SECONDARY LEAD
SMELTING
AP-42 Section 7.11
Reference Number
9

RESEARCH REPORT

Battelle
Columbus Laboratories

72-CI-8

RESEARCH REPORT

on

SECONDARY LEAD PLANT STACK EMISSION SAMPLING AT N. L. INDUSTRIES PLANT, McCOOK, ILLINOIS

Contract No. 68-02-0230
Task Order No. 1

to

ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF AIR PROGRAMS

July 19, 1972

by

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July 25, 1972

Mr. Tom Logan
Emission Testing Branch
Office of Air Programs
Environmental Protection Agency
Mutual Plaza Building
Durham, North Carolina 27711

Dear Tom:

Secondary Lead Plant Stack Emission Sampling
Contract No. 68-02-0230
Task Order No 1

Enclosed are 15 copies of the Research Report on "Secondary Lead Plant Stack Emission Sampling at N.L. Industries Plant, McCook, Illinois". The samples were obtained at this plant on February 9 and 10, 1972.

Yours very truly,

Richard B Engdahl

Richard B. Engdahl
Environmental Systems and
Processes Section

RBE:iw

Encs. (15)

cc: M.P. Hunneycutt (w/enc)
Contracting Officer

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BATTELLE
Columbus Laboratories

July 17, 1972

INTRODUCTION

Gas and particulate emission measurements were made at N. L. Industries secondary lead plant in McCook, Illinois, on February 9 and 10, 1972. 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 reverberatory furnace was sampled in the duct from the process baghouse outlet after particulate removal by three water-cooled cyclones and a baghouse in series. Particulate and SO_2 emissions were determined and Orsat analyses were made for the three runs. The effluent from a second baghouse, which filters the air from hoods over various lead kettles, the furnace feed hopper, and the furnace slag tap, was not sampled.

The reverberatory furnace is used to melt down manufacturers' scrap and worn-out batteries. The furnace is fed almost continuously. Figure 1 is a sketch showing the gas flow through the plant. Particulate and SO_2 samples were obtained from the duct between the baghouse and the exhaust blower.

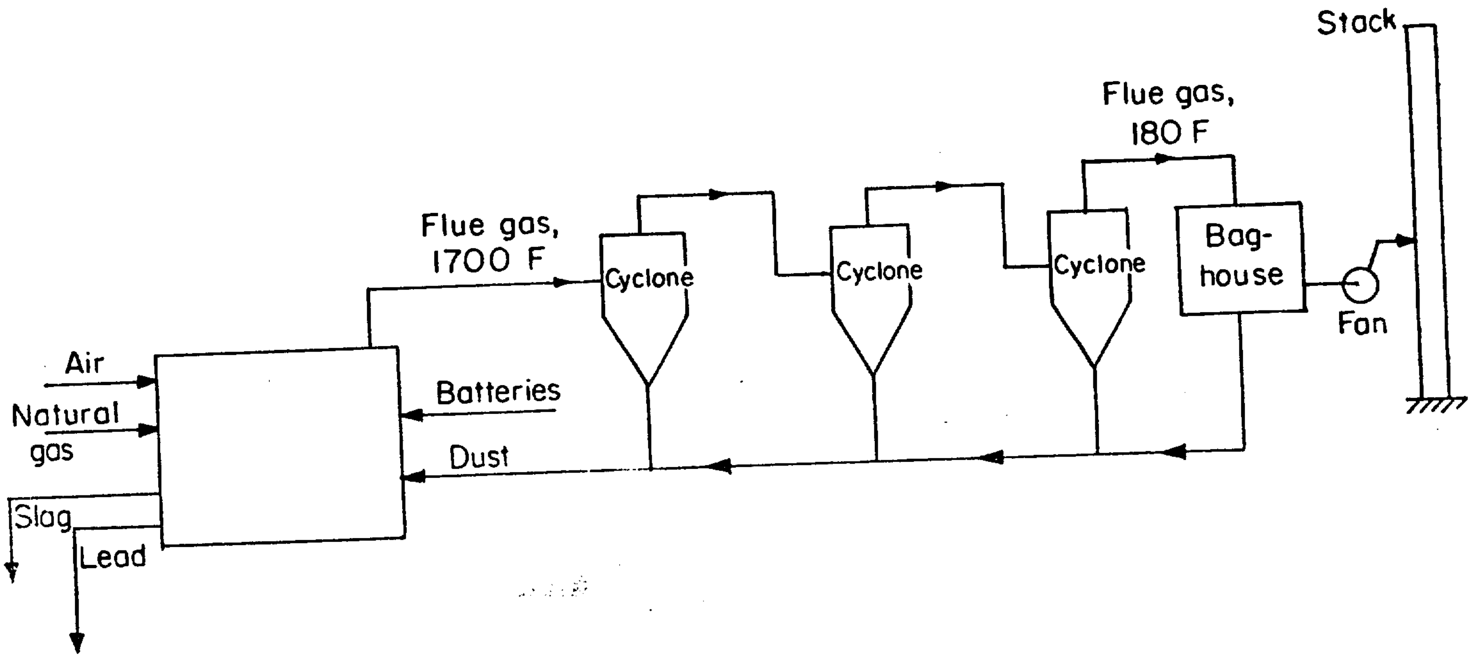

FIGURE 1. DIAGRAM OF GAS FLOW THROUGH REVERBERATORY FURNACE AND GAS-CLEANING DEVICES

SUMMARY

The exhaust duct from the baghouse on the secondary lead reverberatory furnace at N. L. Industries, Inc., in McCook, Illinois, was sampled for particulates, SO_2, CO_2, O_2, and CO in three runs of 2.5 hours each on February 9 and 10, 1972. A summary of the results is presented in Table 1, Appendix K. Due to the method of furnace operation, the measured emissions are expected to be relatively constant at all times of day except for a short duct-cleaning period when little emission is expected.

The particulate caught in the probe and filter for the three runs, averaged 0.0033 gr/scf which is equivalent to an emission of 0.25 lb/ton of lead produced. The lead particulate emission averaged .03 lb/ton. Additional material caught in the impinger section of the particulates train increased the average total emission to 0.01 gr/scf, or 1.07 lb/ton of lead produced.

The SO_2 concentration was measured in four runs, the last three concurrent with particulate sampling. The SO_2 emission was consistent for the three runs, averaging 2030 ppm, or 186 lb/ton of lead produced.

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

A chemical analysis was made of the acetone probe-wash residue. The little metal that was found was predominantly lead. None of the common anions were found. Therefore, the probe residue is probably organic materials which would not be detected in these analyses.

The impinger water residue and its chloroform and diethyl ether extract residues were analyzed for lead by atomic absorption for Run 1.

The lead measured in the residue was 0.0015 gr/scf as compared with 0.00036 gr/scf measured on the filter and probe wash residues. The 0.00036 gr/scf collected on the filter and in the probe wash residues was used to calculate the lead emission. Other metals were not detected in significant quantities. The sulfate ion and nitrogen compounds measured 0.0043 gr/scf and 0.00094 gr/scf, respectively. By material balance about one half of the sample was not detected analytically and presumably was organic compounds as these would not be detected by the methods used.

PROCESS DESCRIPTION

The N. L. Industries Plant in McCook processes battery plates into soft lead ingots. The plant consists of a receiving yard where the battery plates are stored under roof; a reverberatory furnace to melt lead scrap; holding and refining kettles for molten lead; a continuous casting line for ingots; and a shipping section. The plant is automated to an unusual degree and therefore requires a minimum of heavy lifting.

Scrap is received in the form of discarded batteries with the cases but not the separators removed. Lead oxide dust from the second baghouse, and the cyclones and cleanouts in the process duct system are intermittently loaded into the furnace. The dust from the process baghouse is fed continuously to the furnace by a conveyor system. The refining kettle drosses and the reverberatory furnace slag are sent to another plant for processing.

The lead from the reverberatory furnace contains about 0.5 percent antimony. The antimony content is further reduced in kettles by stirring sodium hydroxide and sodium nitrate into the lead to oxidize the antimony. The drosses which contain the antimony are skimmed from the kettle and the purified lead is cast into ingots for sale.

The Lead Reverberatory Furnace

The reverberatory furnace is used to make a soft lead product (pure lead) from scrap lead (battery plates) which averages about 3.5 percent antimony. When lead is melted under a lead oxide slag, the antimony migrates to the slag; the ratio of antimony to lead is about 15 times greater in the slag than in the metal. By controlling the relative amount of slag and metal in the hearth, the absolute compositions can be regulated. At McCook, the relative amounts of metal and slag are regulated by the feed composition. The metal product is about 0.5 percent antimony. The slag product, containing about 60 percent lead and 8 percent antimony is as valuable as the metal product. The slag is reduced in a blast furnace at a different site to extract the metal values.

A sketch of a lead reverberatory furnace is shown in Figure 2. A charge of lead is normally rammed in from one end. A burner is located on each side of the ram. The heat generated from a natural gas flame passes over the hearth and melts the lead; the exhaust gasses pass through a refractory flue. Lead and slag are tapped from the furnace at the end opposite the feed. The hearth at N.L. Industries is about 25-ft long by 8-ft wide with the roof about 3 ft above the melt.

The furnace is fed in 600 to 700 lb increments at regular intervals. On February 9, two stack gas samples were taken, during which time 110 increments were fed during the 8-hr day shift. The furnace operator mentioned that the feed rate is controlled by the buildup of unmelted feed in the front of the furnace. The feed is loaded into a hopper over the feed ram; the ram operates continuously. Baghouse dust is dropped from a conveyor onto the ram just in front of the feed hopper.

The two natural gas burners are operated at full capacity at all times except during a short shutdown each morning when the duct work is cleaned. The firing rate as determined from the outlet gas composition was calculated as 480 cfm which is equivalent to 28,000,000 Btu/hr.

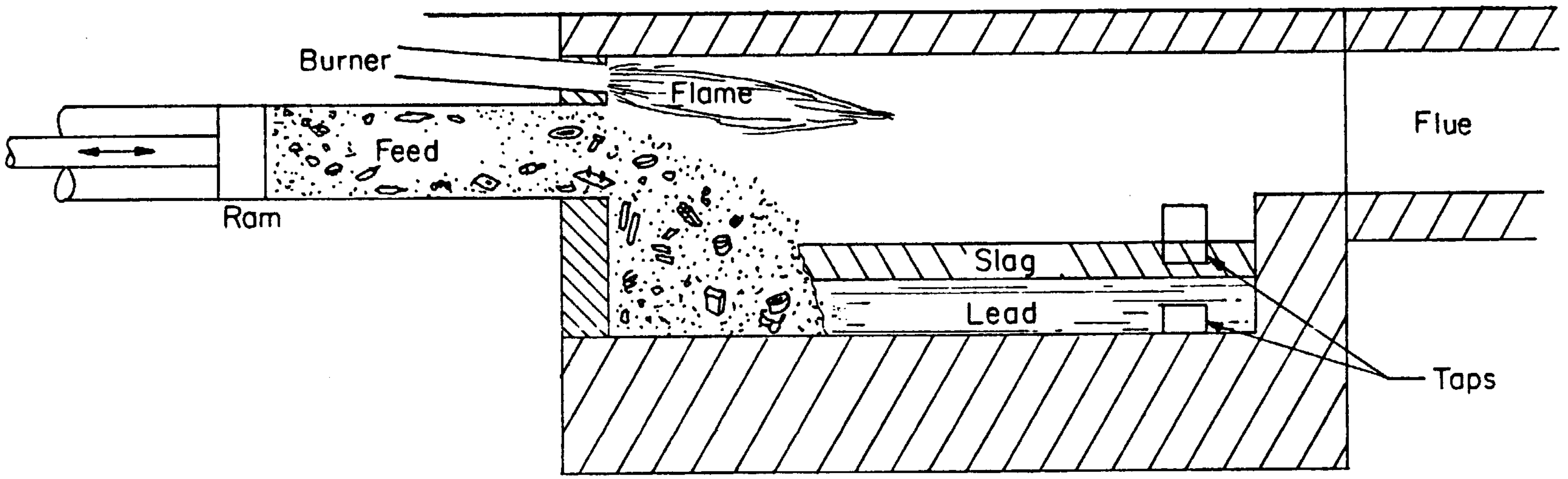

FIGURE 2. SKETCH OF REVERBERATORY FURNACE

The furnace was operated under a slight draft to prevent dust from escaping from the furnace at places other than the flue. The draft was kept to a minimum to limit the quantity of cold air entering the furnace. Dust occasionally was observed puffing from the feed port.

The lead from the furnace is tapped intermittently into a holding kettle. About 2 min is required for a normal tap of 5 tons.

Slag is tapped more or less continuously at a slow rate. While the mold for 1000 pounds was filled once in 10 minutes, a fill time of 30 minutes was typical. The slag tap, at times, was dammed for an hour or two which increased the average fill time to 40 minutes. The tap was always dammed for the 1 to 5 minute interval required to change slag molds.

The furnace fumes are formed primarily by volatilization of the slag. The amount of fume is not measured since the dust from the cyclones is returned to the furnace by a Payloader without being weighed. The dust from the baghouse also is conveyed to the furnace without being weighed. A reasonable estimate would be 5-10 ton/day of dust collected in the cyclones and baghouse.

Description of Gas Cleaning Equipment

Two separate gas cleaning systems are used at N. L. Industries. The system sampled collects dust from the reverberatory furnace exhaust gas. A second system is used to collect dust from the top of the lead kettles, the slag tap on the blast furnace, and the furnace feed.

The sampled gas stream leaving the reverberatory furnace passes through a cooling tower, three water-cooled cyclones which remove particulates and cool the gas stream from 1700 F to about 250 F. A baghouse as shown in Figure 3 removes the remaining dust. The cylindrical sections

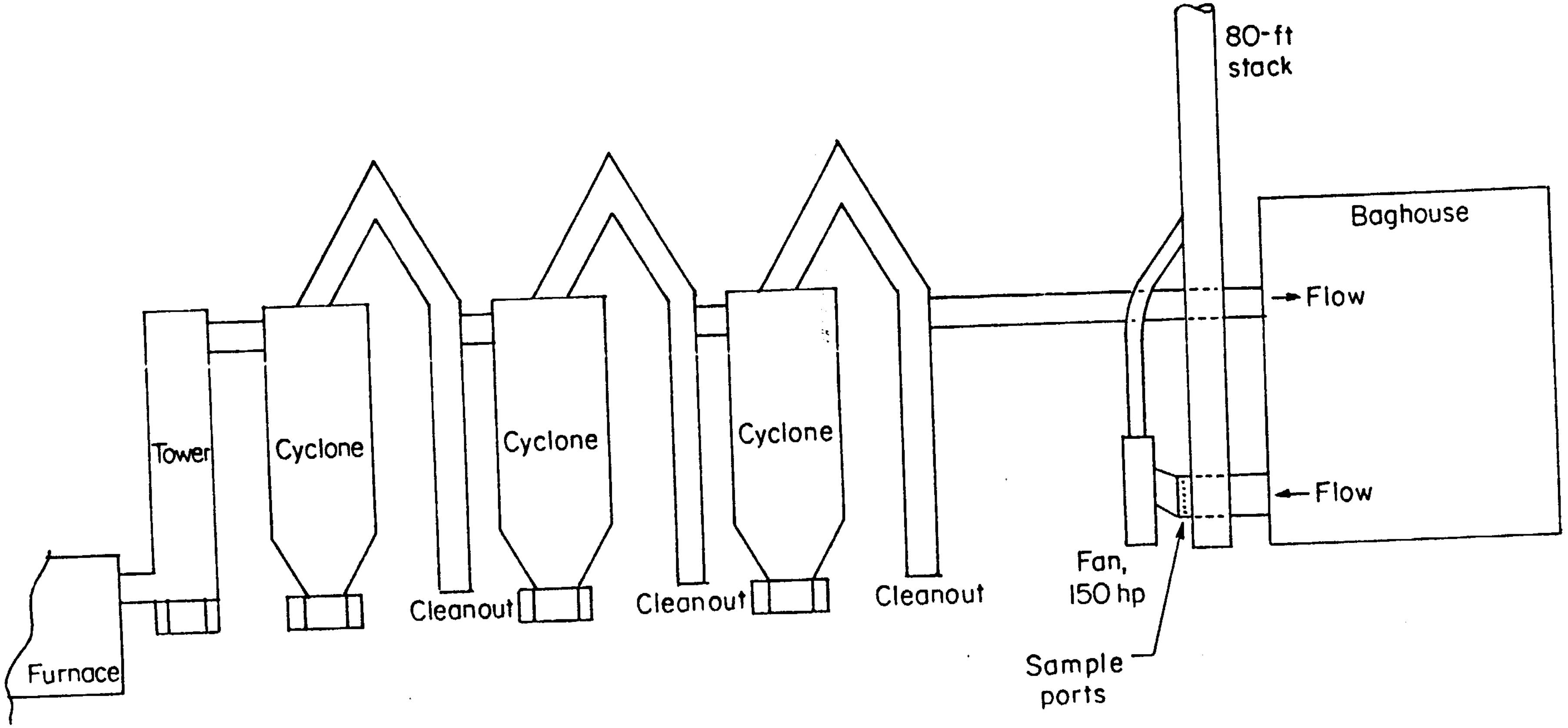

FIGURE 3. FLOW DIAGRAM FOR SECONDARY LEAD REVERBERATORY FURNACE GAS-CLEANING SYSTEM, McCOOK, ILLINOIS

of the tower and cyclones are watercooled. Cleanout ports are located at the bottom of the tower and of each cyclone and at the bottom of legs which are used to collect dust which settles in the ductwork as can be seen in Figure 3. The cyclones have a 14-ft high water cooled cylindrical side and a 6-ft high conical hopper through which the dust discharges into a 4-ft high bin. The cyclones are 12, 11, and 10 ft in diameter.

