

10445

FINAL OK

COAL PREPARATION PLANT EMISSION TESTS

Note: This is a reference cited in AP 42, *Compilation of Air Pollutant Emission Factors, Volume I Stationary Point and Area Sources*. AP42 is located on the EPA web site at www.epa.gov/ttn/chief/ap42/

The file name refers to the reference number, the AP42 chapter and section. The file name "ref02_c01s02.pdf" would mean the reference is from AP42 chapter 1 section 2. The reference may be from a previous version of the section and no longer cited. The primary source should always be checked.

TEST NO. 73-CCL-1
U.S. STEEL #50
Pineville, West Virginia

Prepared for

ENVIRONMENTAL PROTECTION AGENCY
Research Triangle Park
North Carolina 27711

Contract No 68-02-0233



SCOTT RESEARCH LABORATORIES, INC.

PLUMSTEADVILLE, PENNSYLVANIA 18949

SRL 1281 45 0972

Environmental Protection Agency
Test No. 73-CCL-1
Thomas E. Ward, Project Test Officer
U.S. Steel #50
Pineville, West Virginia, Norman R. Troxel, P.E.

September 1972

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1.0 INTRODUCTION

Scott Research Laboratories, Inc. performed source sampling tests at the U.S. Steel #50 plant in Pineville, West Virginia during the week of August 28, 1972. The plant uses an American Air Filter venturi scrubber to control the exhaust gas emissions from a coal cleaning and preparation operation.

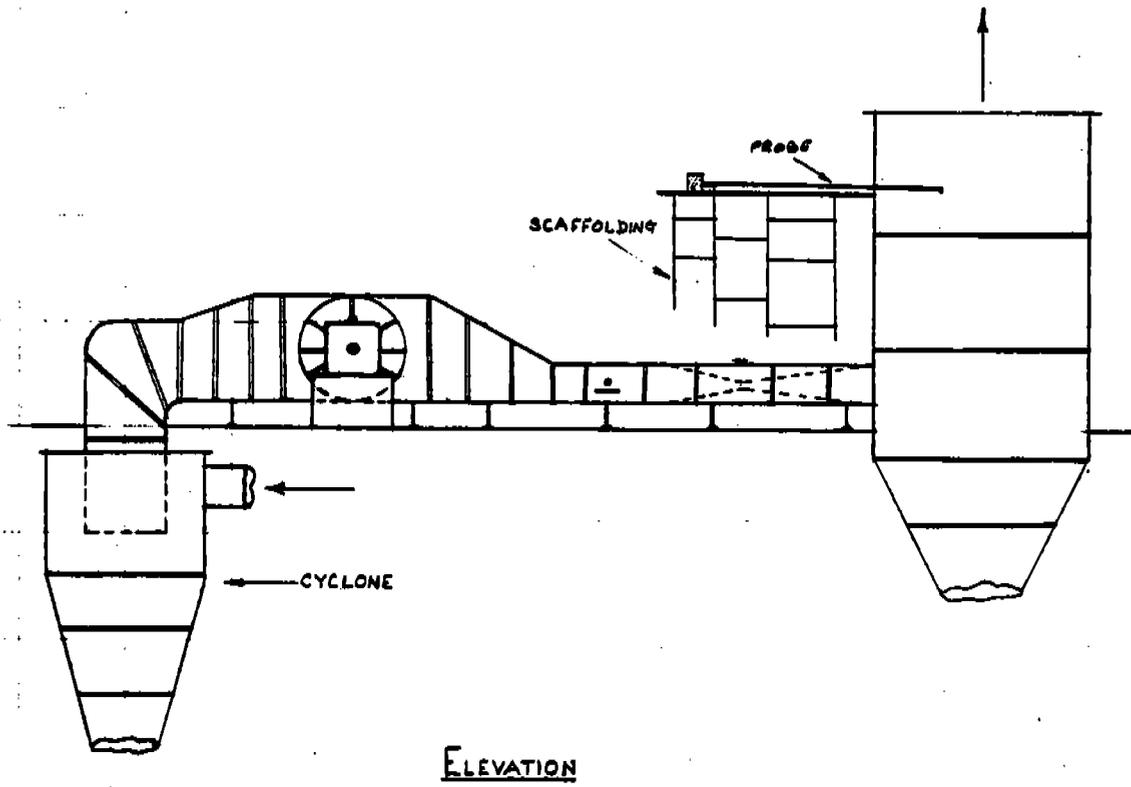
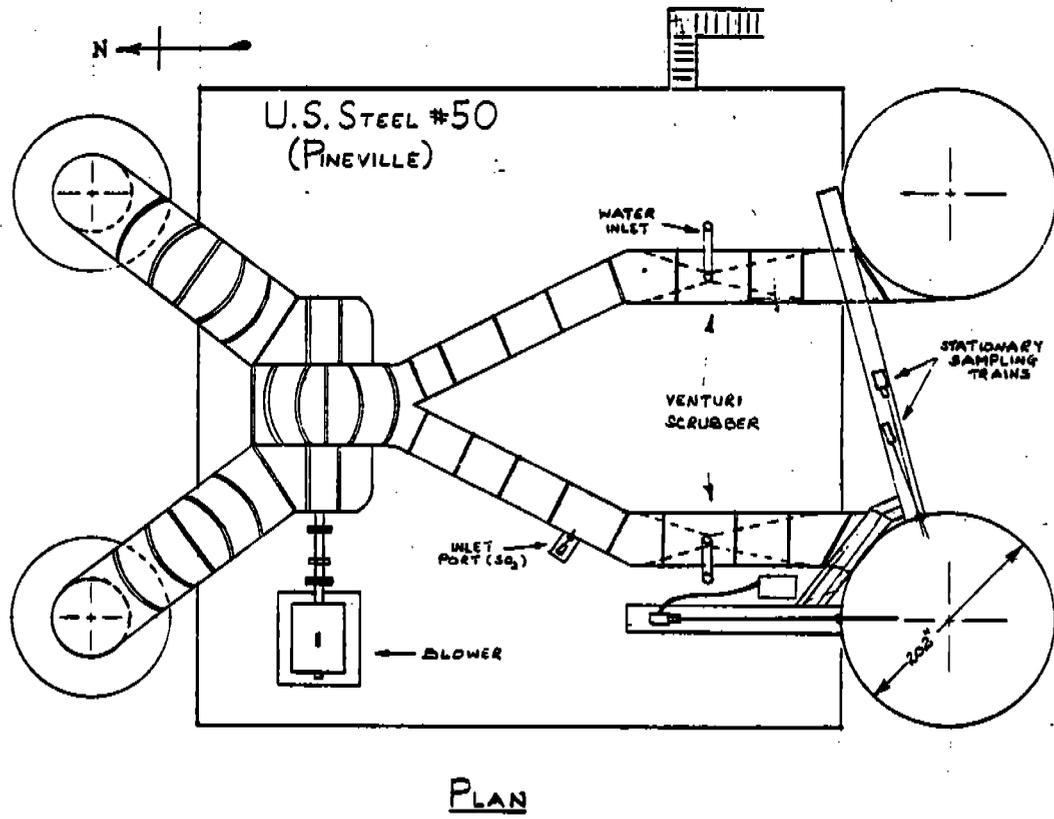
The outlet exhaust gases, as they were being emitted to the atmosphere, were sampled and analyzed for the determination of total particulate loading, oxides of nitrogen, sulfur dioxide, carbon dioxide, carbon monoxide, and oxygen concentrations. Samples of the exhaust gases before they entered the venturi scrubber were also collected for the determination of sulfur dioxide concentration.

In addition, experimental particulate tests were performed measuring only the amount of particulate trapped in the probe and on the filter while sampling continuously at only one traverse point. In one instance the nozzle opening was directed perpendicular to the direction of the maximum flow while with the other sampling train, the nozzle was vertical to the effluent path direction of the longitudinal axis of the duct.

One stationary nozzle particulate test was performed on August 29, 1972. On August 30, 1972 two stationary nozzle particulate tests and one complete and one half of an EPA particulate tests were performed. The second half of the EPA particulate test was completed on August 31, 1972. Also, the first half of the third EPA particulate test was run on August 31, 1973, after which testing had to be stopped because the plant ran out of coal. Also on August 31, 1972, two additional stationary particulate tests were conducted. Figure 1 shows the location of the sampling points at the plant.



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FIGURE 1 SAMPLE POINT LOCATIONS



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2.0 SUMMARY OF RESULTS

A summary of test results is presented in Table 1. The particulate weights are summarized and shown in Table 2 with all of the particulate results included as Appendix A. Appendix B presents all of the gaseous results, and the raw data sheets are included as Appendix C.

From the results it can be seen that the results of the first two EPA tests agree very well while the third test gave loadings considerably lower. It is noted that the third test is actually the result of sampling on only one traverse and was made at a time when the plant was running low on coal. In fact, the test had to be stopped because the plant ran out of coal for processing. From Table 2 it appears that proportionately there was less material caught on the filter during the third test than during the first two tests.

The moisture content recorded is somewhat low because the condensation which occurred in the 18' long probe was not taken into consideration. The true moisture content is estimated to be in the 13 to 14% range. Consequently the true isokinetic rates are approximately 6% higher than those reported.

The sulfur dioxide and nitrogen oxide values compare favorably for the first two tests. The results from the third test vary somewhat from those of the first two tests.

From visual observations it was seen that water droplets were forming around the probe and nozzle during the sample periods. The upward vector of the effluent gases is such that it will not support the weight of the water droplets, thus, it is very possible that the high particulate loadings are a result of pulling these droplets into the sample line.



TABLE 1 SUMMARY OF TEST RESULTS

	Stationary Samples					
	135° 1 8/29/72	180° 2 8/30/72	125° 3 8/30/72	180° 4 8/31/72	125° 5 8/31/72	180° 5 8/31/72
Nozzle Angle						
Run Number			3 (3)			
Sample Date	8/30/72	8/30/72 8/31/72	8/31/71	8/30/72	8/30/72	8/31/72
Sample Gas Vol., SCF.	82.13	79.16	47.04	68.60	77.64	37.71
Moisture, % (1)	7.07	8.56	6.59	13.5*	13.5*	13.5*
Stack Gas Temp., °F	130	130	130	130	130	130
Stack Gas Vel., fpm	2067	2089	2137	3490	3490	3402
Stack Gas Vol., SCFM	269,035	267,488	159,775	579,611	579,611	565,471
Particulate Collected						
Probe, Cyclone, Filter, mg.	1106.2	1101.3	409.5	740.0	139.9	993.8
Total, mg.	1136.7	1169.3	426.5	-	-	-
Particulate Concentration						
Probe, Cyclone, Filter, gr/scf.	0.207	0.214	0.134	0.166	0.028	0.406
Total, gr/scf.	0.213	0.227	0.140	-	-	0.026
Particulate Emissions						
Probe, Cyclone, Filter, lb/hr.	478.20	491.10	183.60			
Total, lb/hr.	491.40	521.40	191.20			
Percent Isokinetic (2)	94.56	96.25	91.19			
Carbon Monoxide, %	0.0	0.0	0.0			
Carbon Dioxide, %	1.7	1.0	1.1			
Oxygen, %	18.6	18.9	19.1			
Sulfur Dioxide Outlet, ppm	0.0	0.0	0.0			
Sulfur Dioxide Inlet, ppm	2.3	2.8	7.7			
NO _x , ppm	72.1	70.7	64.6			

* Assumed

(1) True moisture content is more like 13%. The amount shown does not include the water condensed in the 18' long probe.
 (2) These values are between 5 and 6% lower than actual if the correct moisture is used for calculation.
 (3) These values are based on only one traverse.



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TABLE 2 PARTICULATE WEIGHTS SUMMARY

Run Number	1	2	3
Container 1, mg.	33.2	33.3	14.5
Container 2, mg.	1073.0	1068.0	395.0
Container 3a, mg.	1.0	0.0	0.0
Container 3b, mg.	23.5	60.0	14.0
Container 5, mg.	6.0	8.0	3.0
Probe, Cyclone, Filter, mg.	1106.2	1101.3	409.5
Total, mg.	1136.7	1169.3	426.5



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The results of the stationary sampling trains varied considerably. The loadings were considerably lower where the nozzle was turned into the direction of the highest flow compared to where the sample probe was at the same point but the nozzle was directed in the usual vertical direction. It is believed this is due to the water droplets which were formed on the nozzles having a greater chance to be pulled into the vertical nozzles than into the slanted nozzles.



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3.0 PROCESS DESCRIPTION AND OPERATION

A coal thermal dryer is operated at the United States Steel Pinnacle No. 50 coal preparation plant in Pineville, West Virginia. The emissions from the operation pass through an American Air Filter venturi scrubber before being emitted to the atmosphere.

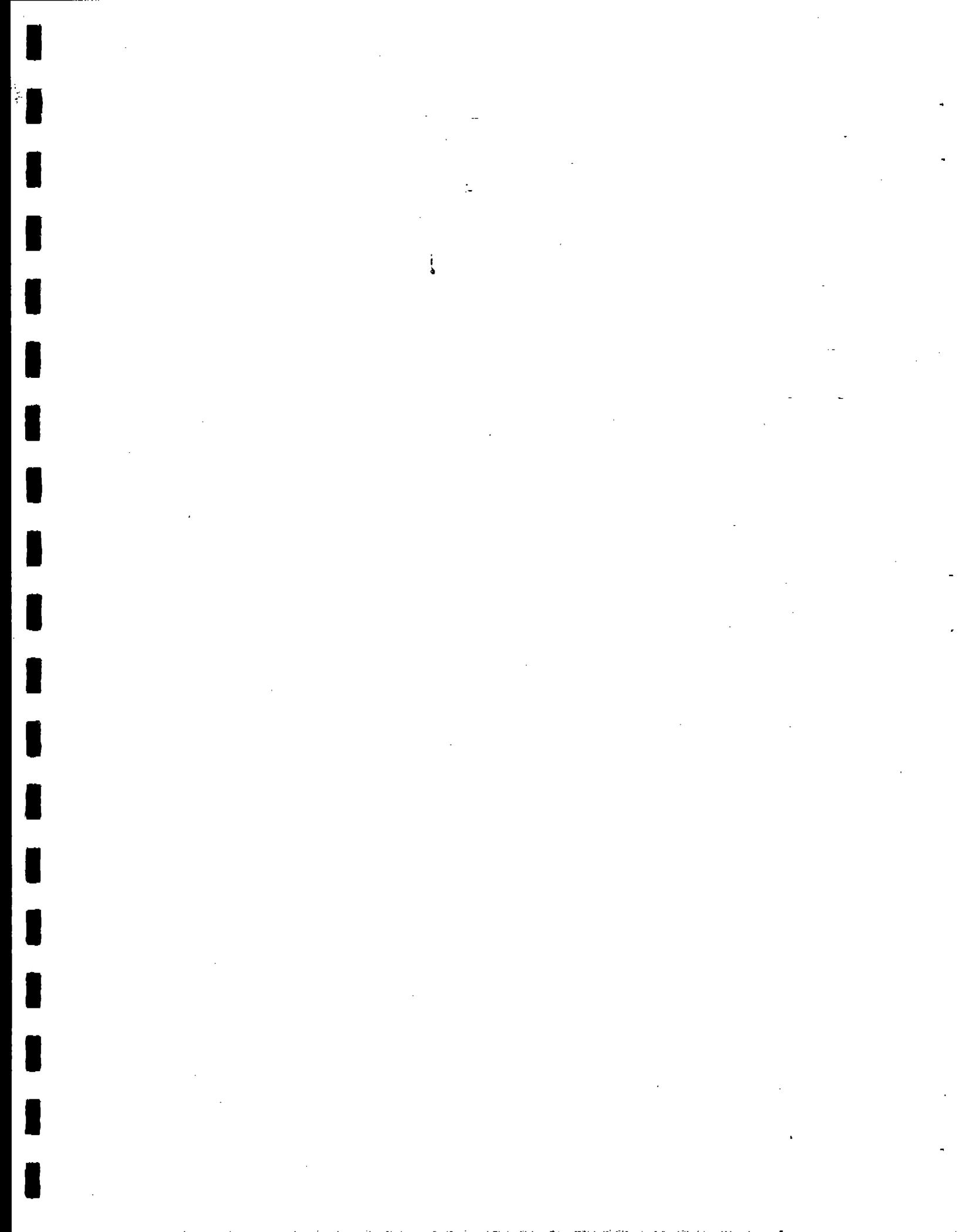
The coal being processed was Pocahontas #3 with approximately 105 grindability. During the tests it was being dried to 5.6% moisture.

The control panel in the plant was monitored during the tests and the following data was taken:

Run No.:	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Raw Coal, TPH	NA	976	946	975
Inlet fan amperage	79	70	70	70
Outlet fan amperage	230	225	225	225
Exhaust gas temp., °F	160	155	155	160
Drying chamber temp., °F	143	140	145	160
Combustion chamber temp, °F	950	860	840	850
Calculated coal feed				
Rate to dryer, TPH:	NA	465	450	465
% Capacity	NA	95	93	95
Filter cake to dryer, TPH	NA	41	41	41
% Capacity	NA	100	100	100
Scrubber inlet static press. in. H ₂ O	23.75	23.75	23.75	23.75

Although the measured scrubber pressure drop was only 23.75" w.g., the outlet fan was drawing maximum amperage. With the filter cake being dried as designed this is the maximum ΔP generated by the 200 H.P. fan. Thus, the plant was tested at its highest pollution potential with the highest energy scrubber possible at the operating conditions.





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4.0 LOCATION OF SAMPLING POINTS

The exhaust gases from the coal cleaning operation pass through an American Air Filter venturi scrubber and are then emitted to the atmosphere through a 202 inch diameter stack.

