <sub>m</sub> - average gas meter temperature, F	103	86	89
V <sub>m</sub> - volume of dry gas sampled @ std. standard conditions, ft <sup>3</sup> , dry	51.9	110.9	105.5
V <sub>w</sub> - total H <sub>2</sub> O collected, impingers and silical gel., ml.	42.4	56.2	71.1
V <sub>w</sub> - volume of water vapor collected ft <sup>3</sup> @ standard conditions(a)	2.01	2.66	3.37
V <sub>total</sub> - total gas volume, standard conditions, ft <sup>3</sup>	53.94	113.59	108.86
Moisture in the stack gas volume, percent	3.7	2.3	3.1
F <sub>d</sub> - mole fraction of dry gas	0.96	0.98	0.97

(a) 70 F, 29.92 in. Hg

PARTICULATE EMISSION DATA (Cont'd)

Run No.	1	2	3
CO <sub>2</sub> , dry, percent	2.2	2.1	1.5
O <sub>2</sub> , dry, percent	19.0	19.0	19.5
CO, dry, percent	0.5	0.2	0.4
N <sub>2</sub> , dry, percent	78.3	78.7	78.6
M W <sub>d</sub> - molecular weight of dry stack gas	29.1	29.1	29.0
M W - molecular weight of stack gas	28.7	28.8	28.7
T <sub>s</sub> - stack temperature, F	176	182	177
ΔP <sub>s</sub> x (T <sub>s</sub> + 460)	15.951	15.762	15.870
P <sub>s</sub> - stack pressure, in. Hg absolute	29.2	29.0	29.0
V <sub>s</sub> - stack velocity @ stack conditions, fpm	2396	2371	2393
A <sub>s</sub> - stack area, in <sup>2</sup>	1780	1780	1780
Q <sub>s</sub> - stack gas volume @ standard conditions, (a) ft <sup>3</sup> dry	23,200	22,903	23,120
T <sub>t</sub> - net time of test, min.	91	188	186
D <sub>n</sub> - sampling nozzle diameter, in.	0.235	0.235	0.235
Percent I - percent isokinetic	100	105	100
m <sub>f</sub> - particulate, probe and filter, mg	9.4	19.2	15.8
m <sub>t</sub> - particulate, total, mg	(b)	339.3	271.6
C <sub>an</sub> - particulate, probe and filter, gr/scf, dry	0.0028	0.0027	0.0023
C <sub>ao</sub> - particulate, total, gr/scf	(b)	0.0471	0.0396

(a) 70 F, 29.92 in. Hg

(b) Not available.

PARTICULATE EMISSION DATA (Cont'd)

Run	1	2	3
$C_{at}$ - particulate; probe and filter, gr/ft <sup>3</sup> @ stack conditions	0.0022	0.0021	0.0018
$C_{au}$ - particulate; total, gr/ft <sup>3</sup> @ stack conditions	(a)	0.0368	0.0310
$C_{aw}$ - particulate; probe and filter, 1b/hr	0.5541	0.5231	0.4570
$C_{ax}$ - particulate; total, 1b/hr	(a)	9.2439	7.8566
$C_p$ - particulate; probe and filter, 1b/ton lead	0.1498	0.2092	0.1523
$C_{pt}$ - particulate; total 1b/ton lead	(a)	3.6976	2.6189
$C_{ls}$ - lead emission; probe and filter, gr/scf (b)	(a)	0.00035	0.00033
$C_{la}$ - lead emission; probe and filter, gr/ft <sup>3</sup>	(a)	0.00027	0.00026
$C_{lh}$ - lead emission; probe and filter, 1b/hr	(a)	0.0679	0.0648
$C_{lt}$ - lead emission; probe and filter, 1b/ton	(a)	0.0272	0.0216

(a) Not available.

(b) 70 F, 29.92 in. Hg

EXAMPLE PARTICULATE CALCULATIONS

## SAMPLE NUMBER 3

1. Volume of dry gas sampled at standard conditions - 70 F, 29.92 in. Hg, ft<sup>3</sup>

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

$$= \frac{17.7 \times 112.4 \left( 29.03 + \frac{1.09}{13.6} \right)}{112.4 + 460} \quad \text{dry}$$

$$= 105.5 \text{ ft}^3$$

2. Volume of water vapor at 70 F and 29.92 in. Hg, ft<sup>3</sup>

$$V_{w\text{gas}} = 0.0474 \times V_w$$

$$V_{w\text{gas}} = 0.0474 \times 71.1$$

$$= 3.37 \text{ ft}^3$$

3. Percent moisture in stack gas

$$\% M = \frac{100 \times V_{w\text{gas}}}{V_{m\text{std}} + V_{w\text{gas}}}$$

$$= \frac{100 \times 3.37}{105.5 + 3.37}$$

$$= 3.1 \text{ percent}$$

4. Mole fraction of dry gas

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

$$= \frac{100 - 3.1}{100}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd).

## 5. Average molecular weight of dry stack gas

$$\begin{aligned}
 M\ W_d &= (CO_2 \times \frac{44}{100}) + (O_2 \times \frac{32}{100}) + (N_2 + CO) \times \frac{28}{100} \\
 &= (1.5 \times 0.44) + (19.5 \times 0.32) + (79 \times 0.28) \\
 &= 29.0
 \end{aligned}$$

## 6. Molecular weight of stack gas

$$\begin{aligned}
 M\ W &= M\ W_d \times M_d + 18 (1 - M_d) \\
 &= 29.0 \times 0.97 + 18 (1 - 0.97) \\
 &= 28.7
 \end{aligned}$$

## 7. Stack velocity @ stack conditions, fpm

$$\begin{aligned}
 V_s &= 4350 \times \sqrt{\Delta P_s \times (T_s + 460)} \quad \left( \frac{1}{P_s \times M\ W} \right)^{1/2} \text{ fpm} \\
 &= 4350 \times 15.87 \quad \left[ \frac{1}{29.0 \times 28.7} \right]^{1/2} \\
 &= 2393 \text{ fpm}
 \end{aligned}$$

## 8. Stack gas volume at standard conditions, scfm

$$\begin{aligned}
 Q_s &= \frac{0.123 \times V_s \times A_s \times \frac{P_s}{T_s + 460} M_d}{M_d} \text{ scfm} \\
 &= \frac{0.123 \times 2393 \times 1780 \times 29.0 \times 0.97}{177 + 460} \\
 &= 23,120 \text{ scfm}
 \end{aligned}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

9. Sampling velocity, percent of isokinetic

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

$$= \frac{1032 \times (177 + 460) \times 105.5}{2393 \times 186 \times 29.0 \times 0.97 \times (0.235)^2}$$

$$= 100 \text{ percent}$$

10. Percent excess air at sampling point

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

$$= \frac{100 \times 19.5}{(0.236 \times 78.6) - 19.5}$$

$$= 1385 \text{ percent}$$

11. Particulate - probe and filter, gr/scf

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

$$= 0.0154 \times \frac{15.8}{105.5}$$

$$= 0.0023 \text{ gr/scf}$$

12. Particulate - total, gr/scf

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

$$= 1.54 \times 10^{-2} \frac{271.6}{105.5}$$

$$= 0.0326 \text{ gr/scf}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

13. Particulate - probe and filter, gr/ft<sup>3</sup> at stack conditions

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

$$= \frac{17.7 \times 0.0023 \times 29.0 \times 0.97}{177 + 460}$$

$$= 0.0018 \text{ gr/ft}^3$$

14. Particulate, total, gr/ft<sup>3</sup> at stack conditions

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

$$= \frac{17.7 \times 0.0396 \times 29.0 \times 0.97}{177 + 460}$$

$$= 0.0310 \text{ gr/ft}^3$$

15. Particulate - probe and filter

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

$$= 0.00857 \times 0.0023 \times 23,120$$

$$= 0.4570 \text{ lb/hr}$$

16. Particulate - total, lb/hr

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

$$= 8.57 \times 10^{-3} \times 0.0396 \times 23,120$$

$$= 7.8566 \text{ lb/hr}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

17. Particulate; probe and filter, 1b/ton lead

$$\begin{aligned} C_p &= C_{aw}/R \\ &= 0.457/3.0 \\ &= 0.1523 \text{ lb/ton lead} \end{aligned}$$

18. Particulate; total 1b/ton lead

$$\begin{aligned} C_{pt} &= C_{ax}/R \\ &= 7.8566/3.0 \\ &= 2.6189 \text{ lb/ton lead} \end{aligned}$$

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

$$\begin{aligned} C_{1s} &= 0.0154 \times \frac{m_1}{V_m \text{ std}} \\ &= 0.0154 \times \frac{2241}{105.5} \\ &= 0.00033 \text{ gr/scf} \end{aligned}$$

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

$$\begin{aligned} C_{1a} &= \frac{17.7 \times C_{1s} \times P_s \times M_d}{(T_s + 460)} \\ &= \frac{17.7 \times 0.00033 \times 29.0 \times 0.97}{177 + 460} \\ &= 0.00026 \text{ gr/scf} \end{aligned}$$

21. Lead emission; probe and filter, 1b/hr

$$\begin{aligned} C_{1h} &= 8.57 \times 10^{-3} \times C_{1s} \times C_s \\ &= 8.57 \times 10^{-3} \times 0.00033 \times 23,120 \\ &= 0.0648 \text{ lb/hr} \end{aligned}$$

22. Lead emission; probe and filter, 1b/ton lead

$$\begin{aligned} C_{1t} &= C_{1h}/R \\ &= 0.0648/3.0 \\ &= 0.0216 \text{ lb/ton lead} \end{aligned}$$

APPENDIX B

COMPLETE GASEOUS RESULTS WITH  
SAMPLE CALCULATIONS

## APPENDIX B

COMPLETE GASEOUS RESULTS WITH SAMPLE CALCULATIONSSO<sub>2</sub> EMISSION DATA

Run No.	1	2	3
Date (1971)	11/17	11/18	11/18
T <sub>m</sub> - average gas meter temperature, F	77	64	67
P <sub>b</sub> - barometric pressure, in. Hg absolute	29.2	29.0	29.0
V <sub>m</sub> - volume of dry gas sampled @ meter conditions, ft <sup>3</sup>	9.6	7.5	7.5
V <sub>m</sub> standard	9.24	7.35	7.31
V <sub>t</sub> - volume barium perchlorate, ml	9.6	4.55	3.50
V <sub>tb</sub> - volume barium perchlorate blank, ml	0	0	0
N - normality of barium perchlorate	0.01	0.01	0.01
V <sub>soln</sub> - volume SO <sub>2</sub> soln, ml	100	100	100
V <sub>a</sub> - volume aliquot, ml	1	1	1
C <sub>SO<sub>2</sub></sub> - lb/ft <sup>3</sup> x 10 <sup>-5</sup>	7.26	4.37	3.38
C <sub>SO<sub>2</sub></sub> - ppm (dry)	440	264	204
W <sub>SO<sub>2</sub></sub> - lb/hr	101	59	46

SO<sub>2</sub> EXAMPLE CALCULATIONS

## RUN 2

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

$$= 7.5 \times \frac{530}{524} \times \frac{29.0}{29.9}$$

$$= 7.35 \text{ ft}^3$$

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

$$= \frac{7.05 \times 10^{-5} \times 4.55 \times 0.01 \times 100}{7.35 \times 1}$$

$$= 4.37 \times 10^{-5} \text{ lb/ft}^3$$

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

$$= 6.05 \times 10^6 \times 4.37 \times 10^{-5}$$

$$= 264 \text{ ppm}$$

$$4. \quad W_{SO_2} = C_{SO_2} \times Q_s \times 60 \text{ lb/hr}$$

$$= 4.37 \times 10^{-5} \times 22899 \times 60$$

$$= 59.0 \text{ lb/hr}$$

APPENDIX C

FIELD DATA

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

## APPENDIX C

PRELIMINARY FIELD DATASTACK GEOMETRYPlant N.L. Industries, Beech Grove, IndianaTest No. 1Wall thickness, in. 1/4Location Baghouse outletInside diameter of stack 47.5Date 11/17/71Stack area  $\frac{\pi}{4} (47.5)^2 = 1780 \text{ in.}^2$   
12.4 ft}^2

Point	Percent Diameter for Circular Stack	Distance from Outside of Sample Port, in.
1	1.8	$7/8 \sim 1 + .75 = 1.75$
2	5.7	$2.75 + .75 = 3.50$
3	9.9	$4.75 + .75 = 5.50$
4	14.6	$7.0 + .75 = 7.75$
5	20.1	$9.5 + .75 = 10.25$
6	26.9	$12.75 + .75 = 13.50$
7	36.6	$17.5 + .75 = 18.25$
8	63.4	$30.5 + .75 = 31.25$
9	73.1	$35.0 + .75 = 35.75$
10	79.9	$38.25 + .75 = 39.0$
11	85.4	$41.0 + .75 = 41.75$
12	90.1	$43.25 + .75 = 44.0$
13	94.3	$45.25 + .75 = 46.0$
14	98.2	$47.125 + .75 = 47.75$

VELOCITY TRAVERSE FIELD DATAPlant N.L. Industries, Beech Grove, IndianaTest Preliminary velocity traverseDate 11/16/71Operator Paul R. WebbMeter  $\Delta H$  1.84

Clock Time	Point	$P_s$ in., $H_2O$
6:53 pm	14E	0.3
	13	0.35
	12	0.4
	11	0.4
	10	0.4
	9	0.4
	8	0.5
	7	0.5
	6	0.5
	5	0.5
	4	0.45
	3	0.45
	2	0.35

PARTICULATE CLEANUP SHEETPlant N.L. Industries, Beech Grove, IndianaDate November 17 and 18, 1971Operators P.R. Webb, B.E. Campbell

Run No.	1	2	3
Impinger water volume, ml	232	238	253
Final	<u>200</u>	<u>200</u>	<u>200</u>
Initial	<u>32</u>	<u>38</u>	<u>53</u>
Collected			
Drierite, weight, gm	517.3	529.1	530.1
Final	<u>506.9</u>	<u>510.9</u>	<u>512.0</u>
Initial	<u>10.4</u>	<u>18.2</u>	<u>18.1</u>
Gain			
Total moisture, gm plus ml	42.4	56.2	71.1
Probe, acetone wash residue, mg (a)	1.5	5.9	3.0
Filter weight, mg	287.2	290.7	308.6
Final	<u>299.3</u>	<u>277.4</u>	<u>295.8</u>
Tare	<u>7.9</u>	<u>13.3</u>	<u>12.8</u>
Gain			
Total particulate weight, probe residue and filter, mg	9.4	19.2	15.8
Weight residue from chloroform-ether extract, mg			
Impinger water	(b)	70.2	75.6
Impinger wash water	(b)	<u>32.1</u>	<u>74.9</u>
Total	(b)	102.3	150.5
Weight residue from aqueous phase, mg (a)			
Impinger water	(b)	160.5	75.2
Impinger wash water	(b)	<u>9.1</u>	<u>5.0</u>
Total	(b)	169.6	80.2
Weight residue from acetone wash, mg (a)	(b)	48.2	25.1
Total weight, back half residue, mg	(b)	320.1	255.8
Total weight, front half plus back half, mg	9.4	339.3	271.6

(a) Water and acetone blanks subtracted; water blank = 0.021 mg/ml.  
acetone blank = 0.027 mg/ml.

(b) Not available.





## PARTICULATE FIELD DATA RUN No. 2 (Cont'd)

Plant N.L. Industries, Beech Grove, Indiana  
 Date 11/18/71  
 Run No. 2 (Cont'd)  
 Operator P.R. Webb, B.E. Campbell  
 Sample Box No. 1  
 Meter Box No. 1  
 Meter  $\Delta H$  @ 1.84  
 C Factor 1.0

Ambient Temperature, F 69  
 Barometric Pressure, in. Hg 28.99  
 Assumed Moisture, Percent 7  
 Heater Box Setting, F 255  
 Probe Tip Diameter, Inches 0.235  
 Probe Length 48 in.  
 Probe Heater Setting 5 - 252 F  
 Average  $\Delta P$  - Average  $\Delta H$

Point	Clock Time	Dry Gas Meter, CF	Pitot, in. H <sub>2</sub> O $\Delta P$	Orifice H, in. H <sub>2</sub> O Desired Actual	Dry Gas Temp., F	Pump Vacuum, in. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press., in. Hg	Stack Temp., F
14S	10:52 am	170.5	0.24	0.66	88	82	5.1	244	56	(a) 180
13	10:59 am	175.0	0.25	0.70	95	86	5.2	245	58	(a) 185
12	11:06 am	178.0	0.30	0.85	98	87	5.9	248	56	(a) 186
11	11:13 am	181.0	0.32	0.90	100	89	5.9	250	56	(a) 186
10	11:20 am	185.0	0.34	0.96	102	91	6.1	252	58	(a) 191
9	11:27 am	189.5	0.40	1.11	104	91	6.7	252	58	(a) 191
8	11:34 am	194.0	0.50	1.4	102	92	7.8	254	58	(a) 191
7	11:41 am	200.0	0.62	1.7	99	88	9.0	250	58	(a) 191
6	11:48 am	205.0	0.53	1.5	98	88	8.0	248	58	(a) 185
5	11:55 am	210.0	0.50	1.4	96	86	7.4	246	57	(a) 187
4	12:02 pm	215.0	0.48	1.35	95	87	7.4	245	58	(a) 187
3	12:09 pm	220.0	0.45	1.25	94	85	7.1	245	59	(a) 182
2	12:16 pm	225.0	0.33	0.93	93	85	6.0	245	57	(a) 177
1	12:23 pm	230.2								182
Average			0.40		1.13	93	82			
Total		117.7								

Comments: Windy, overcast; did not take No. 1 point; to close to stack.

(a) Atmospheric.

## PARTICULATE FIELD DATA

Plant	<u>N.L. Industries, Beech Grove, Indiana</u>	
Date	<u>11/18/71</u>	
Run No.	<u>3</u>	
Operator	<u>P.R. Webb, P.E. Campbell</u>	
Sample Box No.	<u>1</u>	
Meter Box No.	<u>1</u>	
Meter ΔH (a)	<u>1.84</u>	
C Factor	<u>1</u>	

Ambient Temperature, F	70
Barometric Pressure, in. Hg	29.03
Assumed Moisture, Percent	7
Heater Box Setting, F	245
Probe Tip Diameter, Inches	0.235
Probe Length	48 in.
Probe Heater Setting	5 - 236 F
Average $\Delta P$	

(a) Atmospheric.

**Comments:** Probe temperature, 232-236 F.  
Run No. 3 continued on next page.

Plant N. No. Industries, Beech Grove, Indiana

Date 11/18/71

Run No. 3 (Cont'd)

Operator P.R. Webb

Sample Box No. 1

Meter Box No. 1

Meter  $\Delta H @$  1.84

C Factor 1.1

Ambient Temperature, F 70

Barometric Pressure, in. Hg 29.03

Assumed Moisture, Percent 7

Heater Box Setting, F 245

Probe Tip Diameter, Inches 0.235

Probe Length 48 in.

Probe Heater Setting 5 - 236 F

Average  $\Delta P$  Average  $\Delta H$ 

Point	Clock Time	Dry Gas Meter, CF	Pitot, in. H <sub>2</sub> O $\Delta P$	Orifice H, in. H <sub>2</sub> O Desired Actual	Dry Gas Temp., F Inlet Outlet	Pump Vacuum, in. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press., in. Hg	Stack Temp., F
28 E	3:30 pm	285.9	0.32		92 86	5.1	250	63	(a)	180
27	3:37 pm	290.0	0.36		96 87	5.4	255	63	(a)	180
26	3:44 pm	293.7	0.38		1.0 98	5.9	255	63	(a)	180
25	3:51 pm	297.5	0.40		1.08 99	6.1	254	63	(a)	184
24	3:58 pm	301.7	0.42		1.12 100	6.1	255	62	(a)	181
23	4:05 pm	305.7	0.45		1.20 100	6.2	255	61	(a)	181
22	4:12 pm	310.7	0.48		1.30 101	6.6	256	61	(a)	181
21	4:19 pm	318.0	0.52		1.38 103	6.8	256	61	(a)	181
20	4:26 pm	322.4	0.52		1.38 103	6.9	258	61	(a)	181
19	4:33 pm	327.5	0.45		1.20 103	6.1	257	61	(a)	181
18	4:40 pm	332.6	0.45		1.20 103	6.1	256	61	(a)	181
17	4:47 pm	337.6	0.43		1.15 103	6.0	255	61	(a)	181
16	4:54 pm	342.6	0.30		0.81 102	4.9	256	61	(a)	178
15	5:01 pm	346.1								
Average					1.09	95	85			
Total		112.4								179

Comments:

(a) Atmospheric.

GAS SAMPLING FIELD DATA

Date 11/18/71 Plant N.L. Industries, Beech Grove, Indiana  
 Material Sampled for SO<sub>2</sub>  
 Barometric Pressure 29.00 in. Hg  
 Ambient Temperature 70 F  
 Run Number 2 Comments:  
 Power Stat Setting Heated  
 Filter Used: Yes X No \_\_\_\_\_  
 Operator W.C. Baytos, D. Hupp

Clock Time	Meter (ft <sup>3</sup> )	Flow Meter Setting (CFH)	Meter Temperature F
9:15 am	0	6.0	64
9:20 am	0.6	6.0	64
9:25 am	1.1	6.0	64
9:30 am	1.5	6.0	64
9:35 am	2.1	6.0	63.5
9:40 am	2.6	6.0	63.5
9:45 am	3.05	6.0	63.5
9:50 am	3.5	6.0	63.0
9:55 am	4.0	6.0	63.0
10:00 am	4.4	6.0	62.5
10:10 am	5.3	6.0	
10:15 am	6.0	6.0	65.0
10:20 am	6.5	6.0	65.0
10:25 am	7.1	6.0	66.0
10:30 am	7.5	6.0	65.0

Comments: Probe temperature, 180 F.  
 Recovered 67 ml - diluted to 100 ml.

Titration		0.01 N Ba(ClO <sub>4</sub> ) <sub>2</sub>
Aliquot, ml	Titer, ml	
1.00	4.52	
1.00	4.58	
blank	0	

GAS SAMPLING FIELD DATA

Date 11/18/71 Plant N.L. Industries, Beech Grove, Indiana  
 Material Sampled for SO<sub>2</sub>  
 Barometric Pressure 29.97 in. Hg  
 Ambient Temperature 70 F  
 Run Number 3 Comments:  
 Power Stat Setting Heated  
 Filter Used: Yes X No \_\_\_\_\_  
 Operator W.C. Baytos, D. Hupp

<u>Clock Time</u>	<u>Meter (ft<sup>3</sup>)</u>	<u>Flow Meter Setting (CFH)</u>	<u>Meter Temperature F</u>
3:40 pm	0	6.0	66
3:45 pm	0.5	6.0	66
3:50 pm	1.0	6.0	66
3:55 pm	1.4	6.0	66
4:00 pm	2.0	6.0	67
4:05 pm	2.5	6.0	67
4:10 pm	3.0	6.0	67
4:15 pm	3.4	6.0	67
4:20 pm	4.0	6.0	67
4:25 pm	4.5	6.0	67
4:30 pm	5.0	6.0	67
4:35 pm	5.5	6.0	67
4:40 pm	6.0	6.0	68
4:45 pm	6.5	6.0	67
4:50 pm	7.0	6.0	66
4:55 pm	7.5	6.0	66

Comments: Probe temperature, 190 F.  
 Recovered 67 ml - diluted to 100 ml.

<u>Titration</u>	
<u>Aliquot, ml</u>	<u>Titer, ml</u>
1.00	3.50
1.00	3.50
blank	0

ORSAT GAS COMPOSITIONPlant N.L. Industries, Beech Grove, IndianaDate November 17, 18, 1971

Run	Date	Time	CO <sub>2</sub> , Percent	O <sub>2</sub> , Percent	CO, Percent	N <sub>2</sub> , Percent
0	11/17/71	2:05 pm	0.6	19.0	0	80.4
1	11/17/71	3:20 pm	2.3	18.5	0.4	78.8
1	11/17/71	4:55 pm	2.0	19.5	0.5	78.0
1	Average		2.2	19.0	0.5	78.3
2	11/18/71	9:30 am	2.8	18.3	0.1	78.8
2	11/18/71	11:40 am	1.3	19.7	0.2	78.8
2	Average		2.1	19.0	0.2	78.7
3	11/18/71	2:35 pm	1.5	19.5	0.6	78.4
3	11/18/71	3:55 pm	1.6	19.4	0.2	78.8
3	Average		1.5	19.5	0.4	78.6

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 N. L. Industries, Beech Grove, Indiana.

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  $\Delta p$  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  $\Delta p$  and temperature variations. Appendix C is a record of the field data for the three tests completed at N. L. Industries, Beech Grove.

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 blank values were relatively high and may have reduced the results by as much as ten percent.

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 TRAVERSSES 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

## PROPOSED RULE MAKING

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.

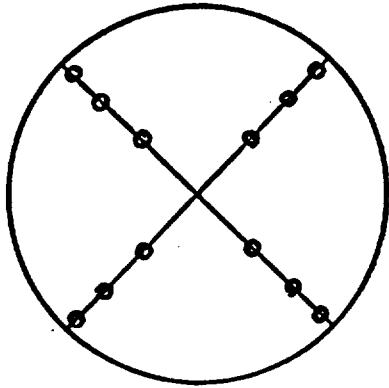
2.1 Selection of a sampling site and minimum number of traverse points.

2.1.1 Select a sampling site that is at least eight duct or duct diameters upstream 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 2.3.2.

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 1-2.

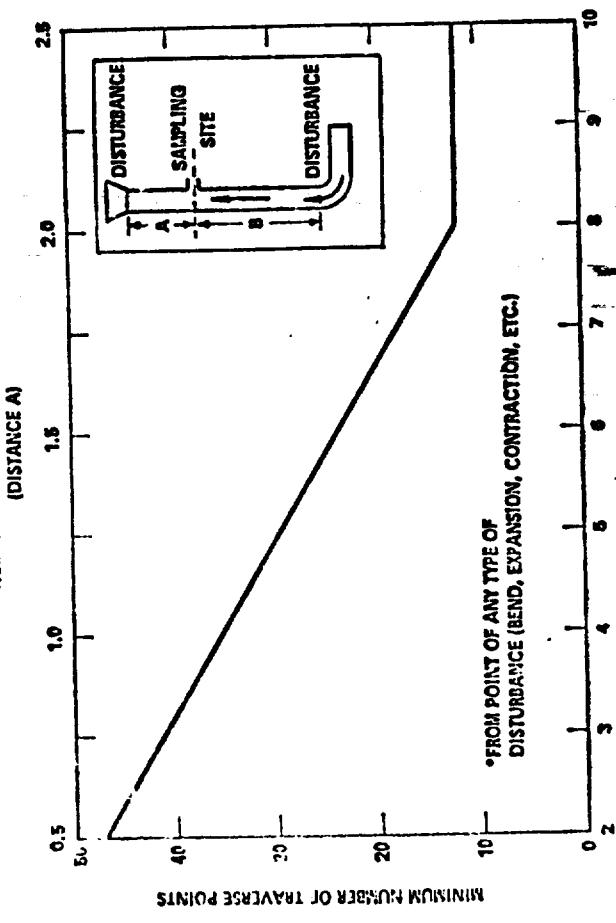
NUMBER OF DUCT DIAMETERS UPSTREAM<sup>a</sup> (DISTANCE A)

Figure 1-1. Minimum number of traverse points.

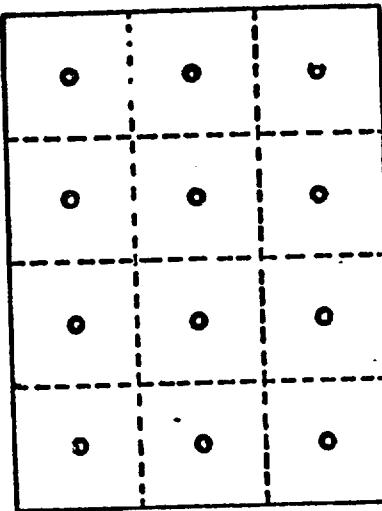


Figure 1-2. Cross section of circular stack showing location of traverse points on perpendicular diameters.



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

## PROPOSED RULE MAKING

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

Traverso 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	57.5	29.6	25.0	21.8	19.4	
9		91.8	82.3	73.1	62.5	38.2	30.3	26.1	23.0	
10		97.5	88.2	79.9	71.7	61.3	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
17								95.6	90.3	85.4
18									98.6	93.3
19										88.4
20										23.9
21										56.1
22										91.3
23										86.8
24										38.7

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

Devorin, Howard, et al. Air Pollution Control 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 Company, Bulletin WP-50. Manufacturing Co., Los Angeles, California, 1963.

Standard Method for Sampling Stacks for Particulate Matter. Iu: 1971 Book of ASTM Standards, Part 23, Philadelphia, 1971. ASTM Designation D-2929-71.

METHOD 8—DETERMINATION OF STACK GAS VELOCITY (TYPE S PILOT 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 (Stauschibe or reverse type) pilot 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 pilot tube should

not be used in the case of nondirectional flow.

2. Apparatus.

2.1. Pilot 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-dilled 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. Pilot tube—Standard type, to calibrate Type S pilot 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.

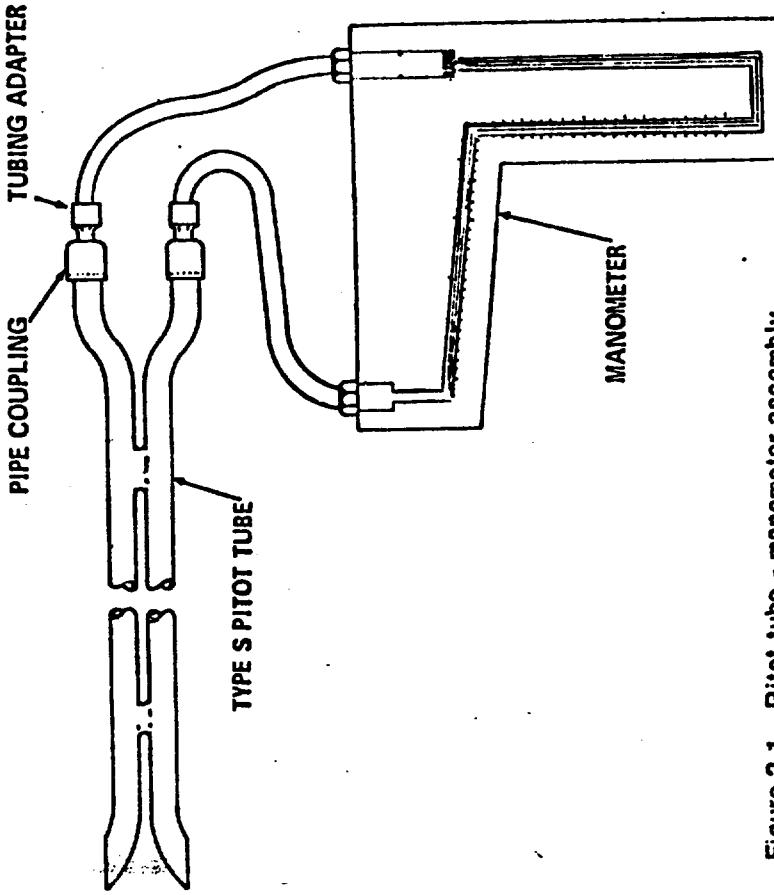


Figure 2-1. Pilot tube - manometer assembly.



## PROPOSED RULE MAKING

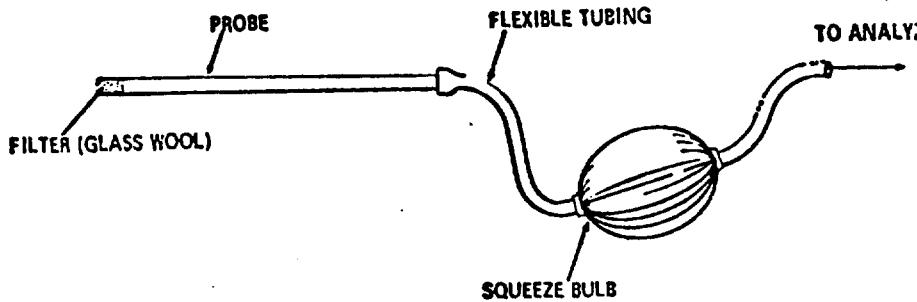


Figure 3-1. Grab-sampling train.

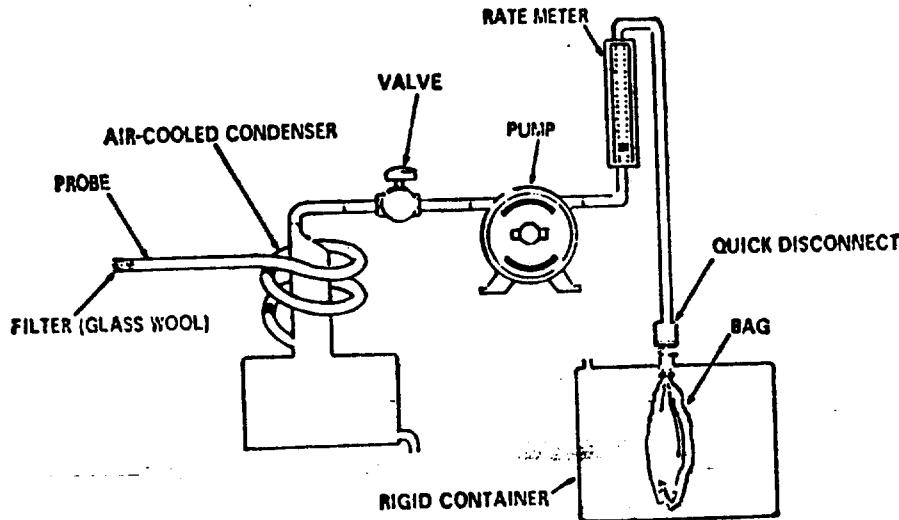


Figure 3-2. Integrated gas-sampling train.

## 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<sub>2</sub>, O<sub>2</sub>, 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<sub>2</sub>.

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.