The long duct which leads from the cyclones to the baghouse is connected with ducts to the other dust-collecting systems and to the other baghouse. The connecting ducts were closed with dampers during the test period and during normal operation.

The baghouse was built by Wheelabrator and consists of four sections of 120 bags each. The bags are 20 ft long and 8 in. in diameter, with a total area of 20,000 ft^2. The design air flow is 30,000 cfm, yielding an air-to-cloth ratio of 1.5. The actual flow is 22,000 cfm for an actual air-to-cloth ratio of 1.1. Each section is shaken for 2 min to remove the dust once every 30 min. The section is closed with dampers during the shake period. The shake cycle starts on the half hour and proceeds through each section sequentially at 2-min intervals until all sections are cleaned. No shaking is performed the last 22 minutes of each half-hour cycle. This shaking cycle probably increases the effluent dust loading during the 8-min shake period. Any variation in dust loading caused by the shake cycle should be averaged for five full cycles, since particulates were sampled for a 2.5-hr period.

LOCATION OF SAMPLING PORTS

It was not possible to install sample ports in the main exhaust stack without disturbing the Gunite lining; the Gunite protects the steel shell against rapid corrosion. The duct between the fan and the stack was not sampled due to the relative geometry of the sampling equipment and the inclined circular duct.

Therefore all of the samples were obtained from a 48-in. square duct located between the baghouse and the exhaust fan, as sketched in Figure 4. The sample points are approximately four equivalent duct diameters downstream from the last exhaust gas entrance; a 24-in. round duct coming from the nearest baghouse section. This duct geometry requires a 36-point traverse. The sample position is 1.5 diameters upstream from the exhaust fan, a distance which requires a minimum of 24-traverse points as indicated by the Federal Register, p 24882, Dec. 23, 1971, in Appendix D. With respect to establishing the number of traverse points, a slight bend and transition to the fan inlet, starting 9 in. downstream from the sampling plane, was not considered significant because of the gentle change in flow direction. The transition reduces the duct from 48 in square to 40 in square. A 34-in diameter round hole opens into the exhaust fan at the outlet end of the duct. The location of the traverse points and the SO_2 sampling point in the sampling plane is shown in Appendix C.

PROCESS OPERATION

The process was operating normally during the sampling period. The tests were made on Wednesday and Thursday following a Monday startup. The startup problems had been eliminated before Wednesday morning. On Tuesday, about 2 in. of water was found in the duct which was to be sampled. This was syphoned out Tuesday and the duct was dry by sample time. The outside temperature was near 0 F during most of the test period. Therefore the gases exhausted from the plant were at the cold limit of operation. This is not expected to have a significant effect on the measured emissions.

Operation of the Reverberatory Furnace

The operation of the reverberatory furnace was typical for normal operation during the three sampling runs and no procedures which would cause variations in the samples were observed. As shown in Appendix I,

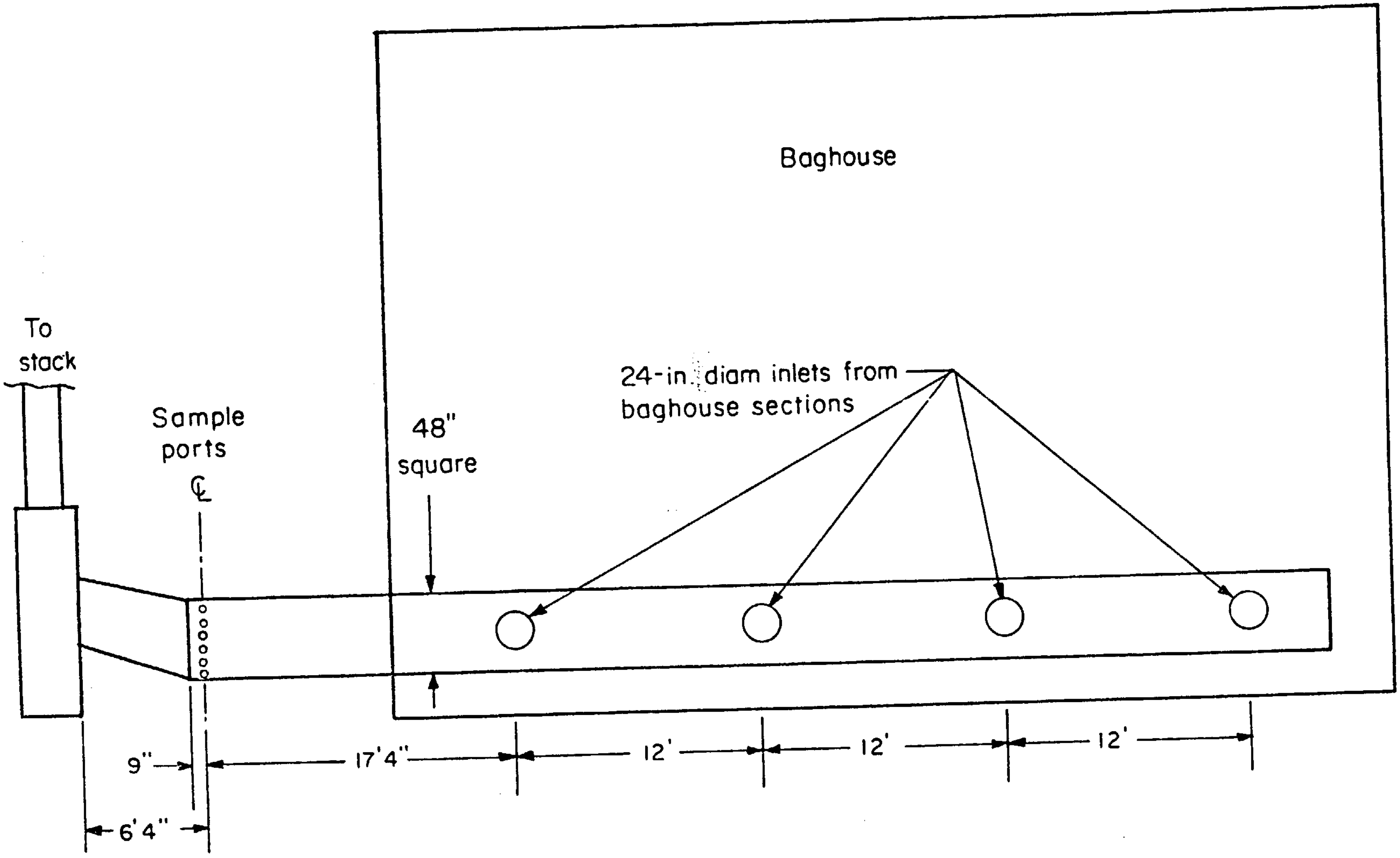

FIGURE 4. SAMPLING POINTS LOCATION

approximately 20,000 lb of battery plates were fed to the furnace during the sampling periods based on the measured output of the furnace. The 5000 lb of slag tapped during Run 1 as compared to the 3000 lb tapped in Runs 2 and 3 is probably a normal variation. The 17.7 tons of lead tapped in Run 2 was caused by a process variation requested by the sampling team. A lead tap into an almost full holding kettle normally would have been made just before the start of the run. The weight of this tap would be lost because the lead in the holding kettle would be pumped into a refining kettle simultaneously with the lead tap so that accurate measurements would be impossible. Therefore, N. L. Industries was requested to modify their procedure so an accurate measurement of the tap could be made, a change which postponed the tap until sampling time. The average furnace production rate was probably normal.

Operation of the Air-Cleaning System

The air-cleaning system was operating normally during all 3 runs. No variations in dust loading were expected because of changes in operation during sampling. The hoppers under the cyclones, tower and dust cleanouts are emptied daily during the period from 8:00 to about 8:40 am. During this period the burners are off and the flow of air through the furnace is reduced, therefore, the dust loading is expected to be less than normal during this short unsampled period.

Because Run 3 was made early in the morning shortly after the duct work had been cleaned, a lower baghouse temperature was recorded. During cleaning, the gas to the furnace is turned off and cold air is aspirated into open cleanout ports which cools off the entire system.

While no measurement was made of the dust collected, probably 500 to 1000 lb per hour are emitted from the furnace. Since about 0.6 lb per hour is emitted from the stack, the overall collection efficiency of the gas cleaning system is about 99.9 percent. The material caught in the impingers was not used in calculating efficiency.

SAMPLING AND ANALYTICAL PROCEDURES

The off gases from the lead reverberatory furnace were sampled for particulates, SO_2, CO_2, and CO by the medthods described in Appendix D, (Federal Register, December 23, 1971). The gas was sampled from a 48-inch square duct using 30 points; 5 rows of 6 points per row. A 5 minute sample was taken at each traverse point. The bottom row of six points was not sampled due to possible sample contamination from dirt on the floor of the duct. A 0.235-inch-diameter probe tip was used for all samples except in Run 1, when a 0.325-inch tip was used for the first three traverse points. The probe tip was changed to insure isokinetic sampling. About 100 cu ft of stack gas was sampled during each run.

The isokinetic sampling rate of 123 percent for Run 1 was due to using a larger nozzle than required for the first three sampling points.

The isokinetic sampling rate of 115 percent for Run 2 did not appear to have altered the dust concentration as compared to Run 1 and Run 3 which suggest that little systematic error was introduced due to the higher sampling rates.

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 solution residue evaporated to a constant weight.

The liquid from the impingers was extracted with chloroform and diethyl ether as described in the Federal Register of December 23, 1971 (Appendix D).

The particulate sampling specification published in the Federal Register of August 17, 1971, but superceded by specifications published December 23, specifies an impinger train following the sampling filter. This impinger train was used in this work, and the results from the impinger catch are reported as well as the results of the probe and filter catch. The standard procedures as set forth by EPA (Federal Register, December 23, 1971) were followed in regards to reporting of solid particulate samples and in this procedure, only the catch on the probe and filter is used to estimate collection efficiencies.

The SO_2 samples were taken simultaneously with the particulate sample for a period of 1/4 hr, during which time a sample volume of about 13 cu ft was passed through the impingers. The impinger liquid was diluted to 100 ml. A 1 ml aliquot of the sample was withdrawn by pipette, then drained into an Erlenmeyer flash for titration against 0.01 normal barium perchlorate. A thorin indicator was used.

An Orsat sample was taken from the duct simultaneously with the particulate sample. The sample was drawn into a 2-ft^3 plastic bag over a period of one hour. After flushing the Orsat with the 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 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.

The amount of the lead in the sample was determined by atomic absorption. The other metallic ions were determined by optical emission spectroscopy. The anions were determined by the wet chemical methods described in Appendix E.

APPENDIX A

COMPLETE PARTICULATE RESULTS WITH SAMPLE CALCULATIONS

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COMPLETE PARTICULATE RESULTS WITH SAMPLE CALCULATIONS

SOURCE TESTING CALCULATION FORMS

Test No.________ No. Runs 3

Name of Firm N. L. Industries

Location of Plant McCook, Illinois

Type of Plant Secondary lead

Control Equipment Cyclone, Baghouse

Sampling Point Locations Inlet to fan

Pollutants Samples Particulates, SO_2 Orsat

Time of Particulate Test:

Run No. 1 Date 2/9/72 Begin 11:00 a.m. End 1:30 p.m.

Run No. 2 Date 2/9/72 Begin 2:15 p.m. End 4:45 p.m.

Run No. 3 Date 2/10/72 Begin 9:20 a.m. End 11:50 a.m.

PARTICULATE EMISSION DATA

Run No.	1	2	3
P_b-barometric pressure, in. Hg Absolute	29.86	29.81	29.85
P_m-orifice pressure drop, in. H_2O	0.65	0.65	0.50
V_m-volume of dry gas sampled @ meter conditions, ft^3	83.4	76.2	61.2
T_m-average gas meter temperature, F	76	79.0	74.0
$V_{m_{std.}}$ - volume of dry gas sampled @ standard conditions, ft^3, dry	82.37	74.7	60.6
V_w- total H_2O collected, impingers and silical gel., ml.	72.4	75.5	56.0
$V_{w_{gas}}$ - volume of water vapor collected ft^3 @ standard conditions(a)	3.43	3.58	2.65
V_{total}- total gas volume, standard conditions, ft^3	85.8	78.3	63.3
Moisture in the stack gas volume, percent	4.0	4.6	4.2
M_d mole fraction of dry gas	0.96	0.95	0.96

(a) 70 F, 29.92 in. Hg

PARTICULATE EMISSION DATA (Cont'd)

Run No.	1	2	3
CO_2, dry, percent	2.4	2.5	2.0
O_2, dry, percent	18.2	18.0	18.3
CO, dry, percent	< 0.1	< 0.1	< 0.1
N_2, dry, percent	79.3	79.4	79.6
$M\,W_d$ - molecular weight of dry stack gas	29.1	29.1	29.1
M W - molecular weight of stack gas	28.7	28.6	28.6
T_s - stack temperature, F	124	132	117
$\sqrt{\Delta P_s \times (T_s + 460)}$	11.527	11.318	9.679
P_s - stack pressure, in. Hg absolute	29.06	29.01	29.05
V_s - stack velocity @ stack conditions, fpm	1,737	1,709	1,461
A_s - stack area, in^2	2,300	2,300	2,300
Q_s - stack gas volume @ standard conditions,(a) ft^3 dry	23,480	22,600	19,940
T_t - net time of test, min.	150	150	150
D_n - sampling nozzle diameter, in.	0.235	0.235	0.235
Percent I - percent isokinetic	124	116	107
m_f - particulate-probe and filter, mg	13.0	15.9	16.0
m_t - particulate-total, mg	63.1	63.6	64.4
C_{an} - particulate, probe, and filter, gr/scf, dry	0.0024	0.0033	0.0042
C_{ao} - particulate, total, gr/scf	0.0118	0.0131	0.0164

(a) 70 F, 29.92 in. Hg

PARTICULATE EMISSION DATA (Cont'd)

Run	1	2	3
C_{at} - particulate; probe and filter, gr/ft^3 @ stack conditions	0.0021	0.0027	0.0036
C_{au} - particulate; total, gr/ft^3 @ stack conditions	0.0100	0.0109	0.0140
C_{aw} - particulate; probe and filter, lb/hr	0.489	0.635	0.721
C_{ax} - particulate; total, lb/hr	2.374	2.540	2.795
C_p - particulate; probe and filter, lb/ton lead	0.2038	0.2646	0.3002
C_{pt} - particulate; total lb/ton lead	0.989	1.058	1.165
C_{ls} - lead emission; probe and filter, gr/scf	0.00034	0.00039	0.00041
C_{la} - lead emission; probe and filter, gr/ft^3	0.00029	0.00033	0.00035
C_{lh} - lead emission; probe and filter, lb/hr	0.0692	0.0763	0.0695
C_{lt} - lead emission; probe and filter, lb/ton	0.0288	0.0318	0.0289