The inlet sulfur dioxide sample was taken from the duct before the gases pass into the venturi scrubber. The sample port was located approximately 4 feet ahead of the venturi scrubber.

The outlet location for sampling the gases prior to being discharged to the atmosphere was in a straight section of the 202 inch diameter stack. Two sampling ports were located at 90° to each other approximately 6 feet upstream from the top of the stack and approximately 7 feet downstream from where the gases entered the stack tangentially. In addition, two other ports were located in the same area.

Special scaffolding and support systems needed to be built in order to accomplish the desired sampling work. Platforms were built out from the stack at both port locations so that the two traverses could be performed. Figure 1 shows the physical layout of the system and the location of the sample ports. In addition, a special 18' long probe had to be prepared in order to accomplish the desired sampling.

Figure 2 shows the traverse points used at the sampling location. There were 48 traverse points sampled for three minutes each. However, a number of the points had zero flow, hence no nozzle dwell time occurred at these points. The traverse points were chosen in accordance with Method 1 published in the Federal Register, Volume 36, No. 24. The two ports were designated as A and B. A was the port on the left and B was the port 90° to the right of A.



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The stationary probe samples were taken with the probe in the stack approximately four inches.



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U.S. STEEL # 5
(PINEVILLE)

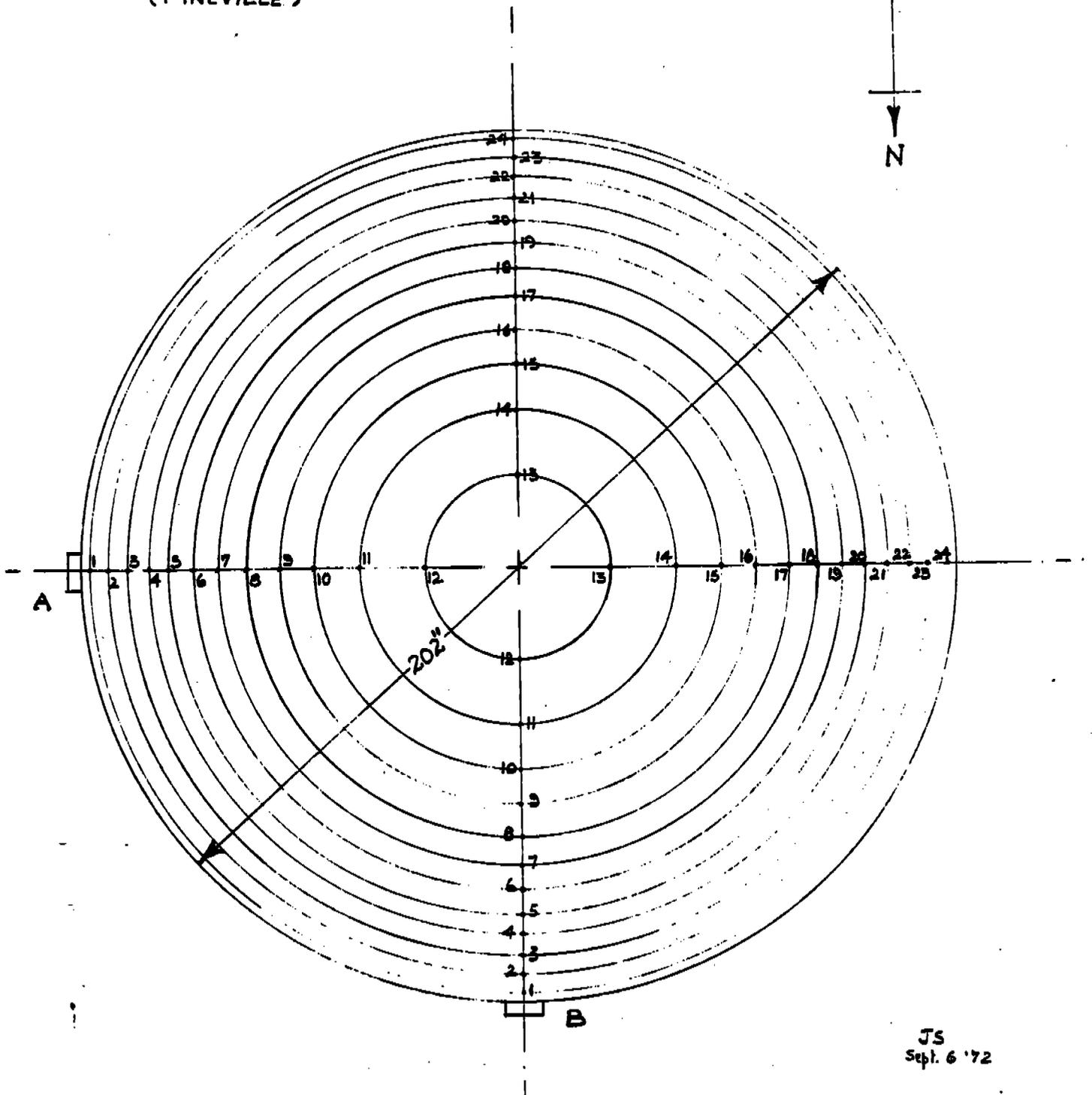
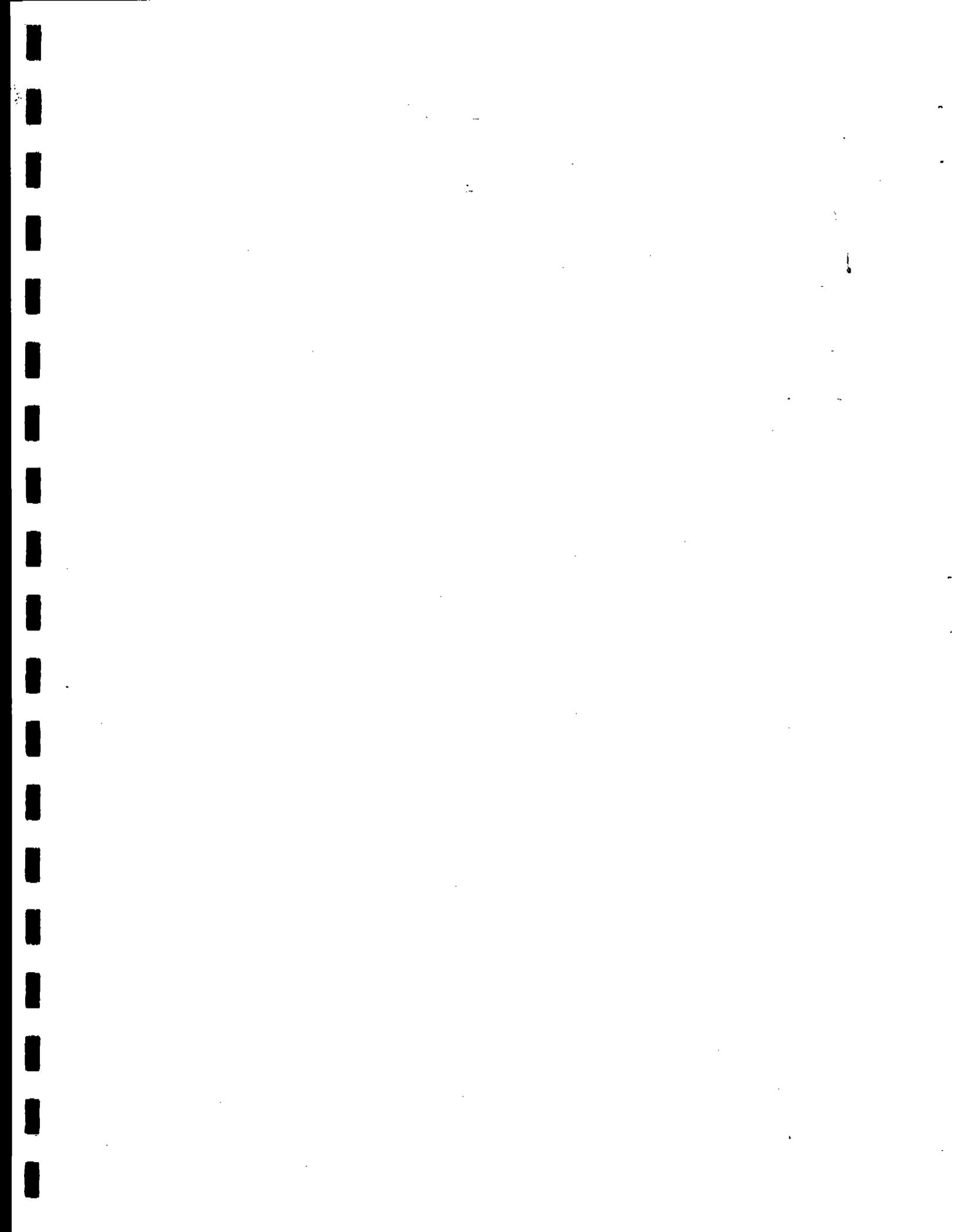


FIGURE 2 TRAVERSE POINT LOCATIONS





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5.0 SAMPLING AND ANALYTICAL PROCEDURES

5.1 PARTICULATE SAMPLING AND ANALYTICAL PROCEDURES

Samples were collected for the determination of particulate matter at the outlet of the venturi scrubber. The sampling and analytical procedures used were the same as those specified by Method 5, "Determination of Particulate Emissions from Stationary Sources", and published in the Federal Register, Volume 36, No. 247, Thursday, December 23, 1971. This method is attached as Appendix D. In addition, the impinger catch was analyzed.

Briefly, the method consists of withdrawing a sample isokinetically from the stack through a heated glass probe into a cyclone, filter, and impinger train. In this case, however, an unheated stainless steel probe had to be used because of the length. The cyclone and filter are contained in a heated box. The sample volume is measured with a dry gas meter, and isokinetic conditions are maintained by monitoring the stack gas velocity with an "S" type pitot tube. After testing is completed, the train is thoroughly washed including the probe. The washings are evaporated, dried, and weighed along with the filter in order to obtain a total weight of particulate matter collected.

The stack gas velocity and flow rate were measured using Method 2, "Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)", and published in the Federal Register. Using both the weight of sample collected and the flow rate determined, a total particulate emission rate was calculated.

Particulate samples were also collected at the outlet of the venturi scrubber using a probe which remained stationary at one point in the traverse. In one instance the pitot tube was rotated to find the



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highest ΔP reading along the traverse. The probe was then situated so that the nozzle was directed in the same direction. A second sampling train was placed into the stack the same distance alongside the other probe, however, in this case the nozzle was directed vertically down into the oncoming gases.

For these samples, only the front half of the EPA train was considered in determining the amount of particulate caught.

5.2 GASEOUS SAMPLING PROCEDURES

Stack gas samples were taken at regular intervals during each particulate sampling traverse to determine the concentration of O_2 , CO , CO_2 , NO_x and SO_2 present in the stack effluent. The sampling location was the same as that used for the particulate samples. In addition, however, SO_2 samples were taken ahead of the venturi scrubber. The sampling and analytical procedures used were in accordance with Federal Register, Volume 36, No. 247, December 23, 1972, "Standards of Performance for New Stationary Sources".

5.3 SO_2 SAMPLING AND ANALYTICAL PROCEDURES

All SO_2 samples were taken through a $\frac{1}{2}$ inch O.D. glass probe heated to approximately $250^\circ F$. This was connected to a glass sample train consisting of one bubbler and three impingers connected in series. The bubbler contained 15 ml. of 80% isopropanol and was used to remove any SO_3 present in the sample stream. The SO_2 was collected in the next two impingers, each containing 15 ml. of 3% H_2O_2 . The third impinger was used to trap any overflow from the two SO_2 impingers.



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Each sampling period was 30 minutes in duration, and the sampling rate was maintained at approximately 1 liter per minute with an in-line flowmeter. A temperature compensated dry gas meter was used to measure the total volume of gas sampled.

Following each test, the SO_2 samples were transferred to polyethylene bottles with distilled water washes. All samples were then returned to the laboratory where they were diluted to volume in a 50 or 100 ml. volumetric flask. A suitable aliquot of each sample was then titrated with a 0.01 N barium perchlorate solution in the presence of thorin indicator. The results were reported as parts per million SO_2 .

5.4 NO_x SAMPLING AND ANALYTICAL PROCEDURES

The NO_x samples were taken using the same heated glass probe described in Section 5.3. Each sample was drawn through this probe into a previously evacuated 2 liter flask containing 25 ml. of NO_x absorbing solution. The flasks were shaken for 5 minutes following each sampling period and then allowed to stand for at least 16 hours. Following this, the samples were shaken again for 2 minutes just prior to measuring the final flask pressure. The samples were then transferred to glass shipping bottles with distilled water washes and neutralized with 1.0 N sodium hydroxide. At the end of the test period, all samples were returned to the laboratory for analysis.

The samples were analyzed via a phenoldisulfonic acid procedure described in the aforementioned Federal Register. The absorbances were measured with a Bausch and Lomb Spectronic 20 Colorimeter. The results were reported as parts per million NO_2 .



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5.5 ORSAT SAMPLING AND ANALYTICAL PROCEDURE

Integrated gas samples were taken for Orsat analysis (CO, CO₂ and O₂) during each particulate sampling period. The sampling apparatus consisted of a ¼ inch O.D. stainless steel probe, a stainless steel coiled tube condenser, a glass water trap, a carbon vane pump, a flowmeter and needle valve assembly, a 3 inch #21 stainless steel hypodermic needle, and a 5 liter Tedlar sample bag fitted with a syringe cap.

The sampling procedure was initiated by purging the probe and condenser system with stack air, adjusting the sample flow rates to approximately 80 cc per minute, and inserting the hypodermic needle into the syringe cap on the sample bag. Several integrated samples were taken over 30 minute periods yielding approximately 4.8 liters of sample for analysis.

At the end of each test day, the sample bags were analyzed by Orsat for CO, CO₂ and O₂. Repetitive analyses were performed on each bag to insure satisfactory duplication. The results were reported in percentages.



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APPENDIX A

COMPLETE PARTICULATE RESULTS WITH EXAMPLE CALCULATIONS



FORM NO. 1281-45

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SOURCE TESTING CALCULATION FORMS

Test. No. 4 (EPA Tests)No. Runs 3Name of Firm U.S. SteelLocation of Plant Pineville, W. VirginiaType of Plant Coal CleaningControl Equipment Cyclone / Venturi ScrubberSampling Point Locations Outlet, 202" dia; 2 posts, 3 minutes each point *Pollutants Sampled Particulate, Orsat, Nox, SO₂

Time of Particulate Test:

Run No. <u>1</u>	Date <u>8/30/72</u>	Begin <u>0945</u>	End <u>1146</u>
Run No. <u>2</u>	Date <u>8/30-8/31/72</u>	Begin <u>1243-0854</u>	End <u>1343-0947</u>
Run No. <u>3</u>	Date <u>8/31/72</u>	Begin <u>1013</u>	End <u>1122</u>

PARTICULATE EMISSION DATA

Run No.	1	2	3			
P _b barometric pressure, "Hg Absolute	28.79	28.80	28.80			
P _m orifice pressure drop, "H ₂ O	2.658	2.689	2.711			
V _m volume of dry gas sampled @ meter conditions, ft. ³	88.628	85.414	51.347			
T _m Average Gas Meter Temperature, °F	93.62	93.78	100.3			
V _{m std.} Volume of Dry Gas Sampled @ Standard Conditions, ft. ³	82.132	79.164	47.039			
V _w Total H ₂ O collected, ml., Impingers & Silical Gel.	131.8	156.3	70.0			
V _{w gas} Volume of Water Vapor Collected ft. ³ @ Standard Conditions*	6.247	7.409	3.318			

* 70°F, 29.92" Hg.

* Run 1 & Run 2 has 35 active (non-zero OP) traverse points
 Run 3 has 20 active traverse points.

PARTICULATE EMISSION DATA (cont'd)

Run No.	1	2	3
M - % Moisture in the stack gas by volume	7.07	8.56	6.59
M _d - Mole fraction of dry gas	.9293	.9144	.9341
% CO ₂	1.70	1.03	1.13
% O ₂	18.60	18.87	19.12
% N ₂	79.70	80.10	79.75
MW _d - Molecular weight of dry stack gas	29.016	28.920	28.945
MW - Molecular weight of stack gas	28.237	27.985	28.224
ΔPs - Velocity Head of stack gas, In.H ₂ O	-	-	-
T _s - Stack Temperature, °F	130	130	130
$\Delta P_s \times (T_s + 460)$	13.54	13.62	14.00
P _s - Stack Pressure, "Hg. Absolute	28.75	28.75	28.75
V _s - Stack Velocity @ stack conditions, fpm	2067.0	2088.6	2137.2
A _s - Stack Area, in. ²	* 2336.8	* 2336.8	* 1355.3
Q _s - Stack Gas Volume @ Standard Conditions. * SCFM	26975	26748	15977.5
T _t - Net Time of Test, min.	105	100	60
D _n - Sampling Nozzle Diameter, in.	.302	.302	.302
%I - Percent isokinetic	94.56	96.25	91.19
m _f - Particulate - probe, cyclone and filter, mg.	1106.2	1101.3	409.5
m _t - Particulate - total, mg.	1136.7	1169.3	426.5
C _{an} - Particulate - probe, cyclone, and filter, gr/SCF	.207416	.214239	.134065
C _{ao} - Particulate - total, gr/SCF	.213135	.227467	.139651
C _{at} - Particulate - probe, cyclone, & filter, gr/cf @ stack conditions	.166248	.168964	.108011

* These are effective areas dependent on the number of active (non-zero) traverse points. True area = 32047 in²

Run No.	1	2	3			
C _{cu} - Particulate, total, -gr/cf @ stack cond.	.170832	.179396	.112495			
C _{aw} - Particulate, wet, cyclone, and filter, lb/hr.	478.2	491.1	183.6			
C _{ax} - Particulate - total, lb/hr.	491.4	521.4	191.2			
% EA - % Excess air @ sampling point	715.3	774.4	913.3			

* 70°F. 29.92" Hg.

