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.

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Monsanto Research Corp.

Monsanto Research Corp.

August 1974

(34)

PARTICULATE, SULFUR DIOXIDE, AND NITROGEN OXIDES EMISSION MEASUREMENTS FROM LIME KILNS

EMB Projects Report No.
75-LIM-8

Plant Tested

Woodville Lime and Chemical Company
Woodville, Ohio
August 6, 7, and 8, 1974

Prepared for

Environmental Protection Agency
Office of Air Quality Planning and Standards
Emission Measurement Branch
Research Triangle Park
North Carolina 27711

by

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Dayton, Ohio 45407

Report Reviewed by Clyde E. Riley

Contract No. 68-02-1404, Task No. 2

(15) T.P., SO_2, NO_x (6) ESP outlet Aug 13

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SECTION I
INTRODUCTION

Under the Clean Air Act of 1970, the Environmental Protection Agency is given the responsibility of establishing performance standards for new installations or modifications to existing installations in stationary source categories. As a contractor, Monsanto Research Corporation (MRC), under the Environmental Protection Agency's "Field Sampling of Atmospheric Emissions" Program, was asked to provide emission data from the Woodville Lime and Chemical Company of Woodville, Ohio.

The field test work was directed by Clyde E. Riley, Field Testing Section, Emission Measurement Branch. The sampling was performed by Monsanto Research Corporation with Darrell L. Harris as Team Leader.

This report tabulates the data collected at the No. 1 lime kiln of the Woodville Lime Company during the sampling program of August 6, 7, and 8, 1974. This kiln is equipped with a Buell electrostatic precipitator to control particulate emissions. The feed for the kiln is a dolomitic stone, quarried at the site, and sized at one inch to two and one-fourth inches. Feed rate is approximately 700 tons per day and product rate is approximately 350 tons per day. The kiln is fired with a mixture of natural gas and a number 6 fuel oil, and no preheater is used. The product is cooled in a Neims cooler before storage and no crushing is performed.

Sampling was performed at the outlet of the electrostatic precipitator. The outlet was measured for particulate concentrations according to procedures described in the Federal Register[1] Method 5, "Determination of Particulate Emissions from Stationary Sources." Method 1, "Sample and Velocity Traverses for Stationary Sources"; Method 2, "Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)"; and Method 3, "Gas Analysis for Carbon Dioxide, Excess Air and Dry Molecular Weight" are other procedures that were required for the Method 5 tests. Other tests that were performed were a combination of Method 8 and Method 6, "Determination of Sulfur Dioxide Emissions from Stationary Sources"; Method 7, "Determination of Nitrogen Oxide Emissions from Stationary Sources"; and Method 9, "Visual Determination of the Opacity of Emissions from Stationary Sources." All of the above methods are described in the Federal Register.[2]

No modifications were necessary to prepare the kiln stack for sampling. Sampling performed previously by another contractor required an extension of the stack and the erection of scaffolding for placement of the sampling devices. These modifications were unchanged for this program (see EMB report 74-LIM-3A).

The following sections of this report include: (1) summary of results, (2) description of the process, (3) location of sampling points and traverse data, (4) process operating conditions, and (5) sampling and analytical procedures. Appendices include all field data from this sampling project. Process production rates are not included in this report but will be available in a future publication.

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[1]Federal Register, Vol. 36, No. 159, August 17, 1971.
[2]Federal Register, Vol. 36, No. 247, December 23, 1971.

SECTION II
SUMMARY OF RESULTS

Data on particulate emissions from the lime kiln are summarized in Tables 1 and 2. Emissions of filterable particulate, as measured by the probe and filter catch, averaged 7.43 pounds per hour (lb/hr) at a concentration of 0.0303 grains per dry standard cubic foot (grains/dscf). Total particulate emissions averaged 12.2 pounds per hour at a concentration of 0.0500 grains/dscf. Emissions from the first test (P-1) were obviously much higher than the other two runs. While the reason for this difference is not apparent, it can probably be attributed to variations in process operations and to problems encountered with control equipment operating parameters, described in Section IV, "Process Operation." In addition, it should be pointed out that this increase in emissions is concurrent with the visible emission data.

Data on the oxides of nitrogen emissions are summarized in Table 3. These data show an average NO_2 concentration of 279 ppm by volume and an hourly emission rate of 55.5 pounds per hour.