EXAMPLE PARTICULATE CALCULATIONS

SAMPLE NUMBER 3

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

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

$$= \frac{17.7 \times (61.2) \left(29.85 + \frac{0.5}{13.6}\right)}{74 \div 460} \quad \text{dry}$$

$$= 60.6 \text{ scf, dry}$$

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

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

$$V_{w_{gas}} = 4.74 \times 10^{-2} \times 56.0$$

$$= 2.65 \text{ ft}^3$$

3. Percent moisture in stack gas

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

$$= \frac{100 \times 2.65}{60.7 + 2.65}$$

$$= 4.2 \text{ percent}$$

4. Mole fraction of dry gas

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

$$= \frac{100 - 4.2}{100}$$

$$= 0.96$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

5. Average molecular weight of dry stack gas

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

$$= (2.0 \times \frac{44}{100}) + (18.3 \times \frac{32}{100}) + 79.7 \qquad \times \frac{28}{100}$$

$$= 29.1$$

6. Molecular weight of stack gas

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

$$= 29.03\ (0.958) + 18\ (1 - 0.958)$$

$$= 28.6$$

7. Stack velocity @ stack conditions, fpm

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

$$= (4350) \times 9.7 \qquad \left[\frac{1}{29.05\ (28.6)}\right]^{1/2}$$

$$= 1461 \text{ fpm}$$

8. Stack gas volume at standard conditions, scfm

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

$$= \frac{0.123 \times 1461\ (2300)(29.05)(0.958)}{577}$$

$$= 19{,}940 \text{ scfm}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

9. Sampling velocity, percent of isokinetic

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

$$= \frac{(1032)\ (577)\ 60.6}{1460(150)(29.05)(0.958)(0.055)}$$

$$= 107 \text{ percent}$$

10. Percent excess air at sampling point

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

$$= \frac{100 \times 18.3}{0.266\ (79.7) - 18.3}$$

$$= 630$$

11. Particulate - probe and filter, gr/scf

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

$$= .0154 \times \frac{16.0}{60.7} \text{ (dry)}$$

$$= 0.0042 \text{ gr/scf}$$

12. Particulate - total, gr/scf

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

$$= 1.54 \times 10^{-2}\ \frac{64.4}{60.7}$$

$$= 0.0164 \text{ gr/scf}$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

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

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

$$= \frac{17.7 \times 4.2 \times 10^{-3} (29.05) \times 0.96}{577}$$

$$= 0.0036 \ gr/ft^3$$

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

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

$$= \frac{17.7 \ (1.64 \times 10^{-2})(29.05)(0.958)}{117 + 460}$$

$$= 0.014 \ gr/ft^3$$

15. Particulate - probe and filter

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

$$= 0.00857 \times 0.0042 \ (19{,}940)$$

$$= 0.72 \ lb/hr$$

16. Particulate - total, lb/hr

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

$$= 8.57 \times 10^{-3} (0.0164) \ (19{,}940)$$

$$= 2.8 \ lb/hr$$

EXAMPLE PARTICULATE CALCULATIONS (Cont'd)

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

$C_p = C_{aw}/R$

$= 0.72/2.4$

$= 0.3$ lb/ton

18. Particulate; total lb/ton lead

$C_{pt} = C_{ax}/R$

$= 2.8/2.4$

$= 1.16$ lb/ton

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

$$C_{1s} = 0.0154 \times \frac{M_1}{V_{m\ std}}$$

$= 0.0154 \times 1.5/60.63$

$= 0.00041$ gr/scf

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

$$C_{1a} = \frac{17.7 \times C_{1s} \times P_s \times M_d}{(T_s + 960)}$$

$$= \frac{17.7 \times 0.00041 \times 29.05 \times 0.96}{150 + 460}$$

$= 0.00035$ gr/ft^3

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

$C_{1h} = 8.57 \times 10^{-3} \times C_{1s} \times Q_s$

$= 8.57 \times 10^{-3} \times 4.1 \times 10^{-4} \times 1.994 \times 10^4$

$= 0.069$ lb/hr

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

$C_{1t} = C_{1h}/ R$

$= 0.069/2.4$

$= 0.03$ lb/ton

APPENDIX B

COMPLETE GASEOUS RESULTS WITH SAMPLE CALCULATIONS

COMPLETE GASEOUS RESULTS WITH SAMPLE CALCULATIONS

SO_2 EMISSION DATA

Run No.	Preliminary	1	2	3
Date (1972)	2/8	2/9	2/9	2/10
T_m - average gas meter temperature, F	58	78	69	54
P_b - barometric pressure, in. Hg absolute	29.66	29.82	29.80	29.84
V_m - volume of dry gas sampled at meter conditions, ft^3	8.10	13.53	12.87	12.89
V_m standard	8.21	13.27	12.83	13.25
V_t - volume barium perchlorate, ml	46.72	64.10	63.50	59.95
V_{tb} - volume barium perchlorate, blank, ml	0	0	0	0
N - normality of barium perchlorate	0.01	0.01	0.01	0.01
V_{soln} - volume SO_2 soln, ml	100	100	100	100
V_a - volume aliquot, ml	1	1	1	1
C_{SO_2} - $lb/ft^3 \times 10^{-5}$	40.1	34.1	34.9	31.9
C_{SO_2} - ppm (dry)	2420	2060	2110	1930
W_{SO_2} - lb/hr	570	480	473	382

SO_2 EXAMPLE CALCULATIONS

RUN 2

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

$$= 12.87 \times \frac{530}{529} \times \frac{29.8}{29.9}$$

$$= 12.83 \text{ scf}$$

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

$$= \frac{7.05 \times 10^{-5} \text{ s } 63.50 \times 0.01 \times 100}{(12.83 \; (1)}$$

$$= 34.9 \times 10^{-5} \text{ lb/scf}$$

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

$$= 6.05 \times 10^6 \times 34.9 \times 10^{-5}$$

$$= 2110 \text{ ppm}$$

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

$$= 3.49 \times 10^{-5} \times 22{,}600 \times 60$$

$$= 473 \text{ lb/hr}$$

APPENDIX C

FIELD DATA

Note: Original Field Data Sheets are on file at Battelle-Columbus Laboratories, Columbus, Ohio.

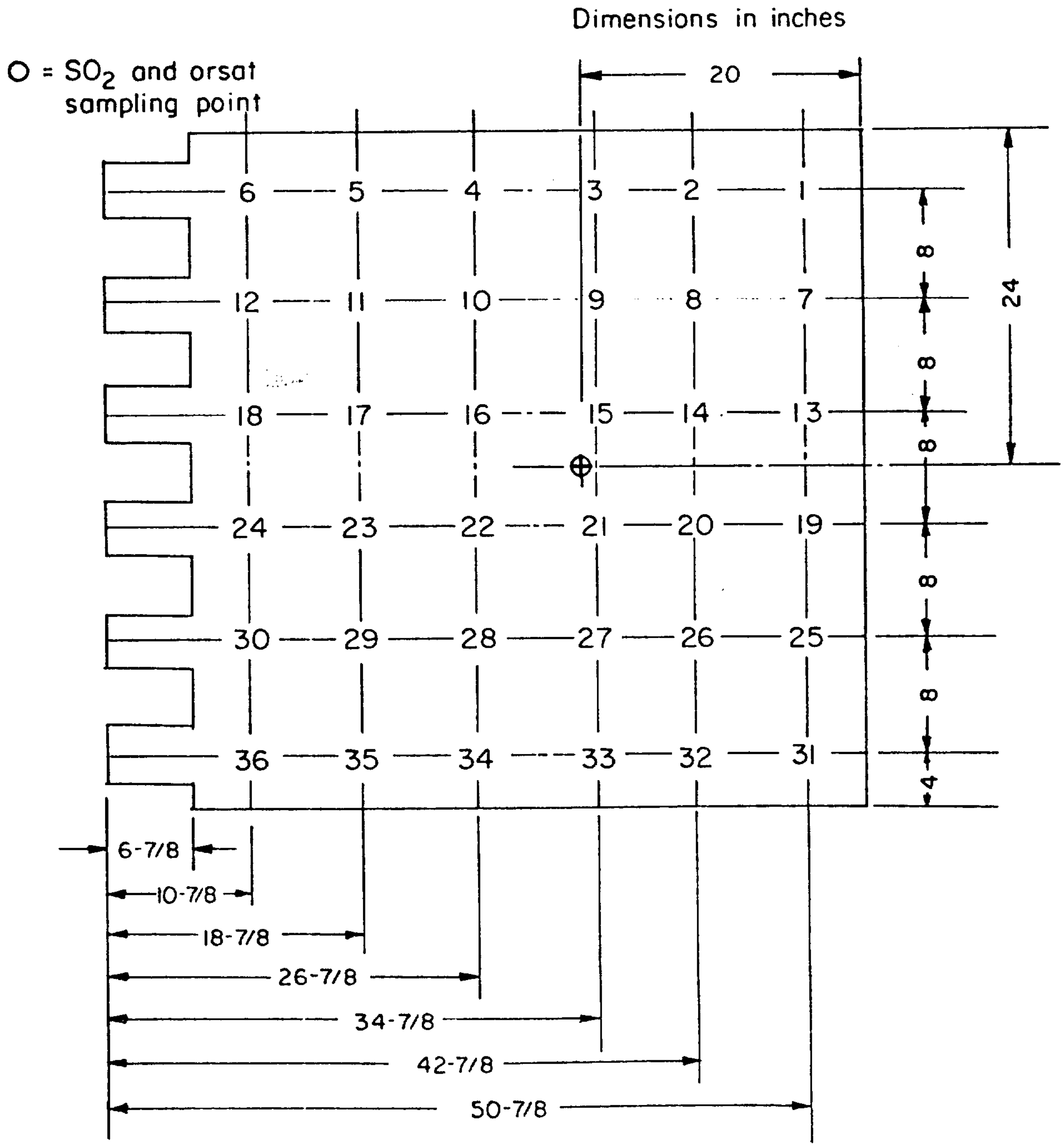
PRELIMINARY FIELD DATA

Stack Geometry

Plant - N.L. Industries, McCook, Illinois

Sample from square horizontal duct.
Duct, 4 ft sq; A-2300 in^2= 16 ft^2

SAMPLE POINT LOCATIONS



VELOCITY TRAVERSE FIELD DATA

Plant N.L. Industries, McCook, Illinois

Test Preliminary velocity traverse

Date 2/8/72

Operator P.R. Webb

Meter ΔH 1.84

Point	ΔP_s	Point	ΔP_s
1	0.28	19	0.23
2	0.30	20	0.25
3	0.25	21	0.22
4	0.30	22	0.20
5	0.32	23	0.20
6	0.32	24	0.18
7	0.32	25	0.27
8	0.30	26	0.27
9	0.28	27	0.20
10	0.28	28	0.18
11	0.28	29	0.15
12	0.25	30	0.11
13	0.30	31	0.25
14	0.28	32	0.25
15	0.20	33	0.15
16	0.20	34	0.15
17	0.20	35	0.10
18	0.20	36	0.09

PARTICULATE CLEANUP SHEET

Plant N.L. Industries, McCook, Illinois

Operators P.R. Webb, H. Leonard

Run No.	1	2	3
Impinger water volume, ml			
Final	257	263	243
Initial	200	200	200
Collected	57	63	43
Drierite, weight, gm			
Final	491.4	487.5	495.0
Initial	476.0	475.0	482.0
Gain	15.4	12.5	13.0
Total moisture, gm plus ml	72.4	75.5	56.0
Probe, acetone wash residue, mg (a)	3.3	7.1	9.6
Filter weight, mg			
Final	284.2	284.1	280.0
Tare	274.5	275.3	273.0
Gain	9.7	8.8	7.0
Total particulate weight, probe residue and filter, mg	13.0	15.9	16.6
Weight residue from chloroform-ether extract, mg			
Impinger water	27.6	27.5	26.9
Impinger wash water	1.8	4.7	1.8
Total	29.4	32.2	28.7
Weight residue from aqueous phase, mg (a)			
Impinger water	13.8	7.5	15.1
Impinger wash water	4.2	3.5	1.6
Total	18.0	11.0	16.7
Weight residue from acetone wash, mg (a)	2.7	4.5	2.4
Total, back half residue, mg	50.1	47.7	47.8
Total, front half plus back half, mg	63.1	63.6	64.4

(a) Acetone and water blank subtracted;
Acetone blank = 0.016 mg/ml, Water blank = 0.014 mg/ml.

PARTICULATE FIELD DATA

lant N.L. Industries, McCook, Illinois
ute 2/9/72
un No. 1
perator P.R. Webb, H.G. Leonard
ample Box No. 1
leter Box No. 1
leter ΔH @ 1.84
Factor 1.0

Ambient Temperature, F 60
Barometric Pressure, in. Hg 29.86
Assumed Moisture, Percent 5
Heater Box Setting, F
Probe Tip Diameter, Inches 0.325 - 0.235
Probe Length 8 ft.
Probe Heater Setting 290 F
Average ΔP
Average ΔH

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H_2O ΔP	Orifice H, In. H_2O		Dry Gas Temp., F		Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press, In. Hg	Stack Temp., F
				Desired	Actual	Inlet	Outlet					
1	11:00 am	209.8	0.32	3.5	3.0	82	58	11	260	47	-0.8	128
2	11:05	216.7	0.35	3.8	2.7	85	58	11.5	255	62	-0.8	120
3	11:10	222.8	0.36	4.0	2.7	87	62	11.5	250	76	-0.8	120
4	11:15	228.2	0.30	---	0.9	85	64	---	250	70	-0.8	120
5	11:20	230.0	0.27	---	0.8	84	65	5.0	245	68	-0.8	120
6	11:25	232.7	0.22	---	0.55	84	65	3.5	245	64	-0.8	120
7	11:30	235.2	0.27	---	0.80	82	65	5.0	242	58	-0.8	128
8	11:35	237.2	0.30	---	0.90	84	67	5.5	243	56	-0.8	120
9	11:40	240.3	0.25	---	0.75	84	66	5.0	245	57	-0.8	118
10	11:45	243.3	0.27	---	0.80	85	68	5.0	246	58	-0.8	120
11	11:50	245.9	0.25	---	0.75	86	69	5.0	248	59	-0.8	122
12	11:55	248.0	0.20	---	0.61	86	70	4.1	250	60	-0.8	122
13	12:00	252.0	0.27	---	0.80	86	70	5.2	255	57	-0.8	122
14	12:05	254.0	0.27	---	0.80	86	70	5.2	255	55	-0.8	122
15	12:10	256.0	0.22	---	0.66	86	70	4.3	253	54	-0.8	122
16	12:15	259.0	0.20	---	0.62	84	70	4.3	248	53	-0.8	122

Comments:

PARTICULATE FIELD DATA

lant N.L. Industries, McCook, Illinois
ate 2/9/72
un No. 1 (Cont'd)
perator
ample Box No.
eter Box No.
eter Δ 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 Δ P Average Δ H

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H_2O Δ P	Orifice H, In. H_2O		Dry Gas Temp., F		Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press, In. Hg	Stack Temp., F
				Desired	Actual	Inlet	Outlet					
17	12:20 pm	261.1	0.20	---	0.6	84	70	4.1	244	50	-0.8	122
18	12:25	264.1	0.18	---	0.55	83	69	4.0	230	50	-0.8	122
19	12:30	266.0	0.24	---	0.72	83	70	5.0	246	49	-0.8	130
20	12:35	268.4	0.24	---	0.72	83	68	5.0	250	48	-0.8	125
21	12:40	271.1	0.20	---	0.60	83	69	4.2	255	48	-0.8	125
22	12:45	273.0	0.18	---	0.54	83	68	4.0	254	48	-0.8	125
23	12:50	275.5	0.18	---	0.54	83	69	4.0	245	49	-0.8	130
24	12:55	277.7	0.14	---	0.42	83	70	4.0	253	48	-0.8	130
25	1:00	280.2	0.20	---	0.60	83	70	4.8	256	48	-0.8	135
26	1:05	282.5	0.25	---	0.72	84	70	4.8	256	58	-0.8	130
27	1:10	285.0	0.20	---	0.60	84	69	4.2	258	47	-0.8	130
28	1:15	287.9	0.15	---	0.45	83	70	3.5	258	48	-0.8	130
29	1:20	289.4	0.15	---	0.45	83	70	3.5	260	48	-0.8	130
30	1:25	291.4	0.11	---	0.34	83	69	3.0	260	47	-0.8	130
End	1:30	293.2										
TOTAL	150 min.	83.4 cf										
AVERAGE					0.65	84	67.6					124

Comments: Very little water at 11:00 am. No water in Bottom Point at 1:30 pm .