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SOURCE TESTING CALCULATION FORMS

Test. No. 4 (Stationary Tests)

No. Runs 5

Name of Firm U. S. Steel

Location of Plant Pineville, W. Virginia

Type of Plant Coal Cleaning

Control Equipment Cyclone / Venturi Scrubber

Sampling Point Locations Outlet Stack, 202" dia. Single point, *

Pollutants Sampled Particulate, orsat analysis, SO₂(inlet), NO_x, Hydrocarbons

Time of Particulate Test:

Run No. <u>1</u>	Date <u>8/29</u>	Begin <u>1/30</u>	End <u>1255</u>
Run No. <u>2 + 3</u>	Date <u>8/30</u>	Begin <u>0945</u>	End <u>1143</u>
Run No. <u>4 & 5</u>	Date <u>8/31</u>	Begin <u>1015</u>	End <u>1125</u>

PARTICULATE EMISSION DATA

Run No.	1	2	3	4	5
<i>Nozzle Angle:-</i>	<u>135°</u>	<u>180°</u>	<u>125°</u>	<u>180°</u>	<u>125°</u>
P _b barometric pressure, "Hg Absolute	<u>28.70</u>	<u>28.78</u>	<u>28.78</u>	<u>28.80</u>	<u>28.80</u>
P _o orifice pressure drop, "H ₂ O	<u>1.240</u>	<u>1.351</u>	<u>1.351</u>	<u>1.231</u>	<u>1.231</u>
V _m volume of dry gas sampled @ meter conditions, ft. ³	<u>40.73</u>	<u>73.41</u>	<u>83.08</u>	<u>40.39</u>	<u>48.66</u>
T _m Average Gas Meter Temperature, °F	<u>87</u>	<u>87</u>	<u>87</u>	<u>87</u>	<u>87</u>
V _m std. Volume of Dry Gas Sampled @ Standard conditions, ft. ³	<u>37.91</u>	<u>68.60</u>	<u>77.64</u>	<u>37.71</u>	<u>45.49</u>
V _w Total H ₂ O collected, ml., Impingers & Silical Gel.	<u>-</u>	<u>-</u>	<u>-</u>	<u>-</u>	<u>-</u>
V _w gas Volume of Water Vapor Collected ft. ³ @ Standard Conditions*	<u>-</u>	<u>-</u>	<u>-</u>	<u>-</u>	<u>-</u>

* 70°F, 29.92" Hg.

* nozzle posn. changed. Nozzle angle is with reference to vertical.

PARTICULATE EMISSION DATA (cont'd)

Run No.	nozzle angle	RAC	RAC	GI	RAC	GI
		1 135°	2 180°	3 125°	4 180°	5 125°
XH - % Moisture in the stack gas by volume		13.5	13.5	13.5	13.5	13.5
M _d - Mole fraction of dry gas		.865	.865	.865	.865	.865
% CO ₂		2.67	1.70	1.70	1.13	1.13
% O ₂		18.0	18.6	18.6	19.12	19.12
% N ₂		79.33	79.7	79.7	79.75	79.75
MW _d - Molecular weight of dry stack gas		29.147	29.016	29.016	28.945	28.945
MW - Molecular weight of stack gas		27.642	27.529	27.529	27.467	27.467
ΔP _s - Velocity Head of stack gas, In.H ₂ O		.5225	.8630	.8630	.8190	.8190
T _s - Stack Temperature, °F		130	130	130	130	130
(ΔP _s X (T _s + 460))		17.558	22.565	22.565	21.982	21.982
P _s - Stack Pressure, "Hg. Absolute		28.66	28.74	28.74	28.76	28.76
V _s - Stack Velocity @ stack conditions, fpm		2713.6	3489.7	3489.7	3402.2	3402.2
A _s - Stack Area, in. ²		32047	32047	32047	32047	32047
Q _s - Stack Gas Volume @ Standard Conditions. * SCFM		449453	579611	579611	565471	565471
T _t - Net Time of Test, min.		85	118	115	70	70
D _n - Sampling Nozzle Diameter, in.		.201	.201	.194	.201	.194
%I - Percent isokinetic		99.92	100.99	125.90	95.93	124.22
m _f - Particulate - probe, cyclone and filter, mg.		156.0	740.0	139.9	993.8	77.0
m _t - Particulate - total, mg.		-	-	-	-	-
C _{an} - Particulate - probe, cyclone, and filter, gr/SCF		.06337	.1661	.0273	.4058	.02607
C _{ao} - Particulate - total, gr/SCF		-	-	-	-	-
C _{at} - Particulate - probe, cyclone, & filter, gr/cf @ stack conditions		.04913	.1239	.02070	.3029	.01946

* Moisture assumed in all cases to be 13.5%

PARTICULATE EMISSION DATA (cont'd)

Run No.	Nozzle angle	1 RAC	2 RAC	3 UGI	4 RAC	5 UGI
C _{cu}	- Particulate, total, lb/hr @ stack end.	—	—	—	—	—
C _{aw}	- Particulate, probe, cyclone, and filter, lb/hr.	244.1	825.1	137.8	1966.5	126.3
C _{ax}	- Particulate - total, lb/hr.	—	—	—	—	—
% EA	- % Excess air @ sampling point	580.3	715.3	715.3	913.3	913.3

* 70°F. 29.92" Hg.

PARTICULATE CALCULATIONS

2001 (7/14 Test)

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

$$V_{m_std} = \frac{17.7 \times V_m \left(P_B + \frac{P_m}{13.6} \right)}{(T_m + 460)} = \frac{17.7 \times 88.628 \left(28.79 + \frac{2.658}{13.6} \right)}{(93.62 + 460)}$$

$$= 82.132 \text{ ft}^3$$

2. Volume of water vapor at 70°F & 29.92" Hg, Ft.³

$$V_{W_gas} = 0.0474 \times V_m = \text{Ft.}^3 = 0.0474 \times 131.8 = 6.247 \text{ ft}^3$$

3. % moisture in stack gas

$$\%M = \frac{100 \times V_{W_gas}}{V_{m_std} + V_{W_gas}} = \frac{100 \times 6.247}{82.132 + 6.247} = 7.07 \%$$

4. Mole fraction of dry gas

$$M_d = \frac{100 - \%M}{100} = \frac{100 - 7.07}{100} = 0.9293$$

5. Average molecular weight of dry stack gas

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

$$= (1.70 \times 0.44) + (18.6 \times 0.32) + (79.70 \times 0.28)$$

$$= 29.016$$

6. Molecular weight of stack gas A-9

$$M W = M W_d \times H_d + 18(1 - H_d) = 29.016 \times 0.9293 + 18(1 - 0.9293) \\ = \underline{28.237}$$

7. Stack velocity @ stack conditions, fpm

$$V_s = 4350 \sqrt{\frac{\Delta P_s \times (T_s + 460)}{P_s \times M W}}^{1/2} = \text{fpm} = 2067 \text{ fpm} \\ \text{(Averaged over 35 active data points)}$$

8. Stack gas volume @ standard conditions, SCFM

$$Q_s = \frac{0.123 \times V_s \times A_s \times M_d \times P_s}{(T_s + 460)} = \text{SCFM} = \frac{0.123 \times 2067 \times 23368 \times 9293 \times 28.75}{(130 + 460)} \\ = \underline{269035 \text{ SCFM}}$$

9. Per cent isokinetic

$$\%I = \frac{1032 \times (T_s + 460) \times V_{m \text{ std.}}}{V_s \times T_t \times P_s \times M_d \times (D_{11})^2} = \% = \frac{1032 (130 + 460) \times 82.132}{2067 \times 105 \times 28.75 \times 9293 \times (302)^2} \\ = \underline{94.56\%}$$

10. Particulate - probe, cyclone, & filter, gr/SCF

$$C_{\text{an}} = 0.0154 \times \frac{M_f}{V_{m \text{ std}}} = \text{gr/SCF} = 0.0154 \times \frac{1106.2}{82.132} \\ = \underline{0.207416 \text{ gr/SCF}}$$

11. Particulate total, gr/SCF

A-10

$$C_{ao} = 0.0154 \times \frac{M_t}{V_{m, std}} = \text{gr/SCF} = 0.0154 \times \frac{1136.7}{82.132} = \underline{0.213135 \text{ gr/SCF}}$$

12. Particulate - probe, cyclone & filter, gr/CF at stack conditions

$$C_{at} = \frac{17.7 \times C_{an} \times P_s \times M_d}{(T_s + 460)} = \text{gr/CF} = \frac{17.7 \times .207416 \times 28.75 \times 0.9293}{(130 + 460)} = \underline{0.166248 \text{ gr/CF}}$$

13. Particulate - total, gr/CF @ stack conditions

$$C_{au} = \frac{17.7 \times C_{ao} \times P_s \times M_d}{(T_s + 460)} = \text{gr/CF} = \frac{17.7 \times .213135 \times 28.75 \times 0.9293}{(130 + 460)} = \underline{0.170832 \text{ gr/CF}}$$

14. Particulate - probe, cyclone, & filter, lb/hr.

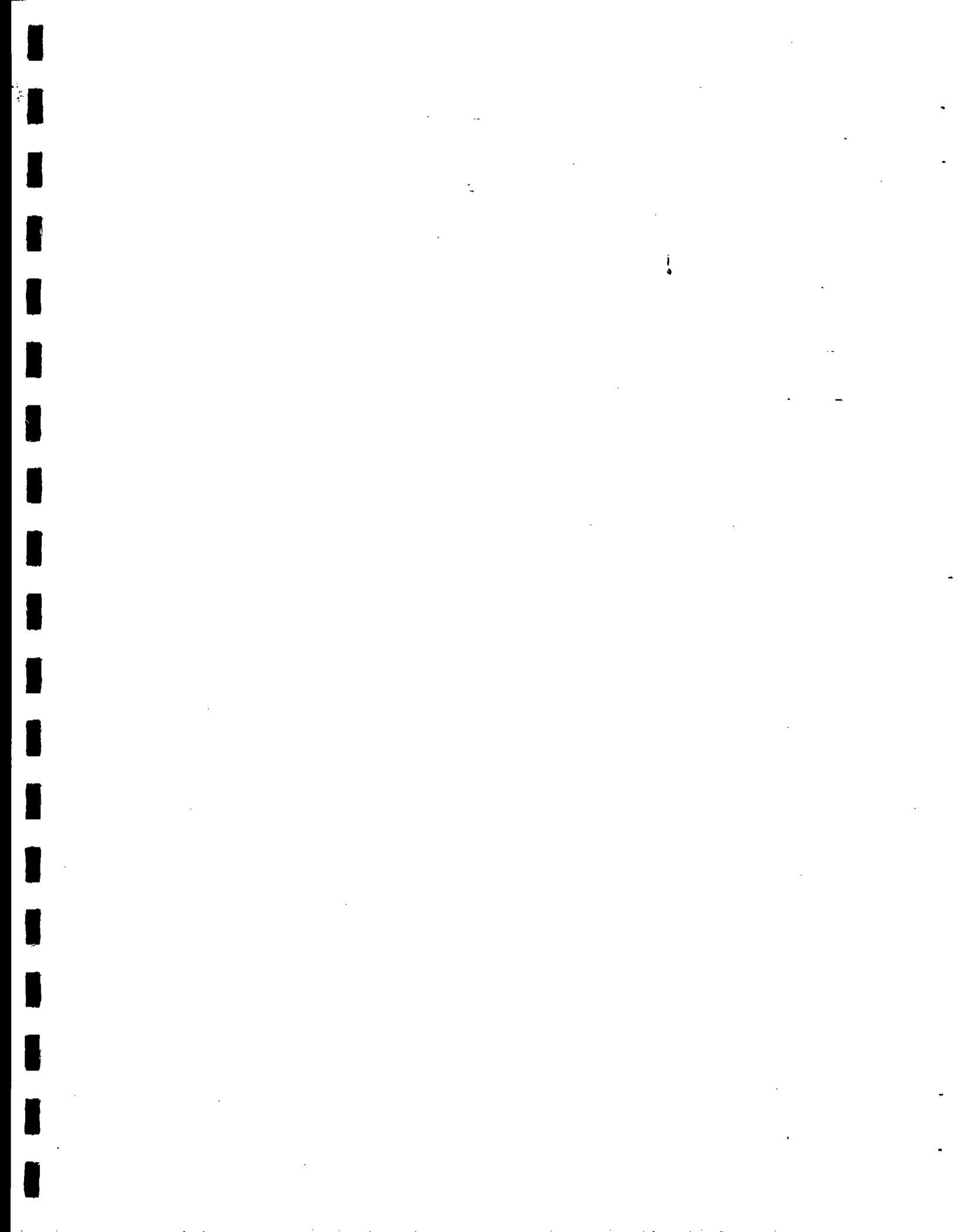
$$C_{aw} = 0.00857 \times C_{an} \times Q_s = \text{lb/hr.} = 0.00857 \times .207416 \times 269035 = \underline{478.2 \text{ lb/hr}}$$

15. Particulate - total, lb/hr.

$$C_{ax} = 0.00857 \times C_{ao} \times Q_s = \text{lb/hr.} = 0.00857 \times .213135 \times 269035 = \underline{491.4 \text{ lb/hr}}$$

10. % excess air at stepping point

$$\% \text{ EA} = \frac{100 \times \% \text{ O}_2}{0.266 \times \% \text{ H}_2 - \% \text{ O}_2} = \% = \frac{100 \times 18.6}{0.266 \times 79.7 - 18.6}$$
$$= \underline{715.3 \%}$$



SRL 1281 45 0972

APPENDIX B

COMPLETE GASEOUS RESULTS WITH EXAMPLE CALCULATIONS



SO₂ EMISSION DATA

Run No.	Test #0		Test #1		Test #3	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Date	29	Aug 72	30	Aug 72	31	Aug 72
mg SO ₂	0.191	0.000	0.220	0.000	0.602	0.000
T _m - Average Gas Meter Temperature, °F	74	74	65	65	74	74
P _b - Barometric Pressure, "Hg abs.	28.70	28.70	28.79	28.79	28.80	28.80
V _m - Volume of dry gas sampled @ meter conditions, ft. ³	1.13	1.06	1.06	1.06	1.06	1.06
ppm SO ₂	2.306	0.00	2.775	0.00	7.721	0.00

Test #0 (Inlet)

$$\text{ppm SO}_2 = \frac{0.7332 \times \text{mg SO}_2 \times (T_m + 460)}{P_b \times V_m} = \frac{0.7332 \times 0.191 \times (74 + 460)}{28.70 \times 1.13} = \underline{2.306} \text{ ppm SO}_2$$

NO_x EMISSION DATA

Run No.	Test # 0 Outlet	Test # 1 Outlet		Test # 3 Outlet		
Date	29 Aug 72	30 Aug 72	30 Aug 72	30 Aug 72	31 Aug 72	31 Aug 72
mg NO ₂	0.1511	0.1406	0.2386	0.1922	0.1571	0.1496
T _f - Flask Temperature, °F	74	65	65	65	74	74
V _f - Flask Volume, liters	2.0404	2.0425	2.0392	2.0498	2.0330	2.0430
P _i - Initial Flask Vacuum, "Hg.	19.00	21.00	20.00	24.00	17.00	23.00
P _f - Final Flask Vacuum, "Hg.	2.75	0.75	1.00	1.00	1.00	1.00
ppm NO ₂	72.11	52.88	95.80	63.42	76.42	52.66

Test # 0

$$\begin{aligned}
 \text{ppm NO}_2 &= \frac{29.63 \times \text{mg NO}_2 \times (T_f + 460)}{V_f \times (P_i - P_f)} \\
 &= \frac{29.63 \times 0.1511 \times (74 + 460)}{2.0404 \times (19.00 - 2.75)} \\
 &= \underline{72.11} \text{ ppm NO}_2
 \end{aligned}$$

C-1

SRL 1281 45 0972

APPENDIX C

FIELD DATA



SCOTT RESEARCH LABORATORIES, INC.