Data on sulfur dioxide emissions are summarized in Table 4. These data show an average SO_2 concentration of 15.3 ppm by volume and an hourly emission rate of 4.21 pounds per hour.

Table 1

SUMMARY OF PARTICULATE RESULTS

(English Units)

Run Number	P-1	P-2	P-3	average
Date	8-6-74	8-7-74	8-8-74	
Method Type	EPA-5	EPA-5	EPA-5	
Volume of gas sampled-dscf[a]	105.92	128.16	119.80	117.96
Percent moisture by volume	9.61	11.05	10.81	10.49
Average stack temperature-°F	611	661	655	642
Stack volumetric flow rate-dscfm[b]	28986	27704	27559	28083
Stack volumetric flow rate-acfm[c]	65320	66488	65754	65854
Percent isokinetic	98.7	104.5	97.4	100.2
Duration of run-minutes	192	192	192	192
Particulates-probe, cyclone and filter catch				
mg	422.9	119.1	117.3	219.8
grains/dscf[d]	0.0615	0.0143	0.0152	0.0303
lb/hr	15.3	3.40	3.59	7.43
Particulates-total catch				
mg	667.8	253.3	173.5	364.9
grains/dscf[d]	0.0971	0.0304	0.0225	0.0500
lb/hr	24.1	7.23	5.31	12.21
Percent impinger catch	36.7	53.0	32.4	40.7

[a]Dry standard cubic feet @ 70°F, 29.92 in. Hg

[b]Dry standard cubic feet per minute @ 70°F, 29.92 in. Hg

[c]Actual cubic feed per minute-stack conditions

[d]Grains per dry standard cubic feet

Table 2

SUMMARY OF PARTICULATE RESULTS

(Metric Units)

Run Number	P-1	P-2	P-3	Average
Date	8-6-74	8-7-74	8-8-74	
Method Type	EPA-5	EPA-5	EPA-5	
Volume of gas sampled-Nm^3 [a]	3.000	3.630	3.364	3.331
Percent moisture by volume	9.61	11.05	10.81	10.49
Average stack temperature-°C	322	349	346	339
Stack volumetric flow rate-Nm^3/sec	13.68	13.08	13.01	13.26
Stack volumetric flow rate-$acfm^3$/sec	30.83	31.38	31.04	31.08
Percent isokinetic	98.7	104.5	97.4	100.2
Duration of run-minutes	192	192	192	192
Particulates-probe, cyclone and filter catch				
mg	422.9	119.1	117.3	219.8
mg/Nm^3	140.9	32.81	34.87	69.53
Kg/hr	6.934	1.543	1.630	3.369
Particulates-total catch				
mg	667.8	253.3	173.5	364.9
mg/Nm^3	222.6	69.78	51.58	114.7
Kg/hr	10.95	3.281	2.412	5.548
Percent impinger catch	36.7	53.0	32.4	40.7

[a]Normal cubic meters at 21.1°C, 760 mm Hg

Table 3

SUMMARY OF NO_x RESULTS

Date	Run #	Time	ppm[a]	lb/DSCF x 10^{-5}	grams/Nm^3	lb/hr[b]	Kg/hr[b]
8-6-74	N-1A	1540	174	2.06	0.330	35.8	16.3
8-6-74	N-1B	1630	226	2.68	0.430	46.6	21.2
8-6-74	N-1C	1820	202	2.39	0.383	41.6	18.9
8-6-74	N-1D	1920	200	2.37	0.380	41.2	18.7
N-1 Average		--	201	2.38	0.382	41.3	18.8
8-7-74	N-2A	1200	341	4.04	0.648	67.2	30.5
8-7-74	N-2B	1240	355	4.21	0.675	70.0	31.8
8-7-74	N-2C	1415	436	5.16	0.827	85.8	39.0
8-7-74	N-2D	1510	320	3.78	0.606	62.8	28.5
N-2 Average		--	363	4.28	0.686	71.4	32.4
8-8-74	N-3A	1045	253	2.99	0.479	49.4	22.4
8-8-74	N-3B	1130	276	3.27	0.524	54.1	24.6
8-8-74	N-3C	1230	292	3.45	0.553	57.0	25.9
8-8-74	N-3D	1335	276	3.27	0.524	54.1	24.6
N-3 Average		--	274	3.24	0.519	53.7	24.4
N-1, N-2, and N-3 Average		--	279	3.30	0.529	55.5	25.2