PARTICULATE FIELD DATA

Plant N.L. Industries, McCook, Illinois
te 2/9/72
Run No. 2
Operator P.R. Webb H.G. Leonard
Sample Box No. 1
Meter Box No. 1
Meter Δ H @ 1.84
C Factor 1.0

Ambient Temperature, F 60
Barometric Pressure, in. Hg 29.81
Assumed Moisture, Percent 5
Heater Box Setting, F 250
Probe Tip Diameter, Inches 0.235
Probe Length 8 ft
Probe Heater Setting
Average Δ P
Average Δ H

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H_2O Δ P	Orifice H, In. H_2O		Dry Gas Temp., F		Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press, In. Hg	Stack Temp., F
				Desired	Actual	Inlet	Outlet					
1	2:15 pm	293.5	0.30	---	0.90	74	64	5.5	232	36	-0.8	130
2	2:20	297.4	0.30	---	0.90	76	65	6.5	252	40	-0.8	130
3	2:25	300.0	0.34	---	1.0	79	65	7.5	275	42	-0.8	134
4	2:30	303.0	0.27	---	0.8	81	66	6.2	280	44	-0.8	134
5	2:35	306.3	0.23	---	0.7	80	66	5.8	270	44	-0.8	135
6	2:40	308.5	0.22	---	0.64	79	65	5.5	260	45	-0.8	130
7	2:45	311.0	0.29	---	0.86	78	66	7.3	250	45	-0.8	130
8	2:50	314.0	0.29	---	0.86	82	68	7.0	246	45	-0.8	130
9	2:55	316.5	0.27	---	0.80	82	68	65	242	46	-0.8	132
10	3:00	320.0	0.25	---	0.75	83	68	60	245	46	-0.8	132
11	3:05	322.1	0.23	---	0.67	82	69	5.5	246	45	-0.8	132
12	3:10	324.8	0.20	---	0.60	85	70	5.0	246	47	-0.8	132
13	3:15	328.0	0.26	---	0.77	86	72	6.5	250	48	-0.8	132
14	3:20	330.0	0.22	---	0.66	88	72	5.5	250	48	-0.8	132
15	3:25	332.6	0.20	---	0.59	88	73	5.2	252	49	-0.8	132
16	3:30	335.0	0.16	---	0.48	86	73	4.5	250	48	-0.8	132

Comments:

PARTICULATE FIELD DATA

lant N.L. Industries, McCook, Illinois
ate 2/9/72
un No. 2 (Cont'd)
perator
ample Box No.
eter Box No.
eter ΔH @
C 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 ΔP
Average ΔH

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H_2O ΔP	Orifice H, In. H_2O		Dry Gas Temp., F		Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press, In. Hg	Stack Temp., F
				Desired	Actual	Inlet	Outlet					
17	3:35 pm	337.1	0.16	---	0.49	88	74	5.0	253	48	-0.8	142
18	3:40	339.2	0.16	---	0.49	88	74	5.0	255	48	-0.8	130
19	3:45	341.9	0.26	---	0.70	88	76	6.5	255	49	-0.8	130
20	3:50	345.0	0.24	---	0.70	91	76	6.2	254	49	-0.8	134
21	3:55	347.4	0.20	---	0.60	91	76	5.5	254	49	-0.8	134
22	4:00	349.0	0.19	---	0.57	92	77	5.2	252	48	-0.8	134
23	4:05	351.9	0.14	---	0.43	91	78	4.3	255	49	-0.8	135
24	4:10	354.0	0.14	---	0.43	91	78	4.2	255	48	-0.8	135
25	4:15	356.0	0.19	---	0.57	88	77	4.3	260	48	-0.8	135
26	4:20	358.6	0.25	---	0.74	90	78	7.0	260	47	-0.8	135
27	4:25	361.4	0.22	---	0.65	92	78	5.5	255	48	-0.8	135
28	4:30	363.6	0.19	---	0.57	92	78	5.2	255	48	-0.8	135
29	4:35	366.0	0.12	---	0.37	91	78	4.0	255	47	-0.8	130
30	4:40	367.7	0.11	---	0.34	88	78	4.0	255	47	-0.8	130
End	4:45	369.7										
TOTAL	180 min	76.2 cf										
AVERAGE					0.65	86	72					132

PARTICULATE FIELD DATA

lant N.L. Industries, McCook, Illinois
ate 2/10/72
un No. 3
perator P.R. Webb, H.G. Leonard
ample Box No. 1
eter Box No. 1
eter ΔH @ 1.84
Factor 1.0

Ambient Temperature, F 62
Barometric Pressure, in. Hg 29.85
Assumed Moisture, Percent 5
Heater Box Setting, F 255
Probe Tip Diameter, Inches 0.235
Probe Length 8 ft
Probe Heater Setting 260 F
Average ΔP ______ Average ΔH ______

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H_2O ΔP	Orifice H, In. H_2O		Dry Gas Temp., F		Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press, In. Hg	Stack Temp., F
				Desired	Actual	Inlet	Outlet					
1	9:20 am	370.3	0.27	---	0.80	66	48	5.5	240	40	-0.8	112
2	9:25	372.8	0.32	---	0.95	73	48	7.0	260	40	-0.8	118
3	9:30	375.2	0.29	---	0.86	77	52	6.2	266	42	-0.8	118
4	9:35	377.5	0.25	---	0.72	79	54	5.5	265	45	-0.8	118
5	9:40	380.0	0.22	---	0.63	79	57	4.8	260	48	-0.8	123
6	9:45	382.7	0.16	---	0.47	78	58	3.8	258	48	-0.8	115
7	9:50	385.0	0.20	---	0.58	78	58	4.0	258	48	-0.8	115
8	9:55	386.2	0.25	---	0.72	80	62	6.1	258	53	-0.8	115
9	10:00	389.4	0.22	---	0.63	83	64	5.1	256	58	-0.8	114
10	10:05	391.3	0.20	---	0.58	83	64	5.0	260	61	-0.8	115
11	10:10	394.0	0.16	---	0.47	84	65	4.2	260	65	-0.8	120
12	10:15	396.4	0.15	---	0.45	84	67	4.2	255	54	-0.8	115
13	10:20	398.3	0.20	---	0.58	83	68	4.2	255	54	-0.8	112
14	10:25	399.5	0.20	---	0.58	84	69	5.2	265	55	-0.8	117
15	10:30	402.0	0.16	---	0.47	86	70	4.2	260	54	-0.8	118
16	10:35	404.3	0.14	---	0.42	86	70	4.2	262	52	-0.8	118

Comments:

PARTICULATE FIELD DATA

Plant N.L. Industries, McCook, Illinois
Date 2/10/72
Run No. 3 (Cont'd)
Operator
Sample Box No.
Meter Box No.
Meter ΔH @
C 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 ΔP Average ΔH

Point	Clock Time	Dry Gas Meter, CF	Pitot, In. H_2O ΔP	Orifice H, In. H_2O		Dry Gas Temp., F		Pump Vacuum, In. Hg Gauge	Box Temp., F	Impinger Temp., F	Stack Press, In. Hg	Stack Temp., F
				Desired	Actual	Inlet	Outlet					
17	10:40 am	406.5	0.14	---	0.42	86	71	4.0	258	53	-0.8	---
18	10:45	408.6	0.11	---	0.32	86	72	3.2	258	50	-0.8	118
19	10:30	410.0	0.18	---	0.52	85	72	45	255	48	-0.8	118
20	10:55	412.8	0.19	---	0.58	86	72	52	256	48	-0.8	118
21	11:00	415.0	0.14	---	0.42	87	72	3.8	256	48	-0.8	118
22	11:05	416.6	0.11	---	0.32	86	72	3.2	256	48	-0.8	118
23	11:10	418.2	0.07	---	0.21	86	72	2.5	255	47	-0.8	118
24	11:15	419.9	0.07	---	0.21	86	72	2.5	256	45	-0.8	118
25	11:20	421.4	0.18	---	0.52	85	71	5.0	260	45	-0.8	118
26	11:25	423.6	0.18	---	0.52	85	72	5.0	256	45	-0.8	118
27	11:30	426.4	0.13	---	0.39	87	72	5.8	256	47	-0.8	118
28	11:35	427.7	0.10	---	0.29	86	72	3.2	256	47	-0.8	118
29	11:40	429.3	0.05	---	0.14	86	72	2.0	256	47	-0.8	118
30	11:45	430.3	0.05	---	0.14	85	71	2.0	256	47	-0.8	118
End	11:50	431.5										
TOTAL	150 min	61.2 cf										
AVERAGE					0.50	82.8	65.9					117

GAS SAMPLING FIELD DATA

Date 2/8/72

Material Sampled for SO_2

Barometric Pressure 29.66 in. Hg

Ambient Temperature 70 F

Run Number Preliminary

Power Stat Setting Heated

Filter Used: Yes X No ___

Operator W.C. Baytos, G.W. Keigley

Plant N.L. Industries, McCook, Illinois

Comments: Recovered 95.0 ml- dilute to 100 ml.
Probe temperature, 180 F.

Titration	
Aliquot, ml	Titer, ml
1.0	47.2
Blank	0

0.01 N $Ba(ClO_4)_2$

Clock Time	Meter, ft^3	Flow Meter Setting (cfh)	Meter Temperature, F
2:25 pm	0	6.0	58
2:30	0.50	6.6	58
2:35	1.04	6.6	58
2:40	1.57	6.6	58
2:45	2.09	6.6	58
2:50	2.71	6.6	68
2:55	3.13	6.6	58
3:00	3.64	6.6	58
3:05	4.10	6.6	58
3:10	4.58	6.7	58
3:15	5.12	6.8	58
3:20	5.67	7.0	58
3:25	6.47	7.3	59
3:30	6.93	7.5	59
3:35	7.52	7.6	60
3:40	8.10	8.0	60

GAS SAMPLING FIELD DATA

Date 2/9/72

Material Sampled for SO_2

Barometric Pressure 29.82 in. Hg

Ambient Temperature 75 F

Run Number 1

Power Stat Setting Heated

Filter Used: Yes X No

Operator W.C. Baytos, G.W. Keigley

Plant N.L. Industries, McCook, Illinois

Comments: Recovered 90 ml - diluted to 100 ml.
Probe temperature, 105 F.

Titration	
Aliquot, ml	Titer, ml
1.00	64.20
1.00	63.90
Blank	0

0.01 N $Ba(ClO_4)_2$

Clock Time	Meter, ft^3	Flow Meter Setting (cfh)	Meter Temperature, F
11:50 am	0	6.6	76
11:55	0.55	6.6	76
12:00 pm	1.10	6.6	76
12:05	1.66	67	77
12:10	2.20	6.7	78
12:15	2.75	6.8	78
12:20	3.31	6.8	78
12:25	3.97	6.8	78
12:30	4.42	6.8	78
12:35	5.09	6.8	78
12:43	5.88	6.8	78
12:45	6.20	6.9	78
12:50	6.65	6.9	78
12:55	7.2	6.9	78
1:00	7.77	7.0	78
1:05	8.33	7.0	79
1:10	8.90	7.0	79
1:15	9.35	7.0	80
1:20	10.02	7.0	80
1:25	10.59	7.0	80
1:30	11.15	7.0	80
1:35	11.74	7.0	80
1:40	12.50	7.0	80
1:45	13.05	7.0	80
1:50	13.53	7.5	79

GAS SAMPLING FIELD DATA

Plant N.L. Industries, McCook, Illinois

Date 2/9/72

Material Sampled for SO_2

Barometric Pressure 29.80 in. Hg

Ambient Temperature 60 F

Run Number 2

Power Stat Setting Heated

Filter Used: Yes X No ___

Operator W.C. Baytos, G.W. Keigley

Comments: Recoverd 89 ml - diluted to 100 ml.
Probe temperature, 120 F.

Titration	
Aliquot, ml	Titer, ml
1.00	62.80
1.00	64.20
Blank	0

0.01 N $Ba(ClO_4)_2$

Clock Time	Meter, ft^3	Flow Meter Setting (cfh)	Meter Temperature, F
3:10 pm	0	7.0	72
3:15	0.57	7.1	72
3:20	1.14	7.1	71
3:25	1.69	6.5	70
3:30	2.23	6.5	70
3:35	2.77	6.5	70
3:40	3.42	6.5	69
3:45	3.95	6.5	68
3:50	4.50	6.4	67
3:55	5.03	6.4	63
4:00	5.59	6.5	66
4:05	6.20	6.5	66
4:10	6.53	6.4	65
4:15	7.05	6.4	65
4:20	7.59	6.5	64
4:25	8.11	6.4	66
4:30	8.63	6.5	66
4:35	9.27	6.5	65
4:40	9.80	6.5	65
4:45	10.32	6.5	66
4:50	10.85	6.5	66
4:55	11.38	6.5	66
5:00	12.10	6.5	67
5:05	12.33	6.5	67
5:10	12:87	6.4	67

GAS SAMPLING FIELD DATA

Date 2/10/72

Material Sampled for SO_2

Barometric Pressure 29.84 in. Hg

Ambient Temperature 53 F

Run Number 3

Power Stat Setting Heated

Filter Used: Yes X No ___

Operator W.C.Baytos, G.W. Keigley

Plant N.L. Industries, McCook, Illinois

Comments: Recovered 96 ml - diluted to 100 ml.
Probe temperature, 180 F.

Titration

Aliquot, ml	Titer, ml
1.00	60.45
1.00	59.65
Blank	0

0.01 N $Ba(ClO_4)_2$

Clock Time	Meter, ft^3	Flow Meter Setting (cfh)	Meter Temperature, F
8:55 am	0	6.0	48
9:00	0.39	5.8	48
9:05	0.95	7.2	48
9:10	1.50	7.2	49
9:15	2.01	7.2	50
9:20	2.55	7.2	50
9:25	3.09	7.2	51
9:30	3.64	7.2	52
9:36	4.38	7.0	53
9:40	4.81	7.0	53
9:46	5.46	7.0	54
9:50	5.89	7.0	54
9:55	6.43	7.0	54
10:00	6.87	7.0	55
10:05	7.41	6.9	56
10:10	7.94	6.9	57
10:15	8.47	6.9	58
10:20	9.00	6.9	58
10:25	9.55	6.8	59
10:30	10.09	6.8	59
10:35	10.62	6.8	60
10:40	11.26	6.8	61
10:45	11.82	6.8	61
10:50	12.35	6.7	62
10:55	12.89	6.7	62

ORSAT GAS COMPOSITION

Plant N.L. Industries, McCook, Illinois

Run	Date	CO_2, Percent	O_2, Percent	CO, Percent	N_2, Percent
1	2/9/72	2.3	18.3	<0.1	79.4
		2.4	18.0	<0.1	79.6
	Average-	2.4	18.1	<0.1	79.5
2		2.4	18.0	<0.1	79.4
		2.6	18.0	<0.1	79.6
	Average-	2.5	18.0	<0.1	79.5
3	2/10/72	2.0	18.3	<0.1	79.7

Note: Detectable limits of CO by Orsat is <0.1 by volume.

APPENDIX D

STANDARD SAMPLING PROCEDURES

APPENDIX D

STANDARD SAMPLING PROCEDURES

The sampling procedures as described in the December 23, 1971, issue of the Federal Resgister, reproduced as a part of this appendix, has been used as a reference for the source emission test conducted at N. L. Industries Plant, McCook, Illinois.

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

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

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

During the sampling period, isokinetic flow was maintained by adjusting the sampling flow rate to compensate for stack Δp and temeperature variations. Appendix C is a record of the field data for the three tests completed at N.L. Industries Plant, McCook, Illinois.

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. They 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 weight of acetone wash residue and the net filter weight now 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.

method(s) prescribed by the manufacturer(s) of such instrument, the instrument shall be subject to manufacturers recommended zero adjustment calibration 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_2 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.^{3} corrected to standard conditions.

(2) The volumetric flow rate of the total effluent shall be determined by using Method 2 and traversing according to Method 1. Gas analysis shall be performed by using the integrated sample technique of Method 3. Moisture content can be considered to be zero.

(d) Acid produced, expressed in tons per hour of 100 percent sulfuric acid shall be determined during each 2-hour testing period by suitable flow meters and shall be confirmed by a material balance over the production system.

(e) For each repetition acid mist and sulfur dioxide emissions, expressed in lb./ton of 100 percent sulfuric acid shall be determined by dividing the emission rate in lb./hr. by the acid produced. The emission rate shall be determined by the equation, lb./hr.=$Q_s \times c$, where Q_s=volumetric flow rate of the effluent in ft.^{3}/hr. at standard conditions, dry basis as determined in accordance with paragraph (c)(2) of this section, and c=acid mist and SO_2 concentrations in lb./ft.^{3} as determined in accordance with paragraph (c)(1) of this section, corrected to standard conditions, dry basis.

APPENDIX—TEST METHODS

METHOD 1—SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

1. *Principle and Applicability.*

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

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

2. *Procedure.*

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

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

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

equation 1-1

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

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

2.1.4 To use Figure 1-1 first measure the distance from the chosen sampling location to the nearest upstream and downstream 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.