4.3 EA =

$$\frac{(\% O_2) - 0.5(\% CO)}{0.2(\% N_2) - (\% O_2) + 0.5(\% CO)} \times 100$$

equation 3-1

where:

$$\begin{aligned} \% EA &= \text{Percent excess air.} \\ \% O_2 &= \text{Percent oxygen by volume, dry basis.} \\ \% N_2 &= \text{Percent nitrogen by volume, dry basis.} \\ \% CO &= \text{Percent carbon monoxide by volume, dry basis.} \\ 0.264 &= \text{Ratio of oxygen to nitrogen in air by volume.} \end{aligned}$$

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:

$$\begin{aligned} M_d &= \text{Dry molecular weight, lb./lb.-mole.} \\ \% CO_2 &= \text{Percent carbon dioxide by volume, dry basis.} \\ \% O_2 &= \text{Percent oxygen by volume, dry basis.} \\ \% N_2 &= \text{Percent nitrogen by volume, dry basis.} \\ 0.44 &= \text{Molecular weight of carbon dioxide divided by 100.} \\ 0.32 &= \text{Molecular weight of oxygen divided by 100.} \\ 0.28 &= \text{Molecular weight of nitrogen divided by 100.} \end{aligned}$$

## 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.<sup>1</sup>

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<sup>1</sup> 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 Pilot 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.

<sup>1</sup> Trade name.

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.



## PROPOSED RULE MAKING

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

2.1.4 Filter holder—Pyrex<sup>1</sup> 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 re-

placing the tip with a 1/2-inch ID glass tube extending to 1/2-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.

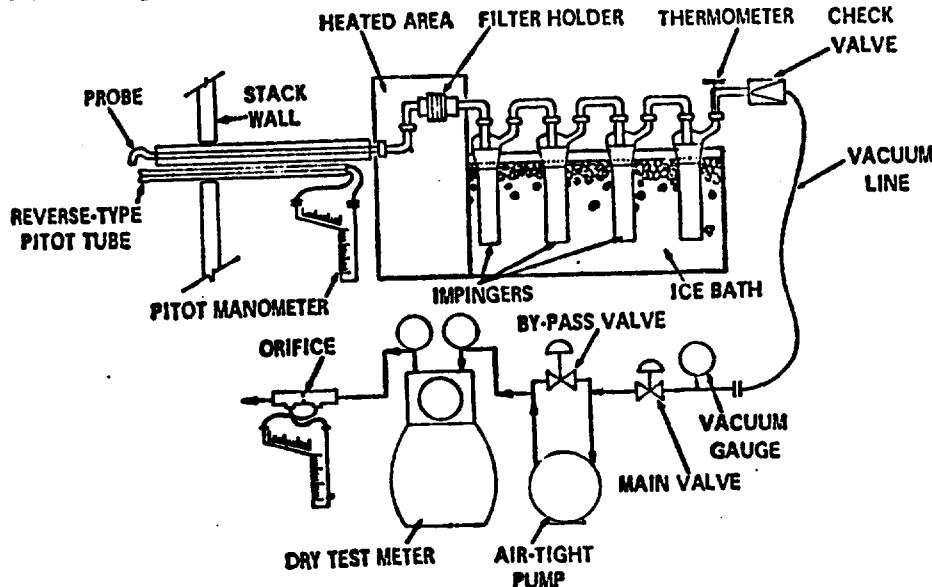


Figure 5-1. Particulate-sampling train.

2.1.7 Barometer—To measure atmospheric pressure to  $\pm 0.1$  in. Hg.

2.3 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  $\pm 0.1$  mg.

2.3.4 Beakers—250 ml.

<sup>1</sup> Trade name.

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

2.3.6 Trip balance—300 g. capacity, to measure to  $\pm 0.05$  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,<sup>2</sup> 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<sup>3</sup> 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.

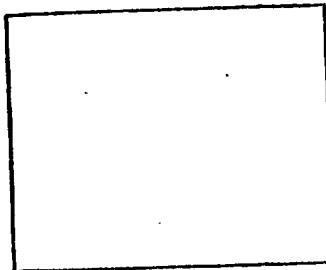
4.1.3 Particulate train operation. For each run record the data required on the example sheet shown in Figure 5-2. Take readings at each sampling point at least every 5 minutes and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position 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.

<sup>2</sup> Dry using Drierite at 70°  $\pm$  10° F.

## PROPOSED RULE MAKING

15715

PLANT \_\_\_\_\_  
LOCATION \_\_\_\_\_  
OPERATOR \_\_\_\_\_  
DATE \_\_\_\_\_  
RUN NO. \_\_\_\_\_  
SAMPLE BOX NO. \_\_\_\_\_  
METER BOX NO. \_\_\_\_\_  
METER  $\Delta H_3$  \_\_\_\_\_  
C FACTOR \_\_\_\_\_



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

### **SCHEMATIC OF STACK CROSS SECTION**

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 washing 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 after

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:

sicate, 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. Dessiccate and dry to a constant weight. Report results to the nearest 0.5 mg.

Coufainer 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 ether. Combine the ether and chloroform extracts, transfer to a tared beaker and evaporate at 70° F. until no solvent remains. Desiccate, 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.

## PROPOSED RULE MAKING

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	
	IMPIINGER 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):

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

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 meter.

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.

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

equation 5-1

where:  
 $V_{\text{sample}}$  = 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.

$T_{\text{sample}}$  = Absolute temperature at standard conditions, 530° R.

$T_m$  = Average dry gas meter temperature, °R.

$P_{\text{bar}}$  = Barometric pressure at the orifice meter, in. Hg.

$\Delta H$  = Pressure drop across the orifice meter, in. Hg.

13.6 = Specific gravity of mercury.

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

6.1.3 Volume of Water vapor.

$$V_{\text{water}} = V_i \left( \frac{\rho_{\text{H}_2\text{O}}}{M_{\text{H}_2\text{O}}} \right) \left( \frac{RT_m}{P_{\text{std}}} \right) = \\ (0.0474 \frac{\text{cu. ft.}}{\text{ml.}}) V_i$$

equation 5-2

where:  
 $V_{\text{water}}$  = Volume of water vapor in the gas sample (standard conditions), cu. ft.

$V_i$  = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.

$\rho_{\text{H}_2\text{O}}$  = Density of water, 1 g./ml.

$M_{\text{H}_2\text{O}}$  = Molecular weight of water, 18 lb./lb. mole.

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

$T_{\text{sample}}$  = Absolute temperature at standard conditions, 530° R.

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

6.1.4 Total gas volume.

$$V_{\text{total}} = V_{\text{sample}} + V_{\text{water}}$$

equation 5-3

where:

$V_{\text{total}}$  = Total volume of gas sample (standard conditions), cu. ft.

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

$V_{\text{water}}$  = 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.

$$c' = \left( 0.0154 \frac{\text{gr.}}{\text{mg.}} \right) \left( \frac{M_p}{V_{\text{total}}} \right)$$

equation 5-4

where:

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

$M_p$  = Total amount of particulate matter collected, mg

$V_{\text{total}}$  = 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



## PROPOSED RULE MAKING

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-arsenophenylazo)-2-naphthol-3, 6-disulfonic acid, disodium salt (or equivalent). Dissolve 0.20 g. in 100 ml. distilled water.

3.3.4 Barium perchlorate (0.01N)—Dissolve 1.95 g. of barium perchlorate  $[\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}]$  in 200 ml. distilled water and dilute to 1 liter with isopropanol. Standardize with sulfuric acid.

3.3.5 Sulfuric acid standard (0.01N)—Purchase or standardize against a primary standard to  $\pm 0.0002\text{N}$ .

#### 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 air 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. Erlenmeyer flask. Add 40 ml. of isopropanol and 2 to 4 drops of thorin indicator. Titrate to a pink endpoint using 0.01N 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.

$$V_{m,014} = V_m \left( \frac{T_{std}}{T_m} \right) \left( \frac{P_{bar}}{P_{std}} \right) = \left( 17.71 \frac{^{\circ}\text{R}}{\text{in. Hg}} \right) \frac{V_m P_{bar}}{T_m} \quad \text{equation 6-1}$$

where:

$V_{m,014}$  = 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.

$$c_{SO_2} = \left( 7.05 \times 10^{-5} \frac{\text{lb.-l.}}{\text{g.-ml.}} \right) \frac{(V_t - V_{tb}) (N) \left( \frac{V_{so_2}}{V_t} \right)}{V_{std}} \quad \text{equation 6-2}$$

where:

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

$7.05 \times 10^{-5}$  = 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./1. lb.-1./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.

$V_{std}$  = Total solution volume of sulfur dioxide, ml.

$V_t$  = Volume of sample aliquot titrated, ml.

$V_{m,014}$  = 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<sub>2</sub> and SO<sub>3</sub> in Flue Gases. Journal of the Institute of Fuel, 24: 237-243, 1961.

Matty, R. E. and E. K. Diehl. Measuring Flue-Gas SO<sub>2</sub> and SO<sub>3</sub>. 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<sup>1</sup> 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<sup>1</sup> 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, 30 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.

<sup>1</sup> Trade name.

APPENDIX E

LABORATORY REPORT

## APPENDIX E

LABORATORY REPORTBACK HALF IMPINGER WATER RESIDUE PHASE  
ANALYSIS BY WET CHEMISTRYPlant N.L. Industries, Beech Grove, IndianaDate November 18, 1971Run No. 2

	Organic, mg	Inorganic, mg	Total, mg
Split Residue Mass Before Analysis	35.1	80.2	115.3
Constituent Mass per Split Residue			
SO <sub>3</sub>	< 0.3	< 0.3	< 0.3
SO <sub>4</sub>	0.7	35.7	36.4
Total Sample Residue Mass	70.2	160.5 162.6(a)	230.7
Constituent Mass per Total Residue			
SO <sub>3</sub>	< 0.6	< 0.6	< 0.6
SO <sub>4</sub>	1.4	71.4	72.8

(a) Water blank subtracted; water blank = 0.021 mg/ml.

Plant N.I. Industries, Beech Grove, Indiana

Date November 17, 18, 1971

FRONT HALF LABORATORY REPORT  
ANALYSIS BY ATOMIC ABSORPTION

Run	Probe Acetone Wash Residue, $\mu\text{g}$ (a)	Pb, $\mu\text{g}$ per Residue, $\mu\text{g}$	Pb, $\mu\text{g}$ per Total Residue, $\mu\text{g}$	Filter Catch, $\mu\text{g}$	Pb, $\mu\text{g}$ per Split Filter Sample, $\mu\text{g}$	Pb, $\mu\text{g}$ per Total Filter Catch
1	1500	(b)	(b)	7900	(b)	(b)
2	5900	0.0869	513	13300	0.1489	1980
3	3000	0.1770	531	12800	0.1336	1710

(a) Acetone blank subtracted; blank = 0.027  $\text{mg}/\text{ml}$ .

(b) Not available.

Note: See analysis flow chart for procedure (E-9).

BACK HALF IMPINGER WATER LIQUID PHASE  
ANALYSIS BY WET CHEMISTRYPlant N.L. Industries, Beech Grove, IndianaDate November 18, 1971Run No. 2

Total Sample Volume - 230 ml

Split Sample Volume Analyzed - 80 ml

Constituent	μg/ml	mg per	
		Split Sample Volume	Total Sample Volume
Total Acid	14	1.12	3.25
SO <sub>3</sub> <sup>=</sup>	310	24.8	71.9
SO <sub>4</sub> <sup>=</sup>	140	11.2	32.5
Cl-	8	0.6	1.86
NH <sub>4</sub> <sup>+</sup>	190	15.2	4.41
pH(a)	2.4	---	---

(a) Value is pH units.

BACK HALF IMPINGER WATER LIQUID PHASE  
ANALYSIS BY WET CHEMISTRYPlant N.L. Industries, Beech Grove, IndianaDate November 18, 1971Run No. 2

Total Sample Volume - 238 ml

Split Sample Volume Analyzed - 120 ml

Constituent	μg/ml	mg per	
		Split Sample Volume	Total Sample Volume
SO <sub>3</sub> <sup>=</sup>	0.268	32.15	64.3
SO <sub>4</sub> <sup>=</sup>	0.265	31.8	63.6
Cl <sup>-</sup>	0.0065	0.78	1.56
NH <sub>4</sub> <sup>+</sup>	0.1442	17.3	34.6
NO <sub>3</sub>	(b)	---	---
pH(a)	2.5	---	---

(a) Value is pH units.

(b) None detected.

LABORATORY REPORTOPTICAL EMISSION SPECTROSCOPY TRACE METAL  
( $\mu\text{g}$  element per total sample)Plant N.L. Industries, Beech Grove, IndianaTotal Sample Volume, Run 1 - 230 ml  
Run 2 - 238 mlSplit Sample Volume Analyzed, Run 1 - 80 ml  
Run 2 - 120 ml

Element	Impinger Water	
	Run 1	Run 2
Hg(a)	< 0.2	< 0.1
Be	< 0.2	< 0.1
Cd	< 12.	< 6.
As	< 12.0	36.
V	< 2.	< 1.
Mn	0.7	< 1.
Ni	12.	< 1.
Sb	< 12.	< 6.
Cr	23.	< 1.
Zn	< 23.	< 12.
Cu	1.0	1.
Pb	< 12.	12.
Se(b)	--	--
B	7.0	5.
F(b)	--	--
Li	< 23.	< 36.
Ag	0.2	< 0.1
Sn	< 12.	12.
Fe	70.	5.
Sr	< 7.0	< 6.
Na	< 23.	< 12
K	< 70.	< 12.
Ca	70.	24.
Si	120.	24.
Mg	2.	6.
Co	70.	< 1.
Ba	< 2.	< 1.
Al	12.	6.
Total Mass, $\mu\text{g}$ (d)	(c)	230,700

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

(b) Not detectable by OES absorption.

(c) Not available.

(d) Water blank subtracted; blank = 0.021 mg/ml.

Note: Symbol &lt; indicates minimal detection limits.

LABORATORY REPORTOPTICAL EMISSION SPECTROSCOPY TRACE METAL  
( $\mu$ g of element per total sample)Plant N.L. Industries, Beech Grove, Indiana

Element	Probe Residue		Filter		
	Run 2.	Run 3	Run 2	Run 3	Blank
Hg <sup>(a)</sup>	--	--	--	--	< 1.
Be	< 1.	< 1.	< 1.	< 1.	< 1.
Cd	< 10.	< 10.	< 20.	< 20.	< 20.
As	20.	20.	60.	60.	< 20.
V	< 1.	< 1.	< 1.	< 1.	< 1.
Mn	2.	< 1.	2.	2.	2.
Ni	< 10.	< 10.	< 20.	< 20.	< 20.
Sb	< 10.	< 10.	< 20.	< 20.	< 20.
Cr	< 10.	< 10.	< 20.	< 20.	< 20.
Zn	< 100.	100.	< 200.	< 200.	< 200.
Cu	2.	5.	0	0	2.
Pb <sup>(b)</sup>	--	--	--	--	50.
Se <sup>(c)</sup>	--	--	--	--	--
B	< 1.	< 1.	0	0	4000.
F <sup>(c)</sup>	--	--	--	--	--
Li <sup>(d)</sup>	--	--	--	--	--
Ag	< 1.	< 1.	6.	< 2.	< 2.
Sn	< 10.	< 10.	20.	20.	< 10.
Fe	500.	20.	100.	100.	100.
Sr	< 1.	< 1.	10.	10.	5.
Na	< 100.	< 100.	10,000.	8,000.	2,000.
K	< 500.	< 500.	1,000.	1,000.	1,000.
Ca	100.	100.	6,000.	4,000.	10,000.
Si	50.	50.	Major	Major	Major
Mg	20.	10.	1,000.	0	4,000.
Ba	< 10.	< 10.	100.	100.	100.
Al	10.	20.	0	0	4,000.
Total Weight, $\mu$ g	5,900. <sup>(e)</sup>	3,000. <sup>(e)</sup>	13,300.	12,800.	

(a) Not determined.

(b) Pb determined by Atomic Absorption.

(c) Not determined by this technique.

(d) Not determined - added buffer element.

(e) Acetone blank subtracted; blank = 0.027 mg/ml.

Note: Symbol &lt; indicates minimal detection limits.

EXAMPLE LEAD DETERMINATION  
FILTER, RUN 2Plant N.L. Industries, Beech Grove, IndianaDate November 18, 1972

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A - Initial Extract Volume per Split Filter Sample, ml	4.5
B - Aliquot, ml	1.0
C - Dilution Factor	10
D - Absorption, Percent	51
E - Absorbance from Atomic Absorbance Tables, Based on Percent Absorption	0.3098
F - Lead, $\mu\text{g}/\text{ml}$ of Diluted Aliquot <sup>(a)</sup>	22.0
G - Lead, $\mu\text{g}/\text{Split Filter Sample}$	990
H - Lead, $\mu\text{g}/\text{Total Filter}$	1980

Example Computation

$$C \times A \times F = G$$

$$10 \times 4.5 \times 22.0 = 990$$

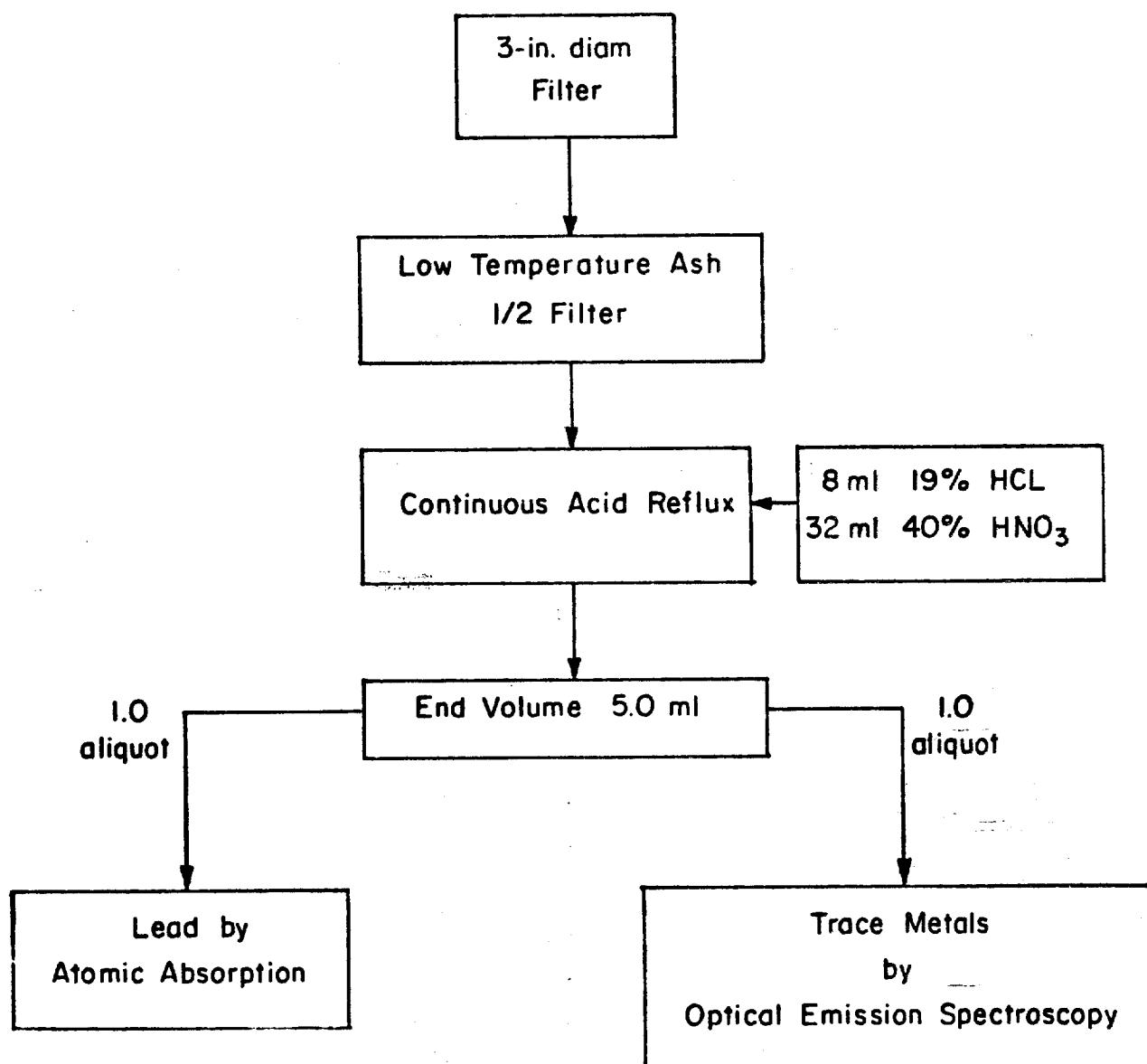
$$2 \times G = H$$

$$2 \times 750 = 1980$$

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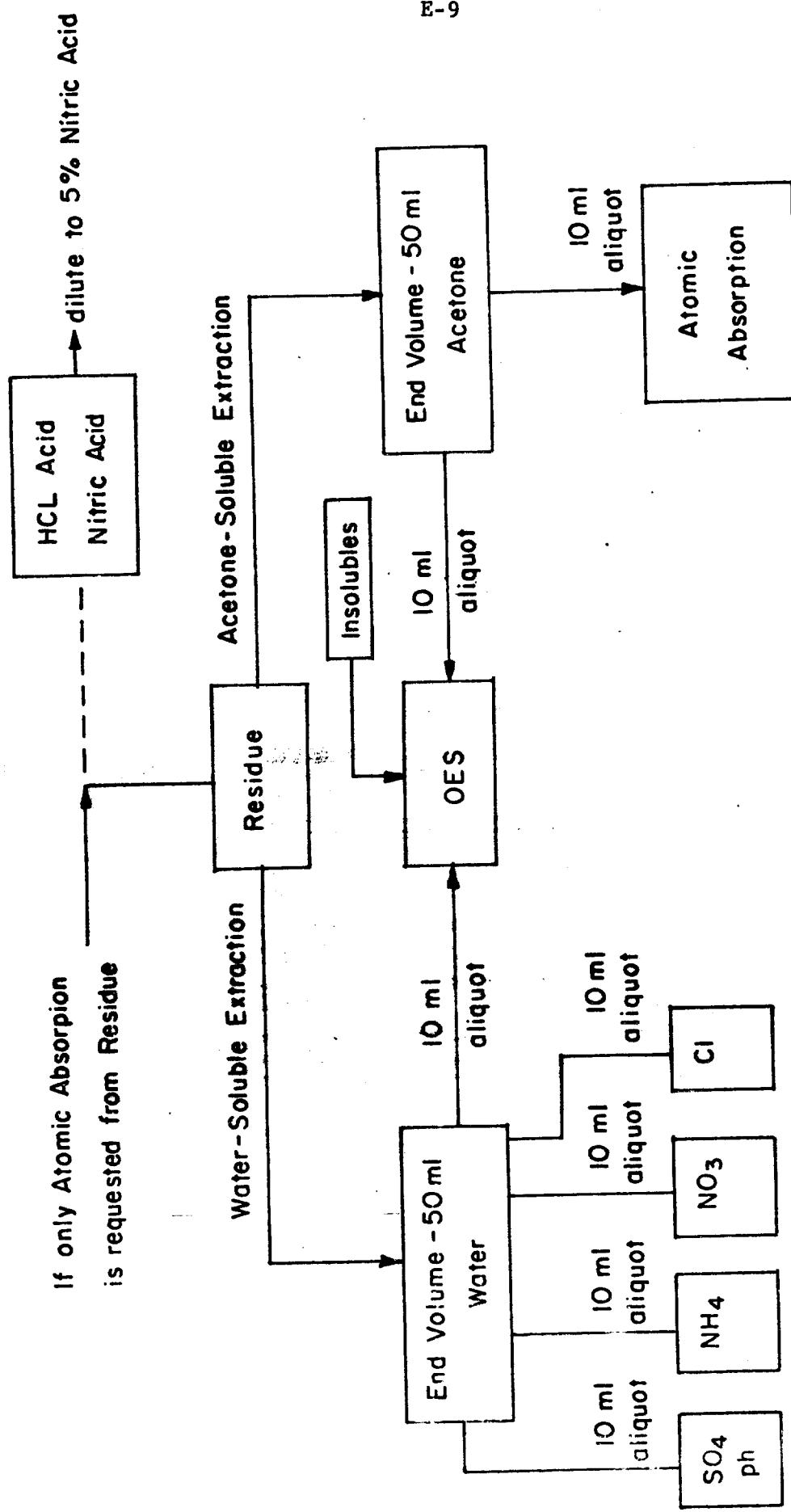
(a) Determined from calibration curve; based on 0.3098 absorbance.

## FILTER ANALYSIS FLOW DIAGRAM



## RESIDUE ANALYSIS FLOW DIAGRAM

If only Atomic Absorption is requested from Residue



APPENDIX F

TEST LOG

APPENDIX F

TEST LOG

November 16, 1971	Arrive on site. Move equipment to roof.
November 17, 1971	Construct weather shelter. Make preliminary run.
November 18, 1971	Make two runs. Project Officer approves use of preliminary run as one of required runs.
November 19, 1971	Remove equipment from site. Remove weather shelter. 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 REVERE SMELTING AND REFINING PLANT, NEWARK, NEW JERSEY

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  
Donald Hupp, Technician

#### Administrative Support

Richard E. Barrett, Senior Mechanical  
Engineer  
John M. Allen, Division Chief  
Irene Whitener, Secretary

APPENDIX I

COMPLETE OPERATIONAL RESULTS

## APPENDIX I

### OPERATIONAL SCHEDULE

#### BLAST FURNACE CHARGING FORMULA

The blast furnace is usually charged in the following sequence:

1. Coke	290 lb
2. Cast iron	250 lb
3. Return slag	600 lb
4. Scrap steel	150 lb
5. Battery plates	900 lb
6. Oxides	700 lb
7. Battery plates	900 lb
8. Slag (purchased)	700 lb
9. Battery plates	900 lb
10. Oxides	700 lb
11. Battery plates	900 lb
12. Oxides	700 lb
13. Bag dust	700 lb.

The weights were estimated by the operator of the loading machine and additions were made when the feed level in the feed hopper permitted. The coke was supplied in about 4-inch size pieces. The cast iron was foundry scrap about 1/2 to 1-1/2 inches in size. The return slag was tapped from the blast furnace, cooled and broken to about minus 4 inches. The scrap steel was detinned tin cans. The "battery plates" were mainly batteries with the acid and case removed, but included other high metallic lead scrap such as automotive tire weights. The oxides appeared to be batteries that had the cases removed by burning. The slag fed at Position 8 in the above feeding sequence was purchased as a powder. The bag dust fed at Position 13 was the material caught in the cyclones and bag-houses.

OBSERVATIONSPlant N.L. Industries, Beech Grove, IndianaDates November 17, and 18, 197111/17/71

1:15 pm Slag had been tapped recently  
Loading coke  
1:16 pm Loading cast iron  
1:17 pm Loading return slag - afterburner temperature 1100 F  
1:18 pm Loading scrap steel - blast furnace pressure, 40  
1:19 pm Loading battery plates  
1:20 pm Loading oxides  
1:35 pm Loading battery plate  
1:40 pm 4-inch  $H_2O$  vacuum at baghouse outlet  
1:45 pm Stack showing smoke estimated at No. 1 Ringelmann  
1:50 pm Slag tapped  
1:55 pm Loading battery plates  
2:00 pm Loading oxides  
2:05 pm No. 7 section of baghouse bypassed  
2:40 pm Baghouse temperature 260 F, pressure 4 inches  
2:45 pm Start run  
Afterburner temperature 1100 F  
Blast furnace air pressure 50 inches,  $H_2O$   
Load battery plates, 900 pounds  
2:55 pm Baghouse temperature, 280 F  
3:40 pm Run started again, false start at 2:45 pm  
4:00 pm Loading cast iron, 250 pounds  
4:05 pm Loading return slag, 600 pounds  
4:06 pm Loading scrap steel  
4:07 pm Furnace pressure, 40 inches  
Afterburner temperature, 1130 F  
4:11 pm Loading oxides, 700 pounds

OBSERVATIONS (Cont'd)

4:12 pm Loading battery plates, 900 pounds  
4:14 pm Loading slag  
4:25 pm Tapping slag  
4:30 pm Rod out air inlet ports  
Load battery plates, 900 pounds  
4:31 pm Load oxides, 700 pounds  
4:35 pm Screw conveyor removing dust from cyclones and baghouse  
is overloaded  
Baghouse temperature, 300 F  
4:40 pm Load battery plates, 900 pounds  
4:45 pm Load battery plates, 900 pounds  
4:46 pm Load battery plates, 900 pounds  
5:10 pm Load coke, 290 pounds  
9 tons lead tapped between 2:45 and 5:10 pm  
Load cast iron, 250 pounds  
5:15 pm Load return slag, 600 pounds  
Tap slag  
End of test.

11/18/71

8:20 am Baghouse temperature, 300 F  
Baghouse outlet pressure, 2 inches  
One section of bags is out for major repairs and will  
not operate today  
One section of bags is out for hand shaking. Each  
section will be hand shaken in turn  
Afterburner Temperature, 1450 F and dropping  
Afterburner temperature, 1650 at 7:30 am  
N.L. Industries reports Blast furnace operating normally  
except afterburner is off  
8:35 am Loading oxides, Position 6  
8:45 am Tap slag  
9:05 am Start test  
Add Delco slag, Position 8  
Furnace Pressure, 30 inches  
Afterburner temperature, 1210 F  
Burner off

OBSERVATIONS (Cont'd)

9:15 am     False start 9:05 - Start test  
              Load battery plates, Position 9  
              31 inch pot depth - 2 tons lead per inch

9:16 am     Load slag, Position 10

9:18 am     Tap slag

9:20 am     Load battery plates, 1800 pounds, Position 11

9:25 am     Afterburner temperature, 1100 F - not on  
              A constant volume of air is fed to the furnace  
              5 sections of baghouse operational  
              Baghouse temperature, 260 F  
              Damper bleeding some air into baghouse,  
              perhaps 2000 CFM  
              Sky cloudy - no visible emission

9:30 am     Feed oxides  
              Afterburner on  
              Scale to weigh feed is inaccurate

9:35 am     Load bag dust  
              Load coke  
              Furnace pressure, 35 inches

9:37 am     Load cast iron

9:45 am     Load scrap steel

9:53 am     Tap slag furnace pressure, 38 inches  
              Afterburner temperature, 1350 F

10:20 am    Adding Delco slag, Position 8  
              Afterburner temperature, 1500 F

10:25 am    Loading battery plates, Position 9

10:30 am    Smoke visible from stack

10:40 am    Screw conveyor from baghouse is blowing dust  
              Loading oxides, Position 12  
              Tap slag  
              Afterburner temperature, 1550 F

OBSERVATIONS (Cont'd)

11:00 am Add bag dust, Position 13  
Afterburner, 1550 F

11:01 am Load cast iron, Position 12

11:02 am Afterburner off

11:03 am Tap slag  
2-inch section of baghouse exhaust  
6-inch section on exhaust from cyclones

11:07 am Adding battery plates, Position 5

11:12 am Damper open to add cooling air to baghouse  
Baghouse temperature, 300 F

11:15 am Adding oxides, Position 6

11:17 am Loading battery plates, Position 7

11:23 am Baghouse temperature, 315 F  
Air damper open

11:35 am Load Delco slag, Position 8  
Afterburner off, temperature, 1660 F

11:53 am Baghouse, 300 F

11:50 am Tapped slag

11:55 am Load oxide, Position 10

11:58 am Afterburner, 1380 F. Had peaked at 1700 F at 11:40 am

12:00 pm Furnace pressure, 48 inches

12:02 pm Load battery plates, Position 11

12:07 pm Load oxide, Position 12

12:15 pm Baghouse temperature, 270 F

12:18 am Afterburner, 1200 F, gas off  
No steel scrap for Position 4  
Feed steel drum of slag at Position 4 and about 700 pounds oxides

12:30 pm Tap slag  
Add slag, Position 8  
27-inch lead in pot  
8 tons lead tapped, 9:15 am - 12:30 pm

1:43 pm Lead 23-1/2 inches in pot

1:50 pm Tap slag

1:55 pm Load oxides, Position 10

OBSERVATIONS (Cont'd)

1:56 pm Load battery plates, Position 11  
1:57 pm Load oxides, Position 12  
1:58 pm Load Baghouse Dust, Position 13  
1:59 pm Load coke, Position 1  
2:00 pm Afterburner temperature, 1200 F - on  
Load cast iron, Position 2  
2:03 pm Load return slag, Position 3  
Baghouse temperature, 300 F  
2:04 pm Add 700 pounds oxides, Position 4  
2:05 pm Add battery plates, Position 5  
2:12 pm Add Delco slag, Position 8  
Air damper on baghouse closed  
2:15 pm Baghouse temperature, 300 F  
Blast air pressure, 64 inches  
Tap slag  
2:20 pm Blast air pressure, 50 inches  
2:22 pm Load battery plates, Position 9  
2:24 pm Load oxides, Position 10  
Load 290 pounds coke, Position 10  
2:30 pm Repair skip hoist  
Baghouse repairs had been completed about 11:00 am.  
7 sections operational from 11:00 am on  
2:45 pm Baghouse temperature, 290 F  
2:47 pm Add baghouse dust, Position 13  
Afterburner on, temperature steady at 1220 F  
2:50 pm Air damper on baghouse almost closed  
2:55 pm Tap slag  
Blast pressure, 65 inches before tapping; 55 inches  
after tapping  
Plug in tap hole leaking  
3:05 pm Load coke, Position 1  
Load scrap iron, Position 2

OBSERVATIONS (cont'd)

3:06 pm Load slag, Position 3  
3:08 pm Load 700 pounds oxides, Position 4  
Baghouse 300 F  
3:25 pm Tap slag  
Add oxides, Position 6  
3:35 pm Load battery plates, Position 7  
Blast pressure, 55 inches  
Afterburner temperature, 1225 F  
3:40 pm Add Delco slag, Position 8  
Add cast iron, Position 8  
Started traverse in other direction  
3:47 pm Tap slag  
3:51 pm Add double load battery plates, Position 9  
3:54 pm Add oxides  
Blast pressure, 55 inches  
Afterburner temperature, 1240 F  
4:12 pm Tap slag  
4:13 pm Blast pressure, 51 inches  
4:15 pm Load oxides  
4:18 pm Load cap, Position 1 (did not load baghouse dust)  
4:20 pm Load coke, Position 2  
4:22 pm Load return slag, Position 3  
4:23 pm Load baghouse dust  
4:24 pm Load scrap steel, Position 4  
4:27 pm Tap furnace  
Blast pressure, 55 inches  
4:30 pm Stop tapping lead  
4:35 pm Load oxides, Position 6  
Blast pressure, 50 inches  
Afterburner temperature, 1220 F  
4:37 pm Tapping lead again  
4:39 pm Load battery plates, Position 7  
4:42 pm Baghouse temperature 300 F  
Damper closed

OBSERVATIONS (Cont'd)

4:43 pm Add Delco slag, Position 8  
Lead level lost because of transfer of lead into  
receiver pot from another pot  
Lead level was 19 inches at shift change at 3:30 pm.  
9 tons lead tapped between 1:43 - 3:30 pm

4:58 pm End run. Still on Position 8  
Furnace is rated at 77 ton/day  
Baghouse has 320 bags  
25 ft - 7 in x 11-1/2 diameter  
24,000 sq ft in 8 sections

**APPENDIX J**

**SUMMARY OF RESULTS**

## APPENDIX J

SUMMARY OF RESULTSPlant N.L. Industries, Beech Grove, Indiana

Run Number	1	2	3	Average
Date	11/17/71	11/18/71	11/18/71	
Test Time - Minutes	91	188	186	155
Lead Production - TPH	3.7	2.5	3.0	3.1
<b>Stack Effluent</b>				
Flow rate - DSCFM	23,200	22,900	23,120	23,070
Flow rate - DSCF/ton	6,270	9,160	7,710	7,710
Temperature - °F	176	182	177	178
Water vapor - Vol. %	3.7	2.3	3.1	3.0
CO <sub>2</sub> - Vol. % dry	2.2	2.1	1.5	1.9
O <sub>2</sub> - Vol. % dry	19.0	19.0	19.5	19.2
CO - Vol. % dry	0.5	0.2	0.4	0.36
CO - 1b/hr	503	199	401	368
SO <sub>2</sub> - ppm dry	443	264	204	304
SO <sub>2</sub> - 1b/hr	101	59	46	69
Visible emissions - % opacity	10-15	10-70	5-15	12
<b>Particulate Emissions</b>				
<u>Probe and filter catch</u>				
gr/DSCF	0.0028	0.0027	0.0023	0.0026
gr/ACF	0.0022	0.0021	0.0018	0.0020
1b/hr	0.5541	0.5231	0.4570	0.5114
1b/ton lead	0.1498	0.2092	0.1523	0.1704
<u>Total Catch</u>				
gr/DSCF	(a)	0.0471	0.0396	0.0434
gr/ACF	(a)	0.0368	0.0310	0.0339
1b/hr	(a)	9.2439	7.8566	8.5503
1b/ton lead	(a)	3.6976	2.6189	3.1583
<b>Lead Emissions</b>				
<u>Probe and filter catch</u>				
gr/DSCF	(a)	0.00035	0.00033	0.00034
gr/ACF	(a)	0.00027	0.00026	0.00027
1b/hr	(a)	0.0679	0.0648	0.0664
1b/ton lead	(a)	0.0272	0.0216	0.0244

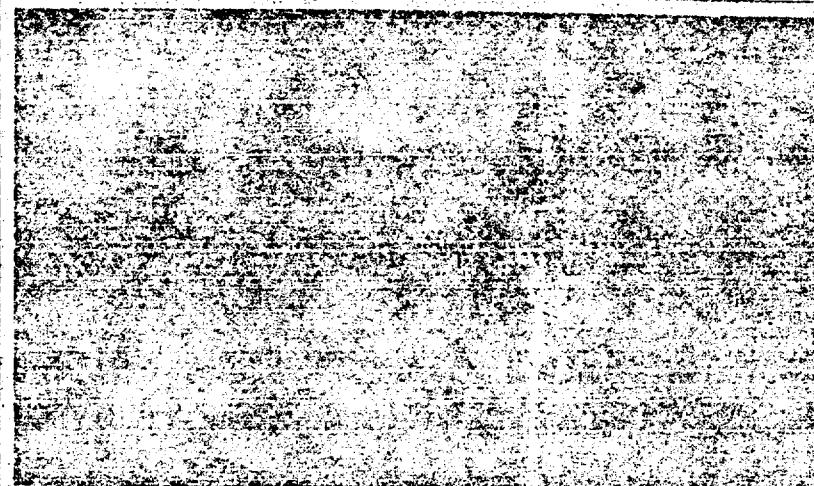
Total Catch

Essentially the same as probe and filter.