[a]Parts per million, by volume

[b]Based on volumetric flow rate from corresponding Method 5 run (DSCFM)

Table 4

SUMMARY OF SO_2 RESULTS

Run #	S-1	S-2	S-3	Average
Date	8-6-74	8-7-74	8-8-74	
Volume of gas sampled (dscf)	146.8	96.14	73.84	105.59
(Nm^3)	4.157	2.723	2.091	2.980
Run Time (min)	187	180	164	177
SO_2 Concentration				
(ppm, by volume)	6.08	26.6	13.1	15.3
(lb/dscf x 10^{-6})	1.01	4.38	2.17	2.52
(mg/Nm^3)	16.1	70.2	34.8	40.4
SO_2 Emission Rate[a]				
(lb/hr)	1.70	7.35	3.59	4.21
(Kg/hr)	0.772	3.34	1.63	1.91

[a]Based on velocity taken from corresponding Method 5 run

Visual emissions measurements were recorded from the No. 1 kiln stack plume independently by two MRC personnel during each particulate test. Data for these opacity measurements are summarized in Table 5. The average opacity for all tests was less than five (5) percent. Periods of high emissions, however, did occur during certain sequential minute intervals for the first test, during which opacity levels exceeded the 5 percent normal range. This normal range value of 5 percent opacity or less was considered typical of the Woodville control device as well as other well-controlled lime-producing operations.

Samples of the limestone feed material in the storage silo, the kiln product, fuel oil, and the dust recovered by the electrostatic precipitator (ESP) were collected near the beginning and end of each of the sampling runs. A composite was prepared of the feed material and fuel oil on which analysis for the total sulfur content was performed along with the individual samples of product and ESP dust. These results are presented in Table 6.

Additional detailed results of the test program are presented in Section V, "Sampling and Analytical Procedures."

Table 5

VISIBLE EMISSIONS SUMMARY

Run No. Date	P-1 8-6-74		P-2 8-7-74		P-3 8-8-74	
	Obs. 1	Obs. 2	Obs. 1	Obs. 2	Obs. 1	Obs. 2
Interval of observations[a]						
Start	1525	1525	1143	1143	1026	1026
End	1925	1925	1522	1522	1357	1357
Duration of observation (min)[b]	197	213	193	194	194	197
Total no. of readings[c]	788	852	773	776	776	788
No. of readings unobservable	0	0	0	7	0	1
No. of readings @ 0% opacity	555	182	682	558	748	717
5%	221	641	89	208	26	68
10%	12	29	2	3	2	2
Percent readings unobservable	0	0	0	0.90	0	0.13
Percent readings @ 0% opacity	70.43	21.36	88.23	71.91	96.39	90.99
5%	28.05	75.23	11.51	26.80	3.35	8.63
10%	1.52	3.41	0.26	0.39	0.26	0.25

[a] 24-hour clock start and end times

[b] Excluding the time that readings were not recorded for period of observation

[c] Readings recorded at 15-second intervals unless otherwise noted

Table 6

TOTAL SULFUR CONTENT OF SILO FEED, FUEL OIL, PRODUCT AND ESP DUST

Date	Sample No.	Material	% Sulfur Found	Avg % Sulfur
8-6-74	S-74-001-788	Silo Feed	0.028, 0.028	0.028
	791	Fuel Oil[a]	1.52	1.52
	792	Kiln Product	0.081, 0.099	0.097
	793	Kiln Product	0.098, 0.108	
	795	ESP Dust	0.572, 0.707	0.614
	796	ESP Dust	0.527, 0.650	
8-7-74	S-74-011-836	Silo Feed	0.034, 0.036	0.035
	839	Fuel Oil[a]	1.36	1.36
	840	Kiln Product	0.40, 0.30	0.23
	841	Kiln Product	0.11, 0.11	
	843	ESP Dust	0.76, 0.87	0.77
	844	ESP Dust	0.70, 0.74	
8-8-74	S-74-001-873	Silo Feed	0.053, 0.048	0.051
	876	Fuel Oil[a]	1.37	1.37
	877	Kiln Product	0.071, 0.077	0.11
	878	Kiln Product	0.149, 0.148	
	880	ESP Dust	0.733, 0.672	0.71
	881	ESP Dust	0.688, 0.732	