FIELD DATA

PLANT US Steel 550
 DATE 8/30/72
 SAMPLING LOCATION Pointe EP17
 SAMPLE TYPE 71
 RUN NUMBER 1
 OPERATOR 740
 AMBIENT TEMPERATURE 65
 BAROMETRIC PRESSURE 28.79
 STATIC PRESSURE (P_s) 15
 FILTER NUMBER (s) 1, 3, 6, 15

PROBE LENGTH AND TYPE 18' SS
 NOZZLE I.D. 3/2" 73
 ASSUMED MOISTURE, %
 SAMPLE BOX NUMBER
 METER BOX NUMBER
 METER ΔH 1.36
 C FACTOR 0.4
 PROBE HEATER SETTING
 HEATER BOX SETTING
 REFERENCE Δp 1.23

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY 3 MINUTES

TRAVERSE POINT NUMBER	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V _m) ft ³	VELOCITY HEAD (ΔP _v) in. H ₂ O	ORIFICE PRESSURE DIFFERENTIAL (ΔH) in. H ₂ O		STACK TEMPERATURE (T _s) °F	DRY GAS METER TEMPERATURE		PUMP VACUUM in. Hg	SAMPLE BOX TEMPERATURE °F	INLET TEMPERATURE (T _{m in}) °F	OUTLET TEMPERATURE (T _{m out}) °F	IMPINGER TEMPERATURE °F
				DESIRED	ACTUAL		INLET (T _{m in}) °F	OUTLET (T _{m out}) °F					
1	0945	202.659	ΔP	2		95	66	65	5		66	65	85
2	3	204.985	124	2		130	73	65	5		73	65	90
3	6	205.03	148	3.7			83	68	8		83	68	90
4	9	211.55	155	4.3			89	68	9.5		89	68	90
5	12	213.00	136	3.9			85	70	1.0		85	70	88
6	15	216.26	117	1.3			87	71	3.0		87	71	88
7	18	217.60	106	1.0			82	72	2.5		82	72	85
8	21	218.085	109	1.3			85	74	2.7		85	74	85
9	24	219.99	103	1.3					2.0				
10			0										
11			0										
12			0										
13			0										
14			0										
15			0										
16	27	220.78	102	1.7			84	76	6.5		84	76	89
17	30	222.125	108	1.6			86	76	2.5		86	76	92
18	33	224.01	116	1.3			90	77	4.0		90	77	90
19	36	226.1575	124	2.3			96	79	6.0		96	79	90
20	39	229.525	134	3.1			104	80	7.0		104	80	90
21	42	233.05	140	4.7			110	82	11.0		110	82	90
22	45	232.00	176	6.0			118	85	17.0		118	85	92
23	48	231.11	110	8.6	6.7		128	86	18.0		128	86	93

COMMENTS:

TRAVERSE POINT NUMBER	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V _m ³)	VELOCITY HEAD (40.2 in. H ₂ O)	ORIFICE DIFFERENTIAL (3/8 in. H ₂ O)		STACK TEMPERATURE (T _s) °F	DRY GAS METER TEMPERATURE (T _m in ³) °F		PUMP VACUUM in Hg	SAMPLE BOX TEMPERATURE °F	IMPINGER TEMPERATURE °F
				DESIRED	ACTUAL		INLET	OUTLET			
24	51	241.11	1.2	5.7	6.2	130	124	58	180		54
1	3	248.07	1.30	2.9	2.9		124	59	7.0		50
2	6	251.02	1.40	3.2	3.2						50
3	9	253.645	1.27	2.1	2.1		125	90	5.5		50
4	12	256.38	1.30	2.4	2.4		124	91	6.0		50
5	15	258.935	1.24	2.3	2.3		114	92	5.0		50
6	18	261.37	1.22	1.8	1.8		114	92	4.5		50
7	21	263.485	1.5	1.25	1.25		112	92	3.0		50
8	24	265.101	1.27	1.58	1.58		110	93	3.0		50
9	27	267.90	1.02	1.7	1.7		106	93	2.0		50
10			0								
11			0								
12			0								
13			0								
14			0								
15			0								
16	30	266.72	1.02	1.7	1.7		101	92	2.0		50
17	33	268.10	1.07	1.59	1.59		100	92	3.0		50
18	36	270.165	1.20	1.60	1.60		103	92	5.0		50
19	39	272.83	1.31	2.6	2.6		108	93	6.5		50
20	42	275.187	1.40	3.3	3.3		113	94	8.0		50
21	45	279.385	1.40	4.8	4.8		119	94	12.5		50
22	48	283.301	1.80	5.7	5.7		124	96	16.6		50
23	51	287.225	1.10	8.0	8.0		127	97	18.5		50
24	54	291.286	1.30	10.0	10.0		130	98	18.5		50

151
Pump

1188

1114

88.628
9/11/11

WATER VOLUME

Run No. 1 Date 8/30/72

Bubbler #1 186 ml Silica Gel No. _____ Wgt. g. 510.7

#2 115 ml

#3 6 ml

Bubbler #4 535.5

Gross 307

Water Added(-) 200 ml

Gross Wgt.(-) ~~510.7~~ 510.7

Net (A) 107 cc

Net (B) 24.8 g

Net (A) 107

Net (+) 24.8
Net (B)

Total Water 131.8 cc

FIELD DATA

PLANT US Steel #5
 DATE 5/30/72
 SAMPLING LOCATION point
 SAMPLE TYPE point
 RUN NUMBER 340
 OPERATOR FW
 AMBIENT TEMPERATURE 74.0
 BAROMETRIC PRESSURE 29.76
 STATIC PRESSURE (P₁) 15
 FILTER NUMBER (s) A2, H305

PROBE LENGTH AND TYPE 18 SS
 NOZZLE I.D. 1.302
 ASSUMED MOISTURE, % 1.3
 SAMPLE BOX NUMBER
 METER BOX NUMBER
 METER ΔH 1.87
 C FACTOR 1.44
 PROBE HEATER SETTING
 HEATER BOX SETTING 350
 REFERENCE ΔP 1.23 (1.25)

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY _____ MINUTES

TRAVERSE POINT NUMBER	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V _m ft ³)	VELOCITY HEAD (ΔP ₃), in. H ₂ O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H ₂ O		STACK TEMPERATURE (T _s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
				DESIRED	ACTUAL		INLET (T _{m in}), °F	OUTLET (T _{m out}), °F			
1	3	291.591	1.26	2.11		195	91	91	5.0		50
2	3	294.01	1.38	3.0		130	98	91	6.3		50
3	6	296.42	1.36	2.19			106	92	6.0		50
4	12	299.81	1.22	2.16			111	92	6.0		50
5	15	302.606	1.24	2.1			114	93	5.0		50
6	18	305.110	1.21	1.7			116	94	4.2		50
7	21	307.70	1.13	1.1			116	95	3.5		50
8	24	311.18	1.08	1.68			114	96	3.0		50
9	27	312.07	1.02	1.6			96	96	2		70
10			0								
11			0								
12			0								
13			0								
14			0								
15			0								
16	30	312.86	1.02	1.6			100	94	2		50
17	33	314.235	1.08	1.62			102	96	2.7		50
18	34	316.195	1.20	1.15			106	97	4.0		50
19	39	318.88	1.33	2.15			111	97	5.5		50
20	42	321.832	1.43	3.1			116	98	7.0		50
21	45	325.133	1.64	4.17			122	99	10.5		50
22	48	329.21	1.78	5.16			126	100	12.5		50
23	51	333.232	1.1	7.8	7.0		131	101	17.5		50

COMMENTS:

1067

721293

A

85.414

1305
1106
0111
1371

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V _m · l _l ³)	VELOCITY HEAD (ω _{ps}), in. H ₂ O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in H ₂ O		STACK TEMPERATURE (T _s), °F	DRY GAS METER TEMPERATURE (T _m in ^l), °F		PUMP VACUUM, in Hg	SAMPLE BOX TEMPERATURE, °F	IRPINGER TEMPERATURE, °F
					DESIRED	ACTUAL		INLET	OUTLET			
24	57	1:00 min	334.593	.98	7	130						50
1	3	0854 8/31/72	331.365	.40	2.19			63	63	7.0		50
2	6	1:00 min	340.28	.41	3.0			71	67	7.0		50
3	9		344.2	.41	3.0			79	67	7.0		50
4	12	2 min	345.95	.33	2.14							
5	15		346.15	.22	1.55							
6	18		350.01	.15	1.15			89	68	4.25		50
7	21		351.285	.06	.76			91	70	4.0		50
8	24		352.07	.02	.16			88	72	3.0		50
9				0						2.0		50
10				0								
11				0								
12				0								
13				0								
14				0								
15				0								
16	27	0920	353.0	.03	1.24			84	73	2.0		50
17	30	0940	354.6	.10	1.75			87	75	3.0		50
18	33		356.345	.16	1.2			92	76	4.0		50
19	36		358.590	.26	1.9			96	78	5.0		50
20	39		361.358	.40	2.9			101	79	6.7		50
21	42		364.650	.60	4.3			105	82	10.0		50
22	45		366.545	.83	5.9			114	84	15.0		50
23	48		372.750	1.1	8.9			120	89	18.0		50
24	51		377.005	1.2	8.7			122	89	18.0		50


 1343
 APPROXIMATE
 OF
 66

1168 9214

WATER VOLUME

Run No. 2

Date 8/30 - 8/31

Bubbler #1 205 ml

Silica Gel No. _____ Wgt. g. 512.7

#2 127 ml

#3 4 ml

Bubbler #4 533.0

Gross 3.36

Water Added(-) 200 ml

Gross Wgt.(-) 512.7
~~212.3~~

Net (A) 136 cc

Net (B) 20.3 g

Net (A) 136

Net (+) (B) 20.3

Total Water 156.3 cc

FIELD DATA

PLANT U.S. STEEL #5
 DATE 8/31/72
 SAMPLING LOCATION PARTICULATE
 SAMPLE TYPE 3
 RUN NUMBER 3
 OPERATOR JOE WILSON
 AMBIENT TEMPERATURE 74°
 BAROMETRIC PRESSURE 28.80
 STATIC PRESSURE (P_s) 0.5
 FILTER NUMBER (S) #3, 3612

PROBE LENGTH AND TYPE 18' S.S.
 NOZZLE I.D. 0.302
 ASSUMED MOISTURE, % 13%
 SAMPLE BOX NUMBER 51396 (RAC)
 METER BOX NUMBER 1.84
 METER Δh_g .94
 C FACTOR 2.50
 PROBE HEATER SETTING 2.50
 HEATER BOX SETTING 0.25
 REFERENCE Δp

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY 3 MINUTES

TRAVERSE POINT NUMBER	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V _m) ³	VELOCITY HEAD (Δp _s) ² , in. H ₂ O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H ₂ O		STACK TEMPERATURE (T _s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
				DESIRED	ACTUAL		INLET (T _{m in}), °F	OUTLET (T _{m out}), °F			
1	3:10:15	377.208	1.37	2.6		130	88	84	6		50
2	3:11	379.970	1.37	3.4			97	85	2.5		50
3	3:12	383.10	1.52	3.8			104	86	5.1		50
4	3:13	386.455	1.41	3.0			109	87	5.0		50
5	3:14	389.425	1.36	1.9							50
6	3:15	391.975	1.17	1.3			108	88	4.0		50
7	3:16	394.150	1.07	1.4			106	88	2.5		50
8	3:17	395.590	1.02	1.6			104	88	1.7		50
9	3:18		1.0								
10	3:19		1.0								
11	3:20		1.0								
12	3:21		1.0								
13	3:22		1.0								
14	3:23		1.0								
15	3:24		1.0								
16	3:25	397.250	1.02	1.6			98	89	1.7		50
17	3:26	398.835	1.19	1.91			100	90	3.0		50
18	3:27	401.020	1.25	1.80			102	90	4.5		50
19	3:28	403.825	1.39	2.90			108	92	6.8		50
20	3:29	407.020	1.50	3.7			114	92	8.5		50
21	3:30	410.485	1.60	4.13			118	93	10.0		50
22	3:31	414.365	1.77	5.14			122	95	13.0		50
23	3:32	418.710	1.91	6.5			126	96	17.5		50

COMMENTS:

109.42 9/16

TRAVERSE POINT NUMBER	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V _m ft ³)	VELOCITY HEAD (V _s) in. H ₂ O	ORIFICE PRESSURE DIFFERENTIAL (ΔH) in. H ₂ O		STACK TEMPERATURE (T _s) °F	DRY GAS METER TEMPERATURE		PUMP VACUUM in Hg	SAMPLE BOX TEMPERATURE °F	IMPINGER TEMPERATURE °F
				DESIRED	ACTUAL		INLET (T _{m in}) °F	OUTLET (T _{m out}) °F			
24	57 1108	421.96	1.1	7.8	6.8	130	130	98			50
1	3 1112	424.20	1.0	7.6	2.2			97	5.5		50
2	6	426.58	12.6	1.9				98	5.0		56
3	9	428.29	11.0	1.5				98			50
4	12 1122	428.615									
5	15										
6	18										
7	21										
8	24										
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											

(27)

RUN 3

WATER VOLUME

Run No. 3

Date 8/31/72

Bubbler #1 147 ml

Silica Gel No. _____ Wgt. g. 507.3

#2 111

#3 ---

Bubbler #4 519.3

Gross .258

Water Added(-) 200

Gross Wgt.(-) 507.3

Net (A) 58 cc

Net (B) 12.0 g

Net (A) 58

Net (+) (B) 12

Total Water 70.0 cc

FIELD DATA

Race (Stationery) Run #1
135°
8-2-9

PLANT U.S. #50
DATE 8-2-9
SAMPLING LOCATION *particulate*
SAMPLE TYPE
RUN NUMBER
OPERATOR *JW*
AMBIENT TEMPERATURE 76
BAROMETRIC PRESSURE 28.7
STATIC PRESSURE (P_s) #4
FILTER NUMBER (s) #4 11315

PROBE LENGTH AND TYPE 8
NOZZLE I.D. .201
ASSUMED MOISTURE, % 13
SAMPLE BOX NUMBER
METER BOX NUMBER 1-05
METER ΔH .08
C FACTOR .80
PROBE HEATER SETTING 250
HEATER BOX SETTING 250
REFERENCE ap

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY _____ MINUTES

TRAVERSE POINT NUMBER	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V _m), ft ³	VELOCITY HEAD (ΔP _s), in. H ₂ O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H ₂ O		STACK TEMPERATURE (T _s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
				DESIRED	ACTUAL		INLET (T _{m in}), °F	OUTLET (T _{m out}), °F			
1130	0	236.1	.5								
1135	5	236.1 237.6	1.0	.32	.32	90	72	70	1		60
1140	10	238.89	1.0	.32	.32	90	72	70	1.5		60
1145	15	240.20	1.0	.75	.75	90	84	72	2.5		
1150	20	242.24	1.0	.95	.95	90	92	72	2.5		
1155	25	244.81	1.0	.95	.95	90	94	74	3.0		
1200	30	247.79	1.0	.89	.89	90	90	76	3.2		
1205	35	250.30	1.0	1.0	1.0	90	94	76	3.5		
1210	40	253.14	1.0	.87	.87	90	102	80	3.5		
1215	45	255.41	1.0	.89	.89	90	102	80	3.5		
1220	50	258.61	1.0	1.15	1.15	90	104	82	4.0		
1225	55	261.70	1.0	1.08	1.08	90	106	84	4.0		
1230	60	264.09	1.0	.78	.78	90	106	84	3.2		
1235	65	267.04	1.0	.72	.72	90	106	84	3.2		
1240	70	269.28	1.0	.82	.82	90	104	88	3.0		
1245	75	271.80	1.0	.82	.82	90	104	88	3.0		
1250	80	274.51	1.0	.87	.87	90	106	88	3.8		
1255	85	276.83	1.0	.87	.87	90	96.63	79.75	6.0	TEST	
1300	90	4.73	1.0			90	87.94				

COMMENTS:

130°

FIELD DATA

PLANT U.S. Steel mine #50
 DATE 8-30
 SAMPLING LOCATION Normal position (1800)
 SAMPLE TYPE particulates
 RUN NUMBER RAC stationary #2
 OPERATOR RAC
 AMBIENT TEMPERATURE _____
 BAROMETRIC PRESSURE 28.78
 STATIC PRESSURE, (P_s) _____
 FILTER NUMBER (s) 45, 3005

PROBE LENGTH AND TYPE 8'
 NOZZLE I.D. 2.01
 ASSUMED MOISTURE, % 13
 SAMPLE BOX NUMBER _____
 METER BOX NUMBER _____
 METER AH® 1.85
 C FACTOR 1.40
 PROBE HEATER SETTING 250
 HEATER BOX SETTING 112
 REFERENCE ΔP _____

SCHEMATIC OF TRAVERSE POINT LAYOUT
 READ AND RECORD ALL DATA EVERY _____ MINUTES

TRAVERSE POINT NUMBER	CLOCK TIME (min)	GAS METER READING (V _m) (ft)	VELOCITY HEAD (ΔP _s) in. H ₂ O	ORIFICE DIFFERENTIAL (ΔH) in. H ₂ O		STACK TEMPERATURE (T _s) °F	DRY GAS METER TEMPERATURE		PUMP VACUUM in. Hg	SAMPLE BOX TEMPERATURE °F	IMPINGING TEMPERATURE °F
				DESIRED	ACTUAL		INLET (T _{m in}) °F	OUTLET (T _{m out}) °F			
9145	0	278.31	1.82	1.23	1.30	95	70	64	2.80	start up	
9150	5	279.99	1.85	1.28	1.30		74	64	2.80	slow	
9155	10	282.7	1.86	1.35	1.35		80	66	3.0		
1000	15	285.4	1.86	1.35	1.35		80	66	3.2		
1005	20	289.12	1.86	1.35	1.35		92	70	3.2		
1010	25	291.9	1.87	1.37	1.35		97	70	3.2		
1015	30	295.34	1.89	1.38	1.38		97	72	3.4		
1020	35	298.60	1.89	1.38	1.38		102	76	3.4		
1025	40	301.4	1.86	1.34	1.24		104	78	3.5		
1030	45	304.81	1.84	1.3	1.3		104	80	3.4		
1035	50	308.18	1.80	1.25	1.25		104	80	3.5		
1040	55	311.2	1.82	1.20	1.20		104	80	3.5		
1045	60	314.58	1.87	1.37	1.37		98	80	4.0		
1050	65	317.30	1.87	1.37	1.37		99	81	4.2		
1055	70	320.40	1.87	1.37	1.37		99	81	4.5		
1100	75	324.0	1.87	1.37	1.37		100	82	4.4		
1105	80	327.8	1.87	1.37	1.37		100	82	4.4		
1110	85	330.40	1.87	1.37	1.37		102	83	4.8		
1115	90	334.15	1.87	1.37	1.37		102	83	4.8		
1120	95	337.0	1.87	1.37	1.37		102	83	4.8		
1125	100	340.56	1.87	1.37	1.37		104	84	5.0		
1130	105	343.90	1.87	1.37	1.37		105	85	5.0		
1135	110	347.08	1.87	1.37	1.37		105	86	5.0		