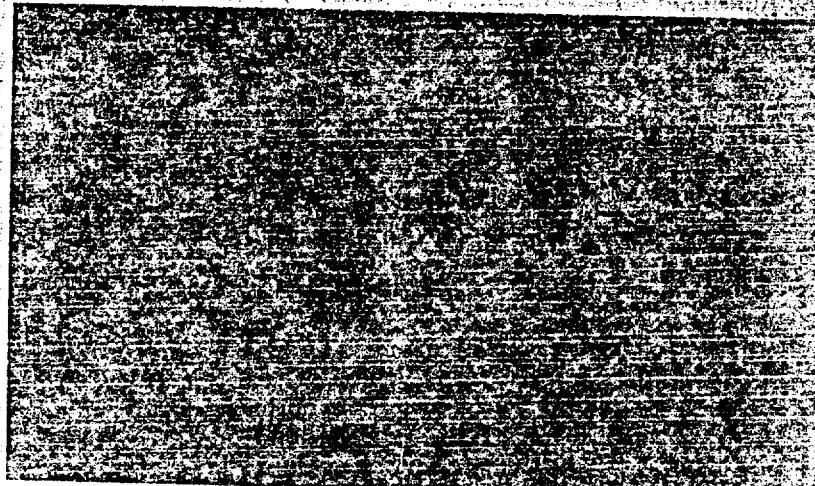
SECONDARY LEAD  
SMELTING  
AP-42 Section 7.11  
Reference Number  
12 (O (b))

A  
B

# RESEARCH REPORT



Environmental  
Science  
Columbus Laboratories



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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

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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

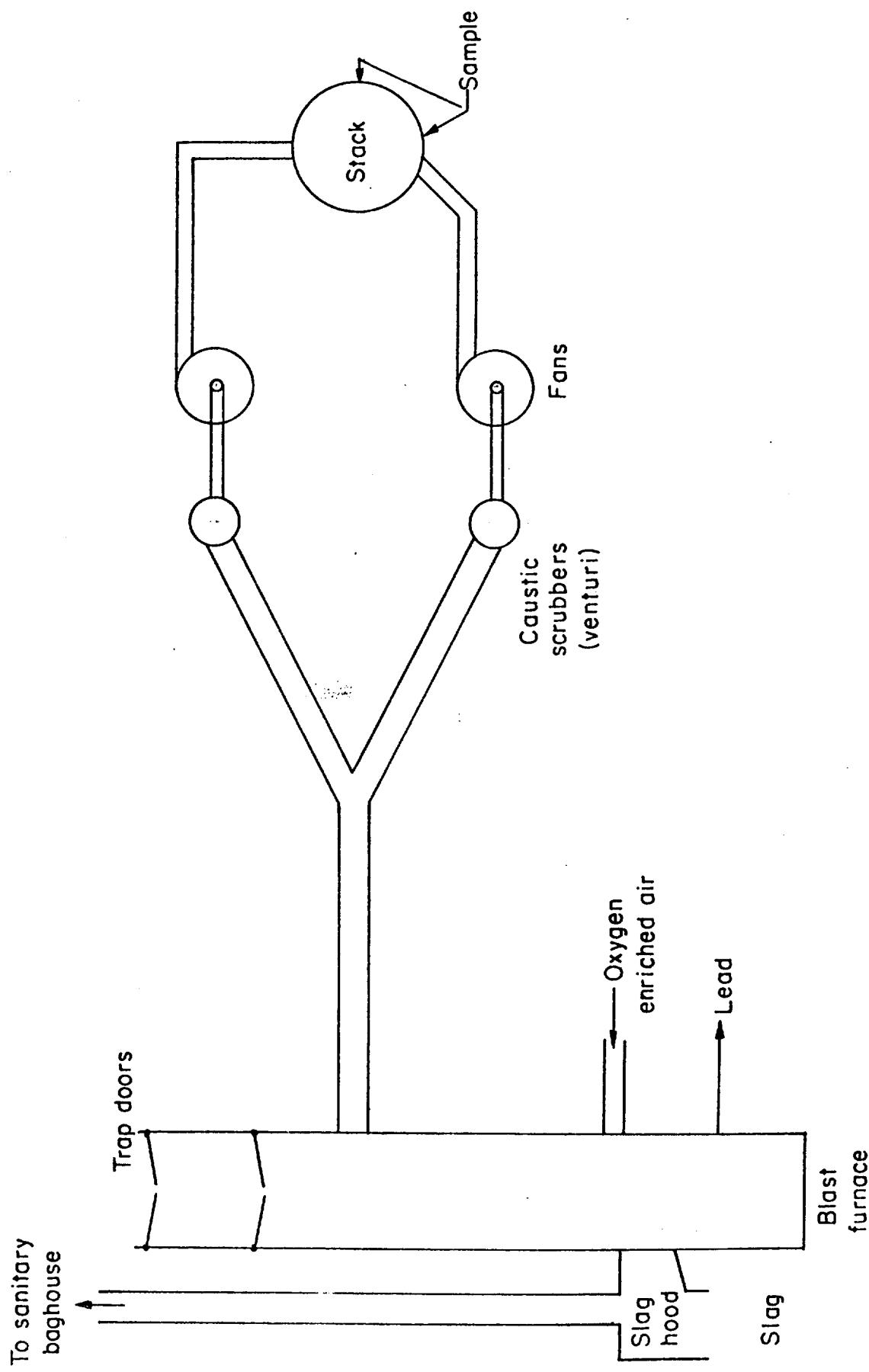


FIGURE 1. GAS FLOW DIAGRAM, REVERE SMELTING AND REF., NEWARK, NEW JERSEY

traverse over a 2-hour period. The  $\text{SO}_2$  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,  $\text{SO}_2$ ,  $\text{CO}_2$ ,  $\text{O}_2$ , and  $\text{CO}$ , 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 ether 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  $\text{SO}_2$  in the stack reached a detectable concentration of 0.1 ppm in only one of the three runs. The  $\text{CO}$  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.

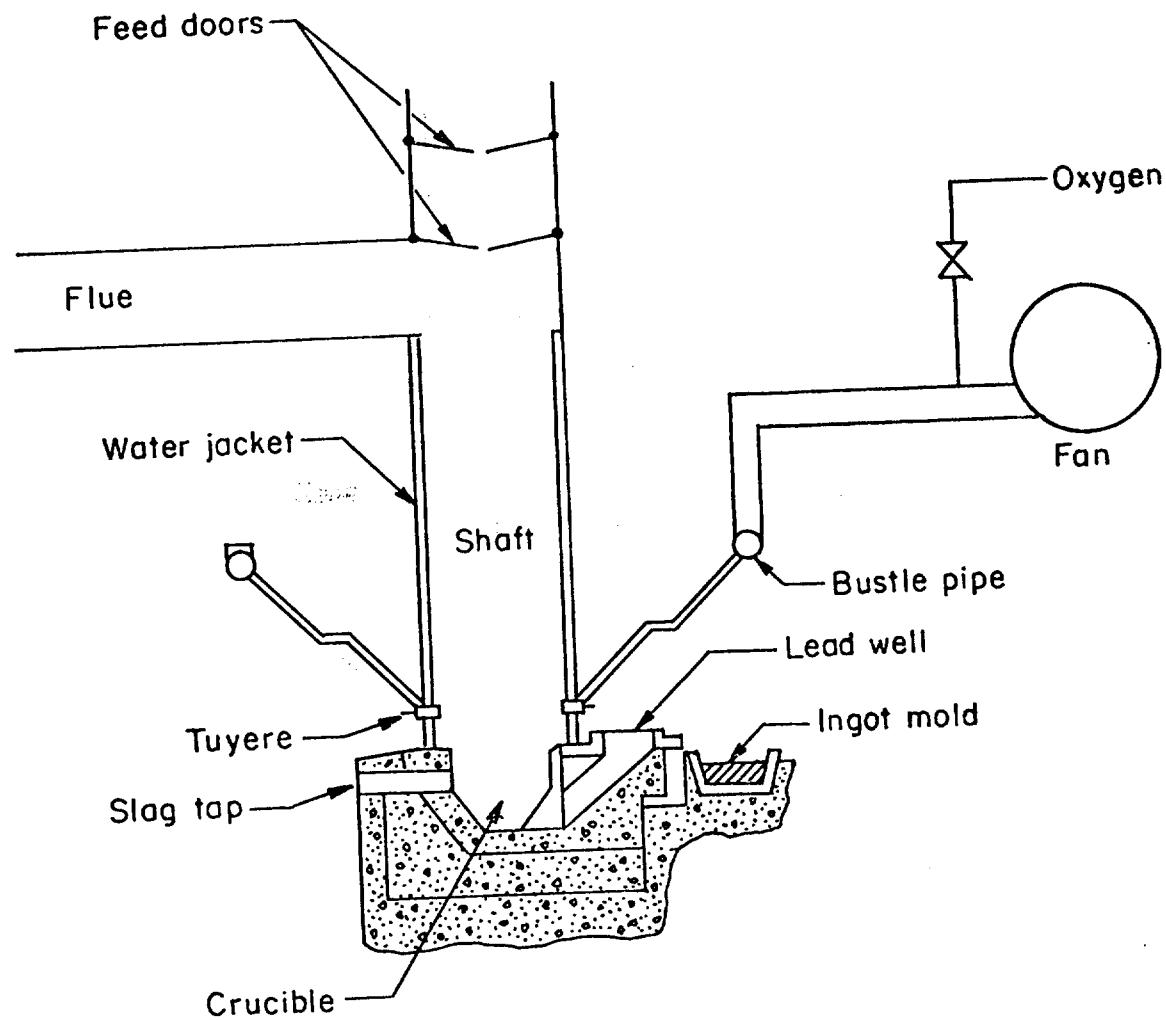


FIGURE 2. LEAD BLAST FURNACE

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<sub>2</sub> and Orsat samples were obtained from a single point at the center of the stack.

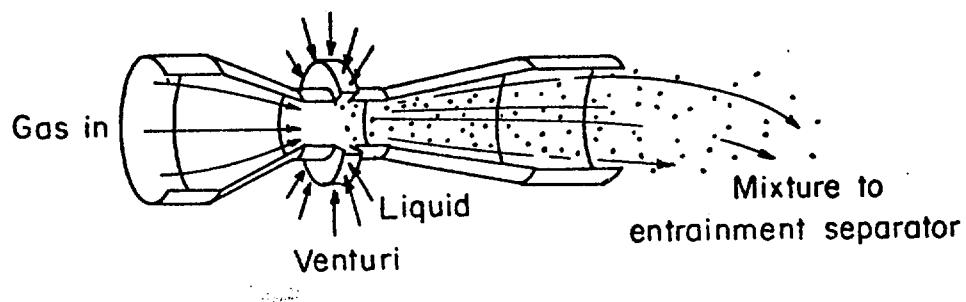
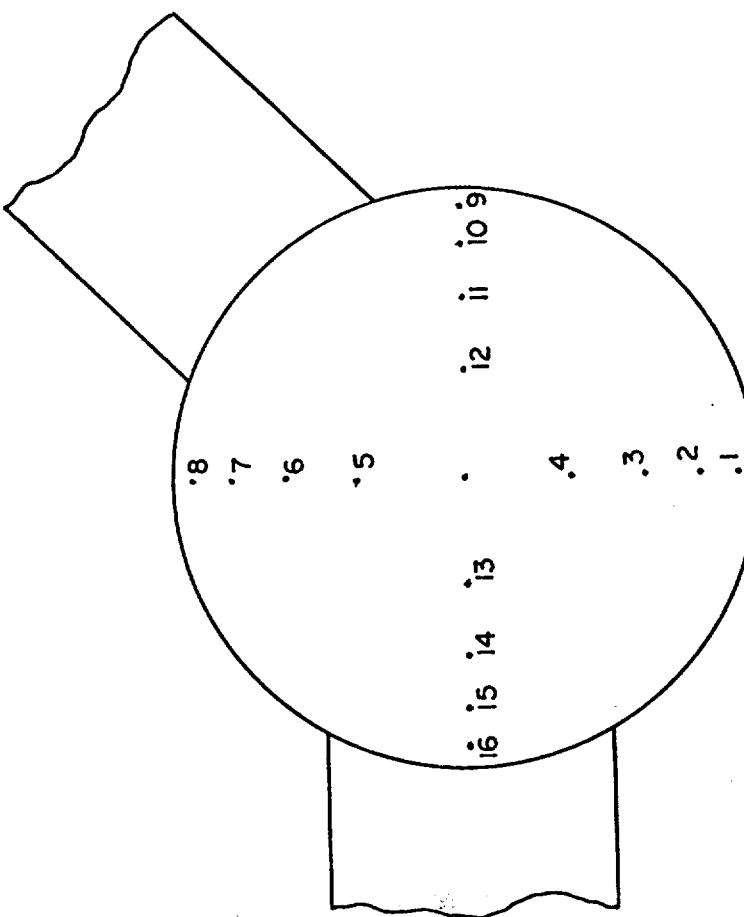


FIGURE 3. VENTURI SCRUBBER



Distance of Sampling Point From Wall

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

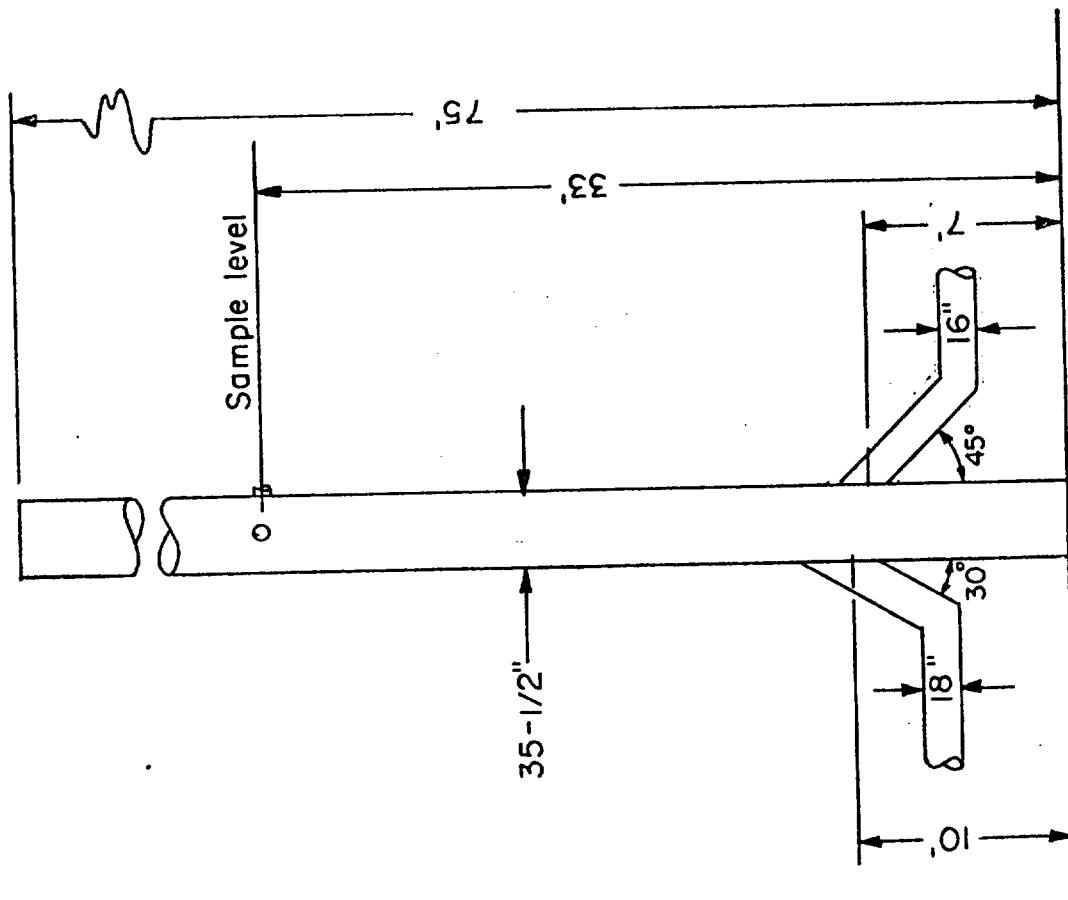


FIGURE 4. LOCATION OF SAMPLING POINTS

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 intermittently 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<sub>2</sub>. 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 dry 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<sub>2</sub>, CO<sub>2</sub>, O<sub>2</sub>, 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.

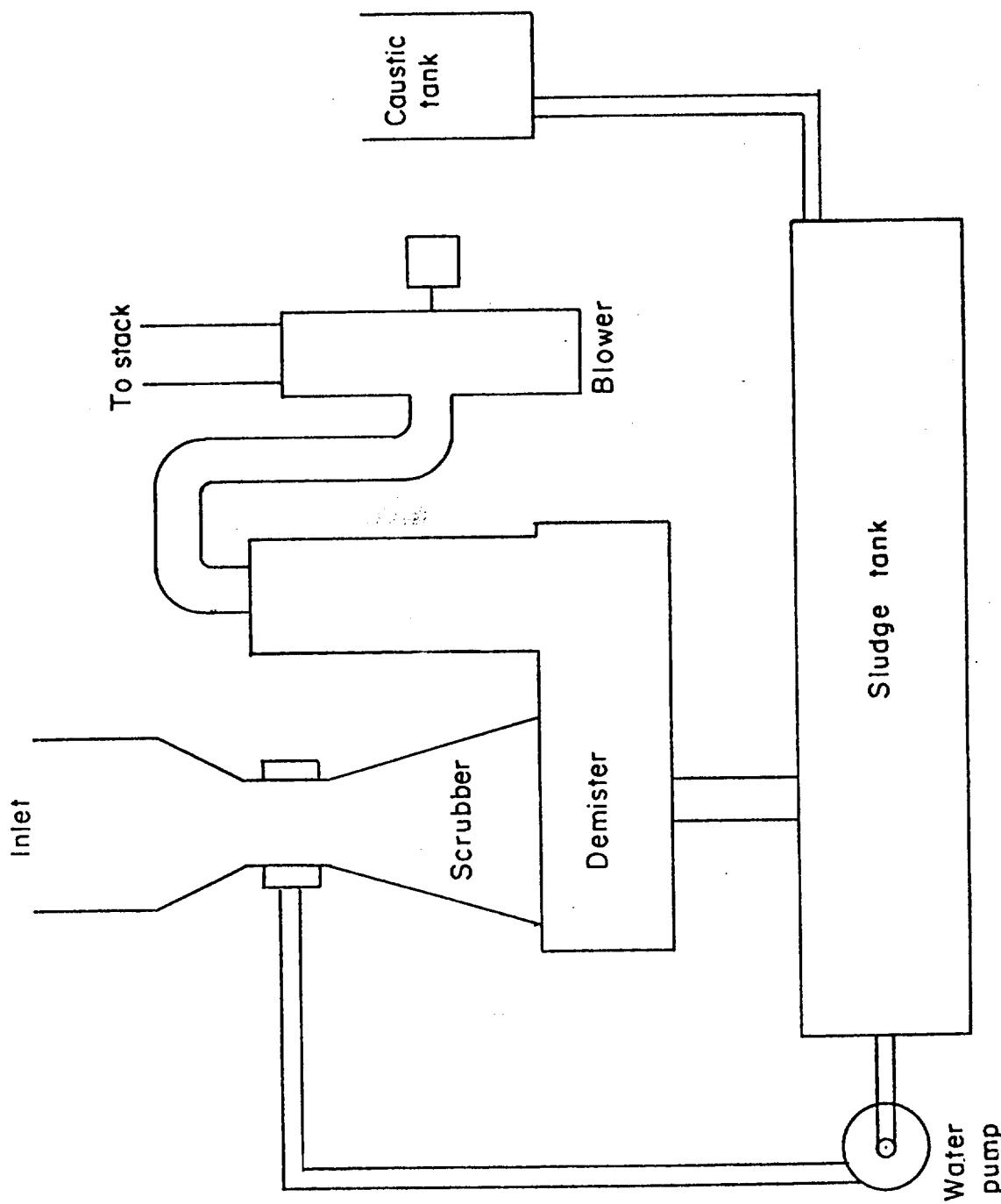


FIGURE 5. VENTURI SCRUBBING SYSTEM

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  $\text{SO}_2$  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 pipetted 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  $\text{CO}_2$  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 CALCULATIONSSOURCE TESTING CLACULATION FORMSTest No. 2, 3, 4No. Runs 3Name of Firm Revere Smelting and Refining CompanyLocation of Plant Newark, New JerseyType of Plant Secondary Lead Blast FurnaceControl Equipment Wet ScrubbersSampling Point Locations StackPollutants Sampled Particulates - SO<sub>2</sub>

Time of Particulate Test:

Run No. <u>2</u>	Date <u>12-8-71</u>	Begin <u>3:10 pm</u>	End <u>5:39 pm</u>
Run No. <u>3</u>	Date <u>12-9-71</u>	Begin <u>9:50 am</u>	End <u>11:55 am</u>
Run No. <u>4</u>	Date <u>12-9-71</u>	Begin <u>12:58 pm</u>	End <u>3:08 pm</u>

PARTICULATE EMISSION DATA

Run No.	2	3	4
P <sub>b</sub> - barometric pressure, in. Hg Absolute	30.18	30.23	30.13
P <sub>m</sub> - orifice pressure drop, in. H <sub>2</sub> O	0.71	0.75	0.80
V <sub>m</sub> - volume of dry gas sampled @ meter conditions, ft <sup>3</sup>	70.2	57.3	59.3
T <sub>m</sub> - average gas meter temperature, F	66	61	64
V <sub>m</sub> <sup>std.</sup> - volume of dry gas sampled @ standard conditions, ft <sup>3</sup> , dry	71.4	59.0	60.5
V <sub>w</sub> - total H <sub>2</sub> O collected, impingers and silical gel., ml.	37.3	21.4	30.0
V <sub>w</sub> - volume of water vapor collected gas ft <sup>3</sup> @ standard conditions (a)	1.8	1.0	1.4
V <sub>total</sub> - total gas volume, standard conditions, ft <sup>3</sup>	73.2	60.0	61.9
Moisture in the stack gas volume, percent	2.4	1.7	2.3
H <sub>2</sub> - 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 <sub>2</sub> , dry, percent	0.5	0.8	0.8
O <sub>2</sub> , dry, percent	20.3	20.2	18.8
CO, dry, percent	0.8	0.3	0.2
N <sub>2</sub> , dry, percent	78.4	78.7	80.2
M W <sub>d</sub> - 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 <sub>s</sub> - stack temperature, F	97	95	93
ΔP <sub>s</sub> x (T <sub>s</sub> + 460)	12.938	13.049	13.300
P <sub>s</sub> - stack pressure, in. Hg absolute	30.18	30.23	30.13
V <sub>s</sub> - stack velocity @ stack conditions, fpm	1914	1925	1970
A <sub>s</sub> - stack area, in <sup>2</sup>	972	972	972
Q <sub>s</sub> - stack gas volume @ standard conditions, (a) ft <sup>3</sup> dry	12100	13330	12450
T <sub>t</sub> - net time of test, min.	120	112	112
D <sub>n</sub> - sampling nozzle diameter, in.	0.235	0.235	0.235
Percent I - percent isokinetic	109	95	96
m <sub>f</sub> - particulate-probe and filter, mg	91.0	32.3	58.7
m <sub>t</sub> - particulate-total, mg	127.3	60.0	92.1
C <sub>an</sub> - particulate, probe, and filter, gr/scf, dry	0.0196	0.0084	0.0149
C <sub>ao</sub> - 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 <sup>3</sup> @ stack conditions	0.0184	0.0080	0.0141
$C_{au}$ - particulate; total, gr/ft <sup>3</sup> @ stack conditions	0.0257	0.0149	0.0221
$C_{aw}$ - particulate; probe and filter, 1b/hr	2.0354	0.8913	1.6061
$C_{ax}$ - particulate; total, 1b/hr	2.8474	1.6556	2.5200
$C_p$ - particulate; probe and filter, 1b/ton lead	1.3570	0.7750	1.2849
$C_{pt}$ - particulate; total 1b/ton lead	1.8983	1.4397	2.0160
$C_{ls}$ - lead emission; probe and filter, gr/scf	0.00127	0.00061	0.00127
$C_{la}$ - lead emission; probe and filter, gr/ft <sup>3</sup>	0.00119	0.00058	0.00120
$C_{lh}$ - lead emission; probe and filter, 1b/hr	0.1320	0.0646	0.1368
$C_{lt}$ - lead emission; probe and filter, 1b/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<sup>3</sup>

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

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

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

2. Volume of water vapor at 70 F and 29.92 in. Hg, ft<sup>3</sup>

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

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

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

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

$$\begin{aligned}
 M\ W_d &= (CO_2 \times \frac{44}{100}) + (O_2 \times \frac{32}{100}) + (N_2 + CO) \times \frac{28}{100} \\
 &= (0.5 \times 0.44) + (20.3 \times 0.32) + 79.2 \times 0.28 \\
 &= 28.9
 \end{aligned}$$

## 6. Molecular weight of stack gas

$$\begin{aligned}
 M\ W &= M\ W_d \times M_d + 18 (1 - M_d) \\
 &= 28.9 \times 0.98 + 18 (1 - 0.98) \\
 &= 28.6
 \end{aligned}$$

## 7. Stack velocity @ stack conditions, fpm

$$\begin{aligned}
 V_s &= 4350 \times \sqrt{\Delta P_s \times (T_s + 460)} \quad \left( \frac{1}{P_s \times M\ W} \right)^{1/2} \text{ fpm} \\
 &= 4350 \times 12.938 \quad \left[ \frac{1}{30.18 \times 28.6} \right]^{1/2} \\
 &= 1914 \text{ fpm}
 \end{aligned}$$

## 8. Stack gas volume at standard conditions, scfm

$$\begin{aligned}
 Q_s &= \frac{0.123 \times V_s \times A_s \times P_s}{(T_s + 460)} M_d \text{ scfm} \\
 &= \frac{0.123 \times 1914 \times 972 \times 30.18 \times 0.98}{97 + 460} \\
 &= 12,100 \text{ scfm}
 \end{aligned}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

9. Sampling velocity, percent of isokinetic

$$\begin{aligned} \%I &= \frac{1032 \times (T_s + 460) \times V_{mstd}}{V_s \times T_t \times P_s \times M_d \times (D_n)^2} \\ &= \frac{1032 \times (97 + 460) \times 71.4}{1914 \times 120 \times 30.18 \times 0.98 \times (0.235)^2} \\ &= 109 \text{ percent} \end{aligned}$$

10. Percent excess air at sampling point

$$\begin{aligned} \%EA &= \frac{100 \times \% O_2}{(0.266 \times \% N_2) - \% O_2} \\ &= \frac{100 \times 20.3}{(0.266 \times 78.4) - 20.3} \\ &= 3690 \text{ percent} \end{aligned}$$

11. Particulate - probe and filter, gr/scf

$$\begin{aligned} C_{an} &= 0.0154 \times \frac{m_f}{V_{mstd}} \\ &= 0.0154 \times \frac{91.0}{71.4} \\ &= 0.0196 \text{ gr/scf} \end{aligned}$$

12. Particulate - total, gr/scf

$$\begin{aligned} C_{ao} &= 1.54 \times 10^{-2} \left( \frac{m_t}{V_{mstd}} \right) \\ &= 1.54 \times 10^{-2} \frac{127.3}{71.4} \\ &= 0.0274 \text{ gr/scf} \end{aligned}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

13. Particulate - probe and filter, gr/ft<sup>3</sup> 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<sup>3</sup> 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, 1b/ton lead

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

18. Particulate; total 1b/ton lead

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

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

$$\begin{aligned} C_{1s} &= 0.0154 \times \frac{M_1}{V_m \text{ std}} \\ &= 0.0154 \times \frac{5.9}{71.4} \\ &= 0.00127 \text{ gr/scf} \end{aligned}$$

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

$$\begin{aligned} C_{1a} &= \frac{17.7 \times C_{1s} \times P_s \times M_d}{(T_s + 960)} \\ &= \frac{17.7 \times 0.00127 \times 30.18 \times 0.98}{(97 + 460)} \\ &= 0.00119 \text{ gr/scf} \end{aligned}$$

21. Lead emission; probe and filter, 1b/hr

$$\begin{aligned} C_{1h} &= 8.57 \times 10^{-3} \times C_{1s} \times Q_s \\ &= 8.57 \times 10^{-3} \times 0.00127 \times 12,100 \\ &= 0.1320 \text{ lb/hr} \end{aligned}$$

22. Lead emission; probe and filter, 1b/ton lead

$$\begin{aligned} C_{1t} &= C_{1h}/R \\ &= 0.1320/1.5 \\ &= 0.0880 \text{ lb/ton lead} \end{aligned}$$