[a]The fuel oil samples were also analyzed for the Btu content with the following results:

S-74-001-791	18236 Btu/lb
S-74-001-839	18343 Btu/lb
S-74-001-876	18404 Btu/lb

SECTION III
PROCESS DESCRIPTION

Limestone consisting primarily of calcium carbonate or combinations of calcium and magnesium carbonate with varying amounts of impurities is quarried at the Woodville Plant. The limestone is calcined or burned to form lime, commonly divided into two basic products--quicklime and hydrated lime. Calcination expels carbon dioxide from the raw limestone, leaving calcium oxide (quicklime). With the addition of water, calcium hydroxide (hydrated lime) is formed.

The basic processes in production are: (1) quarrying the limestone raw material, (2) preparing the limestone for kilns by crushing and sizing, (3) calcining the limestone, and (4) optionally processing the quicklime further by additional crushing and sizing followed by hydration. The majority of lime is produced in rotary kilns which can be fired by coal, oil, or gas. Rotary kilns have the advantage of producing high production per manhour and a more uniform product. They do, however, require higher capital investment and unit fuel costs than most vertical kilns.

The Woodville Lime and Chemical plant has two rotary kilns each equipped with a Buell electrostatic precipitator. The kilns are almost identical. The feed for both is a dolomitic stone, quarried on the site and fed in sizes ranging from 1 inch to 2-1/4 inches at a rate of about 700 tons per day.

There is no preheater. Normally the kiln is fueled with a mixture of 95 percent Number 6 fuel oil and 5 percent natural gas. Both kilns have two heat transfer sections, each 20 feet long. The product, about 350 tons per day, is cooled in a Neims cooler before storage. There is no product crushing, but undersize material is separated and returned to the kiln. The majority of the product is used in the steel industry, mostly in basic oxygen furnaces. None of the product is hydrated.

The electrostatic precipitator on kiln Number 1 was put in operation in July 1971. In this kiln the main process fan is located before the ESP, with a cyclone before the fan to reduce fan blade erosion. The precipitator on kiln Number 2 was put in operation in December 1973. The main process fan is after the ESP, and there is no cyclone.

In both systems the inlet gas to the precipitators is cooled to about 600°F with a combination of water injection and/or tempering air. Each precipitator has 28,800 square feet of collecting surface area, which includes one cell and two fields. Design gas velocity is 1.5 feet per second and treatment time is 10.0 seconds. The plant manager reported that an earlier emission test showed exit loadings of less than 0.005 grains per dry standard cubic foot.

At present the dust collected from the precipitators is disposed of in the quarry. It is expected that in the future the dust will be granulated and used as a component of dry mix fertilizers that are blended in another part of the complex.

At the time of the initial plant inspection (February 8, 1974), the precipitators were working satisfactorily and had been very well maintained. The plant is representative of modern design. Raw materials and products are typical of those in the industry.

SECTION IV
LOCATION OF SAMPLING POINTS

Figure 1 shows the sampling ports used in the Number 1 lime kiln exit stack. The sampling ports were located in a 63.5-inch inside diameter vertical stack, 4 feet (0.75 diameter) from the stack exit, and 12 feet (2.26 diameters) from the nearest upstream disturbance. In order to meet the sampling requirements of Methods 1 and 5 of the Federal Register, Vol. 36, No. 247, it was necessary to use an existing stack extension that had been left on the ESP exhaust outlet from a preceding test program. Forty-eight traverse points (24 along each of two perpendicular diameters) were used as described in the Federal Register, Method 1. The stack was found to be slightly elliptical at this location, measuring 62-3/4 inches on the east-west diameter and 63-1/2 inches on the north-south diameter. The larger diameter was used for calculation of the stack area.

Test Methods 1, 2, 3, 5 and 7 were performed at the set of 90° ports on the stack 146 inches from the outlet of the ESP and 48 inches from the atmospheric outlet of the stack. Additional sampling points in the existing stack at a lower site were used for some of the gas sampling. Modified Method 6 tests were conducted at the west port located 53 inches from the ESP outlet and 141 inches from the stack outlet. Sampling was done at a single point on the west port traverse at a probe insertion depth of 33 inches.