COMMENTS:

863 1351 97.25 77.79

2518

Station 1950
100' from highest

PLANT U.S. STEEL #50 MINE
 DATE 8-30
 SAMPLING LOCATION particular
 SAMPLE TYPE GT #1
 RUN NUMBER OK
 OPERATOR OK
 AMBIENT TEMPERATURE 28.7
 BAROMETRIC PRESSURE 28.7
 STATIC PRESSURE (P_s) 28.7
 FILTER NUMBER (s) 27, 30, 27

PROBE LENGTH AND TYPE 8'
 NOZZLE I.D. .125
 ASSUMED MOISTURE, % 13
 SAMPLE BOX NUMBER
 METER BOX NUMBER
 METER ΔH₀ 1.6
 C FACTOR 1.1
 PROBE HEATER SETTING
 HEATER BOX SETTING
 REFERENCE #

SCHEMATIC OF TRAVERSE POINT LAYOUT
 READ AND RECORD ALL DATA EVERY _____ MINUTES

START 09:45

TRAVERSE POINT NUMBER	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V _m), ft ³	VELOCITY HEAD (ΔP _s), in. H ₂ O	GRIFICE PRESSURE DIFFERENTIAL (ΔH), in. H ₂ O		STACK TEMPERATURE (T _s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
				DESIRED	ACTUAL		INLET (T _{m in}), °F	OUTLET (T _{m out}), °F			
0		807.3	.85	1.4	1.4	130			1		
5		826	.85	1.4	1.4						
10		852.9	.86	1.35	1.35						
15		88.95	.86	1.35	1.35						
20		93.17	.86	1.35	1.35						
25		96.25	.86	1.35	1.35						
30		99.91	.87	1.32	1.37						
35		103.20	.87	1.38	1.38						
40		106.75	.87	1.38	1.38						
45		110.43	.86	1.35	1.35						
50		114.25	.84	1.30	1.30						
55		112.80	.80	1.25	1.25						
60		121.75	.82	1.28	1.30						
65		125.93	.82	1.37	1.37						
70		128.20	.82	1.37	1.37						
75		137.00	.87	1.37	1.37						
80		172.20	.87	1.37	1.37						
85		140.50	.87	1.37	1.37						
90		144.40	.87	1.37	1.37						
95		148.10	.90	1.40	1.40						
100		152.20	.87	1.37	1.37						
105		152.95	.87	1.37	1.37						
110		159.85	.87	1.37	1.37						

COMMENTS:

89.08

FIELD DATA

PLANT US Steel 50 mine
 DATE 8-31
 SAMPLING LOCATION part
 SAMPLE TYPE RAC 3
 RUN NUMBER 180
 OPERATOR gn
 AMBIENT TEMPERATURE 77
 BAROMETRIC PRESSURE 28.8
 STATIC PRESSURE (P) 0.5" H₂O
 FILTER NUMBER (s) 6, 3625

PROBE LENGTH AND TYPE 0'
 NOZZLE I.D. 2
 ASSUMED MOISTURE, % 13
 SAMPLE BOX NUMBER 1015
 METER BOX NUMBER 1015
 METER ΔH 1.97 (21)
 C FACTOR 1.00
 PROBE HEATER SETTING 250
 HEATER BOX SETTING 1.22
 REFERENCE ΔP 1.22

SCHEMATIC OF TRAVERSE POINT LAYOUT
 READ AND RECORD ALL DATA EVERY _____ MINUTES

TRAVERSE POINT NUMBER	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V _m), ft ³	VELOCITY HEAD (Δp), in. H ₂ O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H ₂ O		STACK TEMPERATURE (T _s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	HIPPER TEMPERATURE, °F
				DESIRED	ACTUAL		INLET (T _{m in}), °F	OUTLET (T _{m out}), °F			
1015	0	354.46	.84	1.28	1.28	130°	68	66			start
	5	357.22	.84	1.28	1.28		74	60	3.0		
	10	360.7	.82	1.22	1.22		80	66	3.0		
	15	362.98	.82	1.22	1.22		84	68	3.0		
	20	366.20	.80	1.20	1.20		90	70	3.0		
	25	368.3	.84	1.28	1.28		94	72	3.2		
	30	371.3	.84	1.28	1.28		97	74	3.2		
	35	374.49	.82	1.22	1.22		100	78	3.2		
	40	377.43	.82	1.22	1.22		103	80	3.2		
	45	380.67	.82	1.22	1.22		103	80	3.2		
	50	383.67	.82	1.22	1.22		104	83	3.2		
	55	386.72	.85	1.28	1.28		110	86	4.0		
	60	390.70	.78	1.18	1.18		115	88	4.0		
	65	394.71	.78	1.18	1.18		117	90	3.4		
END	70	394.805	.819	1.231	1.231		97.77	77			end down part of coal
	75										
	80	40.345					87.39				
	85										
	90										

COMMENTS:

CF #2
350

FIELD DATA

PLANT US Steel Mining Co
 DATE 8-31
 SAMPLING LOCATION 9'
 SAMPLE TYPE CF #2
 RUN NUMBER 197
 OPERATOR for 74
 AMBIENT TEMPERATURE 74
 BAROMETRIC PRESSURE 28.8
 STATIC PRESSURE (P_s) 0.5" H₂O
 FILTER NUMBER(S) #8 15625

SCHEMATIC OF TRAVERSE POINT LAYOUT

PROBE LENGTH AND TYPE 8'
 NOZZLE I.D. 1.04
 ASSUMED MOISTURE, % 13
 SAMPLE BOX NUMBER
 METER BOX NUMBER
 METER AH 197
 C FACTOR 98
 PROBE HEATER SETTING
 HEATER BOX SETTING
 REFERENCE AD 1.22

READ AND RECORD ALL DATA EVERY _____ MINUTES

TRAVERSE POINT NUMBER	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V _m) ^{1/2}	VELOCITY HEAD (V _p) ^{1/2} , in. H ₂ O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H ₂ O		STACK TEMPERATURE (T _s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
				DESIRED	ACTUAL		INLET (T _{m in}), °F	OUTLET (T _{m out}), °F			
0	10:15	164.04	.84	1.20	1.28	130	65	66	1.5		
5	10:20	165.86	.84	1.22	1.22		74	66	1.0		
10	10:25	172.6	.82	1.22	1.22		80	66	1.0		
15	10:30	174.86	.82	1.22	1.22		84	68	1.0		
20	10:35	178.30	.80	1.20	1.20		90	70	1.0		
25	10:40	181.22	.84	1.28	1.28		94	72	1.0		
30	10:45	184.8	.84	1.28	1.28		97	74	1.0		
35	10:50	188.63	.82	1.22	1.22		100	78			
40	10:55	192.06	.82	1.22	1.22		103	80			
45	11:00	195.61	.82	1.22	1.22		103	80			
50	11:05	199.25	.82	1.22	1.22		106	83			
55	11:00	202.7	.82	1.28	1.28		110	86			
60	11:15	207.5	.78	1.18	1.18		115	89	1.5		
65	11:20	209.90	.78	1.18	1.18		117	90			
70	11:25	212.709									
75	11:30										
80	11:35										
85	11:40										
90	11:45										

COMMENTS:

end year out of coal

LABORATORY TEST SHEET

LABORATORY

4ND-GEN-112B

TEST OF

Pineville Coal Cleaner

1251-4

TEST ENGINEER

OBSERVERS

DATE

8/29/72

TEST EQUIPMENT

SO₂ - EPA

Sample #	Location	Start Time	Baro Press	Amb Temp	Stop Time	Sample Volume				
Test # 0										
1	out	1137	28.7	74	1208	32L				
1	inlet	1137	28.7	74	1207	30L				
Test # 1										
8/30/72										
1	out	0949	28.79	65	1024	30L				
1	in	0948	28.79	65	1016	30L				
Test # 3										
8/31/72										
1	out	1025	1055	74	28.8	30L				
1	in	1017	1047	74	28.8	30L				

LABORATORY TEST SHEET

LABORATORY

4ND - GEN - 1128

TEST OF

TEST ENGINEER

OBSERVERS

DATE

29 Aug 72

TEST EQUIPMENT

THC & Asat Bag Sampler - EPA

Start Time	Stop Time	Test # 0			
1126	1200				
CO ₂	100.0	100.0		100.0	
	97.3	97.4		97.3	
	2.7 %	2.6 %		2.7 %	
O ₂	97.3	97.4	97.4	97.3	
	79.3	79.6	79.4	79.3	
	18.0 %	18.0 %	18.0 %	18.0 %	
CO	79.3	79.4		79.3	
	79.3	79.4		79.3	
	0.0 %	0.0		0.0 %	
THC		26.0 ppm C ₃ H ₈	→	78.0 ppm C	

LABORATORY TEST SHEET

LABORATORY

4ND - GEN - 1128

TEST OF

Pineville W.Va. Coal Cleaner

TEST ENGINEER

OBSERVERS

DATE

30 Aug 72

TEST EQUIPMENT

JHC's Orsat Sampling Bag - EPA

Sample

Time start Time stop

Test # 1

Sample #	Time start	Time stop	Sample # 1			Sample # 2		
# 1	0955	1055						
# 2	1100	1140						
CO ₂	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
	98.3	98.3	98.2	98.3	98.4	98.4	98.3	98.3
	1.7 %	1.7 %	1.8	1.7 %	1.6 %	1.6 %	1.7 %	1.7 %
O ₂	98.3	98.3	98.2	98.3	98.4	98.4	98.3	98.3
	80.0	79.9	79.9	79.9	79.5	79.6	79.5	79.5
	18.3 %	18.4 %	18.3	18.4 %	18.9	18.8 %	18.8 %	18.8 %
CO	80.0	79.9	79.9	79.9	79.5	79.6	79.5	79.5
	80.0	79.9	79.9	79.9	79.5	79.6	79.5	79.5
	0.0 %	0.0 %	0.0	0.0 %	0.0 %	0.0 %	0.0 %	0.0 %
THC	16.0 ppm-C ₃				16.0 ppm-C ₃			
	→ 48.0 ppm-C				→ 48.0 ppm-C			

LABORATORY TEST SHEET

4ND. GEN. 1128

TEST OF Pineville Coal Cleaner

TEST ENGINEER

OBSERVERS

DATE

31 Aug 72

TEST EQUIPMENT

THC & Ocaf Bag Sampler - EPA

Bag #	Start Time	Stop Time	Test # 2											
			Test # 2			Bag 1			Test # 3			Bag 2		
1	0900	0940												
1	1035	1055												
2	1115	1122												
			Test # 2			Bag 1			Test # 3			Bag 2		
CO ₂	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0		
	99.0	98.9	99.0	98.9	99.0	99.0	98.9	99.0	99.0	98.8	98.7	98.8		
	1.0%	1.1	1.0	1.1	1.0	1.0	1.1	1.0	1.0	1.2	1.3	1.2		
O ₂	99.0	98.9	99.0	98.9	99.0	99.0	98.9	99.0	99.0	98.8	98.7	98.8		
	80.2	80.0	80.1	79.6	79.5	79.6	79.6	79.5	79.6	80.0	79.8	80.0		
	18.8	18.9	18.9	19.3	19.5	19.4	19.3	19.5	19.4	18.8	18.9	18.8		
CO	80.2	80.0	80.1	79.6	79.5	79.6	79.6	79.5	79.6	80.0	79.8	80.0		
	80.2	80.0	80.1	79.6	79.5	79.6	79.6	79.5	79.6	80.0	79.8	80.0		
	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0		
THC	12.0	ppm-C ₃		12.0	ppm-C ₃		10.0	ppm-C ₃		10.0	ppm-C ₃			
	36.0	ppm-C		36.0	ppm-C		30.0	ppm-C		30.0	ppm-C			

SRL 1281 45 0972

APPENDIX D

STANDARD SAMPLING PROCEDURES

The sampling procedures used during the test are the same as those published in the Federal Register, Volume 36, Number 247, Thursday, December 23, 1971. These methods are as follows (Methods 1, 2, 3, 5, 6, and 7). In addition, the impinger catch was analyzed.

The only departure from the method outlined was that a 16 foot stainless steel probe was used rather than a heated glass probe. Heated glass probes, however, were used for the stationary samples.



method(s) prescribed by the manufacturer(s) of such instrument, the instrument shall be subject to manufacturer's 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 SO₂ 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, $lb./hr. = Q_a \times C$, where Q_a = 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 SO₂ 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:

$$\text{equivalent diameter} = 2 \left(\frac{\text{length} \times \text{width}}{\text{length} + \text{width}} \right) \quad \text{equation 1-1}$$

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 0.87.

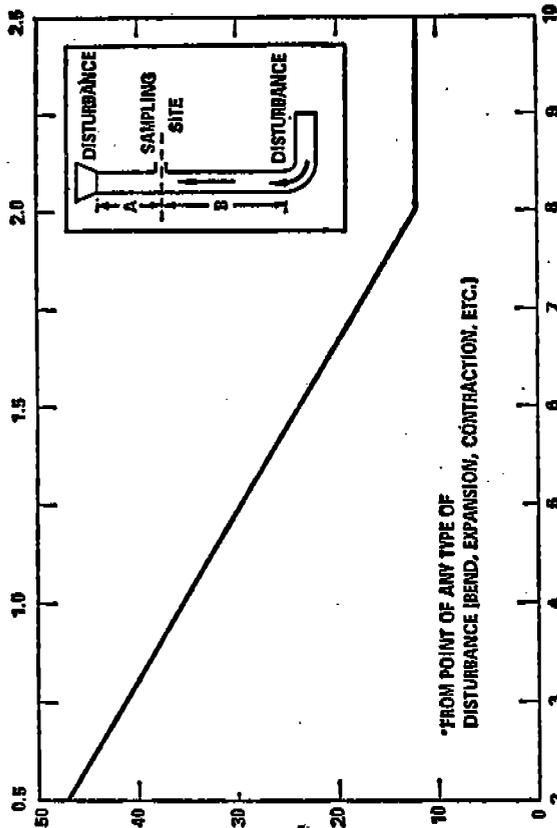
2.1.4 To use Figure 1-1 first measure the distance from the chosen sampling location

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

2.2 Cross-sectional layout and location of traverse points.

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

NUMBER OF DUCT DIAMETERS UPSTREAM*
(DISTANCE A)



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

NUMBER OF DUCT DIAMETERS DOWNSTREAM*
(DISTANCE B)

Figure 1-1. Minimum number of traverse points.

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	34.4	37.5	29.6	25.0	21.8	19.4
9					91.8	82.3	73.1	62.5	38.2	30.6	26.1	23.0
10					97.5	88.2	79.9	71.7	61.8	38.8	31.5	27.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2
14							98.2	91.5	85.4	79.6	73.9	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.5	92.1
22											98.9	94.5
23												96.8
24												98.9

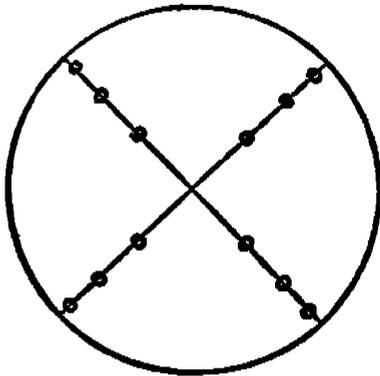


Figure 1-2. Cross section of circular stack divided into 12 equal areas, showing location of traverse points at centroid of each area.

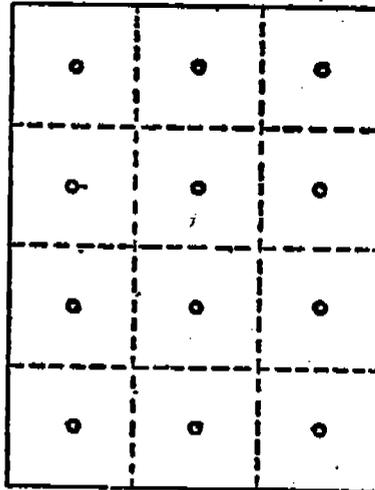


Figure 1-3. Cross section of rectangular stack divided into 12 equal areas, with traverse points at centroid of each area.

RULES AND REGULATIONS

2.2.2 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 28, Philadelphia, Pa. 1971, ASTM Designation D-2928-71.

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

1. Principle and applicability.

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

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

cedures for determining compliance with the New Source Performance Standards.

2. Apparatus.

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

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

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

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

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

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

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

3. Procedure.

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

3.2 Measure the static pressure in the stack.

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

4. Calibration.

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

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

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

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.

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

where:

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

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

C_p = Pitot tube coefficient, dimensionless.

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

$(\sqrt{\Delta P})_{avg.}$ = 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.

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

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

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

Figure 2-3 shows a sample recording sheet for velocity traverse data. Use the averages in the last two columns of Figure 2-2 to determine the average stack gas velocity from Equation 2-2.

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

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

where:

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

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

T_{std} = Absolute temperature at standard conditions, 68°F .