APPENDIX B

COMPLETE GASEOUS RESULTS WITH EXAMPLE CALCULATION

## APPENDIX B

COMPLETE GASEOUS RESULTS WITH EXAMPLE CALCULATIONSSO<sub>2</sub> EMISSION DATA

Run Number	1	2	3	4
Date (1971)	12-8	12-8	12-9	12-9
T <sub>m</sub> - average gas meter temperature, F	56	62	61	63
P <sub>b</sub> - barometer pressure, inches Hg absolute	30.2	30.2	30.2	30.1
V <sub>m</sub> - volume of dry gas sampled at meter conditions, ft <sup>3</sup>	4.9	6.7	9.9	10.6
V <sub>m</sub> standard, ft <sup>3</sup>	5.1	6.9	10.3	10.8
V <sub>t</sub> - volume Ba (ClO <sub>4</sub> ) <sub>2</sub> , ml	0.01	<0.01	<0.01	0.02
V <sub>tb</sub> - volume Ba (ClO <sub>4</sub> ) <sub>2</sub> blank ml	0	0	0	0
N - normality of Ba (ClO <sub>4</sub> ) <sub>2</sub>	0.01	0.01	0.01	0.01
V <sub>soln</sub> - volume of SO <sub>2</sub> soln, ml	50	100	100	100
V <sub>a</sub> - volume aliquot, ml	10	10	10	10
C <sub>SO<sub>2</sub></sub> - 1b/feet <sup>3</sup> x 10 <sup>-5</sup>	0.001	0	0	0.001
C <sub>SO<sub>2</sub></sub> - ppm (dry)	0.1	0	0	0.1
W <sub>SO<sub>2</sub></sub> - 1b/hour	0	0	0	0

SO<sub>2</sub> EXAMPLE CALCULATIONS

Run 4

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

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

$$= 10.8 \text{ ft}^3$$

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

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

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

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

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

$$= 0.1 \text{ ppm}$$

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

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

$$= 0 \text{ lb/hr}$$

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<sup>2</sup>, 6.87 feet<sup>2</sup>

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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 SHEETPlant Revere Smelting and Refining CompanyOperators 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 <sup>(a)</sup>	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 <sup>(a)</sup>			
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 <sup>(a)</sup>	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

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  $\Delta H @$  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  $\Delta P$  \_\_\_\_\_ Average  $\Delta H$  \_\_\_\_\_

C-3

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

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## PARTICULATE FIELD DATA

Lane: Enviro Smelting and Refining Company

Date 12-9-71

Run No. 3

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

Sample Box No. 1

Detector Box No. 1

Meter  $\Delta H @$  1.84

Factor 10

Ambient Temperature, F 52

Barometric Pressure, in. Hg 30.23

Assumed Moisture, Percent 10

Heater Box Setting, F 255

Probe Tip Diameter, Inches 0.235

Probe Length 5 feet

Probe Heater Setting

Average  $\Delta P$  Average  $\Delta H$ 

C-4

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

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



GAS SAMPLING FIELD DATADate 12-8-71Material Sampled for SO<sub>2</sub>Barometric Pressure 30.2 in. HgAmbient Temperature 51 FRun Number 1Power Stat Setting HeatedFilter Used: Yes X No   Operator W.C. Baytos, H. HessPlant 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
10.0	0.01
blank	0

Clock Time	Meter (ft <sup>3</sup> )	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 Plant Revere Smelting and Refining Co.  
 Material Sampled for  $\text{SO}_2$  Comments:  
 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

Titration		
Aliquot, ml	Titer, ml	
10.0	0.01	0.01 N $\text{Ba}(\text{ClO}_4)_2$
10.0	<0.01	
blank	0	

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

Clock Time	Meter (ft <sup>3</sup> )	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 Plant Revere Smelting and Refining Co.  
 Material Sampled for SO<sub>2</sub> Comments: Probe temperature, 180 F.  
 Barometric Pressure 30.1 in. Hg Recovered ml - diluted to 100 ml.  
 Ambient Temperature 53F Titration  
 Run Number 4 Aliquot, Titer,  
 Power Stat Setting Heated ml ml  
 Filter Used: Yes X No    10.0 0.02 0.01 N Ba(C<sub>10</sub>O<sub>4</sub>)<sub>2</sub>  
 Operator W.C. Baytos, H. Hess 0 4.58  
 blank 0

Clock Time	Meter (ft <sup>3</sup> )	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<sub>2</sub>  
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

<u>Titration</u>		
Aliquot, ml	Titer, ml	
10.0	<0.01	0.01 N Ba(ClO <sub>4</sub> ) <sub>2</sub>
blank	0	

Clock Time	Meter (ft <sup>3</sup> )	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 COMPOSITIONPlant Revere Smelting and Refining Company

Run	Date	Time	CO <sub>2</sub> , Percent	O <sub>2</sub> , Percent	CO, Percent	N <sub>2</sub> , 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  $\Delta p$  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  $\Delta p$  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 TRAVERSSES  
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

## PROPOSED RULE MAKING

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:

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

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

2.1.3. Severe 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.

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

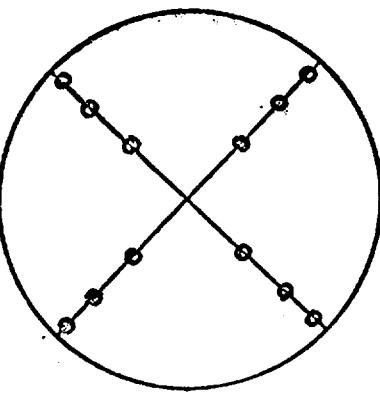
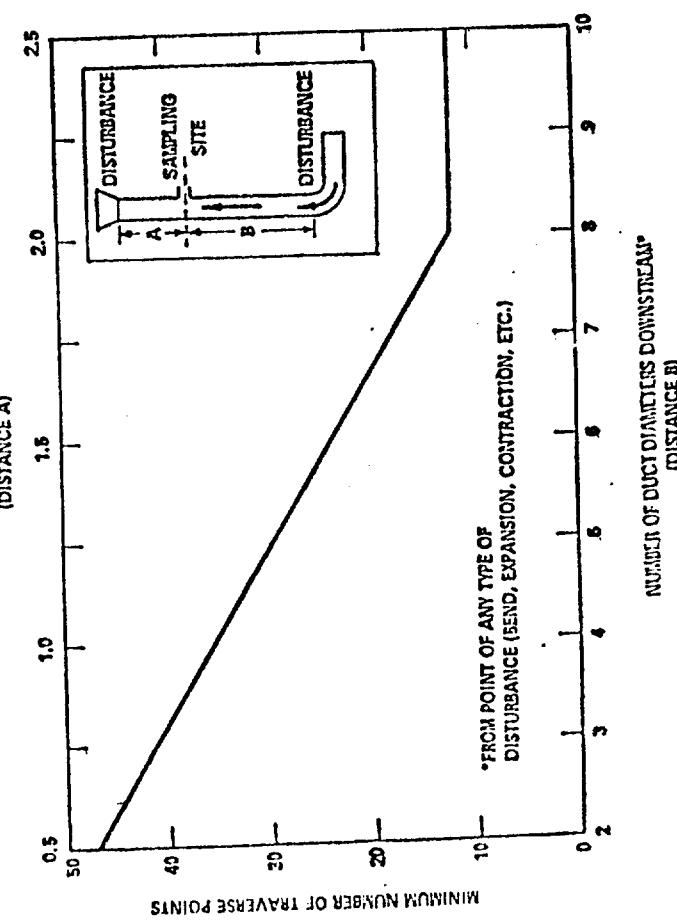


Figure 1-1. Minimum number of traverse points\* from point of any type of disturbance (bend, expansion, contraction, etc.)

NUMBER OF DUCT DIAMETERS UPSTREAM



\*FROM POINT OF ANY TYPE OF DISTURBANCE (BEND, EXPANSION, CONTRACTION, ETC.)

NUMBER OF DUCT DIAMETERS DOWNSTREAM

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

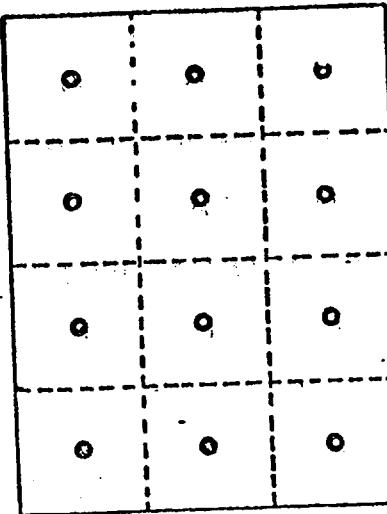


Figure 1-3. Cross section of rectangular stack divided into 12 equal areas, with traverse points at centroid of each area.

## PROPOSED RULE MAKING

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

Traverse point number on 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	14.5	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				93.6	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							93.9					

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 areas 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, 1983.

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 call.

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

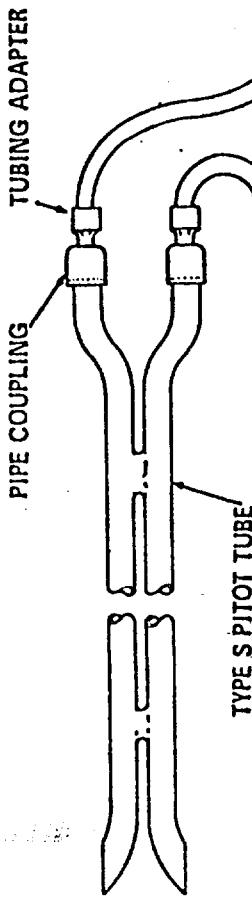


Figure 2-1. Pitot tube - manometer assembly.

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.



## PROPOSED RULE MAKING

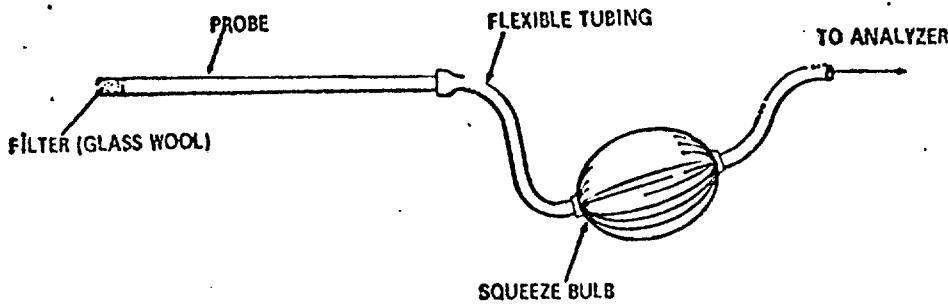


Figure 3-1. Grab-sampling train.

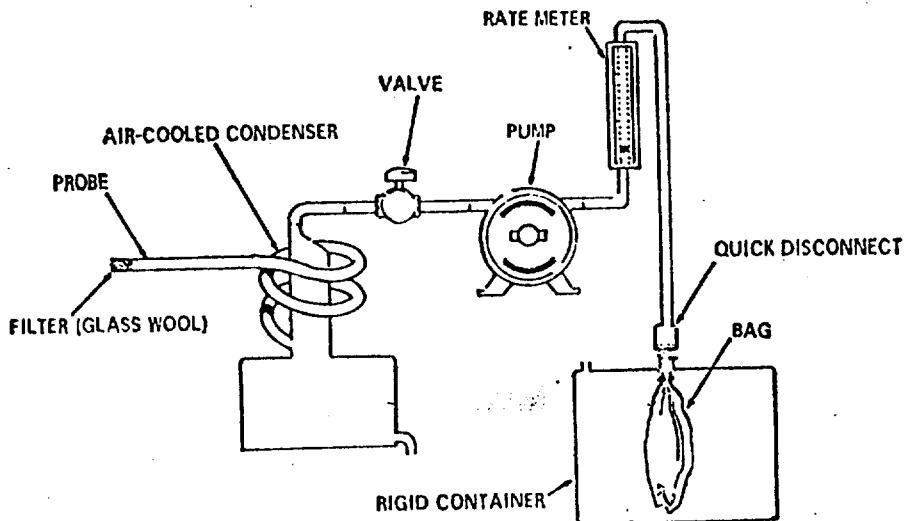


Figure 3-2. Integrated gas-sampling train.

## 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  $\text{CO}_2$ ,  $\text{O}_2$ , and  $\text{CO}$  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  $\text{CO}_2$ .

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.

$\% EA =$

$$\frac{(\% \text{O}_2) - 0.5(\% \text{CO})}{0.261(\% \text{N}_2) - (\% \text{O}_2) + 0.5(\% \text{CO})} \times 100$$

equation 3-1

where:

$$\begin{aligned} \% EA &= \text{Percent excess air.} \\ \% O_2 &= \text{Percent oxygen by volume, dry basis.} \\ \% N_2 &= \text{Percent nitrogen by volume, dry basis.} \\ \% CO &= \text{Percent carbon monoxide by volume, dry basis.} \\ 0.261 &= \text{Ratio of oxygen to nitrogen in air by volume.} \end{aligned}$$

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.

$$M_d = 0.44(\% \text{CO}_2) + 0.32(\% \text{O}_2) + 0.28(\% \text{N}_2 + \% \text{CO})$$

Equation 3-2

where:

$$\begin{aligned} M_d &= \text{Dry molecular weight, lb./lb.-mole.} \\ \% CO_2 &= \text{Percent carbon dioxide by volume, dry basis.} \\ \% O_2 &= \text{Percent oxygen by volume, dry basis.} \\ \% N_2 &= \text{Percent nitrogen by volume, dry basis.} \\ 0.44 &= \text{Molecular weight of carbon dioxide divided by 100.} \\ 0.32 &= \text{Molecular weight of oxygen divided by 100.} \\ 0.28 &= \text{Molecular weight of nitrogen divided by 100.} \end{aligned}$$

## 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.<sup>1</sup>

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<sup>1</sup> 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 Pilot 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 4-1.

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

<sup>1</sup> Trade name.

<sup>2</sup> 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.

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### 4. Calculations.

#### 4.1 Volume of water collected.

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

equation 4-1

where:

$V_{ws}$  = 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.

#### 4.2 Gas volume.

$$V_{m,s} = V_m \left( \frac{P_m}{P_{std}} \right) \left( \frac{T_{std}}{T_m} \right) = (17.71 \frac{\text{ft}^3}{\text{in. Hg}}) \frac{V_m P_m}{T_m}$$

equation 4-2

where:

$V_{m,s}$  = 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.

$$B_{ws} = \frac{V_{ws}}{V_{ws} + V_{m,s}} + B_{wm} = \frac{V_{ws}}{V_{ws} + V_{m,s}} + (0.025)$$

equation 4-3

where:

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

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

$V_{m,s}$  = 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<sup>1</sup> 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.

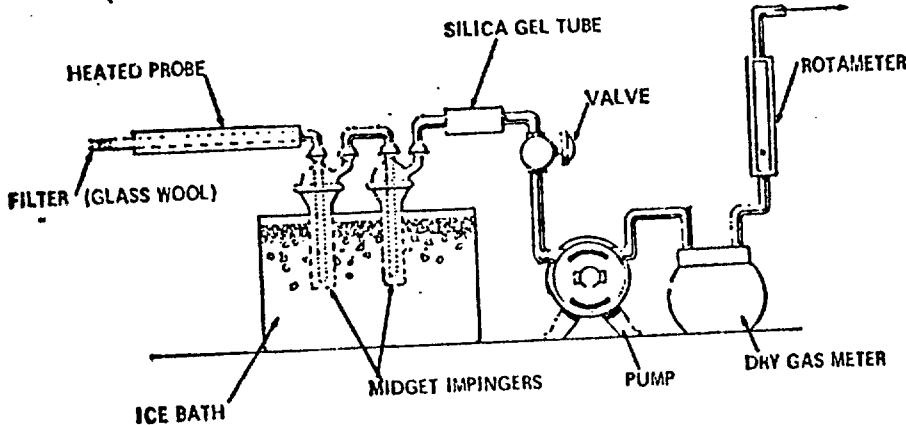


Figure 4-1. Moisture-sampling train.

LOCATION	COMMENTS
TEST	
DATE	
OPERATOR	
BAROMETRIC PRESSURE	

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

Figure 4-2. Field moisture determination.

## PROPOSED RULE MAKING

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

2.1.4 Filter holder—Pyrex<sup>1</sup> 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 re-

placing the tip with a 1/2-inch ID glass tube extending to 1/2-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.

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,<sup>2</sup> 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<sup>3</sup> 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.

<sup>1</sup> Dry using Drierite<sup>1</sup> at 70° ± 10° F.

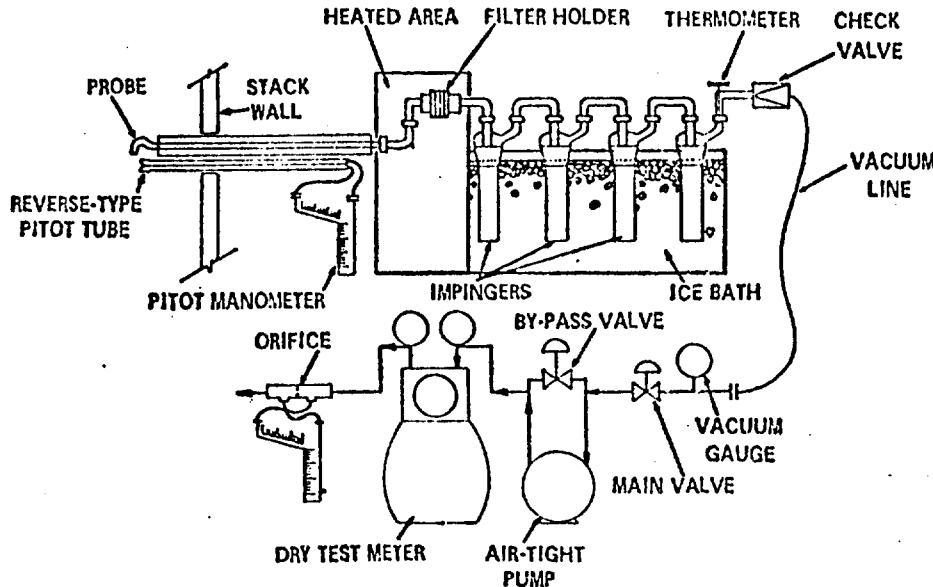


Figure 5-1. Particulate-sampling train.

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, 8 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

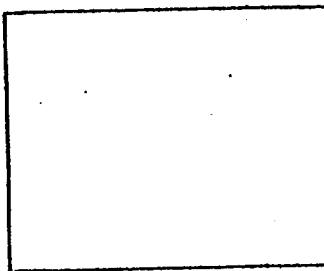
3.2.1 Water—Deionized, distilled.

<sup>1</sup> Trade name.

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PLANT \_\_\_\_\_  
LOCATION \_\_\_\_\_  
OPERATOR \_\_\_\_\_  
DATE \_\_\_\_\_  
RUN NO. \_\_\_\_\_  
SAMPLE BOX NO. \_\_\_\_\_  
METER BOX NO. \_\_\_\_\_  
METER  $\Delta H_3$  \_\_\_\_\_  
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 \_\_\_\_\_

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 add**

silicate, 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. Dissiccate 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. Desiccate, 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.

## PROPOSED RULE MAKING

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.

$$V_{m,14} = V_m \left( \frac{T_{14}}{T_m} \right) \left( \frac{P_{bs} + \Delta H}{P_{std}} \right) = (17.71 \frac{\text{°R}}{\text{in. Hg}}) (V_m) \left( \frac{P_{bs} + \Delta H}{P_{std}} \right)$$

equation 5-1

where:

$V_{m,14}$  = 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.

$T_{14}$  = Absolute temperature at standard conditions, 530° R.

$T_m$  = Average dry gas meter temperature, °R.

$P_{bs}$  = Barometric pressure at the orifice meter, in. Hg.

$\Delta H$  = Pressure drop across the orifice meter, in. H.O.

13.6 = Specific gravity of mercury.

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

## 6.1.3 Volume of Water vapor.

$$V_{w,14} = V_1 \left( \frac{\rho_{H_2O}}{M_{H_2O}} \right) \left( \frac{RT_{14}}{P_{std}} \right) = (0.0474 \frac{\text{cu. ft.}}{\text{ml.}}) V_1$$

equation 5-2

where:

$V_{w,14}$  = Volume of water vapor in the gas sample (standard conditions), cu. ft.

$V_1$  = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.

$\rho_{H_2O}$  = 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.

$T_{14}$  = Absolute temperature at standard conditions, 530° R.

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

## 6.1.4 Total gas volume.

$$V_{total} = V_{m,14} + V_{w,14}$$

equation 5-3

where:

$V_{total}$  = Total volume of gas sample (standard conditions), cu. ft.

$V_{m,14}$  = Volume of gas through dry gas meter (standard conditions), cu. ft.

$V_{w,14}$  = 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.

$$c' = \left( 0.0154 \frac{\text{gr.}}{\text{mg.}} \right) \left( \frac{M_u}{V_{total}} \right)$$

equation 5-4

where:

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

$M_u$  = 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



## PROPOSED RULE MAKING

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-aronophenylazo)-2-naphthol-3, 6-disulfonic acid, disodium salt (or equivalent). Dissolve 0.20 g. in 100 ml. distilled water.

3.3.4 Barium perchlorate (0.01N)—Dissolve 1.95 g. of barium perchlorate [Ba(ClO<sub>4</sub>)<sub>2</sub>·3H<sub>2</sub>O] in 200 ml. distilled water and dilute to 1 liter with isopropanol. Standardize with sulfuric acid.

3.3.5 Sulfuric acid standard (0.01N)—Purchase or standardize against a primary standard to  $\pm 0.0002N$ .

## 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 air 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. erlenmeyer flask. Add 40 ml. of isopropanol and 2 to 4 drops of thorin indicator. Titrate to a pink endpoint using 0.01N 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.

$$V_{m, \text{std}} = V_m \left( \frac{T_{\text{std}}}{T_m} \right) \left( \frac{P_{\text{std}}}{P_m} \right) = (17.71 \frac{\text{in. Hg}}{\text{in. Hg}})^{\frac{V_m P_m}{T_m}} \quad \text{equation 6-1}$$

where:

$V_{m, \text{std}}$  = 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.

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

$T_m$  = Average dry gas meter temperature, °R.

$P_{\text{std}}$  = Barometric pressure at the orifice meter, in. Hg.

$P_m$  = Absolute pressure at standard conditions, 29.92 in. Hg.

## 6.2 Sulfur dioxide concentration.

$$c_{\text{SO}_2} = \frac{7.05 \times 10^{-3} \frac{\text{lb.-l.}}{\text{g.-ml.}} (V_t - V_b) (N) \left( \frac{V_{\text{ref}}}{V_s} \right)}{V_{m, \text{std}}} \quad \text{equation 6-2}$$

where:

$c_{\text{SO}_2}$  = Concentration of sulfur dioxide at standard conditions, dry basis, lb./cu. ft.

$7.05 \times 10^{-3}$  = 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.

$V_b$  = Volume of barium perchlorate titrant used for the blank, ml.

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

$V_{m, \text{std}}$  = Total solution volume of sulfur dioxide, ml.

$V_s$  = Volume of sample aliquot titrated, ml.

$V_{m, \text{std}}$  = 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<sub>2</sub> and SO<sub>3</sub> in Flue Gases. Journal of the Institute of Fuel, 24:237-243, 1961.

Matty, R. E. and E. K. Diehl. Measuring Flue-Gas SO<sub>2</sub> and SO<sub>3</sub>. 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 phenoldisulfone 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<sup>1</sup> 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<sup>1</sup> 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, 30 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.

<sup>1</sup> Trade name.

APPENDIX E

LABORATORY REPORT

## APPENDIX E

LABORATORY REPORTBACK HALF IMPINGER WATER RESIDUE PHASE  
ANALYSIS BY WET CHEMISTRYPlant Revere Smelting and Refining Company, Newark, New JerseyDate December 8, 1971Run 1

	Organic, mg	Inorganic, mg	Total, mg
Split Residue Mass Before Analysis	6.7	12.5 <sup>(a)</sup>	19.2
Constituent Mass per Split Residue			
SO <sub>3</sub>	< 0.3	< 0.3	< 0.3
SO <sub>4</sub>	0.33	8.8	9.13
Total Sample Residue Mass	13.4	25.0 <sup>(a)</sup>	38.4
Constituent Mass per Total Residue			
SO <sub>3</sub>	< 0.6	< 0.6	< 0.6
SO <sub>4</sub>	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, $\mu\text{g}$ (a)	$\frac{\text{Pb, } \mu\text{g per Residue, } \mu\text{g}}{\text{Total Residue}}$	$\frac{\text{Pb, } \mu\text{g per}}{\text{Total Residue}}$	Filter Catch, $\mu\text{g}$	$\frac{\text{Pb, } \mu\text{g per}}{\text{Split Filter Sample, } \mu\text{g}}$	$\frac{\text{Pb, } \mu\text{g per}}{\text{Total Filter Catch}}$
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 CHEMISTRYPlant Revere Smelting and Refining Company, Newark, New JerseyDate December 8, 1972Run 1

Total Sample Volume - 260 ml

Split Sample Volume Analyzed - 130 ml

Constituent	μg/ml	mg per	mg per
		Split Sample Volume	
Total Acid	2	0.26	0.52
SO <sub>3</sub> <sup>=</sup>	160	20.8	41.6
SO <sub>4</sub> <sup>=</sup>	24	3.12	6.24
Cl <sup>-</sup>	2	0.26	0.52
NH <sub>4</sub> <sup>+</sup>	43	5.60	11.20
pH(a)	3.6		

(a) Value is pH units.

LABORATORY REPORTOPTICAL EMISSION SPECTROSCOPY TRACE METAL  
( $\mu\text{g}$  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, $\mu\text{g}$ (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 REPORTOPTICAL EMISSION SPECTROSCOPY TRACE METAL  
( $\mu$ g 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, $\mu$ g	87,000	32,300	58,700	

(a) Filter blank subtracted.

(b) Pb determined by Atomic Absorption.

Note: Symbol &lt; indicates minimal detection limits.

LABORATORY REPORTOPTICAL EMISSION SPECTROSCOPY TRACE METAL  
( $\mu\text{g}$  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, $\mu\text{g}$ (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

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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, $\mu\text{g}/\text{ml}$ of Diluted Aliquot <sup>(a)</sup>	11.1
G - Lead, $\mu\text{g}/\text{Split Filter Sample}$	2500
H - Lead, $\mu\text{g}/\text{Total Filter}$	5000

Example Computation

$$C \times A \times F = G$$

$$50 \times 4.5 \times 11.1 = 2500$$

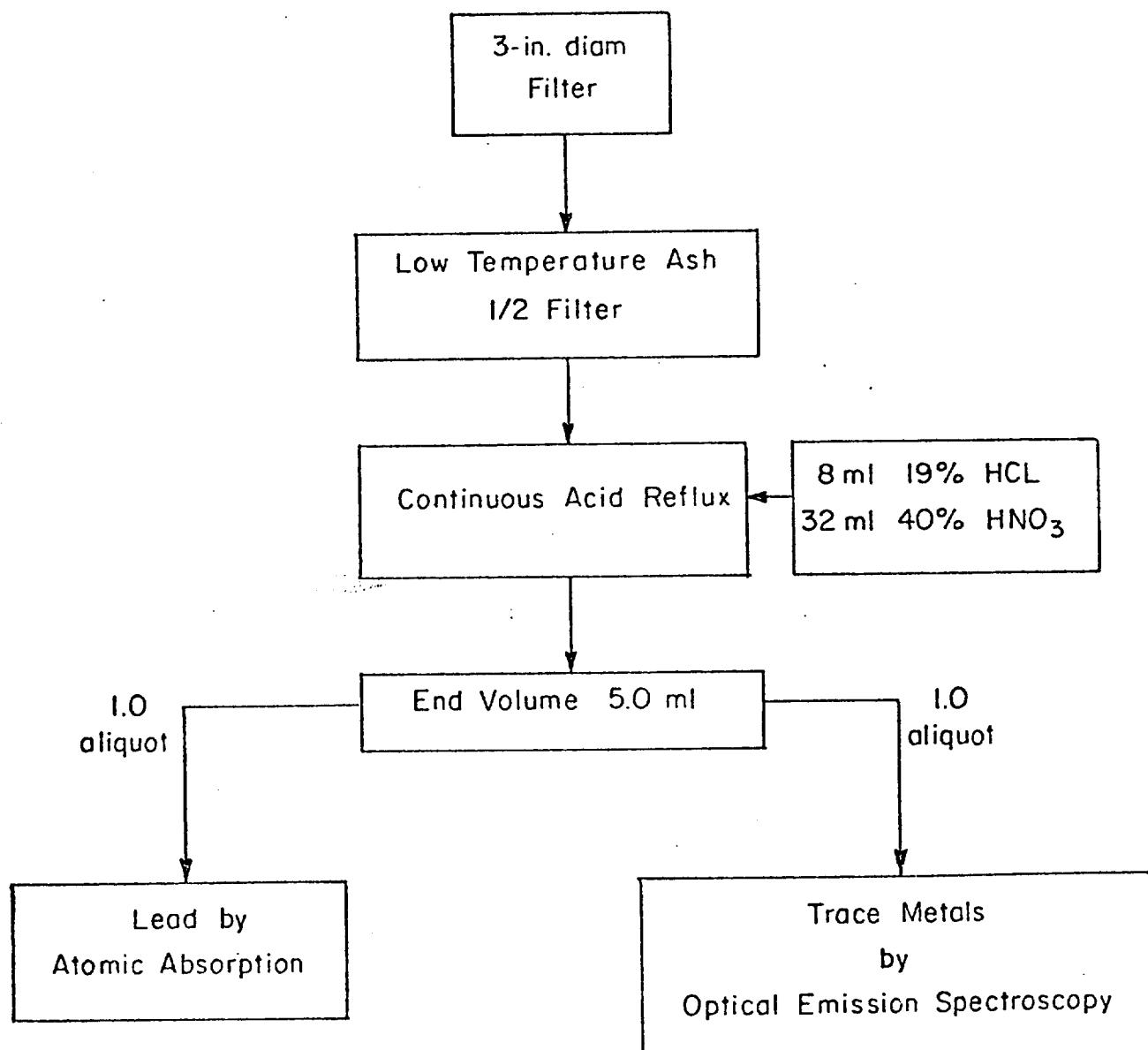
$$2 \times G = H$$

$$2 \times 2500 = 5000$$

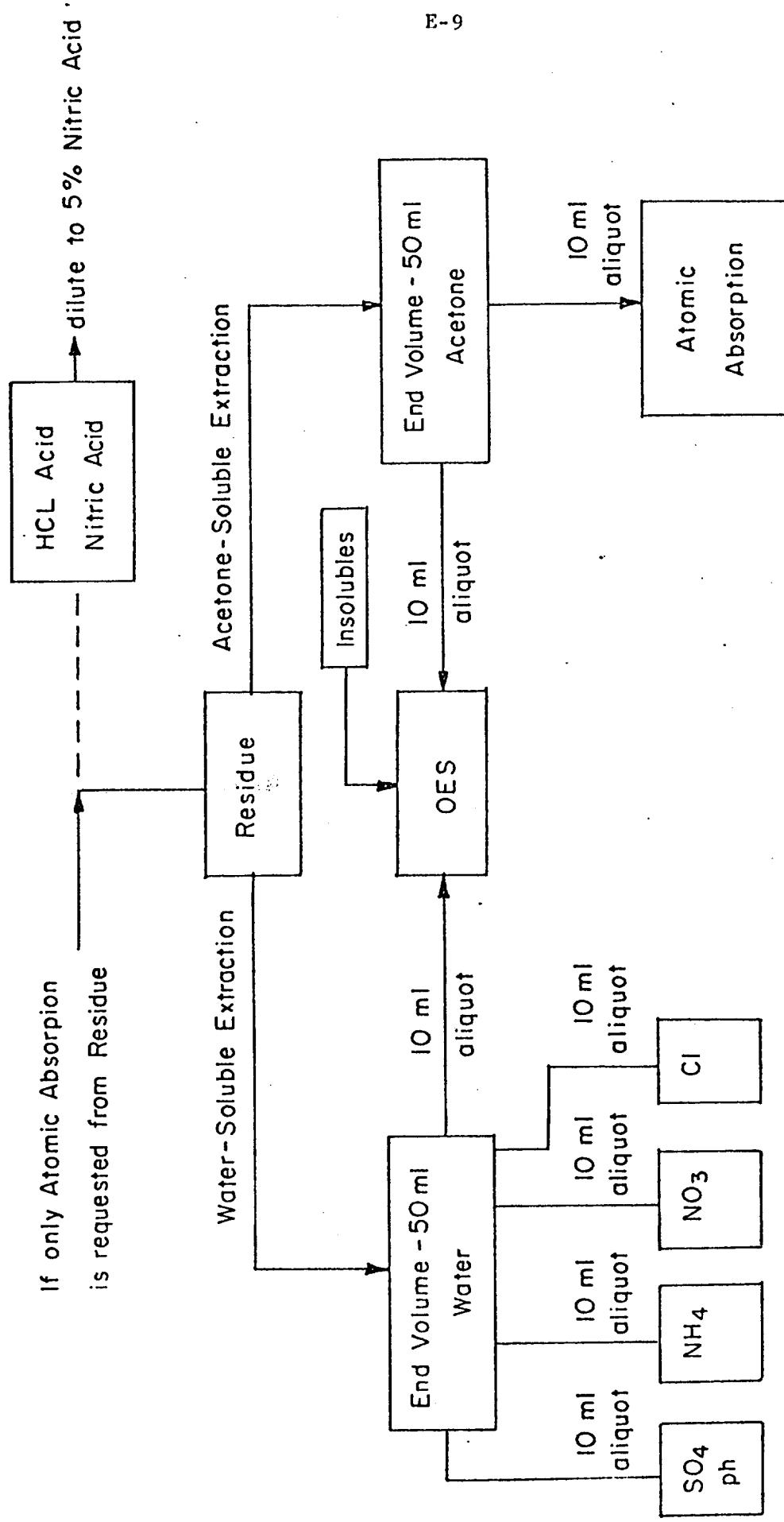
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(a) Determined from calibration curve; based on 0.1707  
absorbance.