2.2 Cross-sectional layout and location of traverse points.

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

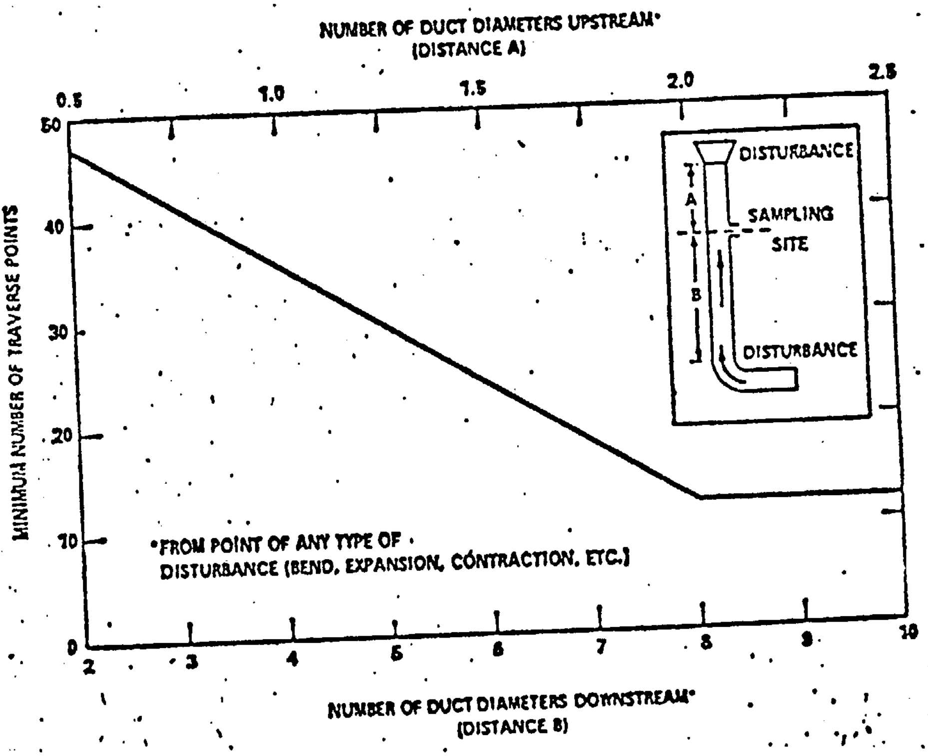


Figure 1-1. Minimum number of traverse points.

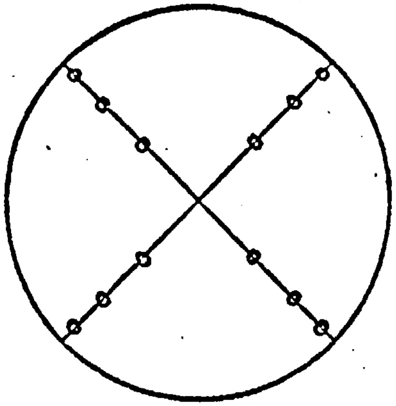


Figure 1-2. Cross section of circular stack divided into 12 equal areas, showing location of traverse points at centroid of each area.

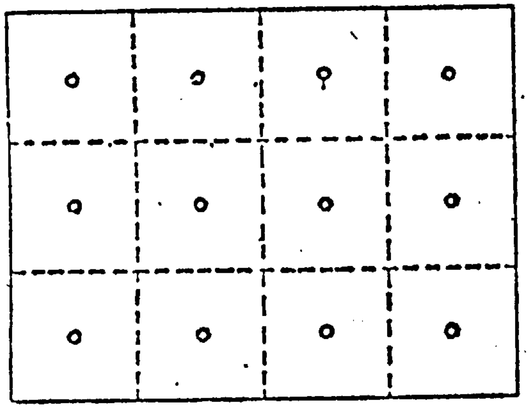


Figure 1-3. Cross section of rectangular stack divided into 12 equal areas, with traverse points at centroid of each area.

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

Traverse point number on a diameter	Number of traverse points on a diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.3	2.5	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.7	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.5	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.5	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.3	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5
6			95.6	80.6	65.8	35.5	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	65.0	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	33.8	31.5	27.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2
14							98.2	91.5	85.4	79.6	73.9	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.5	92.1
22											98.9	94.5
23												96.8
24												98.9

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

3. *References.*

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

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

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

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

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

1. *Principle and applicability.*

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

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

2. *Apparatus.*

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

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

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

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

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

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

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

3. *Procedure.*

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

3.2 Measure the static pressure in the stack.

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

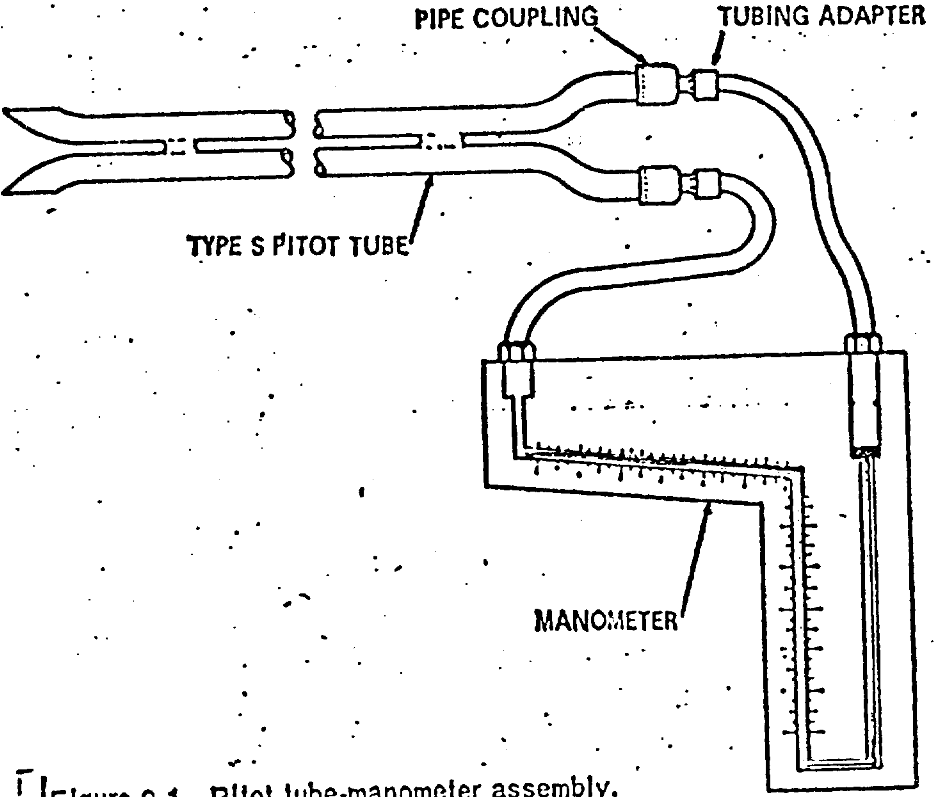


Figure 2-1. Pitot tube-manometer assembly.

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. Calibration should be done in the laboratory and the velocity of the flowing gas stream should be varied over the normal working range. It is recommended that the calibration be repeated after use at each field site.

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

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

where:

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

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

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

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

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

5. *Calculations.*

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

$$(V_s)_{avg.}=K_pC_p(\sqrt{\Delta p})_{avg.}\sqrt{\frac{(T_s)_{avg.}}{P_sM_s}}$$

Equation 2–2

where:

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

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

C_p=Pitot tube coefficient, dimensionless.

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

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

P_s=Absolute stack gas pressure, inches Hg.

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

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

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

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

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

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

$$Q_s=3600\ (1-B_{wo})V_sA\left(\frac{T_{std}}{(T_s)_{avg.}}\right)\left(\frac{P_s}{P_{std}}\right)$$

Equation 2–3

where:

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

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

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

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

6. References.

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

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

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

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

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

PLANT______________________________

DATE______________________________

RUN NO.____________________________

STACK DIAMETER, in.____________________

BAROMETRIC PRESSURE, in. Hg.______________

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

OPERATORS___________________________

SCHEMATIC OF STACK CROSS SECTION

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

Figure 2-2. Velocity traverse data.

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

1. *Principle and applicability.*

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

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

2. *Apparatus.*

2.1 Grab sample (Figure 3–1).

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

2.1.2 Pump—One-way squeeze bulb, or uivalent, to transport gas sample to alyzer.

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

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

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

2.2.3 Needle valve—To adjust flow rate.

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

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

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

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

2.3 *Analysis.*

2.3.1 Orsat analyzer, or equivalent.

3. *Procedure.*

3.1 Grab sampling.

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

3.1.2 Draw sample into the analyzer.

3.2 Integrated sampling.

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

3.2.2 Sample at a rate proportional to the stack velocity.

3.3 Analysis.

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

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

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

4. *Calculations.*

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

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

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

equation 3–1

where:

$\%EA$=Percent excess air.
$\%O_2$=Percent oxygen by volume, dry basis.
$\%N_2$=Percent nitrogen by volume, dry basis.
$\%CO$=Percent carbon monoxide by volume, dry basis.
0.264=Ratio of oxygen to nitrogen in air by volume.

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

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

equation 3–2

where:

M_d=Dry molecular weight, lb./lb-mole.
$\%CO_2$=Percent carbon dioxide by volume, dry basis.
$\%O_2$=Percent oxygen by volume, dry basis.
$\%N_2$=Percent nitrogen by volume, dry basis.
0.44=Molecular weight of carbon dioxide divided by 100.
0.32=Molecular weight of oxygen divided by 100.
0.28=Molecular weight of nitrogen and CO divided by 100.

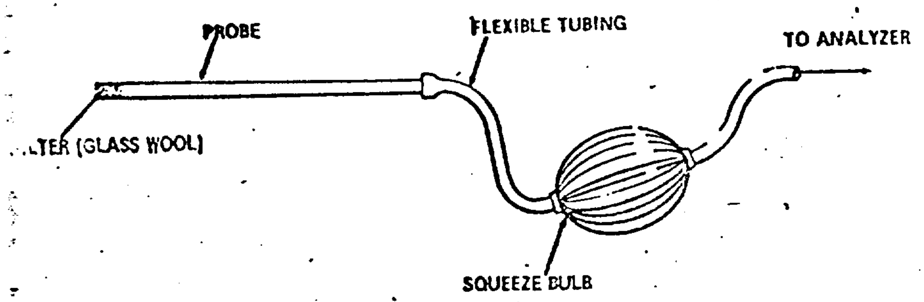


Figure 3-1. Grab-sampling train.

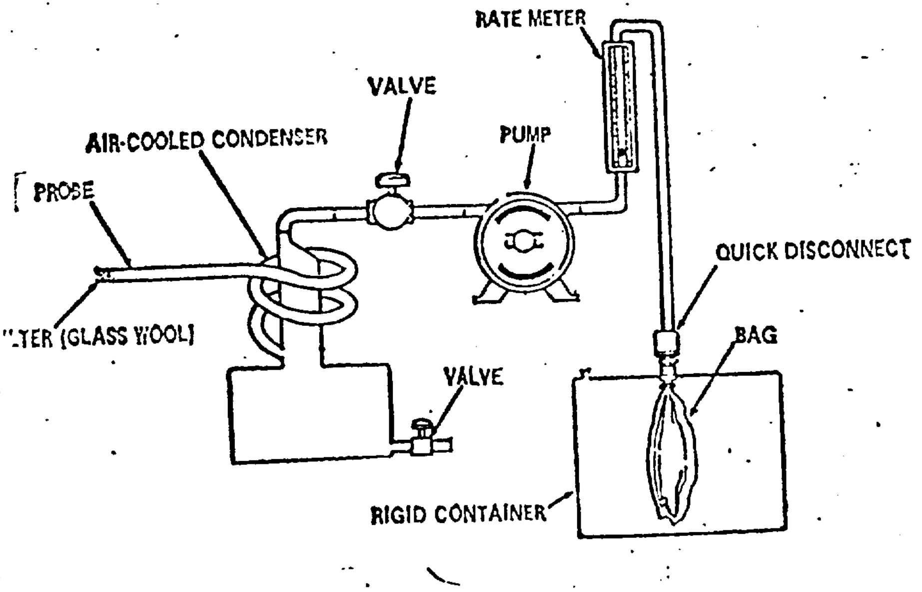


Figure 3-2. Integrated gas - sampling train.

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[1] Trade name.

5. *References.*

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

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

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

METHOD 4—DETERMINATION OF MOISTURE IN STACK GASES

1. *Principle and applicability.*

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

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

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

2. *Apparatus.*

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

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

2.3 Ice bath container—To condense moisture in impingers.

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

2.5 Needle valve—To regulate gas flow rate.

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

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

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

2.9 Graduated cylinder—25 ml.

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

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

3. *Procedure.*

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

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

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

4. *Calculations.*

4.1 Volume of water vapor collected.

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

where:

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

V_f=Final volume of impinger contents, ml.

V_i=Initial volume of impinger contents, ml.

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

ρ_{H_2O}=Density of water, 1 g./ml.

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

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

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

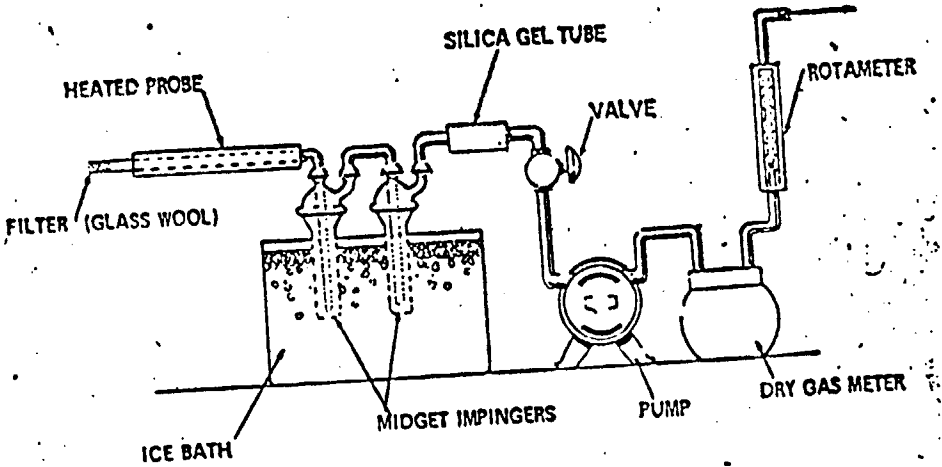

Figure 4-1. Moisture-sampling train.

LOCATION ______________________ COMMENTS
TEST ______________________
DATE ______________________
OPERATOR ______________________
BAROMETRIC PRESSURE ______________________

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

Figure 4-2. Field moisture determination.

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

[2] Trade name.

4.2 Gas volume.

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

where:

$V_{m_{std}}$ =Dry gas volume through meter at standard conditions, cu. ft.

V_m =Dry gas volume measured by meter, cu. ft.

P_m =Barometric pressure at the dry gas meter, inches Hg.

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

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

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

4.3 Moisture content.

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

where:

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

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

$V_{m_{std}}$ =Dry gas volume through meter (standard conditions), cu. ft.

B_{wm}=Approximate volumetric proportion of water vapor in the gas stream leaving the impingers, 0.025.

5. *References.*

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

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

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

METHOD 5—DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

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

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

2. *Apparatus.*

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

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

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

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

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

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

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

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

2.2 Sample recovery.

2.2.1 Probe brush—At least as long as probe.

2.2.2 Glass wash bottles—Two.

2.2.3 Glass sample storage containers.

2.2.4 Graduated cylinder—250 ml.

2.3 Analysis.

2.3.1 Glass weighing dishes.

2.3.2 Desiccator.

2.3.3 Analytical balance—To measure to ±0.1 mg.

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

3. *Reagents.*

3.1 Sampling.

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

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

3.1.3 Water.

3.1.4 Crushed ice.

3.2 Sample recovery.

3.2.1 Acetone—Reagent grade.

3.3 Analysis.

3.3.1 Water.

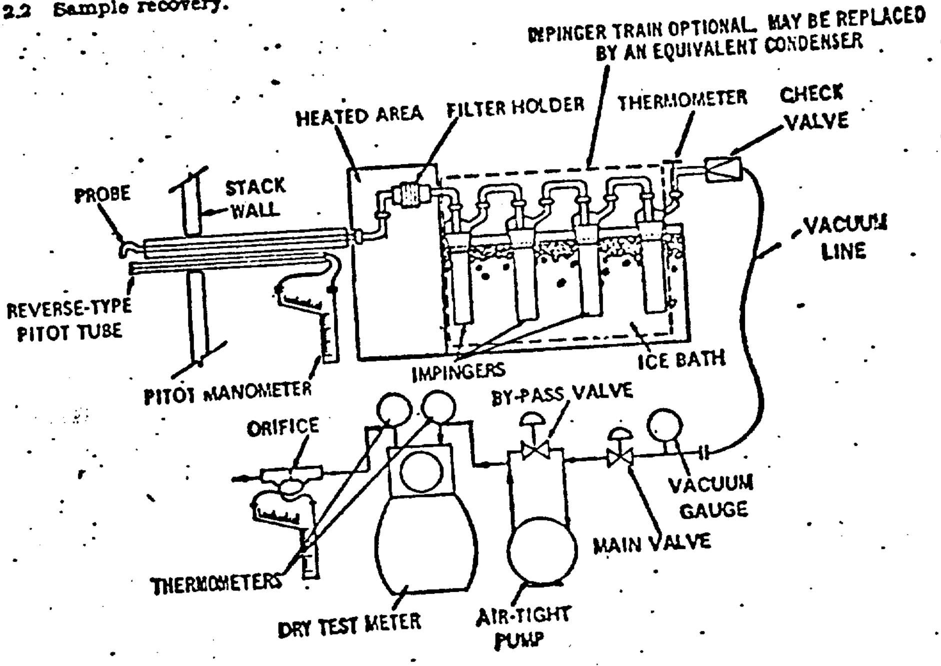

Figure 5-1. Particulate-sampling train.