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

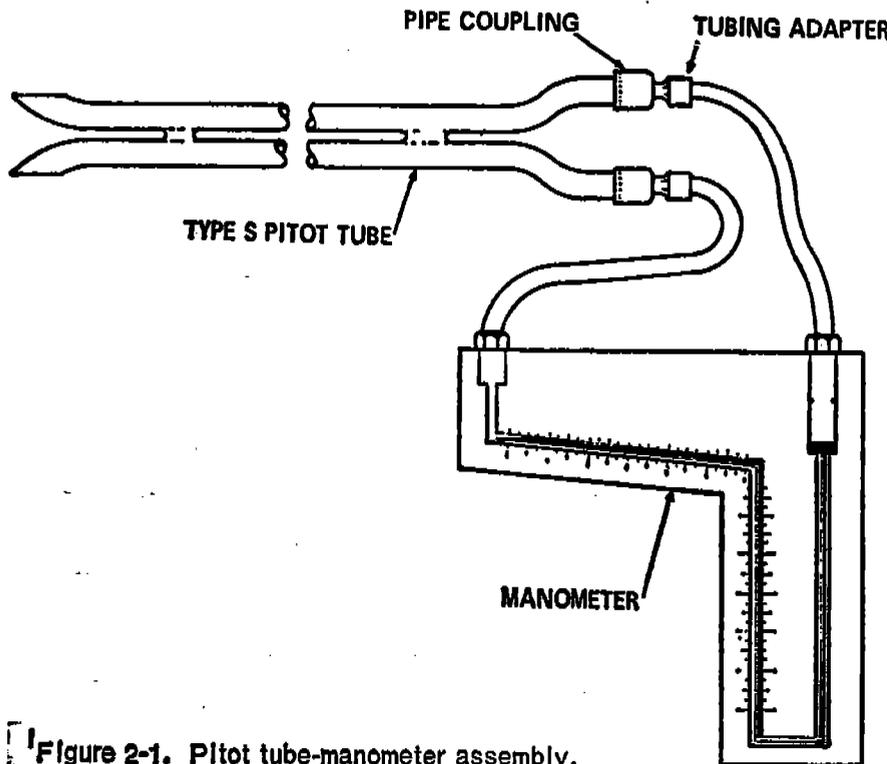


Figure 2-1. Pitot tube-manometer assembly.

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

1. Principle and applicability.

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

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

2. Apparatus.

2.1 Grab sample (Figure 3-1).

2.1.1 Probe—Stainless steel or Pyrex¹ glass, equipped with a filter to remove particulate matter.

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

¹ Trade name.

2.2 Integrated sample (Figure 3-2).

2.2.1 Probe—Stainless steel or Pyrex¹ 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 rate from 0 to 0.035 cfm.

2.2.6 Flexible bag—Tedlar¹ 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 CO₂, O₂, 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% CO₂.

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.

% O₂ = Percent oxygen by volume, dry basis.

% N₂ = 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.

% CO₂ = Percent carbon dioxide by volume, dry basis.

% O₂ = Percent oxygen by volume, dry basis.

% N₂ = 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.

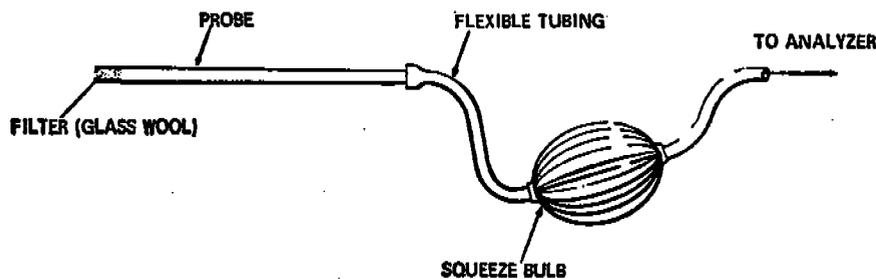


Figure 3-1. Grab-sampling train.

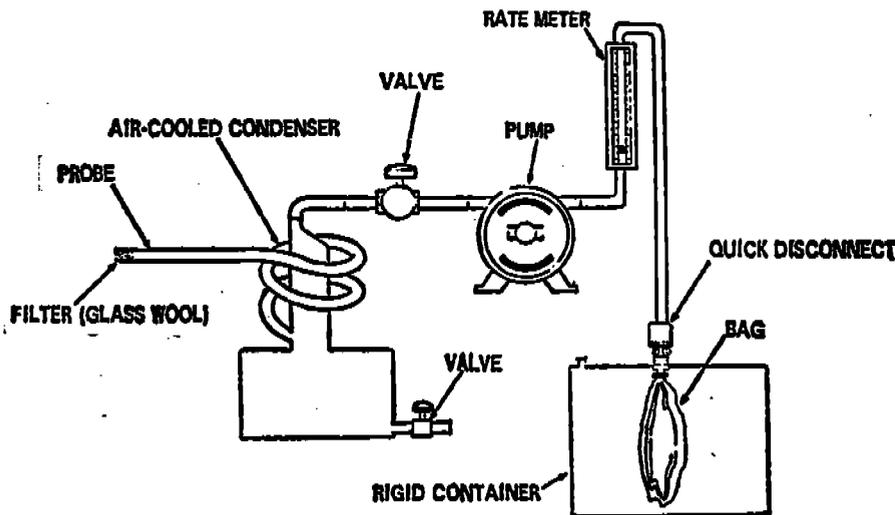


Figure 3-2. Integrated gas sampling train.

5. *References.*
- Altschuler, 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 Control District, Los Angeles, Calif., November 1968.

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 stream¹ 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[®] glass sufficiently heated to prevent condensation

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.

$$V_{w0} = \frac{(V_1 - V_2) P_{H_2O} R T_{std}}{P_{std} M_{H_2O}}$$

where:
 V_{w0} = Volume of water vapor collected (standard conditions), cu. ft.
 V_1 = Final volume of impinger contents, ml.
 V_2 = Initial volume of impinger contents, ml.
 R = Ideal gas constant, 21.83 inches

- 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—26 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,

$$V_{w0} = 0.0474 \frac{ft.^3}{ml.} (V_1 - V_2) \quad \text{equation 4-1}$$

Hg—cu. ft./lb. mole²·R.
 P_{H_2O} = Density of water, 1 g./ml.
 T_{std} = Absolute temperature at standard conditions, 680° R.
 P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.
 M_{H_2O} = Molecular weight of water, 18 lb./lb.-mole.

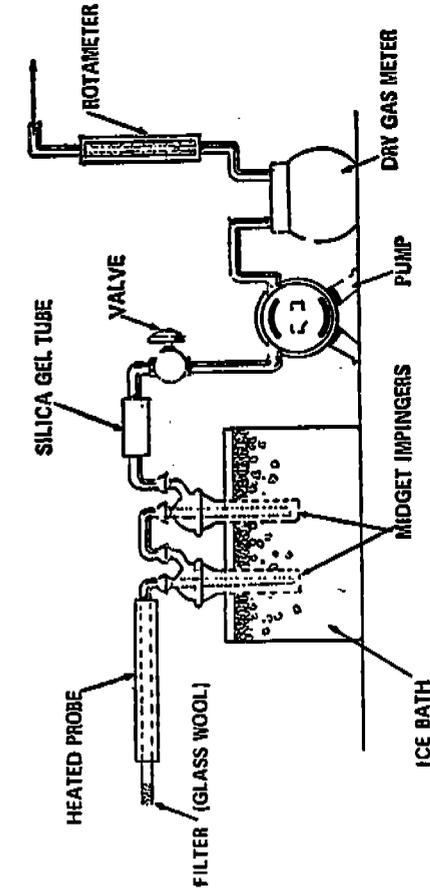


Figure 4-1. Moisture-sampling train.

LOCATION _____ COMMENTS _____
 TEST _____
 DATE _____
 OPERATOR _____
 BAROMETRIC PRESSURE _____

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

Figure 4-2. Field moisture determination.

4.2 Gas volume.

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

where:

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

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

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

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

T_{std} = Absolute temperature at standard conditions, 580° R.

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

4.3 Moisture content.

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

where:

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

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

V_{mo} = 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. DEW, 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, 1963.

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—Pyrex¹ 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 825¹, or equivalent, may be used. Probes for sampling gas streams at temperatures in excess of 600° F. must have been approved by the Administrator.

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

¹ Trade name.

2.1.4 Filter Holder—Pyrex¹ glass with heating system capable of maintaining minimum temperature of 225° F.

2.1.5 Impingers/Condenser—Four impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a 1/2-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 BH¹, 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.

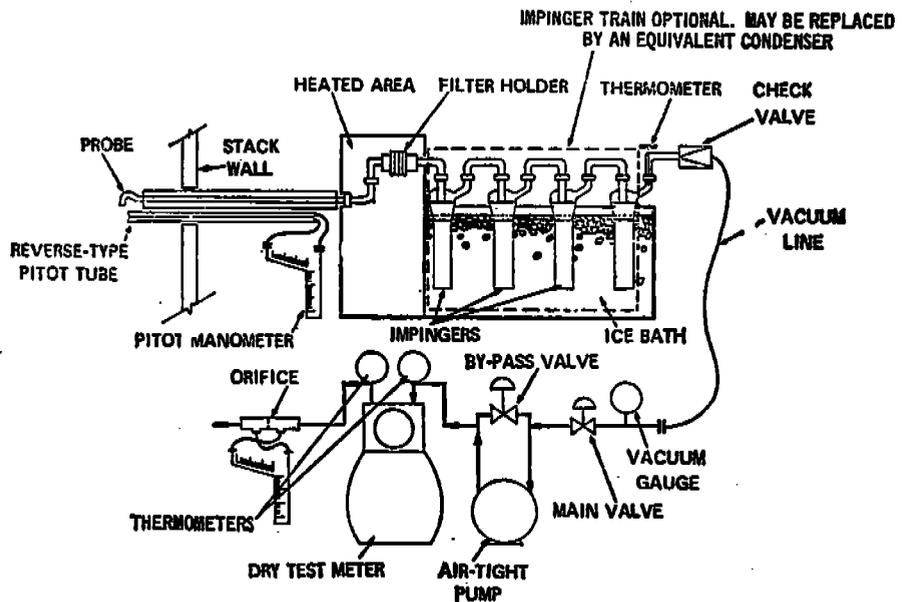


Figure 5-1. Particulate-sampling train.

3.3.2 Desiccant—Drierite¹, 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, desiccate² 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

¹ Trade name.

² Dry using Drierite¹ at 70° F. ± 10° F.

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.

RULES AND REGULATIONS

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 ^a 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.

$$c_s = \frac{\left(\frac{1 \text{ lb.}}{453,600 \text{ mg.}}\right) M_p}{V_{m, std}} = 2.205 \times 10^{-6} \frac{M_p}{V_{m, std}}$$

equation 5-5

where:

c_s = Concentration of particulate matter in stack gas, lb./s.c.f., dry basis.
 453,600 = Mg/lb.

M_p = 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.

$$I = \frac{T_s \left[\frac{V_{10} (\rho_{H_2O}) R}{M_{H_2O}} + \frac{V_m}{T_m} (P_{bar} + \frac{\Delta H}{13.6}) \right]}{\theta V_s P_s A_n} \times 100$$

$$= \frac{(1.667 \frac{\text{min.}}{\text{sec.}}) \left[\left(0.00287 \frac{\text{in. Hg-cu. ft.}}{\text{ml.}^\circ R} \right) V_{10} + \frac{V_m}{T_m} (P_{bar} + \frac{\Delta H}{13.6}) \right]}{\theta V_s P_s A_n}$$

Equation 5-6

where:

- I = Percent of isokinetic sampling.
- V_{10} = 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 H₂O.
- 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 = 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 14-18, 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—Pyrex¹ glass, approximately 5 to 6 mm. ID, with a heating system to prevent condensation and a filtering medium to remove particulate matter including sulfuric acid mist.

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

2.1.3 Glass wool.

2.1.4 Midget impingers—Three.

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

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

2.1.7 Pump—Leak-free, vacuum type.

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

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

2.1.10 Pitot tube—Type S, or equivalent,

¹ Trade names.

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

2.2.1 Glass wash bottles—Two.
 2.2.2 Polyethylene storage bottles—70 store impinger samples.
 2.3 Analysis.

velocity. Take readings at least every five minutes and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the tip of the probe at the first sampling point and start the pump. Sample proportionally throughout the run. At the conclusion of each run, turn off the pump and record the final 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 shipping 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 thorn indicator. Titrate to a pink endpoint using 0.01 N barium perchlorate. Run a blank with each series of samples.

6. Calibration.

6.1 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.

$$V_{m, std} = V_m \left(\frac{T_{std}}{T_m} \right) \left(\frac{P_{bar}}{P_{std}} \right) \quad \text{equation 6-1}$$

where:
 $V_{m, std}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.
 V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.
 T_{std} = Absolute temperature at standard conditions, 530° R.
 T_m = Average dry gas meter temperature, °R.
 P_{bar} = Barometric pressure at the orifice meter, inches Hg.
 P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.2 Sulfur dioxide concentration.

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, 1966.
 Corbett, P. F., The Determination of SO₂ and SO₃ in Flue Gases, Journal of the Institute of Fuel, 24:237-243, 1931.
 Matty, R. E. and E. K. Diehl, Measuring Flue-Gas SO₂ and SO₃, 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, 163 (1968).

METHOD 7—DETERMINATION OF NITROGEN OXIDES
 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

where:
 $C_{SO_2} = (7.05 \times 10^{-6} \frac{lb.-l.}{g.-ml.}) \frac{(V_1 - V_2) N \left(\frac{V_{std}}{V_1} \right)}{V_{m, std}} \quad \text{equation 6-2}$

C_{SO_2} = Concentration of sulfur dioxide at standard conditions, dry basis, lb./cu. ft.
 7.05×10^{-6} = 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_1 = Volume of barium perchlorate titrant used for the sample, ml.
 V_2 = Volume of barium perchlorate titrant used for the blank, ml.
 N = Normality of barium perchlorate titrant, g.-eq./l.
 V_{std} = Total solution volume of sulfur dioxide, 50 ml.
 V_m = 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.

2.1 Glass wash bottles—Two.
 2.2 Polyethylene storage bottles—70 store impinger samples.
 2.3 Analysis.

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 2.3 Analysis.

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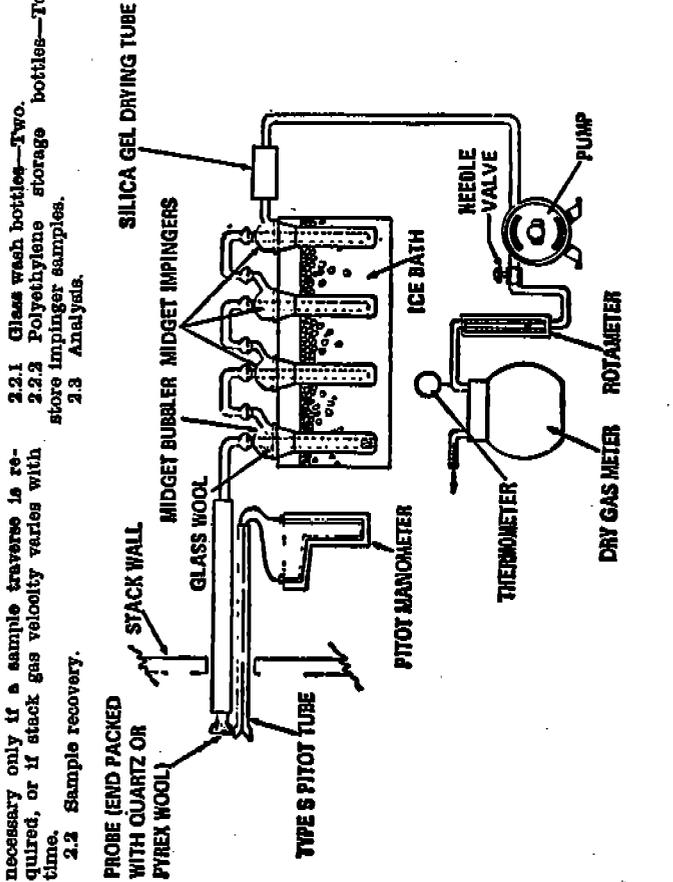


Figure 6-1. SO₂ sampling train.

8.1.1 Pipettes—Transfer type, 5 ml. and 10 ml. sizes (0.1 ml. divisions) and 25 ml. size (0.2 ml. divisions).
 8.1.2 Volumetric flasks—50 ml., 100 ml., and 1,000 ml.
 8.1.3 Burettes—5 ml. and 50 ml.
 8.1.4 Erlenmeyer flask—125 ml.
 8. Reagents.
 8.1.1 Water—Deionized, distilled.
 8.1.2 Isopropanol, 80%—Mix 80 ml. of isopropanol with 20 ml. of distilled water.
 8.1.3 Hydrogen peroxide, 3%—Dilute 100 ml. of 30% hydrogen peroxide to 1 liter with distilled water. Prepare fresh daily.
 8.2 Sample recovery.
 8.2.1 Water—Deionized, distilled.
 8.2.2 Isopropanol, 80%.
 8.3 Analysis.
 8.3.1 Water—Deionized, distilled.
 8.3.2 Isopropanol.
 8.3.3 Thorn indicator—1-(O-arsomophenylazo)-2-naphthol-3,6-disulfonic acid, disodium salt (or equivalent). Dissolve 0.20 g. in 100 ml. distilled water.
 8.3.4 Barium perchlorate (0.01 N)—Dissolve 1.96 g. of barium perchlorate [Ba(ClO₄)₂ · 3H₂O] in 200 ml. distilled water

and dilute to 1 liter with isopropanol. Standardize with sulfuric acid. Barium chloride may be used.
 8.3.5 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

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nitrous oxide, are measure colorimetrically using the phenol-disulfonic 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—Pyrex¹ 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, Pyrex¹ round bottom with short neck and 24/40 standard taper opening, protected against implosion or breakage.

¹ Trade name.