## 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).



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  $\text{BaCl}_2$  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  $\text{BaSO}_4$  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  $\text{BaSO}_4$ . Calculate  $\text{SO}_4^=$  by multiplying by the factor 0.4116. Multiply this result by 5 to get total amount of  $\text{SO}_4^=$  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 representative 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 =      10 ml for      10 ml for      10 ml for  
 pH and  $\text{SO}_4^{=}$      $\text{Cl}^-$              $\text{NH}_4^+$             total nitrogen  
 (Devarda's method)

⑥  $\text{NO}_3^-$  will be calculated from the difference between total nitrogen and  $\text{NH}_4^+$

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  $\sim 100$  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  $\text{SO}_4$ ,  $\text{Cl}$ ,  $\text{NH}_4$ , and  $\text{NO}_3$  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 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 (a) 3:05
Furnace feed times <sup>(c)</sup>	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 NOTESPlant - Revere Smelting and Refining Company  
Newark, New Jersey12/9/71

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 Rod 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<sub>2</sub>O, 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<sub>2</sub>O 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
<u>Stack Effluent</u>				
Flow rate - DSCFM	12,100	13,330	12,540	12,657
Flow rate - DSCF/ton	8,067	11,108	10,450	9,875
Temperature - °F	97	95	93	95
Water vapor - Vol. %	2.4	1.7	2.3	2.13
CO <sub>2</sub> - Vol. % dry	0.5	0.8	0.8	0.7
O <sub>2</sub> - Vol. % dry	20.3	20.2	18.8	19.7
CO - Vol. % dry	0.8	0.3	0.2	0.4
CO - 1b/hr	420	160	109	330
SO <sub>2</sub> - ppm dry	0.06	0.04	0.08	0.06
SO <sub>2</sub> - 1b/hr	0.01	0.01	0.01	0.01
Visible emissions - % opacity	10-15	10-20	5-15	12
<u>Particulate Emissions</u>				
Probe and filter catch gr/DSCF	0.0196	0.0084	0.0149	0.0143
gr/ACF	0.0184	0.0080	0.0140	0.0135
1b/hr	2.0354	0.8913	1.6061	1.5109
1b/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
1b/hr	2.8474	1.6556	2.5200	2.3410
1b/ton lead	1.8983	1.4397	2.0160	1.7847
<u>Lead Emissions</u>				
Probe and filter catch gr/DSCF	0.00127	0.00061	0.00127	0.00105
gr/ACF	0.00119	0.00058	0.00120	0.00099
1b/hr	0.1320	0.0646	0.1368	0.1111
1b/ton lead	0.0880	0.0562	0.1095	0.0846
<u>Total Catch</u>	Essentially the same as probe and filter.			

SECONDARY LEAD  
SMELTING  
AP-42 Section 7.11  
Reference Number  
10C  
✓

RESEARCH REPORT

DP



**Baffelle**

Environmental Laboratories

RESEARCH REPORT

on

SECONDARY LEAD PLANT STACK EMISSION  
SAMPLING AT QUEMETCO, INCORPORATED,  
INDUSTRY, CALIFORNIA

Contract No. 68-02-0230

Task Order No. 1

to

ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF AIR PROGRAMS

August 19, 1972

by

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

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INTRODUCTION

Gas and particulate emission measurements were made at the Quemetco, Incorporated, a subsidiary of St. Joe Minerals Corporation, secondary lead plant in Industry, California, on January 26 and 27, 1972. The results of the measurement at this and other lead plants will be used in determining standards of performance for secondary lead plants. The effluent gas from a lead reverberatory furnace was sampled in the stack from the reverberatory furnace baghouse outlet after particulate removal by a cyclone, three settling chambers, and a baghouse in series. Particulate and SO<sub>2</sub> emissions were determined and an Orsat analysis was made. The fume from the lead and slag taps was treated in a separate baghouse. A blast furnace which was fed exclusively on reverberatory furnace slag, and a lead oxide plant were on the same site, but had separate dust-control systems.

The reverberatory furnace is used to melt down manufacturers' scrap and worn out batteries as well as various lead oxide drosses and dusts. The reverberatory furnace differs, in use, from a lead blast furnace in that it is used to remove antimony from lead in order to produce a soft lead product. The high antimony slag by-product generally is of equal value to the metallic lead. Because of a temporary tight supply of antimony, the slag was the economically important product at the time of testing.

Figure 1 is a sketch showing the gas flow through the reverberatory furnace and its gas cleaning system. The complex ductwork collects most of the dust from the furnace and cools the gas from about 1700 F at the furnace to about 200 F at the baghouse inlet. The baghouse filters out the rest of the dust and the clean gas is exhausted through a short stack which is mounted on the exhaust fan. Samples were taken from near the top of the stack.

#### SUMMARY

The stack on the secondary lead reverberatory furnace at Quemetco, Inc., in Industry, California, was sampled for particulates  $SO_2$ ,  $CO_2$ ,  $O_2$ , and CO in 3 runs of 2 hours each on January 26 and 27, 1972. A summary of the results is presented in Table 1, Appendix I. The furnace was operated on an 8-hour duration batch cycle. The first 2 runs were made during a period of slag tapping when the feed rate was low and the furnace was hot; the third run was made during a period of high feed rate, high firing rate, and at a time when the furnace was cool.

The particulate caught in the probe and filter for the 3 runs was consistent and averaged 0.0035 gr/scf which is equivalent to an emission of 0.21 lb/ton of lead. The lead in the particulate emission averaged 0.0005 gr/scf. Additional material was caught in the impinger section of the particulate train, which increased the average total emission to 0.011 gr/scf or 0.66 lb/ton for the first 2 particulate runs when the furnace was fed slowly, and to 0.021 gr/scf or 1.16 lb/ton during the third particulate run.

The  $SO_2$  concentration was measured in 4 runs: the last 3 concurrent with particulate sampling. The  $SO_2$  emission was consistent for the last 3 runs at 1574 ppm or 109 lb/ton of lead produced. The first run was made at a time of low firing rate and the  $SO_2$  concentration in the stack gas was 685 ppm.

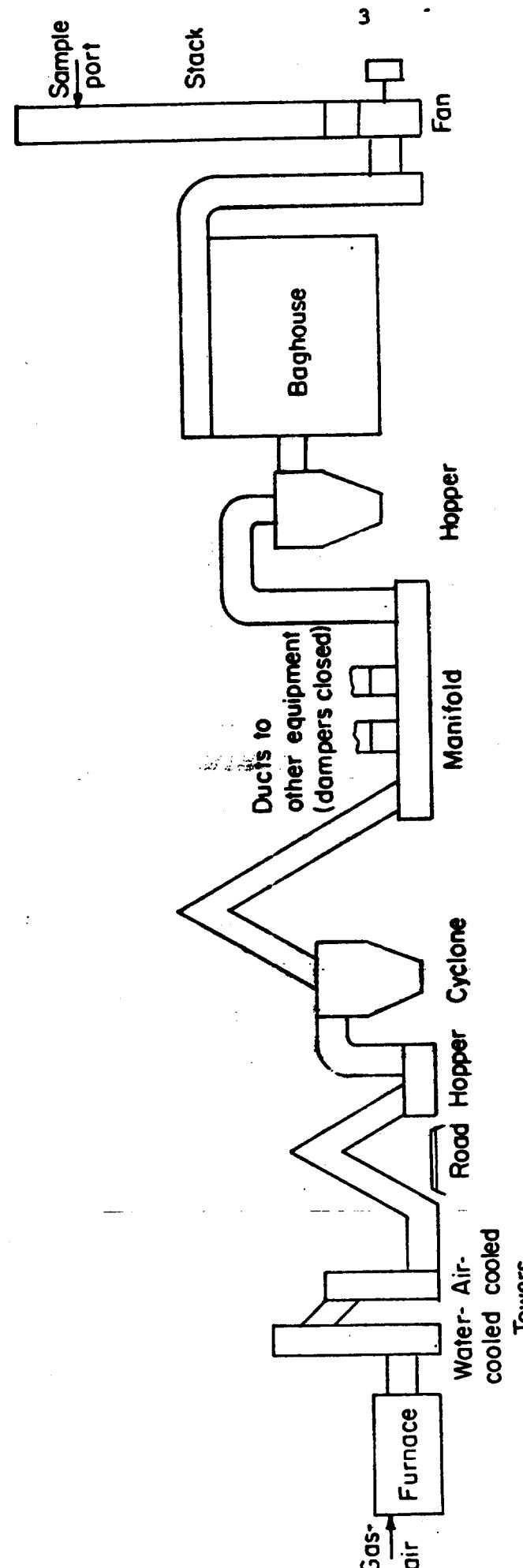


FIGURE 1. DIAGRAM OF REVERBERATORY FURNACE GAS FLOW, QUIMETCO, INC. INDUSTRY, CALIFORNIA

The Orsat analysis shows that the combustion gases are diluted with about 11 volumes of air in the furnace and ductwork following the furnace. Carbon monoxide, if present, was in concentrations below the measuring accuracy of the Orsat (about 0.1 percent).

A chemical analyses was made of the acetone probe wash residue. Few metals were detected; lead being the major metal. None of the more common anions were found and, therefore, it can be concluded that the probe wash residue was generally organic in nature and organics would not be detected by these analyses.

Run 1 impinger water residue and its chloroform - diethylether extract residue were analyzed by optical emission spectroscopy and atomic absorption. Total lead detected by atomic absorption in the two residues was 0.6 mg as compared to 5.6 mg in the front-half residues (lead emission calculations are based only on front-half data). Sulfate was the major element detected by optical emission spectroscopy having a mass of 22.0 mg.

#### PROCESS DESCRIPTION

The Quemetco Plant processes various lead scraps, mainly used automobile batteries, into lead ingots of various compositions and into lead oxides. The plant consists of a large receiving yard, a battery cutting operation, a reverberatory furnace, a blast furnace, lead kettles and casting equipment, and a lead oxide facility. Most of the scrap is received in the form of discarded batteries. Some manufacturers' scrap from battery making operations is also received. The lead ingots are cast to compositions specified by the customer.

The obsolete automobile batteries are fed into the battery cutting operation as they are received. The tops are cut off, the acid dumped and hauled away by a disposal company, the battery plates removed, and the cases ground to coarse pieces. The battery plates are stored for a week or more to drain the acid. A feed of battery plates, ground-up battery cases, and process dust is fed to the reverberatory furnace. The soft lead from the furnace is further refined in kettles, then cast into ingots for sale. The slag from the reverberatory furnace is the only

lead feed to the blast furnace. The high antimony lead is mixed in kettles with soft lead from the reverberatory furnace in preparation for various commercial alloys. Some of the soft lead is used to make lead oxide on the same site.

#### The Reverberatory Furnace

A sketch of a lead reverberatory furnace is shown in Figure 2. A charge of lead is normally rammed in from one end with a burner located on each side of the ram. The natural gas flame passes over the hearth and the heat melts the lead. The exhaust gasses pass through a refractory flue. Lead and slag are tapped from opposite sides of the furnace at the end opposite the feed. The hearth at Quemetco was about 25 feet long by 8 feet wide. The roof was about 3 feet above the melt. The furnace was batch-fed each 8-hour shift. When a new shift arrives, the gas to the furnace is turned off and the collected dust is removed from the ductwork and immediately fed back into the furnace with a Payloader. This operation takes about 1/2 hour. The ductwork is rapped with sledge hammers to knock the dust into the hoppers.

After the ducts are cleaned the gas burners are turned on "high" and the furnace is charged for 1-1/2 to 2 hours, as quickly as possible. At the end of this period a large heel of solid feed material is in the feed end of the furnace and another 30 minutes is required to melt it.

During the meltdown period the furnace is stirred by hand, both to increase the melting rate and to mix the slag with the lead to dissolve the antimony in the slag. Following meltdown, the slag is tapped for 1/2 to 1 hour. The natural gas flow to the burners is reduced to perhaps 50 percent of maximum during this operation.

Three to five tons of slag are tapped at a time into 500-lb cone-shaped blocks called "buttons". Fumes from the hot slag taps from the furnace are collected in a hood system. Several times the slag buttons were

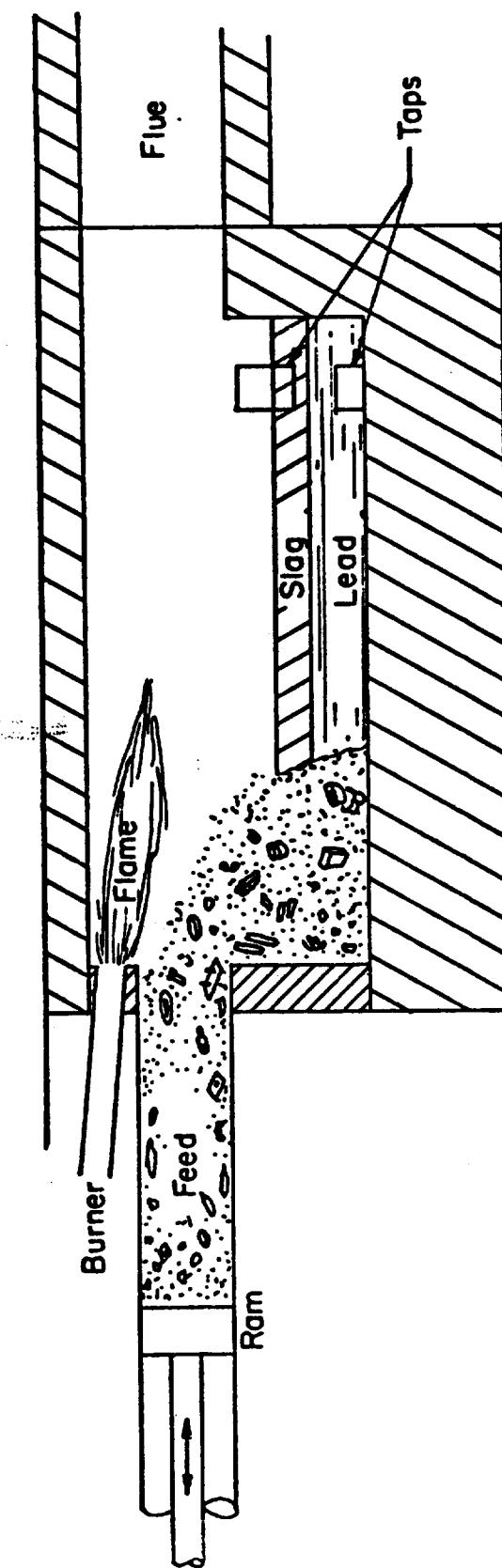


FIGURE 2. SKETCH OF REVERBERATORY FURNACE

removed from the hood before they stopped fuming.

After tapping slag the furnace is allowed to stabilize for an hour, then the crew starts tapping lead into an ingot mold that has compartments for five 1-ton hogs. The furnace is fed at a decreased rate during this time and the gas burners are operated at about 70 percent capacity. While a hood is placed over the lead well, no fumes were observed. The hood is used primarily to exhaust the gas from a burner used to heat the well when lead is not being tapped.

During the last 1/2 hour of the shift, feeding is stopped and the burners are turned to maximum capacity to melt out any heel of solids that may be at the feed end of the furnace.

The feed to the furnace is in 1000 pound increments and is pre-mixed in a large storage pile. It consists of battery plates, flue dust, purchased oxides, and broken battery cases. The battery cases made the furnace slightly reducing and hence some oxides are reduced in the furnace.

A natural gas-air mixture is burned in two pressure burners at the feed end of the furnace. The firing rate to each burner is set by hand-operated valves with a single handle to control both the gas and air flows. At full flow, the air pressure in the line to the burner is about 22 ounces, and at normal firing rate the pressure is about 19 ounces. The burners are frequently adjusted to meet the changing heat demands of the furnace. From the Orsat analysis, assuming all  $\text{CO}_2$  comes from the natural gas, the firing rate is 265 cfm of natural gas or 16,000,000 Btu/hr. From the water-content of the flue gas, a similar calculation indicates a firing rate of 13,000,000 Btu/hr. The vacuum in the furnace is estimated at 1/4 to 1/2 inch of water. Air is pulled into the furnace at high velocity through the two sight and stirring ports on the feed end of the furnace, the feed port, and the slag port. This air is necessary to burn the volatiles in the battery cases.

Gas Cleaning Equipment

A diagram of the gas cleaning equipment is shown in Figure 1. The gas from the furnace is exhausted through a brick flue at about 1700 F and travels into a water cooled tower 8 feet in diameter and 30 feet high. Little dust is collected in the base of this tower. The gases then pass into a steel tower about 5 feet in diameter and 18 feet high. The gases then pass through a 40-inch-diameter duct in the form of an inverted "V" with the top of the "V" about 20 feet high. About 2000 pounds of dust per shift is collected in a hopper under the first leg of the "V" and 2500 pounds per shift in a hopper under the second leg. This second hopper also collects dust from a duct to the following cyclone. Another 2000 pounds per shift is removed from the cyclone. The gases then pass through a manifold where gas from other equipment could be sent to the baghouse on the reverberatory furnace or the furnace off-gas could be sent to other gas cleaning equipment. Dampers in ducts to other equipment were closed. The flue gas then passes through another hopper where about 100 pounds of dust per shift is collected, and into the baghouse which collects another 1500 pounds of dust per shift. All of the equipment between the water cooled tower and the baghouse is constructed of uninsulated steel and the gases passing through it are cooled to about 200 F.

The baghouse, a product of the Rees Blow Pipe Manufacturing Company, was recently insulated to prevent water condensation. The baghouse is constructed in seven sections having a total of 1120 bags; each 6 inches in diameter and 10 feet high. With a total bag area of 17,800 ft<sup>2</sup>, and a gas flow of 15,000 cfm, the air-to-cloth ratio is 0.9. During the first run, the shaking cycle was incorrectly adjusted to give a 10-second shake per section with a 40 second time interval between sections; the total cycle being 6 minutes. Before the second run, the timer was correctly adjusted to give a 35-second shake per section with a 6-1/2-minute time interval between sections; the total cycle being 50 minutes. During the shaking, dampers isolate each section.

LOCATION OF SAMPLING POINT

All of the samples were obtained from the stack at a point 28 feet above ground as shown in Figure 3. The Orsat and SO<sub>2</sub> samples were obtained from the center of the stack, and the particulate sample was obtained from a 20-point traverse and are indicated in Figure 3. The sampling point is seven stack diameters above the square to round transition, a distance which requires a minimum of 20 traverse points. The stack outlet is 1.8 stack diameters above the sampling point, a distance which also requires a minimum of 20 points, as indicated by the Federal Register in Appendix E.

PROCESS OPERATION

The process operation was close to normal. The furnace was started on Monday and the tests were made on Wednesday and Thursday, which may not have been sufficient time to develop smooth furnace operation. A torn bag was found after Run 2 had been completed. The fact that Run 2 had the lowest dust loading of any run suggests that the tear actually occurred after the completion of Run 2. An excessive shake cycle during Run 1 may have contributed to a higher dust loading for that run.

Because of a shortage of antimony ore, the antimonial slag was the more valuable product of the reverberatory furnace. The slag product of the furnace was fed to a blast furnace for the production of hard lead. The blast furnace lead was shipped the same day it was processed. The soft lead from the reverberatory furnace was stored. The desire for slag, rather than lead, is not considered to effect furnace operation or performance.

Furnace Operation

The first sample run was started during the meltdown period following feeding and continued through the slag tap into the initial stages of the lead tap period. From this point in the cycle, the particulate emission would be expected to be near the maximum because the

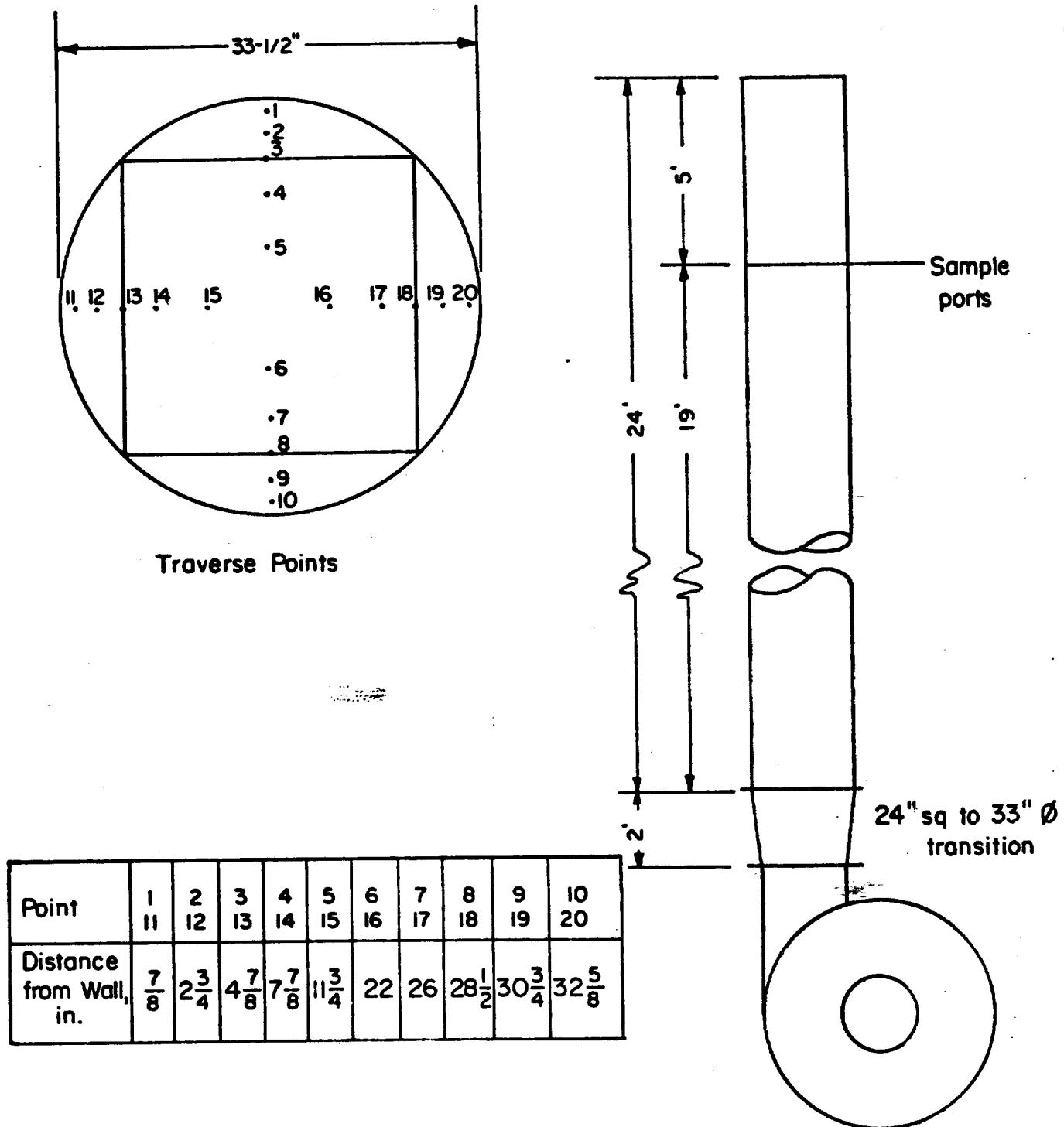


FIGURE 3. SAMPLING POINTS

furnace temperature is at its peak; the gas flows are the highest encountered during the cycle, and because during the meltdown period the solid unmelted charge is near or in contact with high velocity burner gases.

During the second sample run, at the time lead tapping would have been expected, antimony had not migrated into the slag. At this time, additional oxides were fed to the furnace prior to making a second slag tap. Near the end of the run, lead was tapped at a high rate over a short time period. Therefore, during this run the particulates would be expected to be high for the same reasons given for Run 1.

The third sample run was started during the middle of the furnace loading period and continued through the melting and the slag tapping periods. This should be a period of normal to high particulate emission.

The long lead-tapping period was not sampled. The particulate emission is judged to be slightly less during this period because the furnace is relatively cool. Also, the amount of dust in the furnace by visual observation appeared to be less.

Emission samples were not taken during the duct cleaning period, however, the emission may be characterized by long periods of low dust loading interspersed with short, very high, peak loads induced by banging the dust from the ducts with hammers. The amount of particulates caught on the sampling filter was about the same for the three runs. The impinger residue for Run 3 was high in comparison to Runs 1 and 2.

#### Operation of Gas-Cleaning System

During the first sample run, the shaking mechanism on the baghouse was operated excessively and therefore, a higher than normal dust loading was expected, but not observed. Following the second run, a torn bag was found but the expected high dust loading was not observed. Dust from the stack was observed by the plant manager 2 hours after the sample

was taken and the torn bag was then replaced. The gas-cleaning system was operating normally during the third sample run so that average emissions were expected and observed.

About 1000 lb/hr of dust is emitted from the furnace and of this amount about 800 pounds is collected in the ductwork. Nearly all of the remaining 200 pounds is collected in the baghouse. About 0.5 lb/hr is emitted from the stack based on probe and filter collection so that the overall collection efficiency is 99.95 percent. The baghouse efficiency is 99.7 percent. The efficiencies are based on probe and filter catch as required by current specifications published in the Federal Register, December 23, 1971.

#### SAMPLING AND ANALYTICAL PROCEDURES

The off gases from the lead blast furnace were sampled for particulates  $SO_2$ ,  $CO_2$ ,  $O_2$ , and CO by the methods described in Appendix E and the Federal Register of August 17, 1971.

The standard procedures as described in the Federal Register of December 23, 1971, have been followed in reporting data in that only the absolute filter catch is used to estimate collection efficiencies. The particulate emission gas was sampled for 6 minutes at each point using points recommended for a 20 point traverse. A 0.235-inch-diameter probe tip was used for all particulate emission samples. About 100 cubic feet of stack gas was sampled during each run.

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

The  $\text{SO}_2$  samples were taken simultaneously with the particulate sampled for a period of 1-1/4 hours during which time a gas sample volume of about 10 ft<sup>3</sup> was collected. A 1 ml aliquot of the impinger liquid and washings was pippetted into an Erlenmeyer flask and titrated against 0.01 normal barium perchlorate using a thorin indicator.

An Orsat sample was taken from the center of the stack simultaneously with the particulate sample. The sample was drawn into a 2-ft<sup>3</sup> plastic bag over a period of one hour. After flushing the Orsat with collected sample, a 100-ml volume was taken. It was analyzed as follows: the volume was adjusted to 100 ml over acidulated water in the measuring burette; the  $\text{CO}_2$  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 analysis was repeated using the same sample.

**APPENDIX A**

**COMPLETE PARTICULATE RESULTS WITH SAMPLE CALCULATIONS**

## COMPLETE PARTICULATE RESULTS WITH SAMPLE CALCULATIONS

SOURCE TESTING CALCULATION FORMS

Test No. \_\_\_\_\_

No. Runs 3Name of Firm Quemetco, Inc.Location of Plant Industry, CaliforniaType of Plant Secondary LeadControl Equipment BaghouseSampling Point Locations StackPollutants Sampled Particulate, SO<sub>2</sub>, Orsat

## Time of Particulate Test:

Run No. <u>1</u>	Date <u>1/26/72</u>	Begin <u>10:15 am</u>	End <u>12:32 pm</u>
Run No. <u>2</u>	Date <u>1/26/72</u>	Begin <u>1:50 pm</u>	End <u>4:02 pm</u>
Run No. <u>3</u>	Date <u>1/27/72</u>	Begin <u>9:43 am</u>	End <u>11:55 am</u>

PARTICULATE EMISSION DATA

Run No.	1	2	3
P <sub>b</sub> -barometric pressure, in. Hg Absolute	29.58	29.63	29.9
P <sub>m</sub> -orifice pressure drop, in. H <sub>2</sub> O	1.83	1.8	1.55
V <sub>m</sub> -volume of dry gas sampled @ meter conditions, ft <sup>3</sup>	95.8	97.2	87.6
T <sub>m</sub> -average gas meter temperature, F	62.8	65	65
V <sub>m</sub> <sup>std.</sup> - volume of dry gas sampled @ standard conditions, ft <sup>3</sup> , dry	96.38	97.53	88.64
V <sub>w</sub> -total H <sub>2</sub> O collected, impingers and silical gel., ml.	65	60.3	60.5
V <sub>w</sub> - volume of water vapor collected gas ft <sup>3</sup> @ standard conditions(a)	3.08	2.86	2.87
V <sub>total</sub> -total gas volume, ft <sup>3</sup>	99.46	100.39	91.51
Moisture in the stack gas volume, percent	3.1	2.8	3.1
M <sub>d</sub> -mole fraction of dry gas	0.97	0.97	0.97

PARTICULATE EMISSION DATA (Cont'd)

Run No.	1	2	3
CO <sub>2</sub> , dry, percent	1.8	1.8	1.8
O <sub>2</sub> , dry, percent	18.6	18.7	18.7
CO, dry, percent	<0.1	<0.1	<0.1
N <sub>2</sub> , dry, percent	79.5	79.4	79.5
M W <sub>d</sub> - molecular weight of dry stack gas	29.03	29.03	29.03
M W - molecular weight of stack gas	28.7	28.7	28.7
T <sub>s</sub> - stack temperature, F	166	164	175
ΔP <sub>s</sub> x (T <sub>s</sub> + 460)	19.759	20.415	19.389
P <sub>s</sub> - stack pressure, in. Hg absolute	29.58	29.63	29.9
V <sub>s</sub> - stack velocity @ stack conditions, fpm	2950	3044	2879
A <sub>s</sub> - stack area, in <sup>2</sup>	880	880	880
Q <sub>s</sub> - stack gas volume @ standard conditions, (a) ft <sup>3</sup> dry	14,620	15,200	14,200
T <sub>t</sub> - net time of test, min.	120	120	120
D <sub>n</sub> - sampling nozzle diameter, in.	0.235	0.235	0.235
Percent I - percent isokinetic	111	108	105
m <sub>f</sub> - particulate; probe, and filter, mg	26.9	18.0	20.3
m <sub>t</sub> - particulate; total, mg	82.5	54.2	114.9
C <sub>an</sub> - particulate; probe, and filter, gr/scf, dry	0.0043	0.0028	0.0035
C <sub>ao</sub> - particulate; total, gr/scf	0.0132	0.0086	0.0200

(a) 70 F, 29.92 in. Hg

PARTICULATE EMISSION DATA (Cont'd)

Run	1	2	3
$C_{at}$ - particulate; probe and filter, gr/ft <sup>3</sup> @ stack conditions	0.0035	0.0023	0.0028
$C_{au}$ - particulate; total, gr/ft <sup>3</sup> @ stack conditions	0.0107	0.0070	0.0161
$C_{aw}$ - particulate; probe and filter, lb/hr	0.5387	0.3702	0.4297
$C_{ax}$ - particulate; total, lb/hr	1.6520	1.1148	2.4321
$C_p$ - particulate; probe and filter, 1b/ton lead	0.2565	0.1763	0.2046
$C_{pt}$ - particulate; total 1b/ton lead	0.7867	0.5309	1.1581
$C_{ls}$ - lead emission; probe and filter, gr/scf	0.00090	0.00049	0.00045
$C_{la}$ - lead emission; probe and filter, gr/ft <sup>3</sup>	0.00073	0.00040	0.00037
$C_{lh}$ - lead emission; probe and filter, 1b/hr	0.1130	0.0640	0.0553
$C_{lt}$ - lead emission; probe and filter, 1b/ton	0.0538	0.0305	0.0263

EXAMPLE PARTICULATE CALCULATIONS

## SAMPLE NUMBER 1

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

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

$$= \frac{17.7 (95.8) \left( 29.58 + \frac{1.83}{13.6} \right)}{62.8 + 460}$$

$$= 96.38 \text{ ft}^3$$

2. Volume of water vapor at 70 F and 29.92 in. Hg,  $\text{ft}^3$

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

$$V_{w_{gas}} = 4.74 \times 10^{-2} (65)$$

$$= 3.08 \text{ ft}^3$$

3. Percent moisture in stack gas

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

$$= \frac{100 \times 3.08}{96.6 + 3.08}$$

$$= 3.1$$

4. Mole fraction of dry gas

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

$$= \frac{100 - 3.1}{100}$$

$$= 0.97$$

## EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

## 5. Average molecular weight of dry stack gas

$$\begin{aligned}
 M W_d &= (CO_2 \times \frac{44}{100}) + (O_2 \times \frac{32}{100}) + (N_2 + CO) \times \frac{28}{100} \\
 &= (1.8 \times \frac{44}{100}) + (18.6 \times \frac{32}{100}) + 79.6 \times \frac{28}{100} \\
 &= 29.03
 \end{aligned}$$

## 6. Molecular weight of stack gas

$$\begin{aligned}
 M W &= M W_d \times M_d + 18 (1 - M_d) \\
 &= 29.03 (0.97) + 18 (1 - 0.97) \\
 &= 28.7
 \end{aligned}$$

## 7. Stack velocity @ stack conditions, fpm

$$\begin{aligned}
 v_s &= 4350 \times \sqrt{\Delta P_s \times (T_s + 460)} \times \left( \frac{1}{P_s \times M W} \right)^{1/2} \text{ fpm} \\
 &= (4350) \times 19.76 \times \left[ \frac{1}{29.58 (28.7)} \right]^{1/2} \\
 &= 2950 \text{ ppm}
 \end{aligned}$$

## 8. Stack gas volume at standard conditions

$$\begin{aligned}
 Q_s &= \frac{0.123 \times v_s \times A_s \times P_s \times M_d}{(T_s + 460)} \text{ scfm} \\
 &= \frac{0.123 \times 2950 (880) 29.58 (0.97)}{626} \\
 &= 14,600 \text{ scfm}
 \end{aligned}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

9. Sampling velocity, percent of isokinetic

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

$$= \frac{(1032) (626) (96.38)}{(2950) (120) (29.58) (0.97) (0.235)^2}$$

$$= 111 \text{ percent}$$

10. Percent excess air at sampling point

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

$$= \frac{100 \times 18.6}{(0.266 \times 79.5) - 18.6}$$

$$= 730 \text{ percent}$$

11. Particulate - probe and filter, gr/scf

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

$$= 0.0154 \times \frac{26.9}{96.38}$$

$$= 0.0043 \text{ gr/scf}$$

12. Particulate - total, gr/scf

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

$$= 1.54 \times 10^{-2} \frac{82.5}{96.38}$$

$$= 0.0132 \text{ gr/scf}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

13. Particulate - probe and filter, gr/ft<sup>3</sup> at stack conditions

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

$$= \frac{17.7 \times 0.0043 \times 29.58 \times 0.97}{626}$$

$$= 0.0035 \text{ gr/ft}^3$$

14. Particulate, total, gr/ft<sup>3</sup> at stack conditions

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

$$= \frac{17.7 \times 0.0132 \times 29.6 \times 0.97}{626}$$

$$= 0.0107 \text{ gr/ft}^3$$

15. Particulate - probe and filter

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

$$= 0.00857 \times 0.0043 \times 14,600$$

$$= 0.5387 \text{ lb/hr}$$

16. Particulate - total, lb/hr

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

$$= 8.57 \times 10^{-3} \times 0.0132 \times 14,600$$

$$= 1.6520 \text{ lb/hr}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

17. Particulate; probe and filter, 1b/ton lead

$$\begin{aligned} C_p &= C_{aw}/R \\ &= 0.5387/2.1 \\ &= 0.2565 \text{ lb/ton lead} \end{aligned}$$