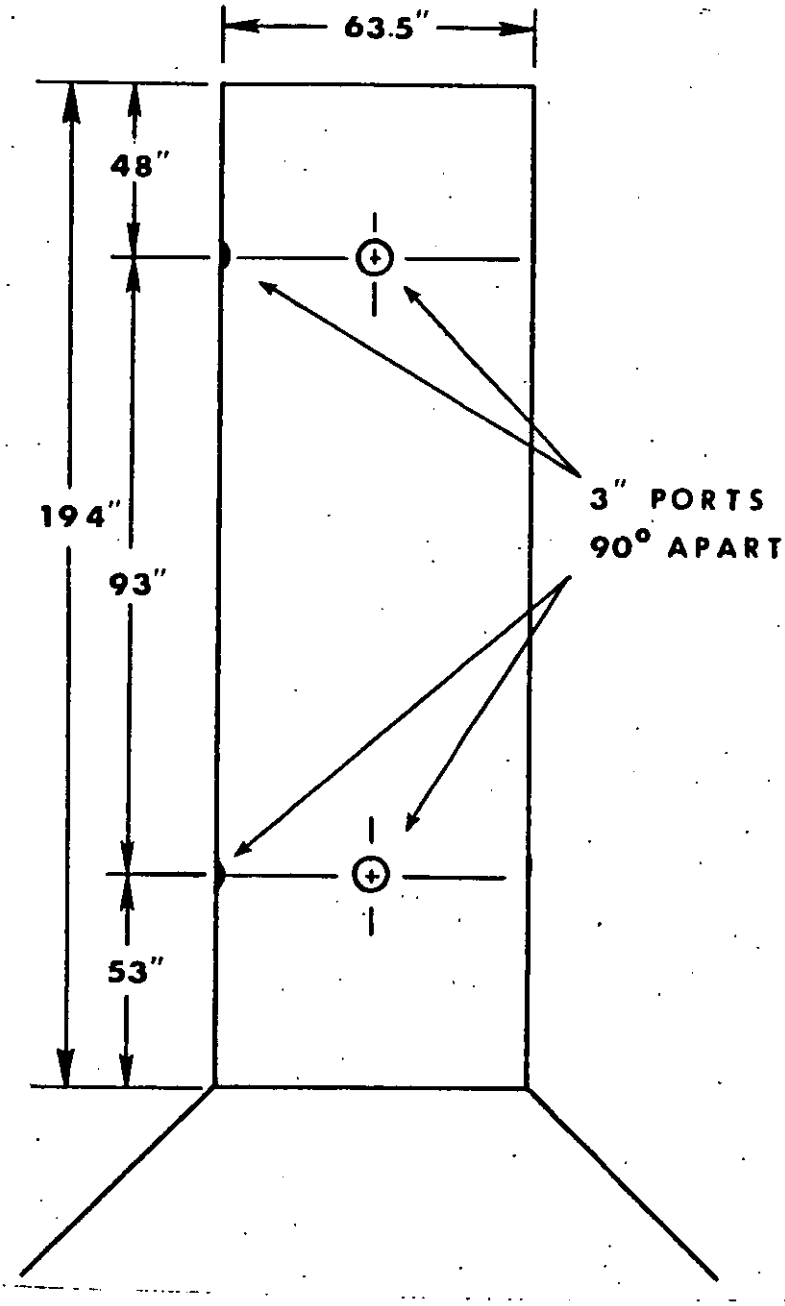


Figure 1. Stack port locations

Figure 2 shows a top view of the stack and the scaffolding location. Figure 3 is a cross-sectional view of the outlet which shows the location of the 48 traverse points.