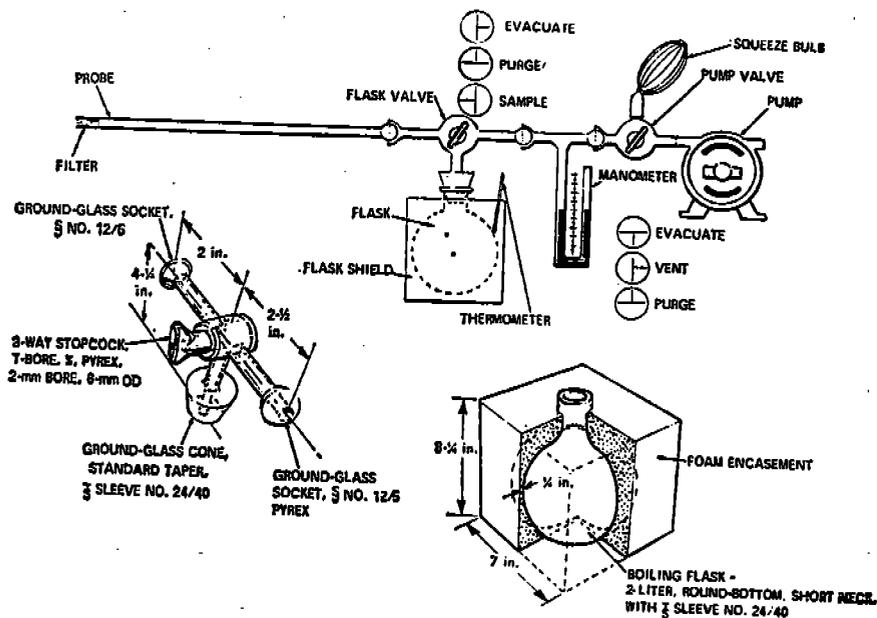


Figure 7-1. Sampling train, flask valve, and flask.

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

3.2.2 Red litmus paper.

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

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

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

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

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

2.1.8 Squeeze bulb—One way.

2.2 Sample recovery.

2.2.1 Pipette or dropper.

2.2.2 Glass storage containers—Cushioned for shipping.

positions. Evacuate the flask to at least 3 inches Hg absolute pressure. Turn the pump valve to its "vent" position and turn off the pump. Check the manometer for any fluctuation in the mercury level. If there is a visible change over the span of one minute, check for leaks. Record the initial volume, temperature, and barometric pressure. Turn the flask valve to its "purge" position, and then do the same with the pump valve. Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and flask valve area, heat the probe and purge until the condensation disappears. Then turn the pump valve to its "vent" position. Turn the flask valve to its "sample" position and allow sample to enter the flask for about 15 seconds. After collecting the sample, turn the flask valve to its "purge" position and disconnect the flask from the sampling train. Shake the flask for 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 (1N) dropwise into both the sample and the blank until alkaline to litmus paper (about 25 to 35 drops in each).

4.3 Analysis.

4.3.1 If the sample has been shipped in a container, transfer the contents to a 250 ml. beaker using a small amount of distilled water. Evaporate the solution to dryness on a steam bath and then cool. Add 2 ml. phenol-disulfonic 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 (1N) dropwise until alkaline to litmus paper (about 25 to 35 drops). Follow the analysis procedure of section 4.3 to collect enough data to draw a calibration curve of concentration in $\mu\text{g. NO}_2$ per sample versus absorbance.

6. Calculations.

6.1 Sample volume.

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 0.5496 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 $\mu\text{g.}$ nitrogen dioxide.

3.3.5 Water—Deionized, distilled.

3.3.6 Phenol-disulfonic 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"

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

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 $\mu\text{g. NO}_2$ for each sample from the plot of $\mu\text{g. NO}_2$ versus absorbance.

$$C = \left(\frac{m}{V_{sc}} \right) \left(\frac{1 \text{ lb.}}{\text{cu. ft.}} \right) = \left(6.2 \times 10^{-5} \frac{\text{lb./s.c.f.}}{\mu\text{g./ml.}} \right) \left(\frac{m}{V_{sc}} \right)$$

equation 7-2

where:

- C = Concentration of NO_2 as NO_2 (dry basis), lb./s.c.f.
- m = Mass of NO_2 in gas sample, $\mu\text{g.}$
- 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—Pyrex¹ 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—Pyrex¹ 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 1/2-inch ID glass tube extending to one-half inch from the bottom of the impinger flask. Similar collection systems, which have been approved by the Administrator, may be used.

2.1.6 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5° 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.

¹ Trade name.

- 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 1108 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, disodium salt (or equivalent). Dissolve 0.20 g. in 100 ml. distilled water.

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

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

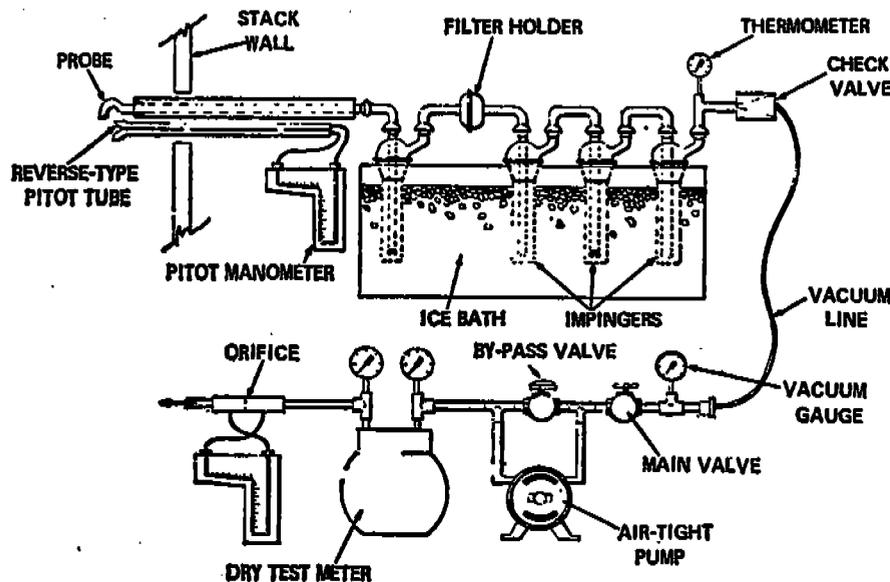


Figure 8-1. Sulfuric acid mist sampling train.

SRL 1281 45 0972

APPENDIX E

LABORATORY REPORT

E.1 ON-SITE HANDLING AND TRANSFER, PARTICULATE

After the completion of a test run, the probe and nozzle were disconnected from the impinger train and all open ends sealed immediately to avoid contamination. At the laboratory facility, the nozzle was disconnected from the probe and very carefully washed with acetone, using a fine bristled brush. All acetone washings were collected in a clean glass jar, the jar itself being placed on a large piece of clean aluminum foil. The probe was then washed using a long handled brush rotated through it under a continuous stream of acetone. The brush was also carefully cleaned, and all washings collected in the glass jar. The probe was finally checked visually for any residue. In some cases this was performed at the test site in order to minimize the time required between tests because of the small amount of coal available for processing.

The impinger train was initially wiped clean on the outside and all glassware connectors, including the filter, removed carefully and all exposed surfaces wiped clean. All the connectors were placed on a piece of aluminum foil ready for washing. The first three impingers were then analyzed for water collection by transferring the water through the outlet port into a graduated cylinder and noting the volume. The impingers were not dismantled and all transfers and washings were performed through the inlet and outlet ports. All of the glassware in the back half of the filter, up to the fourth impinger was then carefully washed with distilled water and the washings collected. This was followed by an acetone wash which was again collected in a separate jar.



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Acetone washings from the glassware in the front half of the filter were collected in the same jar as the probe and nozzle wash. The filter was carefully removed from the holder and placed in a plastic dish which was then sealed with tape. Silica gel in the fourth impinger was weighed in a previously tared glass jar using a triple-beam balance.

All acetone jars had Teflon lined lids. The following designations were used for labeling the containers:

- Container #1: Filter
- Container #2: Acetone wash front half from filter
- Container #3: Water wash back half from filter
- Container #4: Silica gel
- Container #5: Acetone wash back half from filter

In the case of the stationary samples only the first two containers were included in the total particulate catch.

E.2 LABORATORY HANDLING AND ANALYSIS, PARTICULATE

E.2.1 Filter Transfer

Clean plastic dishes were desiccated for 24 hours, labeled and tared on an electronic balance. The filter containers were unsealed and desiccated for 24 hours before carefully transferring the filters to the tared dishes using a fine pair of tweezers. Care was taken to place a piece of aluminum foil under the transfer operation. A "Staticmaster" brush was used to brush any fine particles adhering to the container or foil. All transfers were performed near the balance and the weight reported to the nearest 0.1 mg. The plastic dishes were then sealed for shipment.



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E.2.2 Acetone Washes

The 250 ml. beakers to be used for the acetone wash transfers were leached for 24 hours in 50% nitric acid, washed thoroughly and oven dried overnight. These were then desiccated for 24 hours and tared. Once tared, the beakers were sealed with "parafilm" and handled with tongs or "Kimwipes".

The jars containing the acetone washes were left loosely covered in a hood until the acetone was evaporated. Once the acetone was evaporated, the glass jar was rinsed with acetone, using a rubber policeman, and the washings collected in the tared beaker. When the acetone wash of the front half contained considerable particulate matter, the dried particulate cake was transferred carefully with a spatula into the tared beaker along with the final acetone rinse.

After the acetone had evaporated, the beakers were desiccated for 24 hours and weighed to a constant weight. Where water was present in the acetone wash, it was evaporated in an oven at 90°C after the acetone had all evaporated.

E.2.3 Water Washes

The level of water in the collection bottles was marked for later volume measurement. Each water wash was then transferred into a 2000 ml. separatory funnel and extracted three times with 25 ml. portions of chloroform. Often, where a large volume of water was collected (above 500 ml.) a fourth extraction was used. The chloroform extracts were collected directly in a tared beaker prepared in the same manner as described in the previous section.



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Extraction with three 50 ml. portions of ether followed, collecting the water portion in the original jars. The ether extracts were combined with the chloroform extracts. These were then washed with distilled water in the separatory funnel and returned to the tared beaker for evaporation.

The water portion was transferred to tared beakers, oven dried at 90°C, desiccated, and weighed. All beakers were "parafilm" sealed for shipment. The Project Officer requested that particle size analysis not be performed. A summary of weight measurements is shown in Tables E-1 and E-2.

E.3 ORSAT ANALYSIS

A total of six integrated bag samples were analyzed by Orsat during the three day test period. The Tedlar sample bags had a capacity of about 5 liters and were equipped with Teflon sample tubes fitted with airtight syringe caps. Prior to sampling, each bag was flushed with pure, dry nitrogen and sealed with the syringe cap.

At the end of each sampling day two sample bags (one inlet and one outlet) were returned to the field laboratory where they were analyzed for CO, CO₂, and O₂ by Orsat.

Each bag was connected to the Orsat analyzer by carefully removing the syringe cap and inserting the Teflon tube securely into the Orsat sample tube. The Orsat analyzer was then purged by squeezing the Tedlar bag and forcing the sample through the Orsat bypass. Successive 100 ml. samples were drawn into the Orsat sample burette and then passed



TABLE E-1 SUMMARY OF WEIGHT MEASUREMENTS
(INLET)

	Run 1				Run 2				Run 3				
	Gross (g)	Tare (g)	Net (mg)	Blank (mg)	Gross (g)	Tare (g)	Net (mg)	Blank (mg)	Gross (g)	Tare (g)	Net (mg)	Blank (mg)	Final (mg)
Container #1 (Filter)	1.8262	1.7930	33.2	-	1.8878	1.8545	33.3	-	1.8025	1.7880	14.5	-	14.5
Container #2 (Acetone wash front half)	96.8220	95.7450	1077	4.1	101.9525	100.8813	1071.2	3.5	96.1705	95.7730	395.5	2.5	395
Container #3a (Organic Extract)	99.3680	99.3670	1.0	0.0	98.4755	98.4755	0.0	0.0	100.7020	100.7020	0.0	0.0	0.0
Container #3b (Water after extraction)	95.3170	95.2935	23.5	0.0	97.7770	97.7170	60.0	0.0	95.6875	95.6735	14.0	0.0	14.0
Container #5 (Acetone wash back half)	100.0810	100.0735	7.5	1.6	98.9775	98.9680	9.5	1.4	100.1785	100.1740	4.5	1.4	3.0
Probe, Cyclone, Filter			1106.2		Probe, Cyclone, Filter		1101.3		Probe, Cyclone, Filter		409.5		409.5
Total			1136.7		Total		1169.3		Total		426.5		426.5



TABLE E-2 SUMMARY OF WEIGHT MEASUREMENTS
(OUTLET)

	Run 1 (RAC 135 ^o)				Run 2 (RAC 180 ^o)				Run 3 (GI 125 ^o)			
	Gross (g)	Tare (g)	Net (mg)	Blank Final (mg)	Gross (g)	Tare (g)	Net (mg)	Blank Final (mg)	Gross (g)	Tare (g)	Net (mg)	Blank Final (mg)
Container #1 (Filter)	1.8800	1.8600	20.0	-	1.8160	1.7860	30.0	-	1.8144	1.7825	31.9	-
Container #2 (Acetone wash front half)	99.1805	99.0395	141.0	4.6	98.5755	97.8620	713.5	4.0	93.7133	93.5990	114.3	6.3
Container #3a (Organic Extract)	-	-	-	-	-	-	-	-	-	-	-	-
Container #3b (Water after extraction)	-	-	-	-	-	-	-	-	-	-	-	-
Container #5 (Acetone wash back half)	-	-	-	-	-	-	-	-	-	-	-	-
Probe, Cyclone, Filter			156.0		Probe, Cyclone, Filter		740.0		Probe, Cyclone, Filter		139.9	
Total			156.0		Total		740.0		Total		139.9	



TABLE E-2 SUMMARY OF WEIGHT MEASUREMENTS
(OUTLET)
(continued)

	Run 4 (RAC 180°)				Run 5 (GI 125°)			
	Gross (g)	Tare (g)	Net (mg)	Blank Final (mg)	Gross (g)	Tare (g)	Net (mg)	Blank Final (mg)
Container #1 (Filter)	1.8123	1.7945	17.8	-	1.8005	1.7855	15.0	-
Container #2 (Acetone wash front half)	98.8850	97.9045	980.5	4.2	98.0869	98.0220	64.9	3.0
Container #3a (Organic extract)	-	-	-	-	-	-	-	-
Container #3b (Water after extraction)	-	-	-	-	-	-	-	-
Container #5 (Acetone wash back half)	-	-	-	-	-	-	-	-
Probe, Cyclone, Filter				993.8	Probe, Cyclone, Filter			77.0
Total				993.8	Total			77.0



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through each of the three absorbing solutions (potassium hydroxide - CO_2 , alkaline pyrogallate - O_2 , and cuprous chloride - CO). Repetitive passes were made through each absorbing solution until good duplication of results occurred. At least three 100 ml. samples were analyzed from each Tedlar sample bag. The data recorded for each Orsat analysis is included as Table E-3.

E.4 SO_2 ANALYSIS

A total of six SO_2 gas samples were taken during the course of the test program. Following each sampling period the impinger train was disconnected from the sample probe and purged with ambient air for fifteen minutes at the same flow rate used during the test. The inlet and outlet connections of the impinger train were then sealed with tape to prevent contamination and transported to the field laboratory for transfer. Upon arrival at the field laboratory, the outside surfaces of the impinger train were washed with water and then wiped clean to remove any coal dust that had accumulated during the test. The isopropanol bubbler was then carefully disconnected from the impinger section of the train and its contents discarded. The next two impingers were individually disconnected and the hydrogen peroxide solutions were transferred to separate polyethelene bottles with distilled water washes. The glass connecting tubes were then rinsed with distilled water and the washes added to their respective polyethelene bottles. The final transfer step involved rinsing the third impinger with distilled water and adding this wash to the number two impinger solution. The polyethelene bottles were tightly capped and labeled for shipment to the laboratory.