3.3.2 Desiccant—Drierite,[1] indicating.

4. *Procedure.*

4.1 Sampling

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

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

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

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[1] Trade name.

[1] Trade name.

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

PLANT________
LOCATION________
OPERATOR________
DATE________
RUN NO.________
SAMPLE BOX NO.________
METER BOX NO.________
METER $\Delta H_@$________
C FACTOR________

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE________
BAROMETRIC PRESSURE________
ASSUMED MOISTURE, %________
HEATER BOX SETTING________
PROBE LENGTH, m.________
NOZZLE DIAMETER, in.________
PROBE HEATER SETTING________

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE (P_s), in. Hg	STACK TEMPERATURE (T_s), °F	VELOCITY HEAD (ΔP_s),	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER (ΔH), in. H_2O	GAS SAMPLE VOLUME (V_m), ft³	GAS SAMPLE TEMPERATURE AT DRY GAS METER		SAMPLE BOX TEMPERATURE, °F	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °F
							INLET ($T_{m_{in}}$), °F	OUTLET ($T_{m_{out}}$), °F		
TOTAL							Avg.	Avg.		
AVERAGE							Avg.			

Figure 5-2. Particulate field data.

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

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

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

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

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

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

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

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

5. *Calibration.*

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

6. *Calculations.*

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

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

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

equation 5-1

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.
V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.
T_{std} = Absolute temperature at standard conditions, 530° R.
T_m = Average dry gas meter temperature, °R.
P_{bar} = Barometric pressure at the orifice meter, inches Hg.
ΔH = Average pressure drop across the orifice meter, inches H_2O.
13.6 = Specific gravity of mercury.
P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.3 Volume of water vapor.

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

equation 5-2

where:

$V_{w_{std}}$ = Volume of water vapor in the gas sample (standard conditions), cu. ft.
V_{l_c} = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.
ρ_{H_2O} = Density of water, 1 g./ml.
M_{H_2O} = Molecular weight of water, 18 lb./lb.-mole.
R = Ideal gas constant, 21.83 inches Hg—cu. ft./lb.-mole-°R.
T_{std} = Absolute temperature at standard conditions, 530° R.
P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.4 Moisture content.

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

equation 5-3

where:

B_{wo} = Proportion by volume of water vapor in the gas stream, dimensionless.
$V_{w_{std}}$ = Volume of water in the gas sample (standard conditions), cu. ft.
$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

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

6.6 Concentration.

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

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

equation 5-4

where:

c'_s = Concentration of particulate matter in stack gas, gr./s.c.f., dry basis.
M_n = Total amount of particulate matter collected, mg.
$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

PLANT____________________

DATE____________________

RUN NO.____________________

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

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

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

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

Figure 5-3. Analytical data.

6.6.2 Concentration in lb./cu. ft.

$$c_s = \frac{\left(\frac{1}{453,600}\frac{\text{lb.}}{\text{mg.}}\right)M_n}{V_{m_{std}}} = 2.205\times10^{-6}\frac{M_n}{V_{m_{std}}} \qquad \text{equation 5–5}$$

where:

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

453,600=Mg/lb.

M_n=Total amount of particulate matter collected, mg.

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

6.7 Isokinetic variation.

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

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

where:

I=Percent of isokinetic sampling.

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

ρ_{H_2O}=Density of water, 1 g./ml.

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

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

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

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

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

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

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

θ=Total sampling time, min.

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

P_s=Absolute stack gas pressure, inches Hg.

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

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

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

7. *Reference.*

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

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

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

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

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

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

METHOD 6—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

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

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

2. *Apparatus.*

2.1 Sampling. See Figure 6–1.

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

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

2.1.3 Glass wool.

2.1.4 Midget impingers—Three.

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

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

2.1.7 Pump—Leak-free, vacuum type.

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

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

2.1.10 Pitot tube—Type S, or equivalent,

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[1] Trade names.

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

2.2 Sample recovery.

2.2.1 Glass wash bottles—Two.

2.2.2 Polyethylene storage bottles—To store impinger samples.

2.3 Analysis.

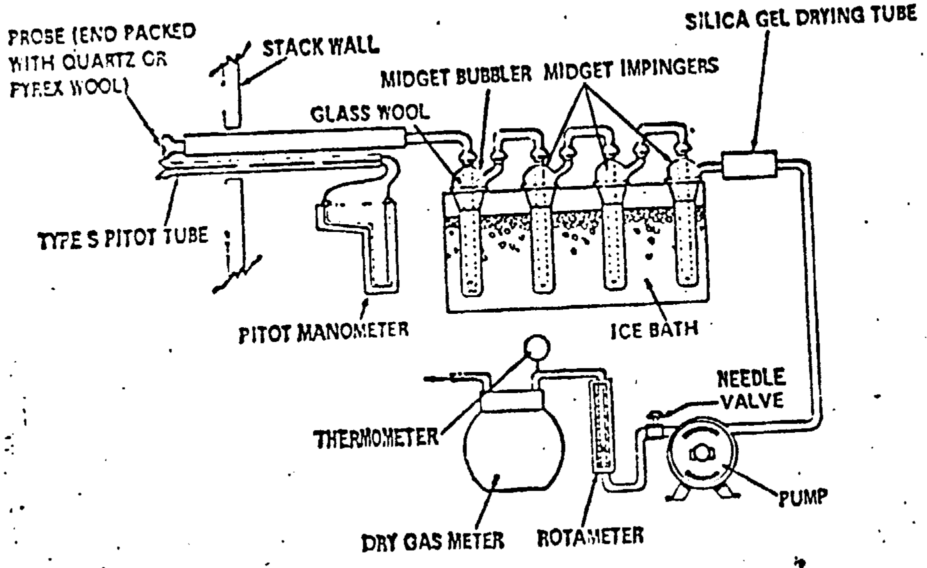

Figure 6-1. SO_2 sampling train.

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

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

2.3.3 Burettes—5 ml. and 50 ml.

2.3.4 Erlenmeyer flask—125 ml.

3. *Reagents.*

3.1 Sampling.

3.1.1 Water—Deionized, distilled.

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

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

3.2 Sample recovery.

3.2.1 Water—Deionized, distilled.

3.2.2 Isopropanol, 80%.

3.3 Analysis.

3.3.1 Water—Deionized, distilled.

3.3.2 Isopropanol.

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

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

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

4. *Procedure.*

4.1 Sampling.

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

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

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

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

5. *Calibration.*

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

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

6. *Calculations.*

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

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

$$17.71\,\frac{°R}{\text{in. Hg}}\left(\frac{V_mP_{bar}}{T_m}\right) \qquad \text{equation 6-1}$$

where:

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

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

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

T_m=Average dry gas meter temperature, °R.

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

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

6.2 Sulfur dioxide concentration.

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

where:

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

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

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

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

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

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

V_a=Volume of sample aliquot titrated, ml.

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

7. *References.*

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

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

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

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

METHOD 7—DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxide, are measure colorimetrically using the phenoldisulfonic acid (PDS) procedure.

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

2. *Apparatus.*

2.1 Sampling. See Figure 7-1.

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

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

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

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

2.1.5 Vacuum line—Tubing capable of withstanding a vacuum of 3 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.

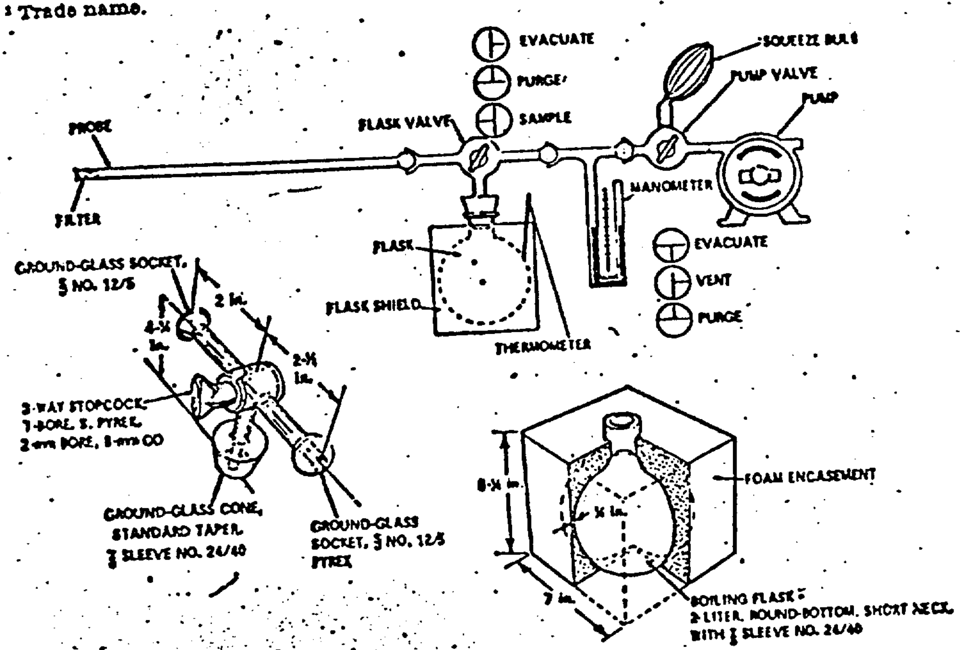

Figure 7-1. Sampling train, flask valve, and flask.

2.2.3 Glass wash bottle.

2.3 Analysis.

2.3.1 Steam bath.

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

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

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

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

2.3.6 Spectrophotometer—To measure absorbance at 420 nm.

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

2.3.8 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 (1*N*)—Dissolve 40 g. NaOH in distilled water and dilute to 1 liter.

3.2.2 Red litmus paper.

3.2.3 Water—Deionized, distilled.

3.3 Analysis.

3.3.1 Fuming sulfuric acid—15 to 18% by weight free sulfur trioxide.

3.3.2 Phenol—White solid reagent grade.

3.3.3 Sulfuric acid—Concentrated reagent grade.

3.3.4 Standard solution—Dissolve 0.5495 g. potassium nitrate (KNO_3) in distilled water and dilute to 1 liter. For the working standard solution, dilute 10 ml. of the resulting solution to 100 ml. with distilled water. One ml. of the working standard solution is equivalent to 25 μg. nitrogen dioxide.

3.3.5 Water—Deionized, distilled.

3.3.6 Phenoldisulfonic acid solution—Dissolve 25 g. of pure white phenol in 150 ml. concentrated sulfuric acid on a steam bath. Cool, add 75 ml. fuming sulfuric acid, and heat at 100° C. for 2 hours. Store in a dark, stoppered bottle.

4. *Procedure.*

4.1 Sampling.

4.1.1 Pipette 25 ml. of absorbing solution into a sample flask. Insert the flask valve stopper into the flask with the valve in the "purge" position. Assemble the sampling train as shown in Figure 7-1 and place the probe at the sampling point. Turn the flask valve and the pump valve to their "evacuate" 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 5 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 (1*N*) 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 5 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 ±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 (1*N*) 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 μg. NO_2 per sample versus absorbance.

6. *Calculations.*

6.1 Sample volume.

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[1] Trade name.

$$V_{sc}=\frac{T_{std}(V_f-V_a)}{P_{std}}\left(\frac{P_f}{T_f}-\frac{P_i}{T_i}\right)=\left(17.71\ \frac{{}^{\circ}R}{in.\ H_g}\right)(V_f-25\ ml.)\left(\frac{P_f}{T_f}-\frac{P_i}{T_i}\right)\quad \text{Equation 7-1}$$

where:

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

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

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

V_f—Volume of flask and valve, ml.

V_a—Volume of absorbing solution, 25 ml.

P_f—Final absolute pressure of flask, inches Hg.

P_i—Initial absolute pressure of flask, inches Hg.

T_f—Final absolute temperature of flask, °R.

T_i—Initial absolute temperature of flask, °R.

6.2 Sample concentration. Read μg. NO_2 for each sample from the plot of μg. NO_2 versus absorbance.

$$C=\left(\frac{m}{V_{sc}}\right)\left(\frac{\frac{1\ lb.}{cu.\ ft.}}{1.6\times10^{7}\frac{\mu g.}{ml.}}\right)=\left(6.2\times10^{-8}\frac{lb./s.c.f.}{\mu g./ml.}\right)\left(\frac{m}{V_{sc}}\right)\quad \text{equation 7-2}$$

where:

C—Concentration of NO_x as NO_2 (dry basis), lb./s.c.f.

m—Mass of NO_2 in gas sample, μg.

V_{sc}—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–1608–60, 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[1] 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[1] 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 ½-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° F., dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume.

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

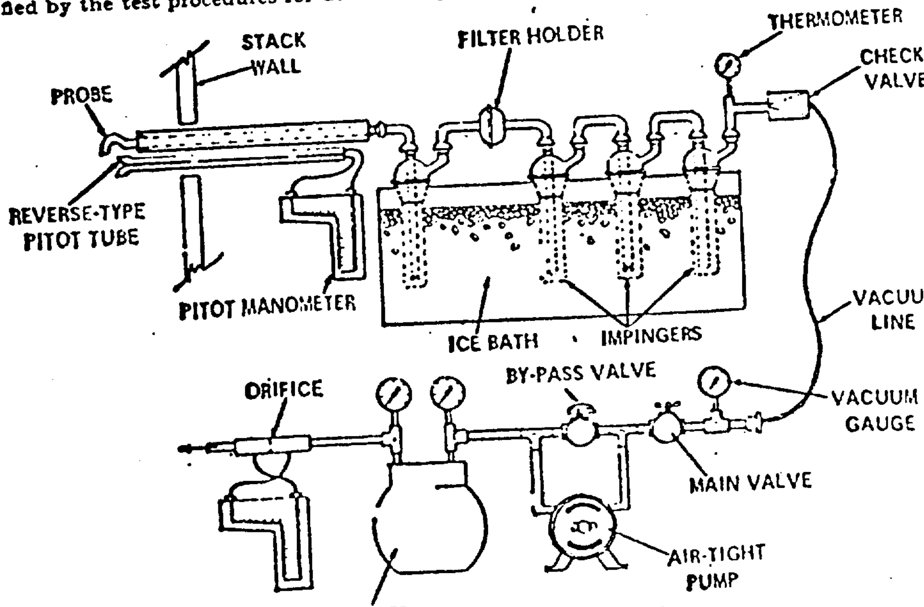

Figure 8-1. Sulfuric acid mist sampling train.

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 ±0.05 g.

2.3.6 Dropping bottle—to add indicator solution.

3. *Reagents.*

3.1 Sampling.

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

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

3.1.3 Water—Deionized, distilled.

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

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

3.1.6 Crushed ice.

3.2 Sample recovery.

3.2.1 Water—Deionized, distilled.

3.2.2 Isopropanol, 80%.

3.3 Analysis.

3.3.1 Water—Deionized, distilled.

3.3.2 Isopropanol.

3.3.3 Thorin indicator—1-(o-arsonophenylazo)-2-naphthol-3, 6-disulfonic acid, d sodium salt (or equivalent). Dissolve 0.2 in 100 ml. distilled water.

3.3.4 Barium perchlorate (0.01*N*)—solve 1.95 g. of barium perchlorate $(CO_4)_2 \cdot 3\ H_2O]$ in 200 ml. distilled water and dilute to 1 liter with isopropanol. Standardize with sulfuric acid.

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

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. 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 Hg vacuum. A leakage rate not in excess of 0.02 c.f.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° F. or less.

4.1.3 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

---

[1] Trade name.

pid adjustment of the sampling rate with-
t other computations. APTD-0576 details
e procedure for using these nomographs.
t the conclusion of each run, turn off the
ump 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.

PLANT ____________
LOCATION ____________
OPERATOR ____________
DATE ____________
RUN NO. ____________
SAMPLE BOX NO. ____________
METER BOX NO. ____________
METER ΔH@ ____________
C FACTOR ____________

SCHEMATIC OF STACK CROSS SECTION

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

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE (P_s), in. Hg	STACK TEMPERATURE (T_s), °F	VELOCITY HEAD (ΔP_s),	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER (ΔH), in. H_2O	GAS SAMPLE VOLUME (V_m), ft³	GAS SAMPLE TEMPERATURE AT DRY GAS METER		SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
							INLET ($T_{m_{in}}$), °F	OUTLET ($T_{m_{out}}$), °F		
							Avg.	Avg.		
TOTAL							Avg.			
AVERAGE										

Figure 8-2. Field data.