18. Particulate; total 1b/ton lead

$$\begin{aligned} C_{pt} &= C_{ax}/R \\ &= 1.6520/2.1 \\ &= 0.7867 \text{ lb/ton lead} \end{aligned}$$

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

$$\begin{aligned} C_{1s} &= 0.0154 \times \frac{M_1}{V_m \text{ std}} \\ &= 0.0154 \times \frac{5.64}{96.38} \\ &= 0.00090 \text{ gr/scf} \end{aligned}$$

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

$$\begin{aligned} C_{1a} &= \frac{17.7 \times C_{1s} \times P_s \times M_d}{(T_s + 960)} \\ &= \frac{17.7 \times 0.0009 \times 29.58 \times 0.97}{626} \\ &= 0.00073 \text{ gr/scf} \end{aligned}$$

21. Lead emission; probe and filter, 1b/hr

$$\begin{aligned} C_{1h} &= 8.57 \times 10^{-3} \times C_{1s} \times Q_s \\ &= 8.57 \times 10^{-3} \times 0.0009 \times 14,600 \\ &= 0.1130 \text{ lb/hr} \end{aligned}$$

22. Lead emission; probe and filter, 1b/ton lead

$$\begin{aligned} C_{1t} &= C_{1h}/R \\ &= 0.1130/2.1 \\ &= 0.0538 \text{ lb/ton lead} \end{aligned}$$

**APPENDIX B**

**COMPLETE GASEOUS RESULTS WITH**  
**SAMPLE CALCULATIONS**

## COMPLETE GASEOUS RESULTS WITH SAMPLE CALCULATIONS

SO<sub>2</sub> EMISSION DATA

Run No.	1	2	3	4
Date (1972)	1/25	1/26	1/26	1/27
T <sub>m</sub> - average gas meter temperature, F	65	57	57	55
P <sub>b</sub> - barometric pressure, in. Hg absolute	29.55	29.66	29.63	29.90
V <sub>m</sub> - volume of dry gas sampled at meter conditions, ft <sup>3</sup>	8.42	11.92	12.18	12.32
V <sub>m</sub> standard	8.39	12.10	12.36	12.66
V <sub>t</sub> - volume barium perchlorate, ml	19.30	56.75	47.00	51.65
V <sub>tb</sub> - volume barium perchlorate blank, ml	0	0	0	0
N - normality of barium perchlorate	0.01	0.01	0.01	0.01
V <sub>soln</sub> - volume SO <sub>2</sub> soln, ml	70	79	94	93
V <sub>a</sub> - volume aliquot, ml	1.0	1.0	1.0	1.0
C <sub>SO<sub>2</sub></sub> - 1b/ft <sup>3</sup> x 10 <sup>-5</sup>	11.35	26.1	25.2	26.8
C <sub>SO<sub>2</sub></sub> - ppm (dry)	685	1580	1525	1620
W <sub>SO<sub>2</sub></sub> - 1b/hr	102	229	230	228

SO<sub>2</sub> EXAMPLE CALCULATIONS

## RUN 2

$$1. V_{m_{std}} = 17.7 \times V_m \left( \frac{P_{BAR}}{T_m + 460} \right)$$

$$= 17.7 \times 11.92 \left( \frac{29.66}{517} \right)$$

$$= 12.10 \text{ ft}^3$$

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

$$= \frac{7.05 \times 10^{-5} \times 56.75 \times 0.01 \times 79}{12.10 \times 1.0}$$

$$= 26.1 \times 10^{-5} \text{ lb/ft}^3$$

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

$$= 6.05 \times 10^6 \times 26.1 \times 10^{-5}$$

$$= 1580 \text{ ppm}$$

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

$$= 26.1 \times 14,600 \times 60$$

$$= 229 \text{ lb/hr}$$

## APPENDIX C

## PRELIMINARY FIELD DATA

STACK GEOMETRYPlant Quemetco, Inc., Industry, CaliforniaTest No. 1Wall thickness, in. 1/4Location Baghouse outletInside diameter of stack. 33.5 in.Date 1/26/72Stack area  $\frac{\pi}{4} (33.5)^2 = 880 \text{ in}^2$   
6.1 ft}^2

Point	Percent Diameter for Circular Stack	Distance from Outside of Sample Port, in.
1	2.5	7/8
2	8.2	2-3/4
3	14.6	4-7/8
4	22.6	7-5/8
5	34.2	11-3/4
6	65.8	22
7	77.4	26
8	85.4	28-1/2
9	91.8	30-3/4
10	97.5	32-5/8

VELOCITY TRAVERSE FIELD DATAPlant Quemetco, Inc., Industry, CaliforniaTest Preliminary velocity traverseDate 1/26/72Operator Paul R. WebbMeter  $\Delta H$  1.84

Point	(1) P. in., $H_2O$
1	0.4
2	0.5
3	0.6
4	0.6
5	0.75
6	0.65
7	0.7
8	0.6
9	0.6
10	0.5

(1)  $\Delta P$ , in.  $H_2O$  Average 0.59.

PARTICULATE CLEANUP SHEETPlant Quemetco, Inc., Industry, CaliforniaOperators P.R. Webb, H. Leonard

Run No.	1	2	3
Impinger water volume, ml			
Final	247	248	247
Initial	<u>200</u>	<u>200</u>	<u>200</u>
Collected	47	48	47
Drierite, weight, gm			
Final	531.7	487.3	488.5
Initial	<u>513.7</u>	<u>475.0</u>	<u>475.0</u>
Gain	18.0	12.3	13.5
Total moisture, gm plus ml	65.0	60.3	60.5
Probe, acetone wash residue, mg <sup>(a)</sup>	5.4	3.7	4.9
Filter weight, mg			
Final	317.5	311.9	308.2
Tare	<u>296.0</u>	<u>297.6</u>	<u>292.8</u>
Gain	21.5	14.3	15.4
Total particulate weight, probe residue and filter, mg	26.9	18.0	20.3
Weight residue from chloroform- ether extract, mg			
Impinger water	36.5	17.5	35.8
Impinger wash water	<u>2.6</u>	<u>2.0</u>	<u>2.4</u>
Total	39.1	19.5	38.2
Weight residue from aqueous phase, mg			
Impinger water	10.5	12.7	47.2
Impinger wash water	<u>3.3</u>	<u>6.7</u>	<u>3.6</u>
Total	13.8	19.4	50.8
Weight residue from acetone wash, mg	2.7	7.3	5.6
Total, back half residue, mg	55.6	36.2	94.6
Total, front half plus back half, mg	82.5	54.2	114.9

(a) Water and acetone blank subtracted; water blank = 0.005 mg/ml,  
acetone blank = 0.011 mg/ml.

PARTICULATE FIELD DATA

Plant Quemetco, Industry, California  
 Date 1/26/72  
 Run No. 1  
 Operator P. R. Webb, H. Leonard  
 Sample Box No. 1  
 Meter Box No. 1  
 Meter  $\Delta H$  @ 1.84  
 Factor 1.0

Ambient Temperature, F 55  
 Barometric Pressure, in. Hg 29.64  
 Assumed Moisture, Percent 5  
 Heater Box Setting, F 255  
 Probe Tip Diameter, Inches 0.235  
 Probe Length 48 in.  
 Probe Heater Setting 5 - 252 F  
 Average  $\Delta P$  — Average  $\Delta H$  —

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H <sub>2</sub> O $\Delta P$	Orifice H, In. H <sub>2</sub> O Desired Actual	Dry Gas Temp., F Inlet Outlet	Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press., In. Hg	Stack Temp., F
10	10:15 am	917.0	0.52	1.6	58 48		240	52	(a)	165
9	10:21	921.9	0.65	1.9	67 50	9	240	57	(a)	170
8	10:27	927.0	0.70	2.0	69 52	9.5	250	69	(a)	180
7	10:33	932.0	0.70	2.0	74 52	9.0	250	72	(a)	175
6	10:39	936.5	0.75	2.2	72 55	9.0	250	70	(a)	175
5	10:45	942.0	0.74	2.2	72 55	9.0	250	70	(a)	175
4	10:51	947.2	0.70	2.0	72 56	9.0	245	62	(a)	175
3	10:57	952.4	0.65	1.9	72 56	9	235	58	(a)	170
2	11:03	957.4	0.50	1.4	73 56	6	235	55	(a)	170
1	11:09	961.9	0.40	1.2	73 58	5.5	245	54	(a)	160
Stop	11:15	965.4	0.7	1.8			255	42	(a)	
Start	11:32	966.4	0.75	2.2	70 58	9.0	255	48	(a)	160
20										
19	11:38	971.8	0.80	2.3	71 58	9	250	49	(a)	160
18	11:44	977.1	0.78	2.2	72 58	9	245	50	(a)	160
17	11:50	981.0	0.80	2.3	70 56	9	235	56	(a)	160
16	11:56	985.0								

(a) Atmospheric.

Comments:

PARTICULATE FIELD DATA

Plant Quemetc, Industry, California

Date 1/26/72

Run No. 1 (Cont'd)

Operator

Sample Box No.

Filter Box No.

Filter  $\Delta H$  @

Factor

Ambient Temperature, F \_\_\_\_\_

Barometric Pressure, in. Hg \_\_\_\_\_

Assumed Moisture, Percent \_\_\_\_\_

Heater Box Setting, F \_\_\_\_\_

Probe Tip Diameter, Inches \_\_\_\_\_

Probe Length \_\_\_\_\_

Probe Heater Setting \_\_\_\_\_

Average  $\Delta P$  \_\_\_\_\_Average  $\Delta H$  \_\_\_\_\_

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H <sub>2</sub> O $\Delta P$	Orifice H, In. H <sub>2</sub> O Desired Actual	Dry Gas Temp., F Inlet Outlet	Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press., In. Hg	Stack Temp., F
15	12:02 pm	991.0	0.67	1.9	70 56	9	235	48	(a)	160
14	12:08	997.0	0.60	1.85	70	56	7	235	49	(a)
13	12:14	1002.0	0.50	1.50	69	56	6	235	49	(a)
12	12:20	1006.0	0.44	1.20	70	58	5	238	48	(a)
11	12:26	1007.0	0.30	0.9	70	58	4	245	48	(a)
End	12:32	1013.8								
TOTAL	120 min	95.8 cf								
AVG.			0.63			1.827	70.2	55.4		
									166	

(a) Atmospheric.

Comments:

PARTICULATE FIELD DATA

Plant Quemetco, Industry, California  
 Date 1/26/72  
 No. 2  
 Operator P.R. Webb, H. Leonard  
 Sample Box No. 1  
 Water Box No. 1  
 Water  $\Delta H @$  1.84  
 Factor 1.0

Ambient Temperature, F 54  
 Barometric Pressure, in. Hg 29.65  
 Assumed Moisture, Percent 5  
 Heater Box Setting, F 255  
 Probe Tip Diameter, Inches 0.235  
 Probe Length 48 in.  
 Probe Heater Setting 5 - 252 F  
 Average  $\Delta P$  — Average  $\Delta H$  —

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H <sub>2</sub> O $\Delta P$	Orifice H, In. H <sub>2</sub> O Desired Actual	Dry Gas Temp., F Inlet Outlet	Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press, In. Hg	Stack Temp., F
20	1:50 pm	014.5	0.65	1.9	68 58	6	225	52	(a)	160
19	1:56	21.0	0.74	2.1	70 56	7	250	56	(a)	160
18	2:02	24.7	0.74	2.1	72 56	7	275	56	(a)	160
17	2:08	29.0	0.84	2.2	72 58	7.4	262	55	(a)	160
16	2:14	36.6	0.88	2.3	72 58	7.4	252	54	(a)	165
15	2:20	41.0	0.72	1.84	75 60	7.4	248	55	(a)	165
14	2:26	47.0	0.63	1.7	76 62	5.5	245	55	(a)	165
13	2:32	50.7	0.58	1.5	74 60	5.0	245	55	(a)	165
12	2:38	55.0	0.48	1.3	75 62	4.8	243	52	(a)	165
11	2:44	59.0	0.40	1.1	75 62	4.0	242	54	(a)	165
Stop	2:50	63.7	—	—	—	—	—	—	—	—
10	3:02 pm	63.8	0.65	1.8	70 58	6	230	50	(a)	165
9	3:08	68.0	0.70	1.84	70 58	6	236	50	(a)	165
8	3:14	74.0	0.74	2.0	69 55	6.5	225	50	(a)	165
7	3:20	79.0	0.72	1.9	72 58	6.5	222	51	(a)	165

Comments:



PARTICULATE FIELD DATA

Plant Quemetcco, Industry, California  
 Date 1/27/72  
 Run No. 3  
 Operator P.R. Webb, H. Leonard  
 Sample Box No. 1  
 Meter Box No. 1  
 Meter  $\Delta H @$  1.84  
 C Factor 1.0

Ambient Temperature, F 54  
 Barometric Pressure, in. Hg 29.90  
 Assumed Moisture, Percent 5  
 Heater Box Setting, F 255  
 Probe Tip Diameter, Inches 0.235  
 Probe Length 48 in.  
 Probe Heater Setting 5 - 252 F  
 Average  $\Delta P$     Average  $\Delta H$    

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H <sub>2</sub> O $\Delta P$	Orifice H, In. H <sub>2</sub> O Desired Actual	Dry Gas Temp., F Inlet Outlet	Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press., in. Hg	Stack Temp., F
10	9:43 am	111.9	0.60	1.5	59 42	6	200	45	(a)	170
9	9:49	116.8	0.68	1.7	68 46	6.2	235	52	(a)	180
8	9:55	122.0	0.70	1.8	70 48	6.5	255	52	(a)	180
7	10:01	125.0	0.72	1.84	74 48	6.5	250	55	(a)	180
6	10:07	130.0	0.75	1.90	74 48	6.5	245	55	(a)	180
5	10:13	135.0	0.68	1.7	78 52	6.5	246	54	(a)	180
4	10:19	139.2	0.65	1.7	80 58	6.5	245	55	(a)	180
3	10:25	143.0	0.55	1.4	82 60	4.8	250	57	(a)	180
2	10:31	148.0	0.40	1.1	83 61	4.2	250	57	(a)	180
1	10:37	152.0	0.32	0.86	82 62	3.5	250	58	(a)	175
End	10:43	155.0								
20	10:55 am	155.0	0.58	1.5	80 64	5.2	255	52	(a)	180
19	11:01	161.0	0.64	1.7	80 64	6	255	53	(a)	180
18	11:07	165.0	0.72	1.84	76 62	6.5	255	55	(a)	180
17	11:13	169.0	0.76	2.0	76 61	7.1	255	55	(a)	175

(a) Atmospheric.

Comments:

## PARTICULATE FIELD DATA

## Quemeticos, California

יובלו רביעי

State 1/27/72 No. 3 (Cont'd)

110.

Plant	Quemetco, Industry, California
Date	1/27/72
Run No.	3 (Cont'd)
operator	
sample Box No.	
meter Box No.	
meter $\Delta H$ @	
Factor	
Ambient Temperature, F	
Barometric Pressure, in. Hg	
Assumed Moisture, Percent	
Heater Box Setting, F	
Probe Tip Diameter, Inches	
Probe Length	
Probe Heater Setting	
Average $\Delta P$	
Average $\Delta H$	

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H <sub>2</sub> O $\Delta$ P	Orifice H, In. H <sub>2</sub> O	Dry Gas Temp., F	Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press., In. Hg	Stack Temp., F
16	11:19 am	174.0	0.76	Desired	Actual	Inlet	Outlet			
15	11:25	179.0	0.66		1.7	76	60	6.2	255	58
14	11:31	184.0	0.62		1.65	70	58	7.0	250	55
13	11:37	189.0	0.46		1.25	70	57	4.5	250	54
12	11:43	193.0	0.44		1.2	68	56	4.2	248	54
11	11:49	197.0	0.30		0.84	68	56	3.2	250	54
End	11:55	199.5								
TOTAL	120 min	87.6 cf								
Avg.			0.599		1.55	74	156			
								175		

### (a) Atmospheric.

### Comments:

GAS SAMPLING FIELD DATADate 1/25/72Plant Quemetco, Industry, Calif.Material Sampled for SO<sub>2</sub>Comments: Gas meter reads 1% low.  
Recovered 70 ml.Barometric Pressure 29.57 in. HgTitration  
Aliquot, Titer,  
ml      ml      0.01N Ba(ClO<sub>4</sub>)<sub>2</sub>Ambient Temperature 61 F1.0      19.40  
1.0      19.20  
Blank      0Run Number 1Power Stat Setting HeatedFilter Used: YES X No   Operator W.C. Baytos, G.W. Keigley

Clock Time	Meter (ft <sup>3</sup> )	Flow Meter Setting (CFH)	Meter Temperature, F
2:25 pm	0.0		67
2:40 pm	0.55	6.0	66
2:45 pm	0.96		66
2:50 pm	1.48		65.5
2:55 pm	2.09	6.0	65.5
3:00 pm	2.49		65.5
3:05 pm	2.94		65.5
3:10 pm	3.93		65.5
3:15 pm	3.93		65.5
3:20 pm	4.93	6.2	65.5
3:25 pm	4.94		65.7
3:30 pm	5.45		65.5
3:35 pm	6.07	6.2	66
3:40 pm	6.60	6.0	66
3:45 pm	7.06		66.5
3:50 pm	7.49		67.0
3:56 pm	8.05	6.0	67.0
4:00 pm	8.34		67.0

GAS SAMPLING FIELD DATADate 1/26/72Material Sampled for SO<sub>2</sub>Barometric Pressure 29.67 in. HgAmbient Temperature 53 FRun Number 2Power Stat Setting HeatedFilter Used: Yes X No   Operator W. C. Baytos, G. W. KeigleyPlant Quemco Inc. Industry, Calif.

Comments:

Recovered 79 ml

Titration

<u>Aliquot</u>	<u>Titer</u>	<u>0.01N Ba(ClO<sub>4</sub>)<sub>2</sub></u>
ml	ml	
1.0	56.90	
1.0	56.70	
blank	0	

<u>Clock Time</u>	<u>Meter (ft<sup>3</sup>)</u>	<u>Flow Meter Setting (CFH)</u>	<u>Meter Temperature, F</u>
9:50 am	0		55
9:55 am	0.38	5.9	54
10:00 am	0.77	5.9	56
10:05 am	1.23	6.1	56
10:10 am	1.79	6.5	56
10:15 am	2.28	6.3	57
10:20 am	2.81	6.3	57
10:25 am	3.33	6.3	58
10:30 am	3.96	6.4	58
10:30 am	4.57	6.3	58
10:42 am	5.20	6.3	59
10:45 am	5.60	6.4	59
10:50 am	6.30		58
10:55 am	6.52	6.1	57
11:00 am	6.95	6.2	57
11:05 am	7.41		58
11:10 am	7.82	6.3	58
11:30 am	8.39	6.1	59
11:40 am	8.88	6.0	58
11:45 am	9.35	6.1	57
11:50 am	9.85		57
11:55 am	10.4	0.0	56
12:00 Noon	10.85	6.0	57
12:05 pm	11.32	6.0	57
12:10 pm	11.80	6.2	58

GAS SAMPLING FIELD DATADate 1/26/72Material Sampled for SO<sub>2</sub>Barometric Pressure 29.63 in. HgAmbient Temperature 54 FRun Number 3Power Stat Setting HeatedFilter Used: Yes X No   Operator W. C. Baytos, G. W. KeigleyPlant Quemetco, Industry, Calif.

Comments: Recovered 94 ml

Titration

Aliquot	Titer	
ml	ml	0.01N Ba(ClO <sub>4</sub> ) <sub>2</sub>
1	47.00	
1	46.90	
Blank	0	

Clock Time	Meter (ft <sup>3</sup> )	Flow Meter Setting (CFH)	Meter Temperature, F
1:15 pm	0	0	57
1:20 pm	0.47	6.0	57
1:25 pm	0.96	6.0	58
1:30 pm	1.46	6.2	58
1:35 pm	1.95	6.1	58
1:40 pm	2.45	6.2	58
1:45 pm	2.95	6.0	58
1:52 pm	3.65	6.1	58
1:55 pm	3.94	6.0	58
2:00 pm	4.45	6.2	58
2:05 pm	4.95	6.2	58
2:10 pm	5.47	6.3	58
2:15 pm	5.97	6.3	58
2:20 pm	6.49	6.2	58
2:25 pm	7.00	6.2	58
2:30 pm	7.59	6.2	58
2:35 pm	8.00	6.2	59
2:40 pm	8.50	6.2	59
2:45 pm	9.00	6.2	59
3:10 pm	9.50	6.1	57
3:15 pm	10.0	6.2	56
3:20 pm	10.5	6.1	57
3:25 pm	11.1	6.1	57
3:30 pm	11.58	6.1	57
3:35 pm	12.06	6.1	57

GAS SAMPLING FIELD DATA

Date 1/27/72 Plant Quemetco Inc. Industry Calif.  
 Material Sampled for SO<sub>2</sub> Comments: Recovered 93 ml  
 Barometric Pressure 29.90 in. Hg Titration  
 Ambient Temperature 54 F Aliquot Titer 0.01N Ba(ClO<sub>4</sub>)<sub>2</sub>  
 Run Number 4 ml ml  
 Power Stat Setting Heated 1.00 51.4  
 Filter Used: Yes X No        1.00 51.9  
 Operator W. C. Baytos, G. W. Keigley Blank 0

Clock Time	Meter (ft <sup>3</sup> )	Flow Meter Setting (CFH)	Meter Temperature, F
09.25 AM	0		51
09:30 AM	0.58	6.1	52
09:35 AM	1.07	6.3	52
09:40 AM	1.55	6.2	52
09:45 AM	2.24	6.2	53
09:50 AM	2.53	6.2	53
09:55 AM	3.02	6.2	54
10:00 AM	3.60	6.2	55
10:05 AM	3.90	6.2	56
10:10 AM	4.40	6.3	56
10:15 AM	4.90	6.3	57
10:20 AM	5.40	6.2	58
10:25 AM	6.00	6.3	58
10:30 AM	6.50	6.4	60
10:35 AM	6.99	6.3	61
11:00 AM	7.68	6.3	64
11:05 AM	8.18	6.2	64
11:10 AM	8.68	6.2	64
11:15 AM	9.25	6.2	64
11:20 AM	9.80	6.2	64
11:25 AM	10.02	6.2	63
11:30 AM	10.69	6.3	63
11:35 AM	11.19	6.3	62
11:40 AM	11.70	6.2	61
11:45 AM	12.20	6.2	61

ORSAT GAS COMPOSITIONPlant Quemetco, Inc. Industry, California

Run	Date	Time	CO <sub>2</sub> , Percent	O <sub>2</sub> , Percent	CO, Percent	N <sub>2</sub> , Percent
1	1/26/72	11:00 AM	2.0	18.5	0	79.5
			1.6	18.8	0	79.6
2	1/26/72	2:30 PM	1.9	18.6	0	79.5
			1.6	18.8	0	79.6
3	1/27/72	10:30 AM	2.0	18.8	0	79.2
			1.8	18.6	0	79.6
			1.7	18.8	0	79.5

**APPENDIX D**

**STANDARD SAMPLING PROCEDURES**

## APPENDIX D

### STANDARD SAMPLING PROCEDURES

The sampling procedures, as described in the December 23, 1972 issue of the Federal Register, reproduced as part of the Appendix, have been used as a reference for the source emission test conducted at Quemetco, Inc., Industry, California.

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  $\Delta p$  and average gas temperature. These data were then used, with the aid of a nomograph, to determine the appropriate nozzle diameter which would allow for 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  $\Delta p$  and temperature variations. Appendix C is a record of the field data for the three tests completed at Quemetco, Inc.

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 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. It must be noted that due to relatively high blank values, emission factors may have been reduced by as much as 10 percent.

**Method 1.** Gas analysis shall be performed by the method(s) prescribed by the manufacturer(s) of such instrument, the instrument shall be subject to manufacturers recommended zero adjustment calibrating procedures at least once per 24-hour operating period unless the manufacturer(s) specified or recommends calibration at shorter intervals. In which case such specifications or recommendations shall be followed. The applicable method specified in the appendix of this part shall be the reference method.

(c) Production rate and hours of operation shall be recorded daily.

(d) The owner or operator of any sulfuric acid production unit subject to the provisions of this subpart shall maintain a file of all measurements required by this subpart. Appropriate measurements shall be reduced to the units of the applicable standard daily and summarized monthly. The record of any such measurement and summary shall be retained for at least 2 years following the date of such measurements and summaries.

#### § 60.85 Test methods and procedures.

(a) The provisions of this section are applicable to performance tests for determining emissions of acid mist and sulfur dioxide from sulfuric acid production units.

(b) All performance tests shall be conducted while the affected facility is operating at or above the maximum acid production rate at which such facility will be operated and under such other relevant conditions as the Administrator shall specify based on representative performance of the affected facility.

(c) Test methods set forth in the appendix to this part or equivalent methods as approved by the Administrator shall be used as follows:

(1) For each repetition the acid mist and SO<sub>2</sub> concentrations shall be determined by using Method 8 and traversing according to Method 1. The minimum sampling time shall be 2 hours, and minimum sampling volume shall be 40 ft<sup>3</sup> corrected to standard conditions.

(2) The volumetric flow rate of the total effluent shall be determined by using Method 2 and traversing according to

2.1.9 When the above sampling site to the nearest upstream and downstream disturbance, determine the corresponding 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 on at least two diameters across the stack. Under no conditions should a sampling point be selected within 1 inch of the stack wall. To obtain the number of traverse points for stacks or ducts with a diameter less than 2 feet, multiply the number of points obtained from Figure 1-1 by 0.67.

2.1.4 To use Figure 1-1 first measure the distance from the chosen sampling location

to the nearest upstream and downstream disturbance. Determine the corresponding number of traverse points for each distance from Figure 1-1. Select the higher of the two numbers of traverse points, or a greater value, such that for circular stacks the number is a multiple of 4, and for rectangular stacks the number follows the criteria of section 2.2.

2.2 Cross-sectional layout and location of traverse points.

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

## D-3 RULES AND REGULATIONS

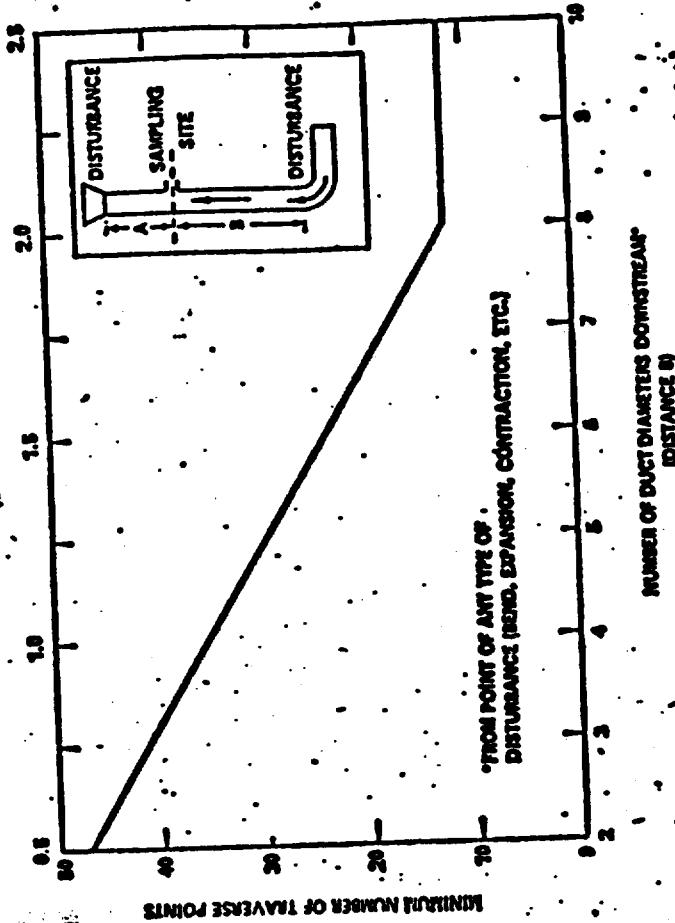


Figure 1-1. Minimum number of traverse points.

$$\text{equation 1-1}$$

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

Traverse point number on a diameter	Number of traverse points on a diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.3	2.5	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.7	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3	75.0	29.5	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5	5.5
4	93.3	70.5	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9	7.9
5	85.3	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5	10.5	10.5
6	95.6	80.6	65.8	35.5	26.9	22.0	18.8	16.5	14.6	13.2	13.2	13.2
7	89.5	77.4	64.5	36.6	28.3	23.6	20.4	18.0	16.1	16.1	16.1	16.1
8	96.7	85.4	65.0	63.4	37.5	29.5	25.0	21.8	19.4	19.4	19.4	19.4
9	91.8	82.3	73.1	62.5	38.2	30.6	26.1	23.0	23.0	23.0	23.0	23.0
10	97.5	88.2	79.9	71.7	61.8	38.8	31.5	27.2	27.2	27.2	27.2	27.2
11	93.3	85.4	78.0	70.4	61.2	39.3	32.3	32.3	32.3	32.3	32.3	32.3
12	97.9	90.1	83.1	76.4	69.4	60.7	49.8	49.8	49.8	49.8	49.8	49.8
13	96.3	87.5	81.2	75.0	68.5	60.2	60.2	60.2	60.2	60.2	60.2	60.2
14	99.2	91.5	85.4	79.6	73.9	67.7	67.7	67.7	67.7	67.7	67.7	67.7
15	95.1	89.1	83.5	78.2	72.8	72.8	72.8	72.8	72.8	72.8	72.8	72.8
16	98.4	92.5	87.1	82.0	77.0	77.0	77.0	77.0	77.0	77.0	77.0	77.0
17	95.6	90.3	85.4	80.6	80.6	80.6	80.6	80.6	80.6	80.6	80.6	80.6
18	98.6	93.3	88.4	83.9	83.9	83.9	83.9	83.9	83.9	83.9	83.9	83.9
19	96.1	91.3	86.8	86.8	86.8	86.8	86.8	86.8	86.8	86.8	86.8	86.8
20	98.7	94.0	89.5	89.5	89.5	89.5	89.5	89.5	89.5	89.5	89.5	89.5
21	96.5	92.1	92.1	92.1	92.1	92.1	92.1	92.1	92.1	92.1	92.1	92.1
22	98.9	94.5	94.5	94.5	94.5	94.5	94.5	94.5	94.5	94.5	94.5	94.5
23	96.8	96.8	96.8	96.8	96.8	96.8	96.8	96.8	96.8	96.8	96.8	96.8
24	98.9	98.9	98.9	98.9	98.9	98.9	98.9	98.9	98.9	98.9	98.9	98.9

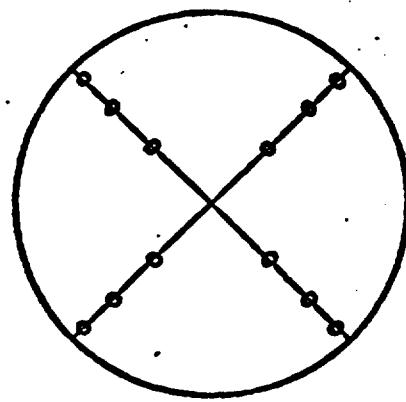


Figure 1-2. Cross section of circular stack divided into 12 equal areas, showing location of traverse points at centroid of each area.

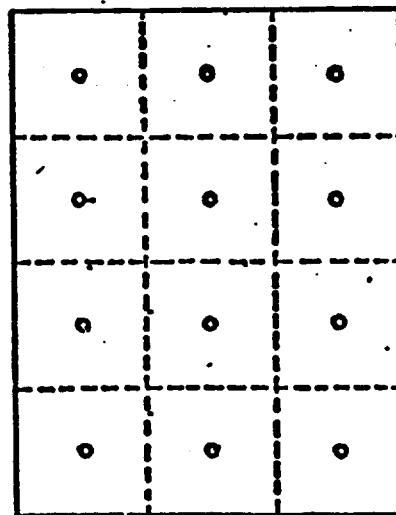


Figure 1-3. Cross section of rectangular stack divided into 12 equal areas, with traverse points at centroid of each area.

## RULES AND REGULATIONS

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

## 3. References.

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

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

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

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

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

## 1. Principle and applicability.

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

1.2 Applicability. This method should be applied only when specified by the test pro-

cedures for determining compliance with the New Source Performance Standards.

## 2. Apparatus.

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

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

2.3 Temperature gauge—Thermocouple or equivalent attached to the pitot tube to measure stack temperature to within 1.6% of the minimum absolute stack temperature.

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

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

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

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

## 3. Procedure.

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

3.2 Measure the static pressure in the stack.

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

## 4. Calibration.

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

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

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

where:

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

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

$\Delta p_{std}$  = Velocity head measured by standard type pitot tube.

$\Delta p_{std}$  = 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.

$$(V_s)_{avg} = K_p C_p \sqrt{\frac{(T_s)_{avg}}{P_s M_s}} \quad \text{Equation 2-2}$$

where:

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

$K_p = 33.43 \frac{\text{ft.}}{\text{sec.}} \left( \frac{\text{lb. mole}^{-1} \text{R}}{\text{lb. mole}} \right)^{1/2}$  when base units are used.

$C_p$  = Pitot tube coefficient, dimensionless.

$(T_s)_{avg}$  = Average absolute stack gas temperature,  $\frac{\text{R.}}{\text{R.}}$

$(\sqrt{\Delta p})_{avg}$  = Average velocity head of stack gas, inches  $H_2O$  (see Fig. 3-2).