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TABLE E-3 ORSAT ANALYSIS DATA

Run #0 - August 29, 1972

Sample Number	Component	Analysis Number	Burette Volume (ml.)		
			Initial	Final	Difference
1	CO ₂	1	100.0	97.3	2.7
		2		97.3	2.7
	O ₂	1	97.3	79.3	18.0
		2		79.3	18.0
	CO	1	79.3	79.3	0.0
		2		79.3	0.0
2	CO ₂	1	100.0	97.4	2.6
		2		97.4	2.6
	O ₂	1	97.4	79.6	17.8
		2		79.4	18.0
		3		79.4	18.0
	CO	1	79.4	79.4	0.0
2			79.4	0.0	
3	CO ₂	1	100.0	97.3	2.7
		2		97.3	2.7
	O ₂	1	97.3	79.3	18.0
		2		79.3	18.0
	CO	1	79.3	79.3	0.0
		2		79.3	0.0

Run #1 - Bag #1 - August 30, 1972

1	CO ₂	1	100.0	98.3	1.7
		2		98.3	1.7
	O ₂	1	98.3	80.0	18.3
		2		80.0	18.3
	CO	1	80.0	80.0	0.0
		2		80.0	0.0
2	CO ₂	1	100.0	98.3	1.7
		2		98.3	1.7
	O ₂	1	98.3	79.9	18.4
		2		79.9	18.4
	CO	1	79.9	79.9	0.0
		2		79.9	0.0
3	CO ₂	1	100.0	98.2	1.8
		2		98.2	1.8
	O ₂	1	98.2	79.9	18.3
		2		79.9	18.3
	CO	1	79.9	79.9	0.0
		2		79.9	0.0



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TABLE E-3 ORSAT ANALYSIS DATA
(continued)

Run #1 - Bag #2 - August 30, 1972

Sample Number	Component	Analysis Number	Burette Volume (ml.)		
			Initial	Final	Difference
1	CO ₂	1	100.0	98.4	1.6
		2		98.4	1.6
	O ₂	1	98.4	79.5	18.9
		2		79.5	18.9
	CO	1	79.5	79.5	0.0
		2		79.5	0.0
2	CO ₂	1	100.0	98.4	1.6
		2		98.4	1.6
	O ₂	1	98.4	79.6	18.8
		2		79.6	18.8
	CO	1	79.6	79.6	0.0
		2		79.6	0.0
3	CO ₂	1	100.0	98.3	1.7
		2		98.3	1.7
	O ₂	1	98.3	79.5	18.8
		2		79.5	18.8
	CO	1	79.5	79.5	0.0
		2		79.5	0.0

Run #2 - August 31, 1972

1	CO ₂	1	100.0	99.0	1.0
		2		99.0	1.0
	O ₂	1	99.0	80.2	18.8
		2		80.2	18.8
	CO	1	80.2	80.2	0.0
		2		80.2	0.0
2	CO ₂	1	100.0	98.9	1.1
		2		98.9	1.1
	O ₂	1	98.9	80.0	18.9
		2		80.0	18.9
	CO	1	80.0	80.0	0.0
		2		80.0	0.0
3	CO ₂	1	100.0	99.0	1.0
		2		99.0	1.0
	O ₂	1	99.0	80.1	18.9
		2		80.1	18.9
	CO	1	80.1	80.1	0.0
		2		80.1	0.0



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TABLE E-3 ORSAT ANALYSIS DATA
(continued)

Run #3 - Bag #1 - August 31, 1972

Sample Number	Component	Analysis Number	Burette Volume (ml.)		
			Initial	Final	Difference
1	CO ₂	1	100.0	98.9	1.1
		2		98.9	1.1
	O ₂	1	98.9	79.6	19.3
		2		79.6	19.3
	CO	1	79.6	79.6	0.0
		2		79.6	0.0
2	CO ₂	1	100.0	99.0	1.0
		2		99.0	1.0
	O ₂	1	99.0	79.5	19.5
		2		79.5	19.5
	CO	1	79.5	79.5	0.0
		2		79.5	0.0
3	CO ₂	1	100.0	99.0	1.0
		2		99.0	1.0
	O ₂	1	99.0	79.6	19.4
		2		79.6	19.4
	CO	1	79.6	79.6	0.0
		2		79.6	0.0

Run #3 - Bag #2 - August 31, 1972

1	CO ₂	1	100.0	98.8	1.2
		2		98.8	1.2
	O ₂	1	98.8	80.0	18.8
		2		80.0	18.8
	CO	1	80.0	80.0	0.0
		2		80.0	0.0
2	CO ₂	1	100.0	98.7	1.3
		2		98.7	1.3
	O ₂	1	98.7	79.8	18.9
		2		79.8	18.9
	CO	1	79.8	79.8	0.0
		2		79.8	0.0
3	CO ₂	1	100.0	98.8	1.2
		2		98.8	1.2
	O ₂	1	98.8	80.0	18.8
		2		80.0	18.8
	CO	1	80.0	80.0	0.0
		2		80.0	0.0



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All SO₂ samples were analyzed by the Barium-Thorin Titration procedure. Each sample was transferred to either a 50 or 100 ml. volumetric flask with distilled water washes and then diluted to volume. A suitable aliquot of either 5 or 10 ml. was chosen and then pipeted to a 250 ml. Erlenmeyer flask. Isopropanol was then added to each sample in 4 to 1 proportions (isopropanol to sample aliquot) by volume. The titration was performed in the presence of four drops of Thorin indicator with a previously standardized solution of 0.0111 N barium perchlorate. A solution blank was titrated with each set of samples analyzed. Each sample was titrated twice or until good duplication of results was obtained. Table E-4 lists all titration data recorded. The titer volumes for each impinger sample pair were then summed and the normality of the sample solution was computed by the following formula:

$$N_s = \frac{V_T \times N_T}{V_s}$$

where: V_T = Volume of titer (ml.)
 N_T = Normality of titer (0.0111)
 V_s = Volume of sample aliquot (ml.)

From this information the milligrams of SO₂ per sample were calculated using the formula:

$$\text{mg SO}_2 = V_d \times N_s \times \text{meq. wt. SO}_2$$

where: V_d = Sample dilution volume (ml.)
 N_s = Normality of sample solution
 meq. wt. SO₂ = 32

The mg SO₂ for each sample were then converted to ppm as shown in Appendix B.



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E.5 NO_x ANALYSIS

Immediately after each NO_x flask sample was taken, the flask containing the absorbing solution and the gas sample was shaken for five minutes. The flask was then allowed to sit until the following morning when it was shaken again for two minutes. Following this final shake, the flask pressure was measured with a mercury manometer. Each flask was then carefully wiped off and the stopcocks removed. The absorbing solutions were then transferred to glass shipping bottles with two 10 ml. washes of distilled water. Just prior to shipping, the samples were neutralized with 1.0 N sodium hydroxide (approximately 40 drops). At this time solution blanks were made for each set of samples. The blanks contained 25 ml. of NO_x absorbing solution and 20 ml. of distilled water and were neutralized with 1.0 N sodium hydroxide. At the end of the test period all samples were transported to the laboratory for analysis.

All NO_x samples were analyzed by the Phenoldisulfonic acid procedure. Prior to analysis, a calibration curve was established for a suitable range of NO_x concentrations. From a standard potassium nitrate solution with an equivalent concentration of 25 µg NO₂ per ml. four aliquots of 4, 8, 12 and 16 ml. were added to respective 250 ml. beakers. Twenty-five ml. of NO_x absorbing solution was added to each of these beakers and the analysis procedure described below was followed. These solutions were read against a blank containing no standard solution and a calibration curve of % absorbance versus µg NO₂ was plotted.



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Upon arrival at the laboratory, each sample was transferred to a 250 ml. beaker and evaporated to dryness on a steam bath. After cooling, 2 ml. of phenoldisulfonic acid was added and each sample was triturated thoroughly with a glass stirring rod. One ml. of distilled water and four drops of concentrated sulfuric acid were added and the samples were returned to the steam bath for three minutes. The samples were then collected and 20 ml. of distilled water was added. Concentrated ammonium hydroxide was then added dropwise until each sample was alkaline to litmus paper. The samples were transferred to 100 ml. volumetric flasks with distilled water and portions of each solution were read at 420 m μ on a Bausch and Lomb Spectronic 20 Colorimeter. The solution blanks run with each set of samples were used for the colorimeter zero reference. The absorbances read for each sample were then converted to $\mu\text{g NO}_2$ via the previously established calibration curve. NO_x concentrations were calculated as ppm NO_2 following the procedure described in Appendix B. Table E-5 lists all the absorbance data for NO_x .

E.6 TOTAL HYDROCARBON ANALYSIS

Immediately following each Orsat analysis the remainder of the sample contained in each Tedlar bag was analyzed for hydrocarbons via a Beckman Model 108-A Total Hydrocarbon Analyzer. This instrument utilized a flame ionization detector and the following operating conditions were maintained during each analysis:

Sample Backpressure:	2.50 psi
Fuel Pressure:	23.0 psi
Oxidant Pressure	11.0 psi
Range:	100 X



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The Scott compressed gases used during each analysis were:

Fuel: 40% hydrogen in nitrogen

Oxidant: Blended Air

Zero: Hydrocarbon Free Air (<0.1 ppm-C)

Span: 99.9 ppm propane ($\pm 2.0\%$ analysis) in nitrogen

Just prior to introducing each sample into the analyzer, the instrument was zeroed and spanned on range 100. The Tedlar sample bag was then connected to the analyzer via a Teflon tube and the sample was drawn into the analyzer until a stable reading was recorded on the meter. The bag was then disconnected and resealed with the syringe cap. The instrument zero and span points were rechecked to insure that the calibration had not changed during the analysis. The complete analytical procedure was then repeated until good duplication of results were obtained. All meter readings recorded for each sample are included as Table E-4.

The meter readings were then converted to parts per million carbon by the following formula:

$$\text{ppm-C} = \text{meter units} \times \frac{99.9 \text{ ppm-C}_3\text{H}_8}{100 \text{ units-Span}} \times \frac{3 \text{ ppm-C}}{\text{ppm-C}_3\text{H}_8}$$

The final data with example calculations are included in Appendix B of this report.



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TABLE E-4 SO₂ ANALYSIS DATA

<u>Sample Location</u>	<u>Total Volume (ml.)</u>	<u>Analysis Number</u>	<u>Sample Aliq. (ml.)</u>	<u>Volume Titer (ml.)</u>	<u>Sample Normality</u>
Run #0 - August 29, 1972					
Inlet	61	1	10	0.1	9.8×10^{-5}
		2	10	0.1	9.8×10^{-5}
Outlet	65.5	1	10	0.0	0.0
		2	10	0.0	0.0
Run #1 - August 30, 1972					
Inlet	70	1	10	0.1	9.8×10^{-5}
		2	10	0.1	9.8×10^{-5}
Outlet	66	1	10	0.0	0.0
		2	10	0.0	0.0
Run #3 - August 31, 1972					
Inlet	64	1	10	0.3	2.94×10^{-4}
		2	10	0.3	2.94×10^{-4}
Outlet	70	1	10	0.0	0.0
		2	10	0.0	0.0



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TABLE E-5 NO_x ANALYSIS DATA

<u>Run No.</u>	<u>Date</u>	<u>Absorbance @ 420 mμ</u>	<u>NO_x Conc. (μg NO₂)</u>
0	8/29/72	0.202	151.1
1	8/30/72	0.188	140.6
1	8/30/72	0.319	238.6
1	8/30/72	0.257	192.2
3	8/31/72	0.210	157.1
3	8/31/72	0.200	149.6



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TABLE E-6 TOTAL HYDROCARBON DATA

<u>Run No.</u>	<u>Date</u>	<u>Sample No.</u>	<u>Range</u>	<u>Meter Units</u>	
				<u>Sample</u>	<u>Span</u>
0	8/29/72	1	100	26.0	100.0
		2	100	26.0	100.0
1-A	8/30/72	1	100	16.0	100.0
		2	100	16.0	100.0
1-B	8/30/72	1	100	16.0	100.0
		2	100	16.0	100.0
2	8/31/72	1	100	12.0	100.0
		2	100	12.0	100.0
3-A	8/31/72	1	100	12.0	100.0
		2	100	12.0	100.0
3-B	8/31/72	1	100	10.0	100.0
		2	100	10.0	100.0



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APPENDIX F

TEST LOG



SCOTT RESEARCH LABORATORIES, INC.

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TEST LOG

On Monday, August 28, 1972, the Scott team arrived at the U.S. Steel Coal plant in Pineville, West Virginia and set up the laboratory and test equipment. Preliminary velocity and temperature traverses were performed and an appropriate nozzle size selected for the EPA test train and for the two stationary trains. All equipment was assembled for testing to begin on Tuesday.

The first run was started at 1130 on Tuesday, August 29. One of the stationary sample trains (the GI unit) developed a leak in the filter holder, and it was decided to abandon this sample train for the day. The leak was traced to the presence of an "O" ring in the filter holder and all subsequent runs were performed satisfactorily without using the "O" ring.

At approximately the second to last point on the first traverse it was observed that something was wrong with the pitot tube reading. Upon inspection it was found that the probe and pitot tube had bent down into the stack. The test was stopped and the probe removed from the stack.

It was decided not to include this as a valid test. The probe was repaired and the equipment was made ready for an attempt to obtain two tests the next day.

Satisfactory SO₂ samples were collected at the inlet and outlet ports from 1137 to 1208. An NO_x sample was collected at 1135 and an Orsat and hydrocarbon sample collected from 1126 to 1200. One stationary train sample (RAC unit) was collected from 1130 to 1255. Fresh glassware and an entire backup system were made ready, and leak tests performed satisfactorily for an early start on Wednesday, August 30.



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On Wednesday a satisfactory EPA test was performed from 0945 to 1146. During this same period satisfactory samples were obtained from the two stationary trains. To expedite the start of the second EPA test, the probe washings were collected carefully at the test site while the rest of the glassware and train were dismantled, sealed and placed in impinger boxes to be transferred later to the laboratory facility. Fresh glassware was assembled for the second test at the site. Two Orsat and hydrocarbon samples were collected from 0955 to 1055 and 1100 to 1140. Inlet and outlet SO_2 samples were obtained from 0949 to 1024 and three NO_x samples collected at 0945, 1038 and 1125.

The second EPA test was progressing satisfactorily when it was learned that the plant had a process upset and it would be unlikely that the test could be finished that day. It was therefore decided to sample at one port, transfer to the other port and continue the test the next day. This first half of the second test was performed from 1243 to 1343.

On Thursday, August 31, the second half of the second test of the EPA train was completed from 0845 to 0947. Glassware transfer for the third EPA test was done on site and the third test started at 1013 along with the two stationary sampling trains. Two Orsat and hydrocarbon samples were collected from 1025 to 1053 and 1115 to 1122. Inlet and outlet SO_2 samples were obtained from 1025 to 1055 and two NO_x samples collected at 1020 and 1122.

By 1125 all the coal in the plant was depleted and the third test had to be ended prematurely halfway through the run. The glassware and sampling trains were transferred to the laboratory site for cleanup,



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and the test equipment was dismantled and packed. The Scott team travelled home on Friday.

Figure F-1 illustrates the test program schedule.



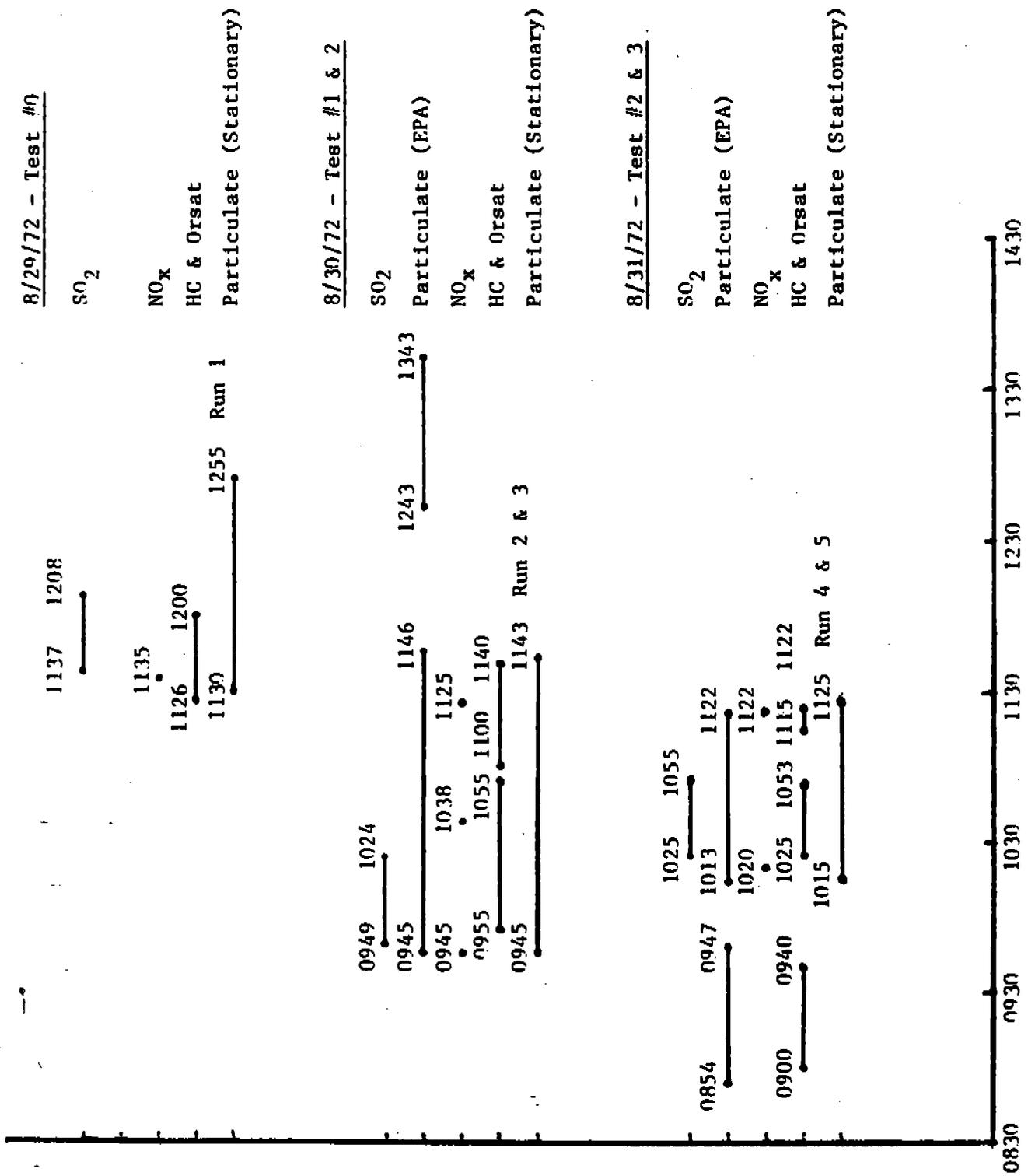


FIGURE F-1 SUMMARY OF TEST PROGRAM



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APPENDIX G

PROJECT PARTICIPANTS AND TITLES

The personnel taking part in the project include:

Thomas Ward	Project Officer - EPA
Charles Sedman	Project Engineer - EPA
Norman Troxel	Senior Engineer - SRL
Joseph Wilson	Field Team Leader - SRL
Jyotin Sachdev	Engineer - SRL
Jack Mycock	Engineer - SRL
William Scott	Technician - SRL
Duane Gulick	Technician - SRL
John Trump	Technician - SRL
Margaret Husic	Chemist - SRL
Louis Reckner	Manager, Atmospheric Chemistry & Industrial Emissions Department