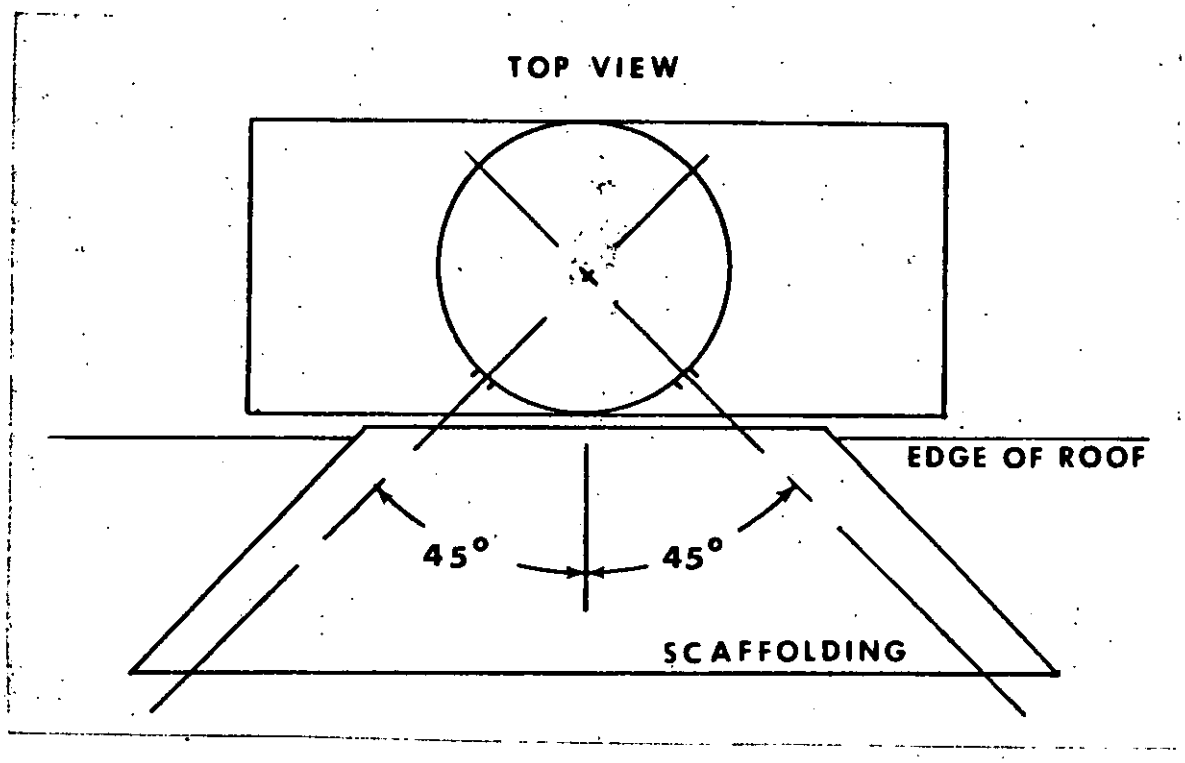


Figure 2. Test site No. 1 kiln precipitator outlet

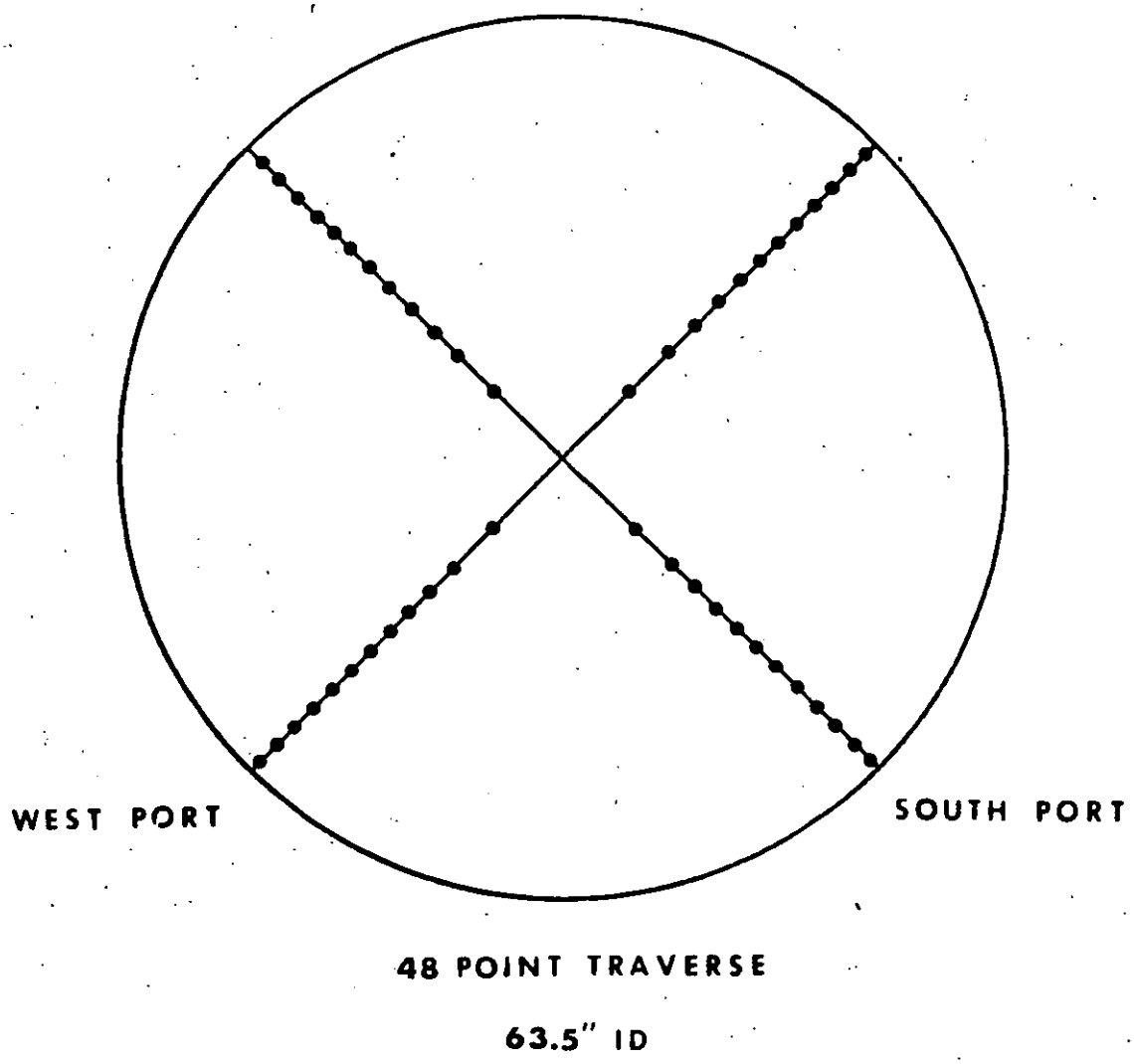


Figure 3. Particulate test traverse cross section

SECTION V
PROCESS OPERATION AND TEST CONDITIONS

From previous plant visits and discussions with plant personnel, it was found that the two electrostatic precipitators operate under normal conditions with essentially no visible emissions. Plant operations appeared to be normal for the Number 1 kiln and electrostatic precipitator on the morning of August 6, 1974, with visible emission opacity readings in the range of 0 to 5 percent.

However, several problems developed during the first day of testing. At 8:10 a.m. the plant manager informed the Project Officer that a fan shutdown was scheduled on the Number 1 kiln. The fan is located on the inlet to the ESP and has to be cleaned of adhering dust material on a routine basis. The shut-down was scheduled for 8:30 a.m. and lasted for only ten minutes.

The first series of tests was started at 3:25 p.m. A probe tip exchange was necessary at 4:15 p.m. due to subisokinetic sampling conditions caused by a low orifice pressure calibration factor. As testing progressed, the opacity readers reported an increasing number of 5 percent readings, with occasional "puffs" as high as 10 percent. Observations of the plume were difficult because of the cloudy overcast sky background as well as the excessive fugitive dust emissions created by trucks loading and unloading ESP dust and quarry

rock in the vicinity of either the ESP unit or the observers.

The oil feed rate to the kiln had to be substantially increased at 2:30 p.m. to maintain the desired kiln temperature. This condition was caused by another kiln starting up which reduced the natural gas pressure on the main feed line. The test was completed at 7:00 p.m.

Near the end of the test, the ESP inlet temperature increased to 710°F at which time (6:00 p.m.), it was noticed that the stack opacity was clearing up. Mr. Judd, the plant chemist, was asked if they could maintain the inlet temperature between 700°F and 710°F for the second test, which would start the following morning.

Upon arrival at the plant the following morning, the stack opacity was observed to be at 0 percent. The process operator stated that the ESP inlet temperature had not dropped below 700°F for the last 16 hours.

The second test was started on August 7 at 11:44 a.m. The majority of visible emissions were recorded to be 0 percent opacity with minor periods of 5 percent opacity through this test sequence. During the test, no sampling or process problems were encountered. The test was completed at 3:22 p.m.