$P_s$  = Absolute stack gas pressure, inches Hg.

$M_s$  = Molecular weight of stack gas (wet basis),  $\frac{\text{lb. mole}}{\text{lb. mole}}$ .

$M_d = M_s(1 - B_{wv}) + 18B_{wv}$

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

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

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

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

$$Q_s = 3600 (1 - B_{wv}) V_s A \left( \frac{T_{std}}{(T_s)_{avg}} \right) \left( \frac{P_s}{P_{std}} \right) \quad \text{Equation 2-3}$$

where:

$Q_s$  = Volumetric flow rate, dry basis, standard conditions,  $\text{ft.}^3/\text{hr.}$

$A$  = Cross-sectional area of stack,  $\text{ft.}^2$

$T_{std}$  = Absolute temperature at standard conditions,  $63^{\circ}\text{F.}$

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

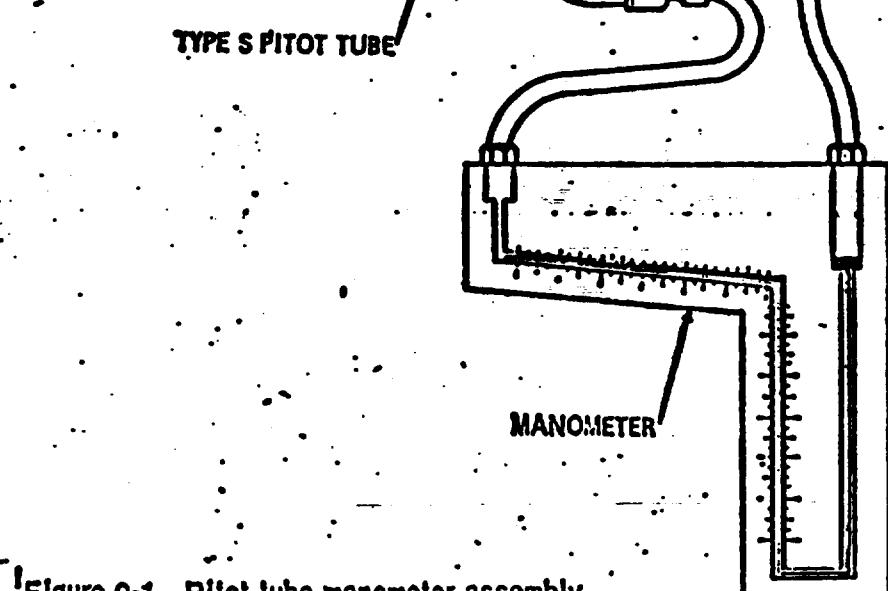


Figure 2-1. Pitot tube-manometer assembly.

## **RULES AND REGULATIONS**

24885.

#### **4. References.**

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

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

Shigebara, R. T., W. F. Todd, and W. S. Smith. Significance of Errors in Stack Sam-

**pling Measurements. Paper presented at the Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14-19, 1970.**

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

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

PLANT \_\_\_\_\_

**DATE** \_\_\_\_\_

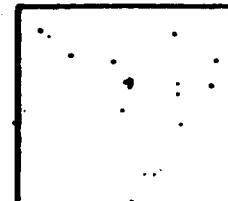
**RUN NO.** \_\_\_\_\_

STACK DIAMETER, in. \_\_\_\_\_

BAROMETRIC PRESSURE, in. Hg. \_\_\_\_\_

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

**OPERATORS** \_\_\_\_\_



## SCHEMATIC OF STACK CROSS SECTION

Figure 2-2. Velocity traverse data.

## RULES AND REGULATIONS

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

## 3.1. Principle and applicability.

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

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

## 3.2 Apparatus.

## 3.2.1 Grab sample (Figure 3-1).

3.2.1.1 Probe—Stainless steel or Pyrex<sup>1</sup> glass, equipped with a filter to remove particulate matter.

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

<sup>1</sup> Trade name.

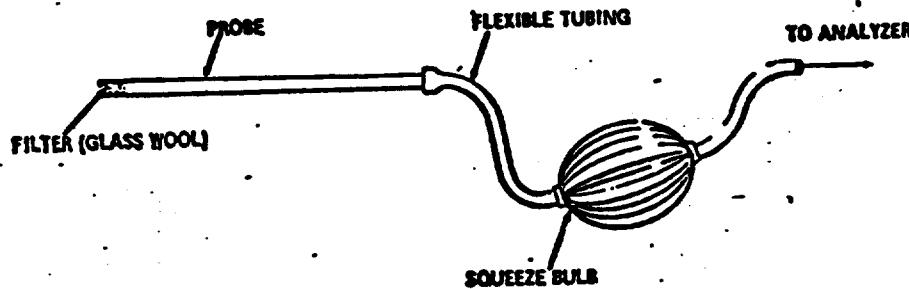


Figure 3-1. Grab-sampling train.

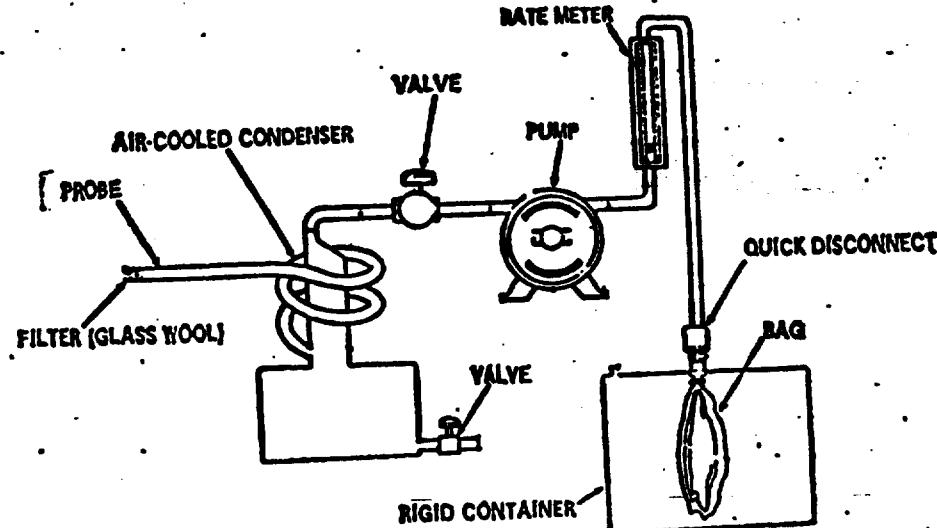


Figure 3-2. Integrated gas-sampling train.

3.2.2 Integrated sample (Figure 3-2).  
 3.2.2.1 Probe—Stainless steel or Pyrex<sup>1</sup> glass, equipped with a filter to remove particulate matter.

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

3.2.2.3 Needle valve—To adjust flow rate.

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

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

3.2.2.6 Flexible bag—Teflon,<sup>2</sup> or equivalent, with a capacity of 2 to 3 cu. ft. Leak test the bag in the laboratory before using.

3.2.2.7 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.

## 3.3 Analysis.

## 3.3.1 Orsat analyzer, or equivalent.

## 3.4 Procedure.

## 3.4.1 Grab sampling.

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

3.4.1.2 Draw sample into the analyzer.

## 3.4.2 Integrated sampling.

3.4.2.1 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.

3.4.2.2 Sample at a rate proportional to the stack velocity.

## 3.4.3 Analysis.

3.4.3.1 Determine the CO<sub>2</sub>, O<sub>2</sub>, and CO concentrations as soon as possible. Make as many passes as are necessary to give constant readings. If more than ten passes are necessary, replace the absorbing solution.

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

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

## 4. Calculations.

4.1 Carbon dioxide. Average the three consecutive runs and report the result to the nearest 0.1% CO<sub>2</sub>.

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

$$\% EA =$$

$$\frac{(\% O_2) - 0.5(\% CO)}{0.204(\% N_2) - (\% O_2) + 0.5(\% CO)} \times 100$$

equation 3-1

where:

% EA = Percent excess air.

% O<sub>2</sub> = Percent oxygen by volume, dry basis.

% N<sub>2</sub> = Percent nitrogen by volume, dry basis.

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

0.204 = 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<sub>d</sub> = Dry molecular weight, lb./lb.-mole.

% CO<sub>2</sub> = Percent carbon dioxide by volume, dry basis.

% O<sub>2</sub> = Percent oxygen by volume, dry basis.

% N<sub>2</sub> = Percent nitrogen by volume, dry basis.

0.44 = Molecular weight of carbon dioxide divided by 100.

0.32 = Molecular weight of oxygen divided by 100.

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



## 4.2 Gas volume.

$$V_{m0} = V_m \left( \frac{P_m}{P_{std}} \right) \left( \frac{T_{std}}{T_m} \right) =$$

$$17.71 \frac{^{\circ}R}{\text{in. Hg}} \left( \frac{V_m P_m}{T_m} \right) \text{ equation 4-2}$$

where:

 $V_m$  = Dry gas volume through meter at standard conditions, cu. ft. $V_{m0}$  = Dry gas volume measured by meter, cu. ft. $P_m$  = Barometric pressure at the dry gas meter, inches Hg. $P_{std}$  = Pressure at standard conditions, 29.92 inches Hg. $T_{std}$  = Absolute temperature at standard conditions, 630° R. $T_m$  = Absolute temperature at meter ( $^{\circ}F + 460$ ), °R.

## 4.3 Moisture content.

$$B_m = \frac{V_{m0}}{V_{m0} + V_{w0}} + B_{w0} = \frac{V_{w0}}{V_{m0} + V_{w0}} + (0.025) \text{ equation 4-3}$$

where:

 $B_{w0}$  = Proportion by volume of water vapor in the gas stream, dimensionless. $V_{w0}$  = Volume of water vapor collected (standard conditions), cu. ft. $V_{m0}$  = Dry gas volume through meter (standard conditions), cu. ft. $B_{w0}$  = 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. 990-AP-40, 1967. Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif., November 1968.

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-0591. Commercial models of this train are available.

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

2.1.2 Probe—Pyrex glass with a heating system capable of maintaining a minimum gas temperature of 250° F. at the exit end during sampling to prevent condensation from occurring. When length limitations (greater than about 8 ft.) are encountered at temperatures less than 600° F., Incoloy 825, or equivalent, may be used. Probes for sampling gas streams at temperatures in excess of 800° F. must have been approved by the Administrator.

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

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

2.1.5 Impingers/Condenser—Four impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a 1/4-inch ID glass tube extending to one-half inch from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip. A condenser may be used in place of the impingers provided that the moisture content of the stack gas can still be determined.

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

2.1.7 Barometer—To measure atmospheric pressure to  $\pm 0.1$  inches Hg.

2.2 Sample recovery.

2.2.1 Probe brush—At least as long as probe.

2.2.2 Glass wash bottles—Two.

2.2.3 Glass sample storage containers.

2.2.4 Graduated cylinder—250 ml.

2.3 Analysis.

2.3.1 Glass weighing dishes.

2.3.2 Desiccator.

2.3.3 Analytical balance—To measure to  $\pm 0.1$  mg.

2.3.4 Trip balance—300 g. capacity, to measure to  $\pm 0.05$  g.

2.4 Reagents.

3.1 Sampling.

3.1.1 Filters—Glass fiber, MSA 1106 BH<sup>1</sup>, or equivalent, numbered for identification and preweighed.

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

3.1.3 Water.

3.1.4 Crushed ice.

3.2 Sample recovery.

3.2.1 Acetone—Reagent grade.

3.2.2 Analysis.

3.2.3 Water.

IMPINGER TRAIN OPTIONAL, MAY BE REPLACED BY AN EQUIVALENT CONDENSER

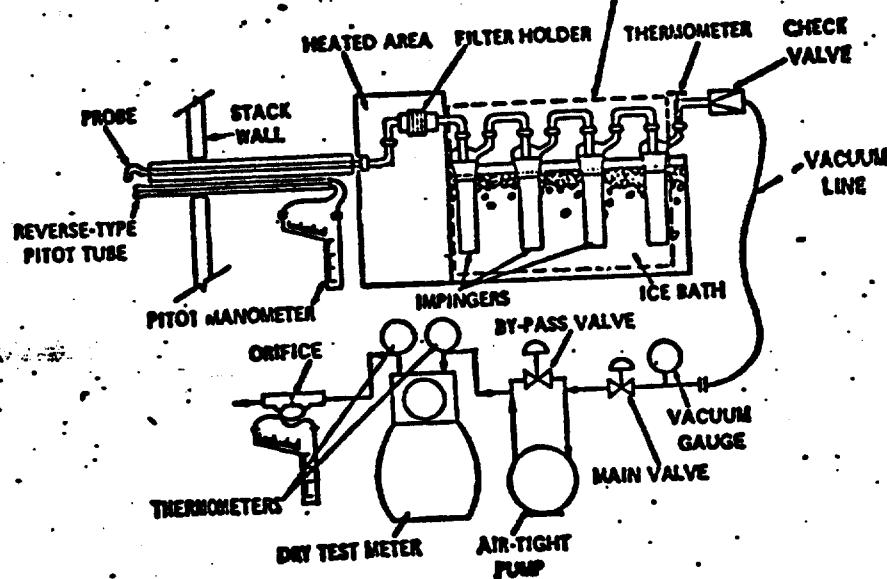


Figure 5-1. Particulate-sampling train.

## 3.2.2 Desiccant—Drierite, 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<sup>2</sup> for at least 24 hours and weigh to the nearest 0.5 mg. in a room where the relative humidity is less than 50%. Place 100 ml. of water in each of the first two impingers, leave the third impinger empty, and place approximately 200 g. of preweighed silica gel in the fourth impinger. Set up the train without the probe as in Figure 5-1. Leak check the sampling train at the sampling site by plugging up the inlet to the filter holder and pulling a 15 in. Hg vacuum. A leakage rate not in excess of 0.02 c.f.m. at a vacuum of 15 in. Hg is acceptable. Attach the probe and adjust the heater to provide a gas temperature of about 250° F. at the probe outlet. Turn on the filter heating system. Place crushed ice around the impingers. Add

more ice during the run to keep the temperature of the gases leaving the last impinger as low as possible and preferably at 70° F. or less. Temperatures above 70° F. may result in damage to the dry gas meter from either moisture condensation or excessive heat.

4.1.3 Particulate train operation. For each run, record the data required on the example sheet shown in Figure 5-2. Take readings at each sampling point, at least every 5 minutes, and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Sample for at least 5 minutes at each traverse point; sampling time must be the same for each point. Maintain isokinetic sampling throughout the sampling period. Nomographs are available which aid in the rapid adjustment of the sampling rate without other computations. APTD-0578 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 6.2.

<sup>1</sup> Trade name.

<sup>2</sup> Dry using Drierite at 70° F.  $\pm 10$ ° F.



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PLANT \_\_\_\_\_  
 DATE \_\_\_\_\_  
 RUN NO. \_\_\_\_\_

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

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

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

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

Figure 5-3. Analytical data.

6.6.2 Concentration in lb./cu. ft.

$$c_0 = \frac{\left( \frac{1}{453,600 \text{ mg.}} \right) M_0}{V_{\text{stack}}} = 2.205 \times 10^{-6} \frac{M_0}{V_{\text{stack}}} \quad \text{equation 5-5}$$

where:  $c_0$  = Concentration of particulate matter in stack gas, lb./cu. ft., dry basis.  
 $453,600 = 31 \text{ g./lb.}$

$M_0$  = Total amount of particulate matter collected, mg.  
 $V_{\text{stack}}$  = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

6.7 Isokinetic variation.

$$I = \frac{T_0 \left[ \frac{V_{\text{stack}} (\rho_{\text{H}_2\text{O}}) R}{M_{\text{H}_2\text{O}}} + \frac{V_{\text{stack}}}{T_0} \left( P_{\text{bar}} + \frac{\Delta H}{13.6} \right) \right]}{\rho V_0 P_0 A_0} \times 100$$

$$= \frac{(1.007 \frac{\text{min.}}{\text{sec.}}) \left[ \left( 0.00207 \frac{\text{in. Hg-cu. ft.}}{\text{ml.}^{\circ}\text{R}} \right) V_{\text{stack}} + \frac{V_{\text{stack}}}{T_0} \left( P_{\text{bar}} + \frac{\Delta H}{13.6} \right) \right]}{\rho V_0 P_0 A_0}$$

Equation 5-6

where:  
 $I$  = Percent of isokinetic sampling.  
 $V_{\text{stack}}$  = Total volume of liquid collected in impingers and silica gel (See Fig. 5-3), ml.  
 $\rho_{\text{H}_2\text{O}}$  = Density of water, 1 g./ml.  
 $R$  = Ideal gas constant, 21.03 inches Hg-cu. ft./lb. mole<sup>o</sup>K.  
 $M_{\text{H}_2\text{O}}$  = Molecular weight of water, 18 lb./lb.-mole.  
 $V_{\text{stack}}$  = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.  
 $T_0$  = Absolute average dry gas meter temperature (see Figure 5-2), °K.  
 $P_{\text{bar}}$  = Barometric pressure at sampling site, inches Hg.  
 $\Delta H$  = Average pressure drop across the orifice (see Fig. 5-2), inches Hg.  
 $T$  = Absolute average stack gas temperature (see Fig. 5-2), °K.  
 $t$  = Total sampling time, min.  
 $V$  = Stack gas velocity, calculated by Method 2, Equation 3-2, ft./sec.  
 $P$  = Absolute stack gas pressure, inches Hg.  
 $A_0$  = Cross-sectional area of nozzle, sq. in.

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

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

7. Reference.

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

Martin, Robert M., Construction Details of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0581.  
 Rom, Jerome J., Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0576.

Smith, W. S., R. T. Shigehara, and W. F. Todd, A Method of Interpreting Stack Sampling Data, Paper presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14-19, 1970.  
 Smith, W. S., et al., Stack Gas Sampling Improved and Simplified with New Equipment, APCA paper No. 67-119, 1967.

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

METHOD 6—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. Principle and applicability.

1.1 Principle. A gas sample is extracted from the sampling point in the stack. The acid mist, including sulfur trioxide, is separated from the sulfur dioxide. The sulfur dioxide fraction is measured by the barium-thorium 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 glass, approximately 5 to 6 mm. ID, with a heating system to prevent condensation and a filtering medium to remove particulate matter including sulfuric acid mist.

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

2.1.3 Glass wool.

2.1.4 Midget impingers—Three.

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

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

2.1.7 Pump—Leak-free, vacuum type.

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

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

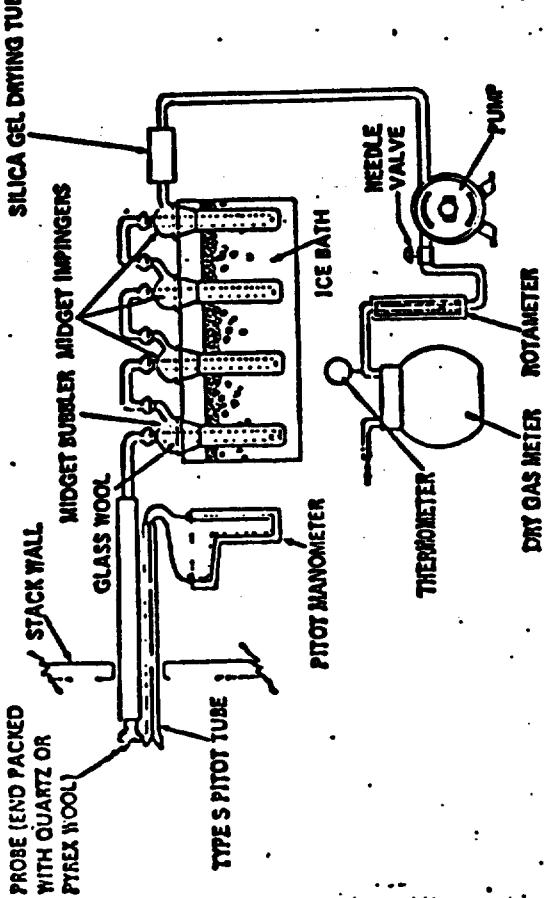
2.1.10 Pitot tube—Type S, or equivalent.

\*Trade names.

## RULES AND REGULATIONS

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

2.2 Sample recovery.

Figure 6-1.  $\text{SO}_2$  sampling train.

2.2.1 Pipettes—Transfer type, 5 ml. and 20 ml. sizes (0.1 ml. divisions) and 25 ml. size (0.2 ml. divisions).

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

2.2.3 Burettes—5 ml. and 50 ml.

2.2.4 Ehrenmeyer flask—125 ml.

## 3. Reagents.

## 3.1 Water—Deionized, distilled.

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

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

## 3.1.3 Water—Deionized, distilled.

3.1.4 Barium perchlorate (0.01 N)—Dissolve 1.95 g. of barium perchlorate ( $\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}$ ) in 200 ml. distilled water.

## 3.2.1 Water—Deionized, distilled.

## 3.2.2 Isopropanol, 80%.

## 3.3 Analysis.

## 3.3.1 Water—Deionized, distilled.

## 3.3.2 Isopropanol.

3.3.3 Thorin Indicator—1-(o-aminophenoxy)-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 ( $\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}$ ) in 200 ml. distilled water.

2.2.1 Glass wash bottles—Two.

2.2.2 Polyethylene storage bottles—to store impinger samples.

2.3 Analysis.

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

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

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

## 5. Calibration.

6.1 Use standard methods and equipment:

6.2 Sulfur dioxide concentration:

$$\text{C}_{\text{SO}_2} = \left( 7.05 \times 10^{-4} \frac{\text{lb.}}{\text{g.-ml.}} \right) \frac{(\text{V}_1 - \text{V}_{\text{so}_2}) \frac{(\text{V}_{\text{so}_2})}{\text{V}_{\text{so}_2}}}{\text{V}_{\text{so}_2}}$$

7.05  $\times 10^{-4}$ —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.

$\text{V}_{\text{so}_2}$ —Volume of barium perchlorate titrant used for the sample, ml.

$\text{V}_1$ —Total solution volume of sulfur dioxide, 50 ml.

$\text{V}_{\text{so}_2}$ —Volume of sample aliquot titrated, ml.

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

6.3 Sample recovery.

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

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

## 6. Calculations.

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

$$\text{V}_{\text{so}_2} = \text{V}_a \left( \frac{\text{T}_{\text{so}_2}}{\text{T}_a} \right) \left( \frac{\text{P}_{\text{so}_2}}{\text{P}_a} \right) -$$

$$17.71 \text{ in. Hg} \left( \frac{\text{V}_{\text{so}_2}}{\text{V}_a} \right) \text{ equation 6-1}$$

where:  $\text{V}_a$ —Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

$\text{T}_{\text{so}_2}$ —Absolute temperature at standard conditions, 530° R.

$\text{T}_a$ —Average dry gas meter temperature, R.

$\text{P}_{\text{so}_2}$ —Barometric pressure at the office meter, inches Hg.

$\text{P}_a$ —Absolute pressure at standard conditions, 29.92 inches Hg.

## 6.2 Sulfur dioxide concentration:

7. References.

Atmospheric Emissions from Sulfuric Acid Manufacturing Processes, U.S. DHEW, PHS, Division of Air Pollution, Public Health Service Publication No. 99-AP-13, Cincinnati, Ohio, 1965.

Corbett, P. F., The Determination of  $\text{SO}_2$  in Flue Gases, Journal of the Institution of Chemical Engineers, 24:237-243, 1961.

Matty, R. E., and E. K. Diehl, Measuring Flue-Gas  $\text{SO}_2$  and  $\text{SO}_3$  Power 101:94-97, November, 1957.

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

7.05  $\times 10^{-4}$ —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.

$\text{V}_{\text{so}_2}$ —Volume of barium perchlorate titrant used for the sample, ml.

$\text{V}_1$ —Total solution volume of sulfur dioxide, 50 ml.

$\text{V}_{\text{so}_2}$ —Volume of sample aliquot titrated, ml.

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

7.05  $\times 10^{-4}$ —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.

$\text{V}_{\text{so}_2}$ —Volume of gas sample through the dry gas meter (standard conditions), cu. ft., see Equation 6-1.

1. Principle and applicability.

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

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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<sup>1</sup> 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<sup>2</sup> round bottom with short neck and 24/40 standard taper opening, protected against implosion or breakage.

<sup>1</sup> Trade name.

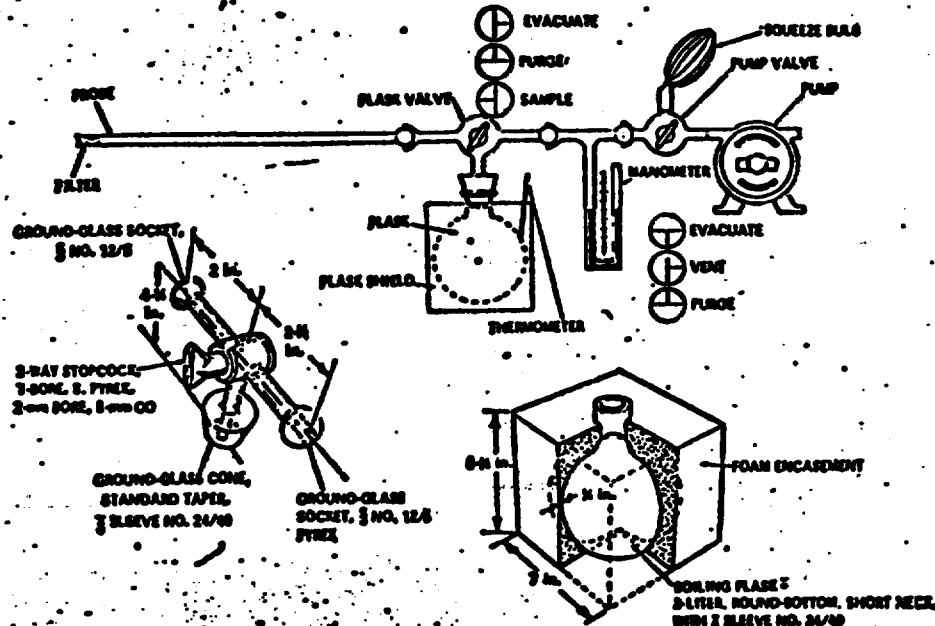


Figure 7-1. Sampling train, flask valve, and flask.

## 2.2.1 Glass wash bottle.

## 2.2 Analysis.

## 2.2.1 Steam bath.

2.2.2 Beakers or casseroles—250 ml., one for each sample and standard (blank).

2.2.3 Volumetric pipettes—1, 2, and 10 ml.

2.2.4 Transfer pipette—10 ml. with 0.1 ml. divisions.

2.2.5 Volumetric flask—100 ml., one for each sample, and 1,000 ml. for the standard (blank).

2.2.6 Spectrophotometer—To measure absorbance at 420 nm.

2.2.7 Graduated cylinder—100 ml. with 1.0 ml. divisions.

2.2.8 Analytical balance—To measure to 0.1 mg.

## 3. Reagents.

## 3.1 Sampling.

3.1.1 Absorbing solution—Add 2.8 ml. of concentrated  $H_2SO_4$  to 1 liter of distilled water. Mix well and add 6 ml. of 3 percent hydrogen peroxide. Prepare a fresh solution weekly and do not expose to extreme heat or direct sunlight.

## 3.2 Sample recovery.

3.2.1 Sodium hydroxide (1N)—Dissolve 40 g. NaOH in distilled water and dilute to 1 liter.

## 3.2.2 Red litmus paper.

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 inches 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 inches 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.

positions. Evacuate the flask to at least 3 inches Hg absolute pressure. Turn the pump valve to its "vent" position and turn off the pump. Check the manometer for any fluctuation in the mercury level. If there is a visible change over the span of one minute, check for leaks. Record the initial volume, temperature, and barometric pressure. Turn the flask valve to its "purge" position, and then do the same with the pump valve. Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and flask valve area, heat the probe and purge until the condensation disappears. Then turn the pump valve to its "vent" position. Turn the flask valve to its "sample" position and allow sample to enter the flask for about 15 seconds. After collecting the sample, turn the flask valve to its "purge" position and disconnect the flask from the sampling train. Shake the flask for 6 minutes.

## 4.2 Sample recovery.

4.2.1 Let the flask set for a minimum of 16 hours and then shake the contents for 2 minutes. Connect the flask to a mercury filled U-tube manometer, open the valve from the flask to the manometer, and record the flask pressure and temperature along with the barometric pressure. Transfer the flask contents to a container for shipment or to a 250 ml. beaker for analysis. Rinse the flask with two portions of distilled water (approximately 10 ml.) and add rinse water to the sample. For a blank use 25 ml. of absorbing solution and the same volume of distilled water as used in rinsing the flask. Prior to shipping or analysis, add sodium hydroxide (1N) dropwise into both the sample and the blank until alkaline to litmus paper (about 25 to 35 drops in each).

## 4.3 Analysis.

4.3.1 If the sample has been shipped in a container, transfer the contents to a 250 ml. beaker using a small amount of distilled water. Evaporate the solution to dryness on a steam bath and then cool. Add 2 ml. phenoldisulfonic acid solution to the dried residue and triturate thoroughly with a glass rod. Make sure the solution contacts all the residue. Add 1 ml. distilled water and four drops of concentrated sulfuric acid. Heat the solution on a steam bath for 3 minutes with occasional stirring. Cool, add 20 ml. distilled water, mix well by stirring, and add concentrated ammonium hydroxide dropwise with constant stirring until alkaline to litmus paper. Transfer the solution to a 100 ml. volumetric flask and wash the beaker three times with 4 to 6 ml. portions of distilled water. Dilute to the mark and mix thoroughly. If the sample contains solids, transfer a portion of the solution to a clean, dry centrifuge tube, and centrifuge, or filter a portion of the solution. Measure the absorbance of each sample at 420 nm, using the blank solution as a zero. Dilute the sample and the blank with a suitable amount of distilled water if absorbance falls outside the range of calibration.

## 5. Calibration.

5.1 Flask volume. Assemble the flask and flask valve and fill with water to the stopcock. Measure the volume of water to  $\pm 10$  ml. Number and record the volume on the flask.

5.2 Spectrophotometer. Add 0.0 to 16.0 ml. of standard solution to a series of beakers. To each beaker add 25 ml. of absorbing solution and add sodium hydroxide (1N) dropwise until alkaline to litmus paper (about 25 to 35 drops). Follow the analysis procedure of section 4.3 to collect enough data to draw a calibration curve of concentration in  $\mu g. NO_2$  per sample versus absorbance.

## 6. Calculations.

## 6.1 Sample volume.

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$$V_{ss} = \frac{T_{std}(V_s - V_0)}{P_{std}} \left( \frac{P_s - P_0}{T_s - T_0} \right) - (17.71 \frac{^{\circ}R}{\text{in. Hg}}) (V_s - 25 \text{ ml.}) \left( \frac{P_s - P_0}{T_s - T_0} \right) \text{ Equation 7-1}$$

where:

$V_{ss}$ —Sample volume at standard conditions (dry basis), ml.

$T_{std}$ —Absolute temperature at standard conditions,  $630^{\circ}$  R.

$P_{std}$ —Pressure at standard conditions, 29.92 inches Hg.

$V_s$ —Volume of flask and valve, ml.

$V_0$ —Volume of absorbing solution, 25 ml.

$$C = \left( \frac{m}{V_{ss}} \right) \left( \frac{1 \text{ lb.}}{\text{cu. ft.}} \right) = \left( 6.2 \times 10^{-1} \frac{\text{lb./s.c.f.}}{\mu\text{g./ml.}} \right) \left( \frac{m}{V_{ss}} \right)$$

equation 7-2

where:

$C$ —Concentration of  $\text{NO}_2$  as  $\text{NO}_2$  (dry basis), lb./s.c.f.

$m$ —Mass of  $\text{NO}_2$  in gas sample,  $\mu\text{g.}$

$V_{ss}$ —Sample volume at standard conditions (dry basis), ml.

### 7. References.

Standard Methods of Chemical Analysis. 6th ed. New York, D. Van Nostrand Co., Inc., 1962, vol. 1, p. 329-330.

Standard Method of Test for Oxides of Nitrogen in Gaseous Combustion Products (Phenoldisulfonic Acid Procedure), In: 1968 Book of ASTM Standards, Part 23, Philadelphia, Pa. 1968, ASTM Designation D-1606-60, p. 725-729.

Jacob, M. B., The Chemical Analysis of Air Pollutants, New York, N.Y., Interscience Publishers, Inc., 1960, vol. 10, p. 351-356.

### METHOD 8—DETERMINATION OF SULFURIC ACID MIST AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

#### 1. Principle and applicability.

1.1 Principle. A gas sample is extracted from a sampling point in the stack and the acid mist including sulfur trioxide is separated from sulfur dioxide. Both fractions are measured separately by the barium-thorin titration method.

1.2 Applicability. This method is applicable to determination of sulfuric acid mist (including sulfur trioxide) and sulfur dioxide from stationary sources only when specified by the test procedures for determining

compliance with the New Source Performance Standards.

#### 2. Apparatus.

2.1 Sampling. See Figure 8-1. Many of the design specifications of this sampling train are described in APTD-0581.

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

2.1.2 Probe—Pyrex<sup>1</sup> glass with a heating system to prevent visible condensation during sampling.

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

2.1.4 Filter holder—Pyrex<sup>1</sup> glass.

2.1.5 Impingers—Four as shown in Figure 8-1. The first and third are of the Greenburg-Smith design with standard tip. The second and fourth are of the Greenburg-Smith design, modified by replacing the standard tip with a  $\frac{1}{2}$ -inch ID glass tube extending to one-half inch from the bottom of the impinger flask. Similar collection systems, which have been approved by the Administrator, may be used.

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

2.1.7 Barometer—To measure atmospheric pressure to  $\pm 0.1$  inch Hg.

<sup>1</sup>Trade name.