4.2 Sample recovery.

4.2.1 Transfer the isopropanol from the first impinger to a 250 ml. graduated cylinder. Rinse the probe, first impinger, and all connecting glassware before the filter with 80% isopropanol. Add the rinse solution to the cylinder. Dilute to 250 ml. with 80% isopropanol. Add the filter to the solution, mix, and transfer to a suitable storage container. Transfer the solution from the second and third impingers to a 500 ml. graduated cylinder. Rinse all glassware between the filter and silica gel impinger with deionized, distilled water and add this rinse water to the cylinder. Dilute to a volume of 500 ml. with deionized, distilled water. Transfer the solution to a suitable storage container.

4.3 Analysis.

4.3.1 Shake the container holding isopropanol and the filter. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample. Pipette a 100 ml. aliquot of sample into a 250 ml. Erlenmeyer flask and add 2 to 4 drops of thorin indicator. Titrate the sample with barium perchlorate to a pink end point. Make sure to record volumes. Repeat the titration with a second aliquot of sample. Shake the container holding the contents of the second and third impingers. Pipette a 25 ml. aliquot of sample into a 250 ml. Erlenmeyer flask. Add 100 ml. of isopropanol and 2 to 4 drops of thorin indicator. Titrate the sample with barium perchlorate to a pink end point. Repeat the titration with a second aliquot of sample. Titrate the blanks in the same manner as the samples.

5. *Calibration.*

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

5.2 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., 29.92 inches Hg) by using Equation 8–1.

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

equation 8–1

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.
V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.
T_{std} = Absolute temperature at standard conditions, 530° R.
T_m = Average dry gas meter temperature, °R.
P_{bar} = Barometric pressure at the orifice meter, inches Hg.
ΔH = Pressure drop across the orifice meter, inches H_2O.
13.6 = Specific gravity of mercury.
P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.2 Sulfuric acid concentration.

$$C_{H_2SO_4}=\left(1.08\times10^{-4}\frac{\text{lb.-l.}}{\text{g.-ml.}}\right)\frac{(V_t-V_{tb})(N)\left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}}$$

equation 8–2

$C_{H_2SO_4}$ = Concentration of sulfuric acid at standard conditions, dry basis, lb./cu. ft.
1.08×10^{-4} = Conversion factor including the number of grams per gram equivalent of sulfuric acid (49 g./g.-eq.), 453.6 g./lb., and 1,000 ml./l., lb.-l./g.-ml.
V_t = Volume of barium perchlorate titrant used for the sample, ml.
V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.
N = Normality of barium perchlorate titrant, g.-eq./l.
V_{soln} = Total solution volume of sulfuric acid (first impinger and filter), ml.
V_a = Volume of sample aliquot titrated, ml.
$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft., see Equation 8–1.

6.3 Sulfur dioxide concentration.

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

equation 8–3

where:

C_{SO_2} = Concentration of sulfur dioxide at standard conditions, dry basis, lb./cu. ft.
7.05×10^{-5} = Conversion factor including the number of grams per gram equivalent of sulfur dioxide (32 g./g.-eq.) 453.6 g./lb., and 1,000 ml./l., lb.-l./g.-ml.
V_t = Volume of barium perchlorate titrant used for the sample, ml.
V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.
N = Normality of barium perchlorate titrant, g.-eq./l.
V_{soln} = Total solution volume of sulfur dioxide (second and third impingers), ml.
V_a = Volume of sample aliquot titrated, ml.
$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft., see Equation 8–1.

7. *References.*

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

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

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

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, 4516/59a.

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.

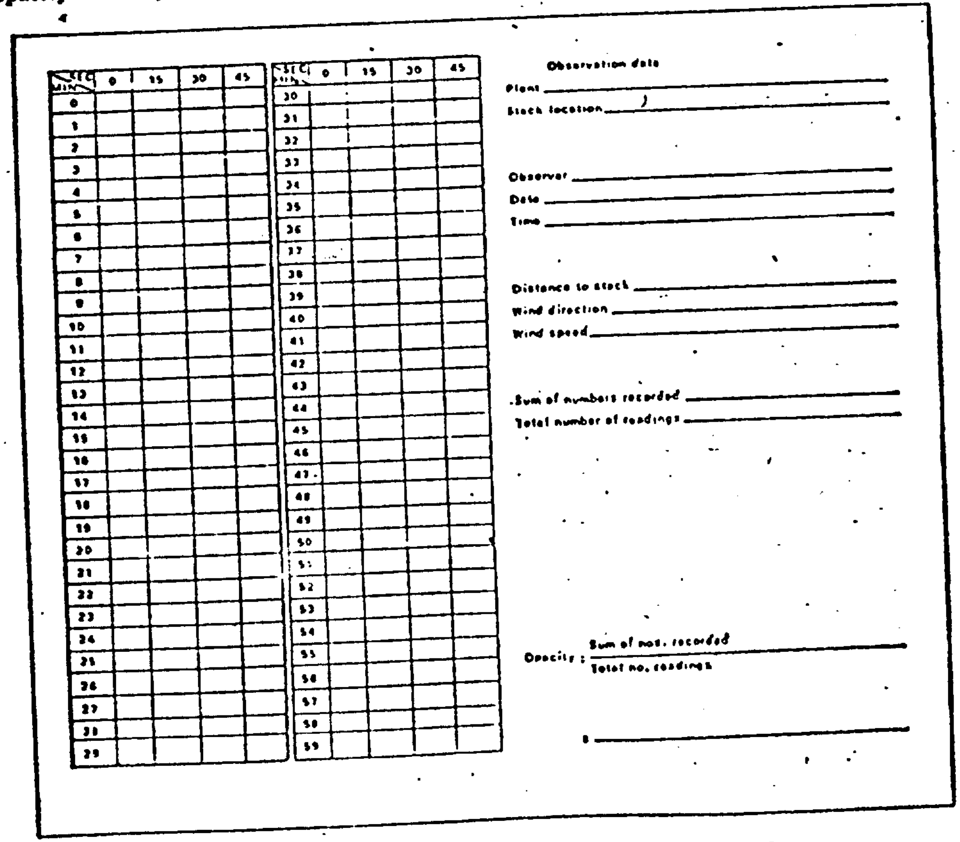
4. *Calculations.*

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.



SEC / MIN	0	15	30	45	SEC / MIN	0	15	30	45
0					30				
1					31				
2					32				
3					33				
4					34				
5					35				
6					36				
7					37				
8					38				
9					39				
10					40				
11					41				
12					42				
13					43				
14					44				
15					45				
16					46				
17					47				
18					48				
19					49				
20					50				
21					51				
22					52				
23					53				
24					54				
25					55				
26					56				
27					57				
28					58				
29					59				

Observation data

Plant ____________________

Stack location ____________________

Observer ____________________

Date ____________________

Time ____________________

Distance to stack ____________________

Wind direction ____________________

Wind speed ____________________

Sum of numbers recorded ____________________

Total number of readings ____________________

Opacity : $\frac{\text{Sum of nos. recorded}}{\text{Total no. readings}}$

% ____________________

Figure 9-1. Field data.

[FR Doc.71-18024 Filed 12-22-71;8:45 am]

APPENDIX E

LABORATORY REPORT

APPENDIX E

LABORATORY REPORT
ANALYSIS BY WET CHEMISTRY

Run No. 1

Plant N.L. Industries, McCook, Illinois

Date February 9, 10, 1972

BACK HALF IMPINGER WATER RESIDUE

Constituents	Aqueous, μg	Oranic, μg	Total, μg
SO_4	8,200	15,000	23,200
Cl	70	< 30	100
NH_4	3,200	< 100	3,300
NO_3	1,700	< 100	1,800
pH(a)	3.5	2.4	---
Total Residue Mass for which anions and pH were determined	13,800(b)	27,600	41,400

(a) Values are pH units.
(b) Water blank subtracted.

Plant N.L. Industries, McCook, Illinois

Date February 9, 10, 1972

ACETONE PROBE WASH RESIDUE

Constituent	Run 1, µg	Run 2, µg	Run 3, µg
SO_4	< 500	< 500	< 500
Cl	260	520	400
NH_4	< 100	< 100	< 100
NO_3	< 100	< 100	< 100
pH(a)	5.0	5.2	5.1
Total Residue Mass from which anions and pH were determined(b)	3300 µg	7100 µg	9600 µg

(a) Values are pH units

(b) Acetone blank subtracted.

Note: < symbol indicates minimal detection limit.

Plant N.L. Industries, McCook, Illinois

Date February 9 and 10, 1972

FRONT HALF LABORATORY REPORT
ANALYSIS BY ATOMIC ABSORPTION

Run	Probe Acetone Wash Residue, μg(a)	Pb, μg per Residue, μg	Pb, μg per Total Residue	Filter Catch, μg	Pb, μg per Split Filter Sample, μg	Pb, μg per Total Filter Catch
1	3,300	0.103	340	9,700	0.155	1500
2	7,100	0.0785	560	8,800	0.154	1350
3	9,600	0.0469	450	7,000	0.164	1150

(a) Acetone blank subtracted.

Note: See analysis flow chart for procedure (E-7).

Plant N.L. Industries, McCook, Illinois

Date February 9, 10, 1972

BACK HALF LABORATORY REPORT
ANALYSIS BY ATOMIC ABSORPTION

Run	Back Half Impinger Water Residue	Pb, ug / Residue, μg	Pb, ug / Total Residue
1	Aqueous residue, μg 13,800[(a)]	0.0376	520
	Organic residue, μg 27,600	0.0091	250

(a) Water blank subtracted.

Plant N.L. Industries, McCook, Illinois

Date February 9, 10, 1972

LABORATORY REPORT

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

	Filter Analysis		
Element	Run 1	Run 2	Run 3
Fe	100	100	60
Zn	< 20	< 20	< 20
Pb(a)	---	---	---
Cu	< 2	< 2	< 2
Se	---	---	---
Sn	40	30	40
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	60	40
Bi	< 6	< 6	< 6
Cd	6	6	2
Sr	10	6	6
Be	< 0.2	< 0.2	< 0.2
Hg	< 60	< 60	< 60
Sb	60	60	40
Total Catch, μg	9,700	8,800	7,000

(a) Pb determined by Atomic Absorption.

NOTE: Filter blank data not available.
< symbol indicates minimal detection limits.

Plant N.L. Industries, McCook, Illinois

Date February 9, 10, 1972

LABORATORY REPORT
OPTICAL EMISSION SPECTROSCOPY TRACE METAL
(ug of element for total sample)

Element	Probe Wash Residue			Impinger Water Residue Run 1		Water Blank
	Run 1	Run 2	Run 3	aqueous	organic	
Be	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	
Cd	5.	5.	< 5.	< 5.	< 5.	
Ag	< 10.	< 10.	< 10.	< 10.	< 10.	
V	< 1.	< 1.	< 1.	< 1.		
Mn	< 1.	< 1.	< 1.	< 1.	< 1.	0.05
Ni	1.	1.	1.	3.	3.	
Sb	100.	50.	10.	30.	10.	
Cr	< 1.	< 1.	< 1.	10.	10.	0.03
Zn	< 10.	10.	10.	10.	< 10.	0.1
Cu	1.	1.	1.	1.	1.	
Se(a)	---	---	---	---	---	---
B	1.	< 1.	2.	10.	< 1.	
F(a)	---	---	---	---	---	---
Li	< 30.	< 30.	< 30.	< 30.	< 30.	
Ag	0.3	0.3	0.1	0.3	0.5	
Sn	10.	10.	< 5.	10.	5.	
Fe	30.	40.	40.	40.	50.	0.75
Sr	< 5.	< 5.	< 5.	< 5.	< 5.	0.05
Na	< 30.	< 30.	< 30.	< 30.	< 30.	
K	10.	10.	10.	10.	10.	
Ca	10.	10.	5.	10.	10.	0.2
Si	30.	30.	30.	100.	10.	0.15
Mg	3.	4.	4.	10.	10.	
Ba	1.	1.	1.	1.	1.	
Al	1.	3.	1.	1.	1.	
Residue Weight,	3,300(b)	7,100(b)	9,600(b)	13,800(c)	27,600(c)	

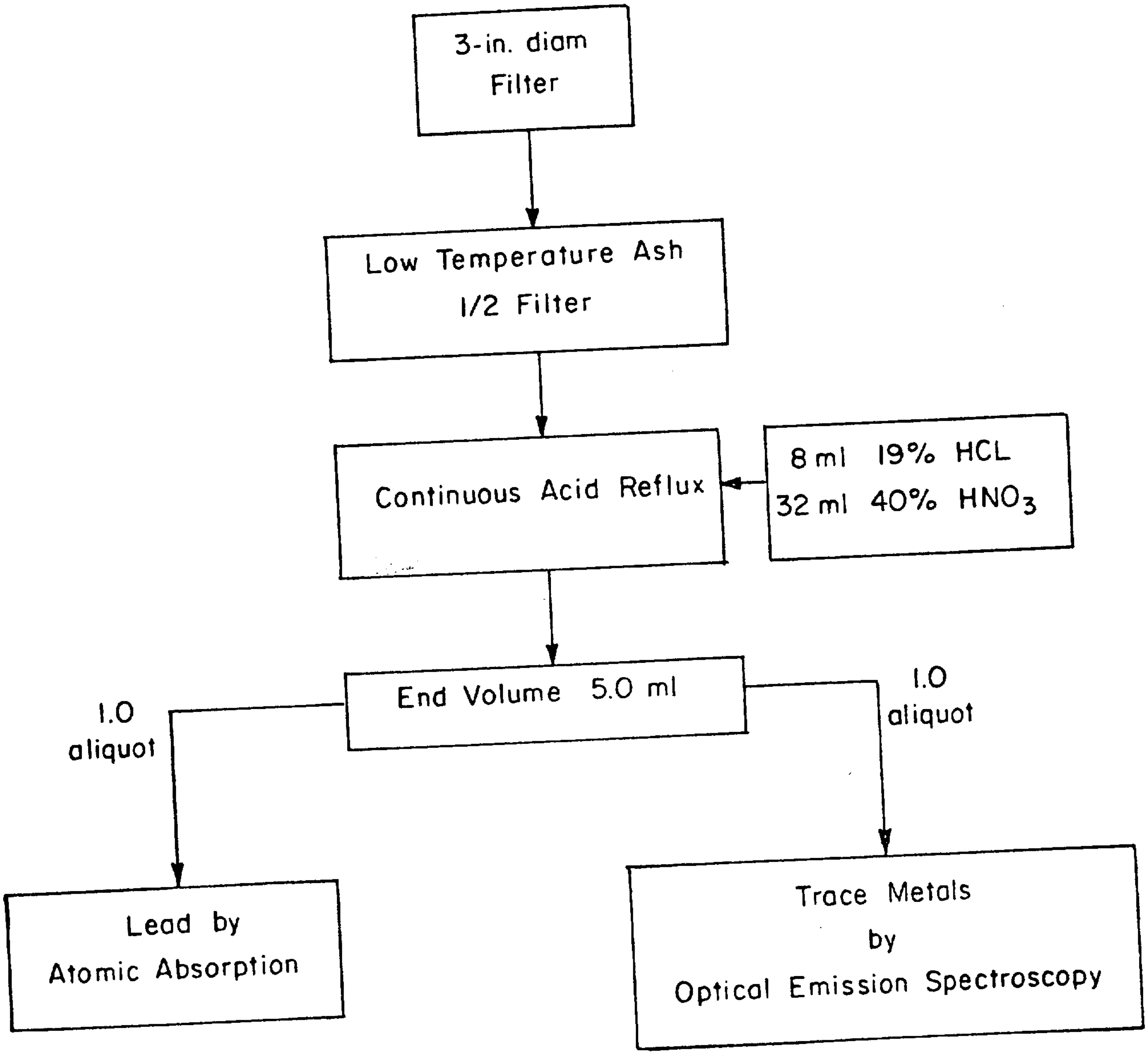
(a) Not detectable by method used.

(b) Acetone blank subtracted.