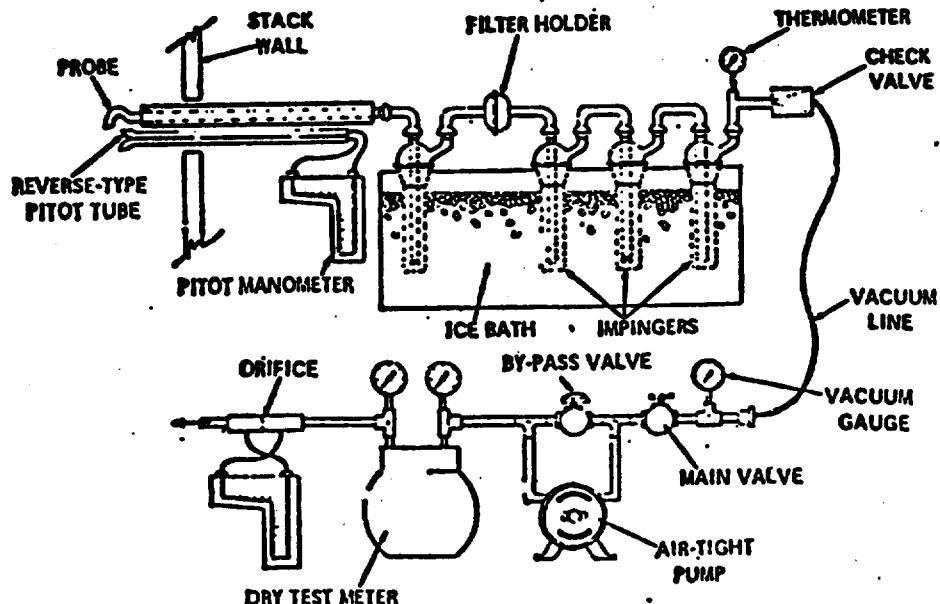


Figure 8-1. Sulfuric acid mist sampling train.

#### 2.2 Sample recovery.

2.2.1 Wash bottles—Two.

2.2.2 Graduated cylinders—250 ml., 500 ml.

2.2.3 Glass sample storage containers.

2.2.4 Graduated cylinder—250 ml.

#### 2.3 Analysis.

2.3.1 Pipette—25 ml., 100 ml.

2.3.2 Burette—50 ml.

2.3.3 Erlenmeyer flask—250 ml.

2.3.4 Graduated cylinder—100 ml.

2.3.5 Trip balance—300 g. capacity, to measure to  $\pm 0.05$  g.

2.3.6 Dropping bottle—to add indicator solution.

#### 2.4 Reagents.

##### 2.4.1 Sampling.

2.4.1.1 Filters—Glass fiber, MSA type 1106 RH, or equivalent, of a suitable size to fit in the filter holder.

2.4.1.2 Silica gel—Indicating type, 6-16 mesh, dried at  $175^{\circ}$  C. (350° F.) for 2 hours.

2.4.1.3 Water—Deionized, distilled.

2.4.1.4 Isopropanol, 80%—Mix 800 ml. of isopropanol with 200 ml. of deionized, distilled water.

2.4.1.5 Hydrogen peroxide, 3%—Dilute 100 ml. of 30% hydrogen peroxide to 1 liter with deionized, distilled water.

2.4.1.6 Crushed ice.

2.4.2 Sample recovery.

2.4.2.1 Water—Deionized, distilled.

2.4.2.2 Isopropanol, 80%.

#### 2.5 Analysis.

2.5.1 Water—Deionized, distilled.

2.5.2 Isopropanol.

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

2.5.4 Barium perchlorate (0.01N)—Dissolve 1.95 g. of barium perchlorate [Ba (CO<sub>3</sub>)<sub>2</sub>·3 H<sub>2</sub>O] in 200 ml. distilled water and dilute to 1 liter with isopropanol. Standardize with sulfuric acid.

2.5.5 Sulfuric acid standard (0.01N)—Purchase or standardize to  $\pm 0.0002$  N against 0.01 N NaOH which has previously been standardized against primary standard potassium acid phthalate.

#### 2.6 Procedure.

##### 2.6.1 Sampling.

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

2.6.1.2 Preparation of collection train. Place 100 ml. of 80% isopropanol in the first impinger, 100 ml. of 3% hydrogen peroxide in both the second and third impingers, and about 200 g. of silica gel in the fourth impinger. Retain a portion of the reagents for use as blank solutions. Assemble the train without the probe as shown in Figure 8-1 with the filter between the first and second impingers. Leak check the sampling train at the sampling site by plugging the inlet to the first impinger and pulling a 15-inch  $\text{Hg}$  vacuum. A leakage rate not in excess of 0.01 c.c.m. at a vacuum of 15 inches Hg is acceptable. Attach the probe and turn on the probe heating system. Adjust the probe heater setting during sampling to prevent any visible condensation. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at  $70^{\circ}$  F. or less.

2.6.1.3 Train operation. For each run, record the data required on the example sheet shown in Figure 8-2. 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. Start the pump and immediately adjust the flow to isokinetic conditions. Maintain isokinetic sampling throughout the sampling period. Nomographs are available which aid in the



## RULES AND REGULATIONS

24895

Rom, Jerome J., Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment, Environmental Protection Agency, Air Pollution Control Office Publication No. APTD-0576.

Shell Development Co. Analytical Department, Determination of Sulfur Dioxide and Sulfur Trioxide in Stack Gases, Emeryville Method Series, 4316/50A.

**METHOD 9—VISUAL DETERMINATION OF THE OPACITY OF EMISSIONS FROM STATIONARY SOURCES**

**1. Principle and applicability.**

1.1 Principle. The relative opacity of an emission from a stationary source is determined visually by a qualified observer.

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

**2. Procedure.**

2.1 The qualified observer stands at approximately two stack heights, but not more than a quarter of a mile from the base of the stack with the sun to his back. From a vantage point perpendicular to the plume, the observer studies the point of greatest opacity in the plume. The data required in

Figure 9-1 is recorded every 15 to 30 seconds to the nearest 5% opacity. A minimum of 25 readings is taken.

**3. Qualifications.**

3.1 To certify as an observer, a candidate must complete a smokereading course conducted by EPA, or equivalent; in order to certify the candidate must assign opacity readings in 5% increments to 25 different black plumes and 25 different white plumes, with an error not to exceed 15 percent on any one reading and an average error not to exceed 7.5 percent in each category. The smoke generator used to qualify the observers must be equipped with a calibrated smoke indicator or light transmission meter located in the source stack if the smoke generator is to determine the actual opacity of the emissions. All qualified observers must pass this test every 6 months in order to remain certified.

**4. Calculations.**

4.1 Determine the average opacity.

**5. References.**

Air Pollution Control District Rules and Regulations, Los Angeles County Air Pollution Control District, Chapter 2, Schedule 6, Regulation 4, Prohibition, Rule 50, 17 p.

Kudluk, Rudolf, Ringelmann Smoke Chart, U.S. Department of Interior, Bureau of Mines, Information Circular No. 8333, May 1967.

0	15	30	45	0	15	30	45
00				00			
01				01			
02				02			
03				03			
04				04			
05				05			
06				06			
07				07			
08				08			
09				09			
10				10			
11				11			
12				12			
13				13			
14				14			
15				15			
16				16			
17				17			
18				18			
19				19			
20				20			
21				21			
22				22			
23				23			
24				24			
25				25			
26				26			
27				27			
28				28			
29				29			
30				30			

Observation data

Plant \_\_\_\_\_

Stack location \_\_\_\_\_

Observer \_\_\_\_\_

Date \_\_\_\_\_

Time \_\_\_\_\_

Distance to stack \_\_\_\_\_

Wind direction \_\_\_\_\_

Wind speed \_\_\_\_\_

Sum of numbers recorded \_\_\_\_\_

Total number of readings \_\_\_\_\_

Calculator:  $\frac{\text{Sum of no. recorded}}{\text{Total no. readings}}$  \_\_\_\_\_

Figure 9-1. Field data.  
[FR Doc. 71-18024 Filed 12-22-71; 8:46 am]

**APPENDIX E**

**LABORATORY REPORT**

APPENDIX E

LABORATORY REPORT

Plant Quemetco, Industry, California

Date January 26, 27, 1972

BACK HALF LABORATORY REPORT  
ANALYSIS BY ATOMIC ABSORPTION

Run	Back Half Impinger Water Residue	Pb, $\mu$ g Residue, $\mu$ g	Pb, $\mu$ g Total Residue
1	Aqueous residue, $\mu$ g 10,500(a)	0.039	410
	Organic residue, $\mu$ g 36,500	0.006	230

(a) Water blank subtracted; blank = 0.005 mg/ml.

LABORATORY REPORT  
ANALYSIS BY WET CHEMISTRY

Run No. 1

Plant Quemetco, Industry, California

Date January 26, 27, 1972

BACK HALF IMPINGER WATER RESIDUE

Constituents	Aqueous, μg	Organic, μg	Total, μg
SO <sub>4</sub>	4,400	17,800	22,200
Cl	30	<30	60
NH <sub>4</sub>	140	80	220
NO <sub>3</sub>	220	<10	230
pH (a)	2.9	2.3	---
Total Residue Mass 10,500(b) for which anions and pH were determined		36,500	47,000

(a) Values are pH units.

(b) Water blank subtracted; blank = 0.005 mg/ml.

Plant Quemetco, Industry, California  
 Date January 26, 27, 1972

**ACETONE PROBE WASH RESIDUE**

Constituent	Run 1, μg	Run 2, μg	Run 3, μg
SO <sub>4</sub>	< 500	< 500	< 500
Cl	580	330	440
NH <sub>4</sub>	< 100	< 100	< 100
NO <sub>3</sub>	< 100	< 100	< 100
pH <sup>(a)</sup>	5.0	5.2	5.1
Total Residue	5400	3700	4900
Mass from which anions and pH were determined <sup>(b)</sup>			

(a) Values are pH units.

(b) Acetone blank subtracted; blank = 0.011 mg/ml.

Note: Symbol < indicates minimal detection limit.

Plant Quemetco, Industry, California

Date January 26, 27, 1972

FRONT HALF LABORATORY REPORT  
ANALYSIS BY ATOMIC ABSORPTION

Run	Probe Acetone Wash Residue, $\mu\text{g}$ (a)	Pb, $\mu\text{g}$ per Residue, $\mu\text{g}$	Pb, $\mu\text{g}$ per Total Residue $\mu\text{g}$	Filter Catch, $\mu\text{g}$	Pb, $\mu\text{g}$ per Split Filter Sample, $\mu\text{g}$	Pb, $\mu\text{g}$ per Total Filter Catch $\mu\text{g}$
1	5400	0.43	2340	21500	0.15	3300
2	3700	0.21	760	14300	0.16	2350
3	4900	0.11	560	15400	0.13	2050

(a) Acetone blank subtracted; blank = 0.011 mg/ml.

Note: See analysis flow chart for procedure (E-8).

Plant Quemetco, Industry, California

EXAMPLE LEAD DETERMINATION FILTER, RUN 1

---

A - Initial Extract Volume per Split Filter Sample, ml	5.0
B - Aliquot, ml	1.0
C - Dilution Factor	25
D - Absorption, Percent	33
E - Absorbance from Atmoc Absorbance Tables, Based on Percent Absorption	0.1739
F - Lead, $\mu\text{g}/\text{ml}$ of Diluted Aliquot <sup>(a)</sup>	13.2
G - Lead, $\mu\text{g}/\text{Split Filter Sample}$	1650
H - Lead, $\mu\text{g}/\text{Total Filter}$	3300

---

Example Computation

$$C \times A \times F = G$$

$$25 \times 25 \times 13.2 = 1650$$

$$2 \times G = H$$

$$2 \times 1650 = 3300$$

---

(a) Determined from calibration curve; based on 0.1739 absorbance.

Plant Quemetco, Industry, CaliforniaLABORATORY REPORTOPTICAL EMISSION SPECTROSCOPY TRACE METAL  
(ug of element for total sample)

Element	Probe Wash Residue			Impinger Water Residue	
	Run 1	Run 2	Run 3	aqueous	organic
Be	<0.1	<0.1	<0.1	<0.1	<0.1
Cd	<5.	<5.	10.	<5.	<5.
Ag	<10.	<10.	<10.	<10.	<10.
V	<1.	<1.	<1.	<1.	<1.
Mn	<1.	<1.	<1.	1.	<1.
Ni	<1.	<1.	1.	<1.	<1.
Sb	20.	10.	100.	200.	10.
Cr	<1.	<1.	<1.	<1.	<1.
Zn	30.	<10.	20.	30.	<10.
Cu	1.	<1.	2.	<1.	<1.
Pb <sup>(a)</sup>	--	--	--	--	--
Se <sup>(b)</sup>	--	--	--	--	--
B	1.	<1.	<1.	<1.	<1.
F <sup>(b)</sup>	--	--	--	--	--
Li	<30.	<30.	<30.	<30.	<30.
Ag	<0.1	<0.1	0.3	<0.1	<0.1
Sn	10.	5.	30.	5.	30.
Fe	10.	2.	40.	30.	3.
Sr	<5.	<5.	<5.	<5.	<5.
Na	100.	30.	30.	30.	<30.
K	20.	<10.	10.	10.	<10.
Ca	60.	2.	10.	50.	1.
Si	10.	10.	30.	30.	10.
Mg	20.	1.	3.	20.	1.
Ba	1.	<1.	1.	10.	<1.
Al	10.	1.	2.	10.	3.
Total Catch, ug	5,400	3,700	4,900	10,500	36,500

(a) Pb determined by Atomic Absorption.

(b) Not determined by methods used.

Plant Quemetco, Industry, CaliforniaLABORATORY REPORTOPTICAL EMISSION SPECTROSCOPY TRACE METAL  
( $\mu$ g of element for total filter catch)

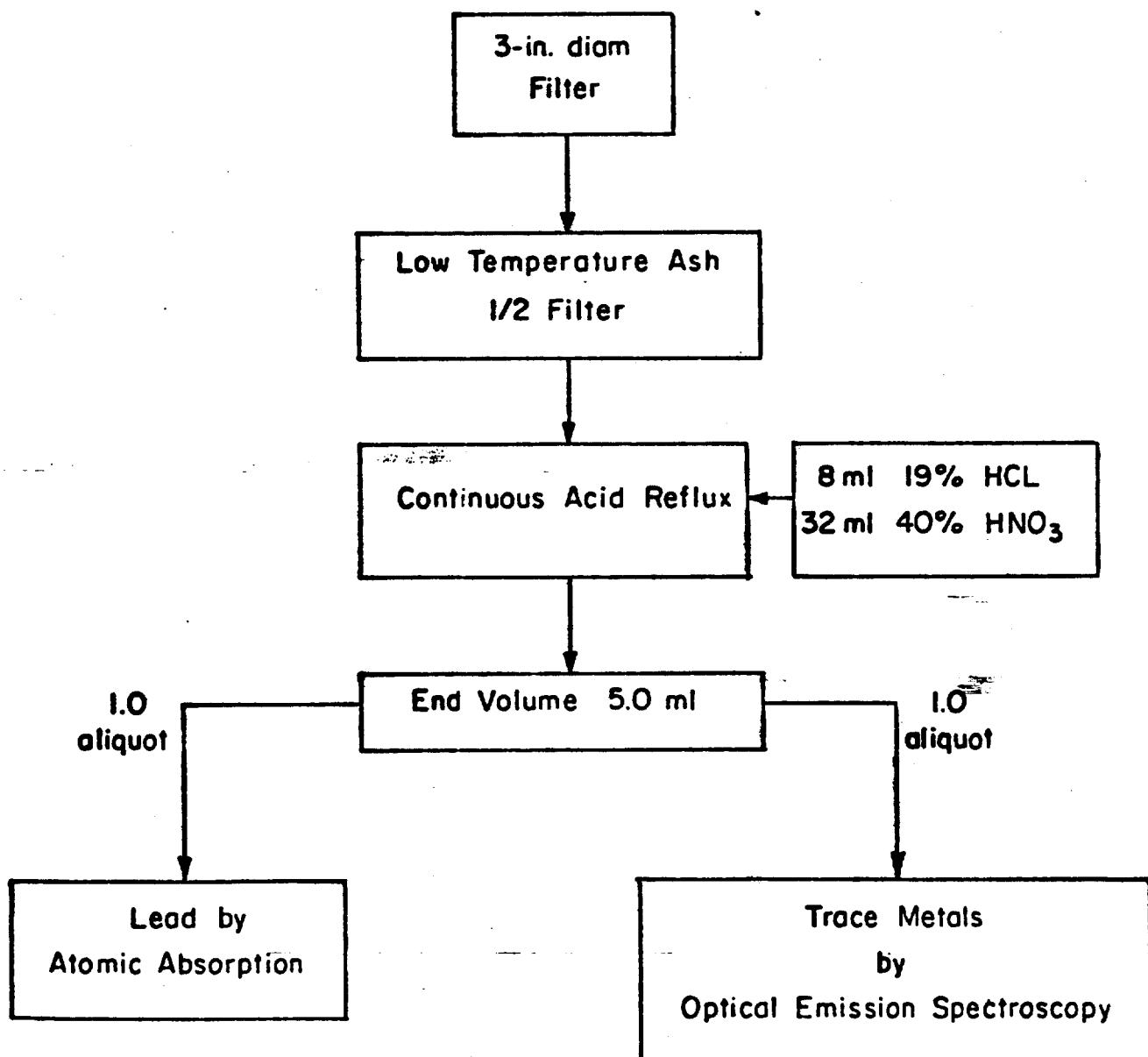
Element	Filter Analysis		
	Run 1	Run 2	Run 3
Fe	100	200	200
Zn	< 20	< 20	< 20
Pb(a)	---	---	---
Cu	4	< 2	< 2
Se	---	---	---
Sn	140	180	30
Ti	< 2	2	< 2
V	< 2	< 2	< 2
Cr	< 2	< 2	< 2
Co	< 6	< 6	< 6
Ni	< 6	6	< 6
Mo	< 2	< 2	< 2
Ba	---	---	---
As	60	100	100
Bi	< 6	< 6	< 6
Cd	40	40	40
Sr	< 0.2	< 0.2	< 0.2
Be	< 60	< 60	< 60
Hg	60	120	120
Total Catch, $\mu$ g	21,500	14,300	15,400

(a) Pb determined by Atomic Absorption.

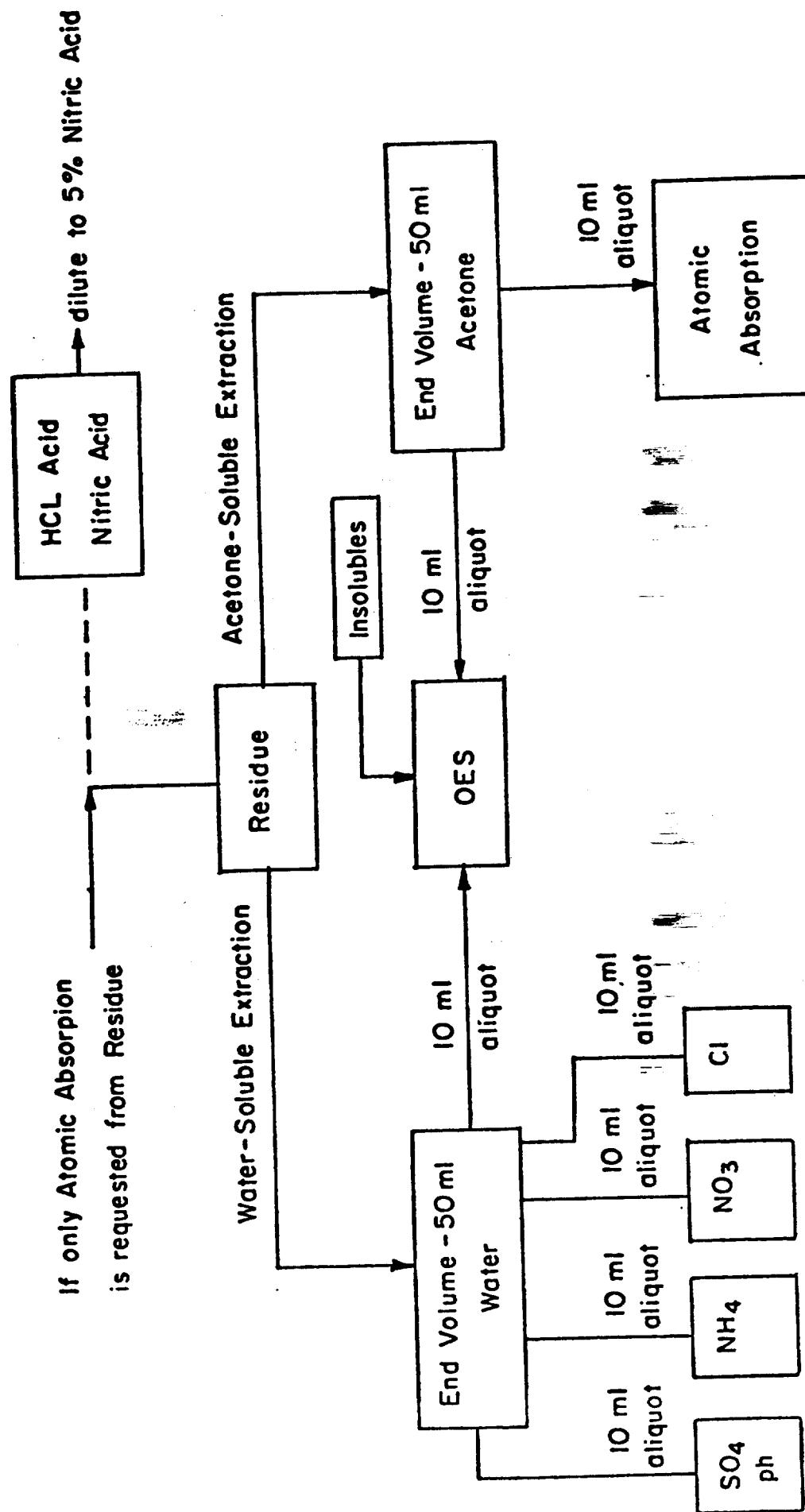
Note: Filter blank data not available.

Symbol &lt; indicates minimal detection limits.

## 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<sub>4</sub>

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<sub>2</sub> 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<sub>4</sub> 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<sub>4</sub>. Calculate SO<sub>4</sub> by multiplying by the factor 0.4116. Multiply this result by 5 to get total amount of SO<sub>4</sub> in the residue.

DISCUSSION OF METHODS

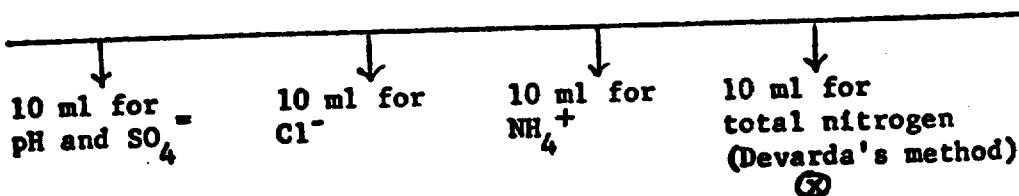
The methods used for the chemical analysis of residues were chosen because they represent reliable procedures used almost routinely in

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 representative 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

$\textcircled{X}$   $\text{NO}_3^-$  will be calculated from the difference between total nitrogen and  $\text{NH}_4^+$

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  $\sim 100$  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  $\text{SO}_4$ ,  $\text{Cl}$ ,  $\text{NH}_4$ , and  $\text{NO}_3$  on the entire filter circle.

**APPENDIX F**

**TEST LOG**

## APPENDIX F

### TEST LOG

January 20, 1972      Ship equipment.

January 24, 1972      Pick up equipment.  
Arrive at test site.

January 25, 1972      Set up equipment,  
SO<sub>2</sub> Run 1.

January 26, 1972      SO<sub>2</sub> Runs 2 and 3.  
Particulates Runs 1 and 2.

January 27, 1972      SO<sub>2</sub> Run 4.  
Particulate Run 3.  
Pack equipment.

January 28, 1972      Ship equipment to next  
test site.

**APPENDIX G**

**RELATED REPORTS**

## APPENDIX G

### RELATED REPORTS

- (1) SECONDARY LEAD PLANT STACK EMISSION SAMPLING AT N. L. INDUSTRIES PLANT, BEECH GROVE, INDIANA
- (2) SECONDARY LEAD PLANT STACK EMISSION SAMPLING AT N. L. INDUSTRIES PLANT, MCCOOK, ILLINOIS
- (3) SECONDARY LEAD PLANT STACK EMISSION SAMPLING AT REVERE SMELTING AND REFINING PLANT, NEWARK, NEW JERSEY
- (4) SECONDARY LEAD PLANT STACK EMISSION SAMPLING AT GENERAL BATTERY CORPORATION, READING, PENNSYLVANIA

**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  
Harold G. Leonard, Senior Technician  
G. William Keigley, Senior Technician  
William C. Baytos, Scientist

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 No.	1	2	3
Start time	10:15 am	1:50 pm	9:43 am
End Time	12:32 pm	4:02 pm	11:55 am
Baghouse temperature, F	151	156	168
Feed, lb	2,000	5,000	6,000
Slag tap, lb	4,600	6,000	5,500
Lead tap, lb	10,000	7,000	0
Slag tap, lb in 24 hr	28,500	28,500	28,500
Lead tap, lb in 24 hr	100,000	100,000	100,000
Lead rate, lb/hr	4,200	4,200	4,200

OPERATIONAL NOTES

Quemetco, Industry, California  
 Tuesday, January 25, 1972

<u>Baghouse Temperatures</u>	
<u>Section</u>	<u>Temperature, F</u>
1	180
2	140
3	180
4	205
5	235
6	210
7	170

2:15 pm Start observing furnace  
 2:18 Load furnace  
 2:25 Load furnace  
 2:30 Complete a lead tap - about 5 tons  
     Mold contains a place for 5,200-lb ingots  
     Same mold used for all taps.  
 2:40 Start filling lead mold  
     Operator reports 5 tons had been tapped before 2:15  
 2:45 Load furnace  
 2:50 Another 5 tons of lead tapped  
 3:15 - 3:30 Tap 5 tons lead  
 3:30 Baghouse temperatures by sections, F:  
     190, 140, 180, 200, 235, 205, 180  
     Furnace valve setting 3 - Air pressure to furnace,  
     10 oz/in<sup>2</sup>  
 4:00 Furnace off for shift change  
     Stop observations

Wednesday, January 26, 1972

Operator reports each night shift.  
 Cast 15 slag buttons and 20 lead hogs.  
 Total for night, 15,000 lb slag, 180,000  
 lb lead. Baghouse temperatures, F:  
 150, 170, 190, 180, 160, 130, 160.

## OPERATIONAL NOTES (Cont'd)

January 26, 1972 (Cont'd)

9:00 am Furnace air setting - 7, 21-oz air pressure  
Load furnace

9:28 Load furnace

9:30 Load furnace

9:35 Furnace setting - 6, 19-oz air pressure

9:45 Load furnace

9:50 Load furnace

10:00 Load furnace  
Plant manager reports 6 min cycle on bag shaking will be changed to a 20 min or 90 min cycle

10:05 Furnace air pressure, 19 oz

10:10 Baghouse temperature by sections - F, 140, 170, 200, 170, 140, 170

10:15-  
10:20 Load furnace

10:50 Preparing to tap slag

11:05 Pressure to gas burners reduced 13 oz on left side, 8 oz on right

11:06 Increase gas burner air pressure 18 oz both sides  
500-lb button slag tapped (No. 1)

11:10 500-lb button slag tapped (No. 2)

11:15 500-lb button slag tapped (No. 3)

11:18 500 lb button slag tapped (No. 4)

11:21 500-lb button slag tapped (No. 5)

11:23 Gas burners off

11:24 500-lb button slag tapped (No. 6)

11:26 500-lb button slag tapped (No. 7)

11:28 Gas burners on, 4 oz air pressure

11:30 500-lb button slag tapped (No. 8)

11:38 Gas burners, 15 oz air pressure

11:44 500-lb button slag tapped (No. 9)

11:45 Gas burners, 20 oz air pressure

11:50 Finish tapping slag, about 100 lb in button No. 10

11:57-  
12:37 Tap 5 tons lead

12:25 Load furnace

## OPERATIONAL NOTES (Cont'd)

January 26, 1972 (Cont'd)

12:25 pm One gas burner off, other 12 oz air pressure  
12:32 Both gas burners on, 20 oz air pressure  
12:34 Load furnace  
12:42 Load furnace  
12:50 Load furnace  
12:55 Load furnace  
1:03 Load furnace  
Baghouse temperatures - 80, 150, 190, 180, 160, 140, 160  
Baghouse shakers off to change timer  
1:15 Load furnace  
1:17 Gas burners, 15 oz one side, 7 oz other  
1:38-  
1:53 Tap 3,000 lb lead  
1:45 Load furnace  
1:50 Gas burners, 17 oz both sides  
1:51 Load furnace  
2:00 Foreman reports furnace was too reducing which means  
antimony is staying in the metal. Extra oxides are  
added to furnace to correct  
2:05 Load furnace  
2:10 Load furnace  
2:20 Load furnace  
2:30 Load furnace  
Baghouse shakers on, new cycle  
2:40 Load furnace  
2:45-  
2:55 Blow air in furnace  
3:00 Start tapping slag  
3:04 Button 1 tapped  
3:08 Button 2 tapped  
3:11 Button 3 tapped  
3:13 Button 4 tapped  
3:15 Button 5 tapped  
3:18 Button 6 tapped  
3:20 Button 7 tapped  
3:21 Button 8 tapped  
3:22 Button 9 tapped

## OPERATIONAL NOTES (Cont'd)

January 26, 1972 (Cont'd)

3:24      Button 10 tapped  
3:26      Button 11 tapped  
3:27      Button 12 tapped - end of tap  
            Gas burners off  
3:34      Gas burners on, air pressure 15 oz  
3:35-  
3:54      Tap 7000 lb lead  
3:57      Gas burner air pressure, 19 oz  
4:00      Baghouse temperatures - F, 150, 160, 180, 170, 150, 130, 150

Thursday, January 27, 1972

8:00 am    Gas burner off to remove dust from air cooling system  
            Remove 500 lb from air cooler (estimate)  
            Remove 1500 lb from hopper under front leg of inverted V-ducts (estimate)  
            Remove 2000 lb from cyclone (estimate)  
            Remove 100 lb from hopper at baghouse inlet (estimate)  
            Remove 2500 lb from hopper under second leg of inverted V  
            Estimate remove 600 lb from baghouse  
            Except for baghouse dust, these materials are charged into furnace as the ductwork is cleaned.

8:30      Cleaning completed, operator reports ducts are cleaned at the beginning of each shift  
            Gas on-air pressure, 20 oz

8:45      Slag stirred into furnace

8:50      Load furnace

8:55      Load furnace

9:00      Blow air into metal through slag tap  
            Remove 600 lb dust from baghouse

9:10      Load furnace

9:20      Load furnace  
            Baghouse temperatures by sections - F, 140, 180, 205, 180, 160, 160, 160

9:25      Load furnace

9:30      Load furnace

9:32      Load furnace  
            Air pressure to gas burner 21 oz

9:35      Load furnace

9:38      Load furnace

## OPERATIONAL NOTES (Cont'd)

January 27, 1972 (Cont'd)

9:38 Load furnace  
 9:43 Air pressure to gas burners  
     12 oz - right side, 18 oz - left side  
 10:00 Load furnace  
     Air pressure to gas burners, 20 oz  
 10:10 Load furnace  
 10:15 Load furnace  
 10:22 Load furnace  
 10:25 Load furnace  
 10:30 Load furnace  
 10:35 Air pressure to gas burners, 20 oz on left - 7 oz on right  
 10:38-  
 10:40 Blow air into bath  
 11:05 Start tapping, slag buttons filled at 11:09, 11:10, 11:18,  
     11:21, 11:24, 11:26, 11:28, 11:31, 11:39, 11:42, 11:54  
     Total - Eleven buttons  
 11:15 Air pressure to gas burners, 20 oz one side, 18 oz other  
 11:54 End of observations

Production record from managers office.

	<u>Shift</u>	<u>Lead Hogs</u>	<u>Slag Buttons</u>
Wednesday	12-8	20	15
	8-4	10	21
	4-12	20	21
Thursday	12-8	20	15

**APPENDIX J**

**SUMMARY OF RESULTS**

## APPENDIX J

SUMMARY OF RESULTS

Run Number	1	2	3	Average
Date	1/26/72	1/26/72	1/26/72	
Test Time - Minutes	120	120	120	120
Lead Production - TPH	2.1	2.1	2.1	2.1
<u>Stack Effluent</u>				
Flow rate - DSCFM	14,600	15,200	14,200	14,667
Flow rate - DSCF/ton	6,952	7,238	6,762	6,984
Temperature - °F	166	164	175	168
Water vapor - Vol. %	3.1	2.8	3.1	3.0
CO <sub>2</sub> - Vol. % dry	1.8	1.8	1.8	1.8
O <sub>2</sub> - Vol. % dry	18.6	18.7	18.7	18.7
CO - Vol. % dry	<0.1	<0.1	<0.1	<0.1
CO - lb/hr	63.5	66.0	61.7	63.7
SO <sub>2</sub> - ppm dry	1580	1525	1618	1574
SO <sub>2</sub> - lb/hr	229	230	228	229
Visible emissions - % opacity	0	0	0	0
<u>Particulate Emissions</u>				
<u>Probe and filter catch</u>				
gr/DSCF	0.0043	0.0028	0.0035	0.0035
gr/ACF	0.0035	0.0023	0.0028	0.0029
lb/hr	0.5387	0.3702	0.4297	0.4462
lb/ton lead	0.2565	0.1763	0.2046	0.2125
<u>Total Catch</u>				
gr/DSCF	0.0132	0.0086	0.0200	0.0139
gr/ACF	0.0107	0.0070	0.0161	0.0113
lb/hr	1.6520	1.1148	2.4321	1.7330
lb/ton lead	0.7867	0.5309	1.1581	0.8252
<u>Lead Emissions</u>				
<u>Probe and filter catch</u>				
gr/DSCF	0.00090	0.00049	0.00045	0.00061
gr/ACF	0.00073	0.00040	0.00037	0.00050
lb/hr	0.1130	0.0640	0.0553	0.0775
lb/ton lead	0.0538	0.0305	0.0263	0.0369

Total Catch

Essentially the same as probe and filter.