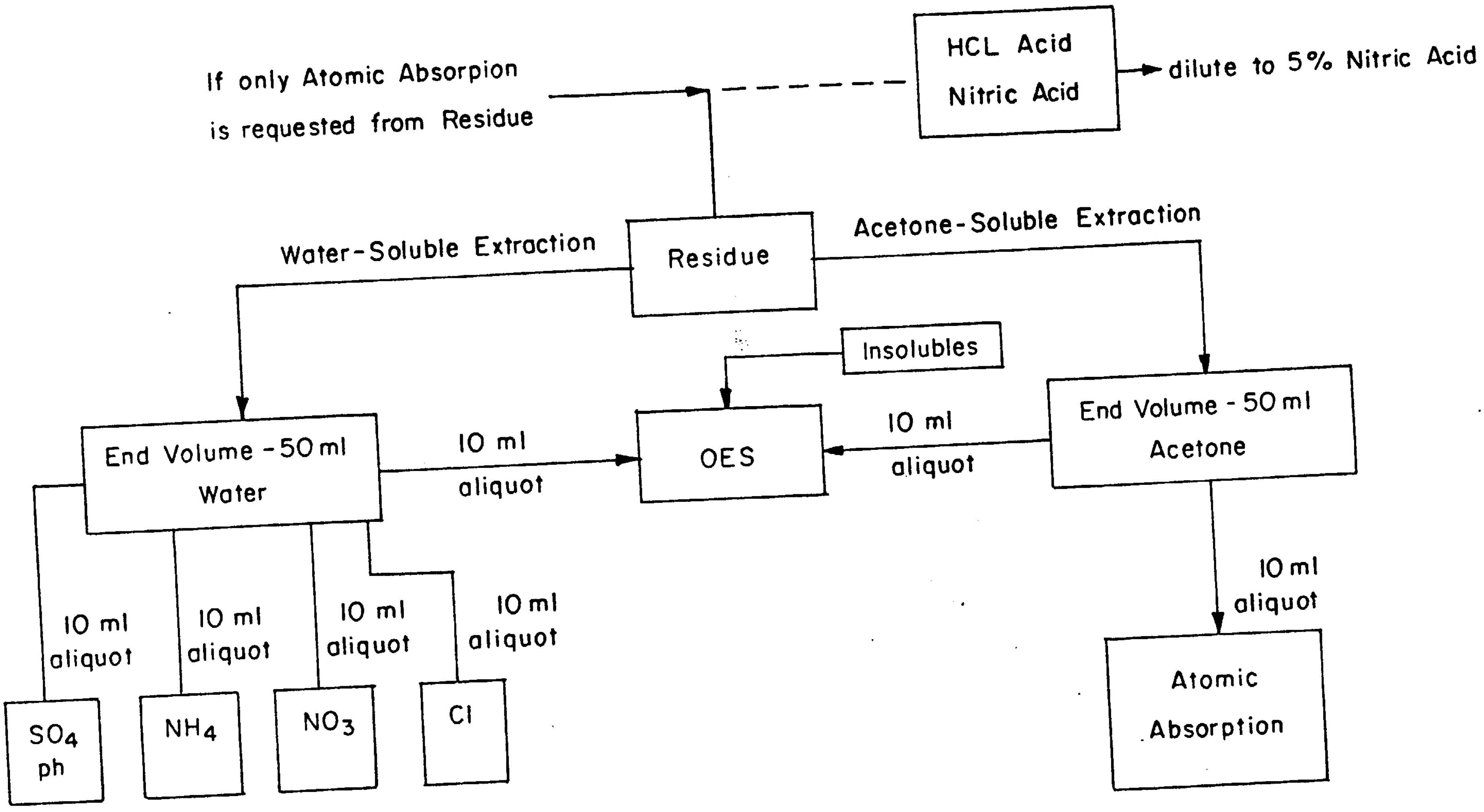
(c) Water blank subtracted.

Note: < symbol indicates minimal detection limits.

FILTER ANALYSIS FLOW DIAGRAM



RESIDUE ANALYSIS FLOW DIAGRAM



Plant N.L. Industries, McCook, Illinois

EXAMPLE 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	18
E - Absorbance from Atomic Absorbance Tables, Based on Percent Absorption	.0862
F - Lead, μg/ml of Diluted Aliquot(a)	6.0
G - Lead, μg/Split Filter Sample	750
H - Lead, μg/Total Filter	1500

Example Computation

$C \times A \times F = G$

$25 \times 5.0 \times 6.0 = 750$

$2 \times G = H$

$2 \times 750 = 1500$

(a) Determined from calibration curve; based on 0.862 absorbance.

CHEMICAL METHODS USED TO ANALYZE EPA RESIDUES

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 if measurements 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

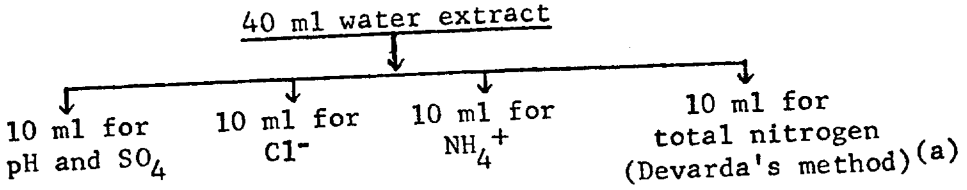
(1) Obtain "water-soluble extract" by extracting residue in beaker with warm water. Adjust volume to 50 ml in a volumetric flask. Remaining materials are "water insoluble" residues.

(2) Obtain "acetone-soluble extract" by extracting "water insoluble" residue in beaker with acetone. Remaining materials are "acetone insoluble" residues. Police beaker thoroughly, making sure that all material is removed, even though insoluble. Adjust volume to 50 ml in a volumetric flask.

(3) Transfer 10 ml of the "water-soluble extract" to crucible for OES. Add 10 ml of the corresponding "acetone-soluble extract" to the 10 ml of "water-soluble extract", being sure to shake the flask 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-soluble extract", filter on Millipore filter, if necessary, and transfer to tared aluminum dish. Dry down at ~100 C. Reweigh vessel and record weight as "acetone-soluble". Multiply result by 2 to convert to original sample.

(5) Proceed with chemical analysis of the "water-soluble extract" as follows:



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[a] NO_3^- will be calculated from the difference between total nitrogen and NH_4^+

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

(6) "Water Insoluble Residue" may be obtained in the following manner, if needed. Wash the remaining 15 ml of acetone-soluble extract into the same dish used in Step (4). Dry down at ~100 C. Reweigh and record weight as "water insoluble, acetone soluble". Multiply by 5/4 to convert to original sample. (If a filtration step was included in Step (4) weigh the filter pad, air dry after filtration is complete, reweigh to obtain weight of retained residue, and add to "water insoluble, acetone soluble" total before multiplying 5/4).

(7) Wash the suspended matter remaining in the 50 ml volumetric flask after acetone extraction into a tared aluminum dish. Dry down at ~100 C. Reweigh and record weight as "water insoluble, acetone insoluble". Total "water insoluble" may be obtained by adding "water insoluble, acetone insoluble" and "water insoluble, acetone insoluble" results.

Preparation of EPA Filter Samples

Filters may be prepared for chemical analysis as follows:

(1) Remove 1/2 of the filter 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) Stop the volumetric flask, mix well, and proceed with the chemical determinations as described for the residue samples.

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

Analysis for Sulfate ($SO_4^=$)[a]

(The 10 ml of 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.

(3) Heat to near boiling temperatue, add 10 ml of 10 percent $BaCl_2$ solution, stir well, and digest at near boiling temperature for an hour.

(4) Remove from the hot-plate and cool to room temperature. Allow the solution to stand at room temperature at least 2 hours - preferably overnight.

(5) Filter off the precipitated $BaSO_4$ through a tight filter paper (Whatman No. 42 or equivalent). Wash the paper and precipitate with hot deionized water until the washings are free of chloride.

(6) Place the paper and precipitate in a crucible, dry under a gas hot-plate, and then heat in a muffle furnace for at least an hour at a temperature of 900 C.

(7) Remove the crucibles from the muffle furnace, cool in a desiccator, and weigh as $BaSO_4$. Calculate SO_4 by multiplying by the factor 0.4116. Multiply this result by 5 to get total amount of $SO_4^=$ in the residue.

(a) For residue analysis, this determination is made on the 10 ml aliquot used for pH measurement; Step (5)-Preparation of EPA Residue Samples.

For filter analysis, 10 ml of the 50 ml volume, Step (3)-Preparation of EPA Filter Samples, is used for this determination.

APPENDIX F

TEST LOG

APPENDIX F

TEST LOG

February 7, 1972	Arrive on test site. Construct shelter.
February 8, 1972	Set up equipment. Make preliminary velocity traverse. Drain water from duct. Make preliminary SO_2 run.
February 9, 1972	Make two runs.
February 10, 1972	Make one run. Pack equipment.
February 11, 1972	Ship equipment. Return home.

APPENDIX G

RELATED REPORTS

APPENDIX G

RELATED REPORTS

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

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

(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

G. William Keigley, Senior Technician

William C. Baytos, Scientist

Harold G. Leonard, Senior Technician

Administrative Support

Richard E. Barrett, Senior Mechanical Engineer

John M. Allen, Division Chief

Irene Whitener, Secretary

APPENDIX I

COMPLETE OPERATIONAL SCHEDULE

COMPLETE OPERATIONAL SCHEDULE

Run No.	1	2	3
Date (1972)	2/9	2/9	2/10
Start Time End Time	11:00 am 1:30 pm	2:15 pm 4:45 pm	9:20 am 11:50 am
Baghouse temperature, inlet, F	200	210	160
Baghouse draft, in. H_2O	9.9	8.8	9.3
Furnace, temperature, F	1825	1850	1700
Battery plates fed, scoops	36	36	39
Battery plates fed, lb(a)	25,200	25,200	27,300
Oxides fed, scoops	1	1	1
Oxides fed, lbs(a) (b)	700	700	700
Lead tapped, tons	5.2	17.7	5.2
Lead tapped, tons/hr	2.1	7.1	2.1
Lead tapped, days average, ton/hr	2.4	2.4	2.4
Slag tapped, lb	5,000	3,000	3,000
Slag tapped, lb/hr	2,000	1,200	1,200
Slag tapped, days average, lb/hr	1,550	1,550	1,550

(a) At 700 lb/scoop, based on N.L. Industries estimate.

(b) Not including baghouse dust fed by conveyor.

APPENDIX J

OPERATIONAL RESULTS

OPERATIONAL NOTES

Plant N. L. Industries, McCook, Illinois

Date February 8, 1972

The furnace was started early Monday (2/7/72) and was operating normally by 4:00 p.m. The baghouse cleans only gas from the reverberatory furnace flue. About 2 inches of water in the duct on 2/7/72.

Slag cast in 1000 lb slabs
Slag tapped continuously
Lead tapped intermittently
Feed not measured; about 700 lb/load
Feed is battery plates or oxides from cyclones or ductwork
Feed opening 27 in. wide, 6 in. high
The number of loads fed to the furnace are counted.

Observations

Note: Battery plates were loaded steadily through all tests. Time of feeding battery plates are listed separately at the end of Observations.

2/9/72

8:30 am Sampling duct is dry
Cleaning out dust lines under cyclones
Not tapping slag
Slag mold is empty
Furnace exhaust temperature, 1600 F
Baghouse temperature (inlet), 160 F
Baghouse pressure (outlet), 9.3 in. H_2O

8:40 am Feed counter on 3
Chart on furnace temperature shows a low of 900 F while cleaning ducts and a high of 1750 F.

9:08 Feed counter on 8

9:29 Tap 6.24 tons lead

9:35 Load oxides, counter on 11

10:23 Load oxides
Furnace temperature, 1650 F

10:20 Start tapping slag

10:25 Load oxides
Baghouse temperature, 170 F
Pressure, 9.3 in. H_2O

10:30 Change slag pots

Observations (Cont'd)

2/9/72 (Cont'd)

10:58 am	Change slag pots Start particulate sampling
11:11	Tap 5.20 tons lead
11:20	Change slag pot
12:04 pm	Change slag pot
12:07	Load oxides
12:10	Start tapping slag after stop at 12:04
12:28	Slag pot changed
1:05	Slag pot changed
1:16	Furnace, 1825 F
1:18	Slag pot changed, stop tapping slag
1:30	First particulate run end
1:35	Start tapping slag
1:43	Change slag pot, stop tapping slag
1:52	Start tapping slag Furnace, 1740 F Baghouse, 200 F, 9.9 in. H_2O vacuum
2:05-2:11	Change slag pot
2:24	Change slag pots, stop tapping
2:44	Tap 10.58 tons lead
3:07	Start tapping slag
3:38-4:05	Change slag pots
4:05	Load oxides
4:14-4:25	Change slag pot
4:34	Tap 7.11 tons lead Furnace, 1850 F
4:45	End of particulate Run 2
5:05	End observations Slag pot 1/2 full Furnace, 1800 F Baghouse, 210 F, 8.8 in. draft.

2/10/72

8:00 am	Cleanout dust under cyclones. Operator reports this is done once a day
8:40	Load oxides

Observations (Cont'd)

2/10/72 (Cont'd)

8:49 am Tap 4.16 tons lead

8:54 Furnace temperature, 1650 F

9:01 Furnace temperature, 1600 F
Baghouse temperature, 135 F
Baghouse pressure, 9.7 in. H_2O

9:20 Start tapping slag
Start particulate Run 3

9:49 Load oxides

9:52 Load oxides

9:55 Change slag pots

10:26-10:32 Change slag pot

10:32 Baghouse temperature, 160 F
Baghouse pressure, 9.3 in. H_2O draft

10:47 Tap 5.20 tons lead

11:12 Change slag pot, stop tapping until 11:48

11:32 Furnace temperature, 1700 F
Baghouse temperature, 175 F
Baghouse pressure, 8.1 in.

Outside temperature was subzero for all three tests.

11:50 End observations
34.3 tons lead tapped, 5:00 pm, 2/9/72

56.43 ton had been tapped betwen 8:00 am, 2/9/72 and 8:00 am, 2/10/72. 37,000 lb slag in same interval.

Battery plates fed into furnace:

2/9/72

11:02 am
11:04
11:06
11:08
11:13
11:20
11:25
11:28
11:32
11:36
11:40
11:43
11:45
11:53
11:55
11:57

(Cont'd)

12:08 pm
12:10
12:15
12:18
12:21
12:23
12:37
12:40
12:49
1:11
1:16
1:18
1:21
1:23
1:25
1:26

(Cont'd)

2:15 pm
2:19
2:22
2:26
2:28
2:50
2:51
2:53
2:57
2:59
3:01
3:03
3:05
3:08
3:26
3:30

(Cont'd)

3:35 pm
3:38
3:40
3:42
3:45
3:47
3:49
3:53
4:08
4:12
4:14
4:15
4:16
4:25
4:30
4:32

(Cont'd)

4:34 pm
4:35
4:45

2/10/72

9:26 am
9:30
9:33
9:45
9:46
9:49
9:52
9:53
9:55
9:57
10:01

(Cont'd)

10:04 am
10:07
10:20
10:22
10:24
10:27
10:29
10:30
10:31
10:32
10:35
10:38
10:41
10:44
10:47
10:48

(Cont'd)

11:05
11:08
11:10
11:28
11:31
11:32
11:34
11:36
11:42
End particulate run (11:50).

APPENDIX K

SUMMARY OF RESULTS

APPENDIX K

SUMMARY OF RESULTS

Run Number	1	2	3	Average
Date	2/9/72	2/9/72	2/10/72	---
Test Time - Minutes	150	150	150	150
Lead Production - TPH	2.4	2.4	2.4	2.4
Stack Effluent				
Flow rate - DSCFM	23,480	22,600	19,940	22,007
Flow rate - DSCF/ton	9,800	9,400	8,300	9,167
Temperature - ^{o}F	124	132	117	124
Water vapor - Vol. %	4.0	4.6	4.2	4.3
CO_2 - Vol. % dry	2.4	2.5	2.0	2.3
O_2 - Vol. % dry	18.2	18.0	18.2	18.1
CO - Vol. % dry	< 0.1	< 0.1	< 0.1	< 0.1
CO - lb/hr	< 102	< 98	< 87	< 96
SO_2 - ppm dry	2,060	2,111	1,930	2,034
SO_2 - lb/hr	480	473	381	445
Visible emissions - % opacity				
Particulate Emissions				
Probe and filter catch				
gr/DSCF	0.0024	0.0033	0.0042	0.0033
gr/ACF	0.0021	0.0027	0.0036	0.0028
lb/hr	0.4890	0.6350	0.7205	0.6148
lb/ton lead	0.2038	0.2646	0.3002	0.2562
Total Catch				
gr/DSCF	0.0118	0.0131	0.0164	0.0138
gr/ACF	0.0100	0.0109	0.0140	0.0860
lb/hr	2.3737	2.5398	2.7950	2.5695
lb/ton lead	0.9890	1.0583	1.1646	1.071
Lead Emissions				
Probe and filter catch				
gr/DSCF	0.00034	0.00039	0.00041	0.00038
gr/ACF	0.00029	0.00033	0.00035	0.00032
lb/hr	0.0692	0.0763	0.0695	0.0717
lb/ton lead	0.0288	0.0318	0.0289	0.0298
Total Catch				

Essentially the same as probe and filter.