The third test was conducted during the following morning (August 8, 1974) starting at 10:27 a.m. The stack plume was reading at 0 percent opacity and the process was operating at a normal production rate. The visible emission readers reported that they were observing a lower percentage of 5 percent readings compared with the second test. Short puffs

of 5 percent were the only readings observed during this testing sequence.

The kiln feed rate was calculated by multiplying a conversion factor times the total indicated tonnage recorded from the control room stone totalizer meter. These data are not available at this time but will be supplied in a future publication. Operating variables for the three test runs are summarized in Table 7. Additional detailed information pertaining to the process operation during the testing periods is presented in Appendix F.

The Woodville Lime and Chemical plant is very representative of modern design and operation, and has a typical raw material and product. During the entire test program conducted between August 6, 1974, and August 8, 1974, the process and electrostatic precipitator were operating at a normal production rate for which the facility was designed. It is therefore recommended that the results of the emission tests conducted at this installation be used in the development of standards of performance for the lime producing industry.

Table 7

SUMMARY OF OPERATING VARIABLES

Date	8-6-74	8-7-74	8-8-74
Particulate test no.	P-1	P-2	P-3
Stone feed rate (ton/hr)[a]	-	-	-
Oil rate (gal/hr)	294	311	316
Firing zone temp (°F)	2649	2662	2600
Mid-kiln temp (°F)	1352	1373	1360
Kiln feed end temp (°F)	1025	1031	1023
Before ESP temp (°F)	702	711	712
Stack temp (°F)	663	673	669
ELECTROSTATIC PRECIPITATOR DATA			
"A" Field			
Primary current (amps)	74	68	69
Primary voltage (volts)	251	257	256
Precipitator current (amps)	.45	.39	.40
"B" Field			
Primary current (amps)	72	70	70
Primary voltage (volts)	238	248	245
Precipitator current (amps)	.44	.44	.44

[a]Obtained by multiplying indicated meter tonnage by a conversion factor

SECTION VI
SAMPLING AND ANALYTICAL PROCEDURES

The outlet gases of the Electrostatic precipitator at the Woodville Lime and Chemical Company lime kiln were sampled for particulate emissions, sulfur dioxide, and nitrogen oxides concentrations. Runs of the particulate series were identified by a number prefixed by "P" followed by the number of the series. SO_2 and NO_x run numbers were preceded with an "S" or "N", respectively.

Sampling procedures were designated by EPA. Analyses of collected samples were performed by Monsanto. Appendix J presents detailed sampling and analytical procedures.

Velocity and Gas Temperature

Gas velocities were measured with a calibrated type S pitot tube and inclined draft gage. Velocities were measured at each sampling point across the stack diameter to determine an average value according to procedures described in the Federal Register[1], Method 2. Temperatures were measured with the use of a thermocouple.

[1]Federal Register, Vol. 36, No. 247, December 23, 1971.

Molecular Weight

A 4-hour integrated sample of the stack gases was collected during each particulate test by pumping the gas into a Tedlar plastic bag at the rate of approximately 0.005 cfm. The sampling train was assembled as shown in Figure 4. This bag sample was then analyzed with an Orsat analyzer for CO_2, O_2, and CO as described in the Federal Register, Method 3.

Particulates

Concentrations of particulate matter in stack gases were measured by Method 5 as described in the Federal Register.[1] A rigid train consisting of a heated glass-lined probe, a 3-inch diameter glass-fiber filter, and a series of Greenburg-Smith impingers was used for particulate sampling, as shown in Figure 5.

Sampling was conducted under isokinetic conditions by monitoring stack-gas velocity with a pitot tube and adjusting the sampling rate accordingly.

The three particulate runs were performed on three successive days. All runs used two 24-point traverses, for a total of 48 points, and each point was sampled 4 min for a total run time of 192 min. On the first run (P-1), a .374 inch nozzle was used to start the run. At the eleventh point of the first traverse, it became obvious that the flow rate would not be sufficient to maintain an isokinetic rate with the .374 inch nozzle throughout the run. Preparation was made for a nozzle change. Between the 13th and 14th points (52 min into the

[1] Federal Register, Vol. 36, No. 159, August 17, 1971.

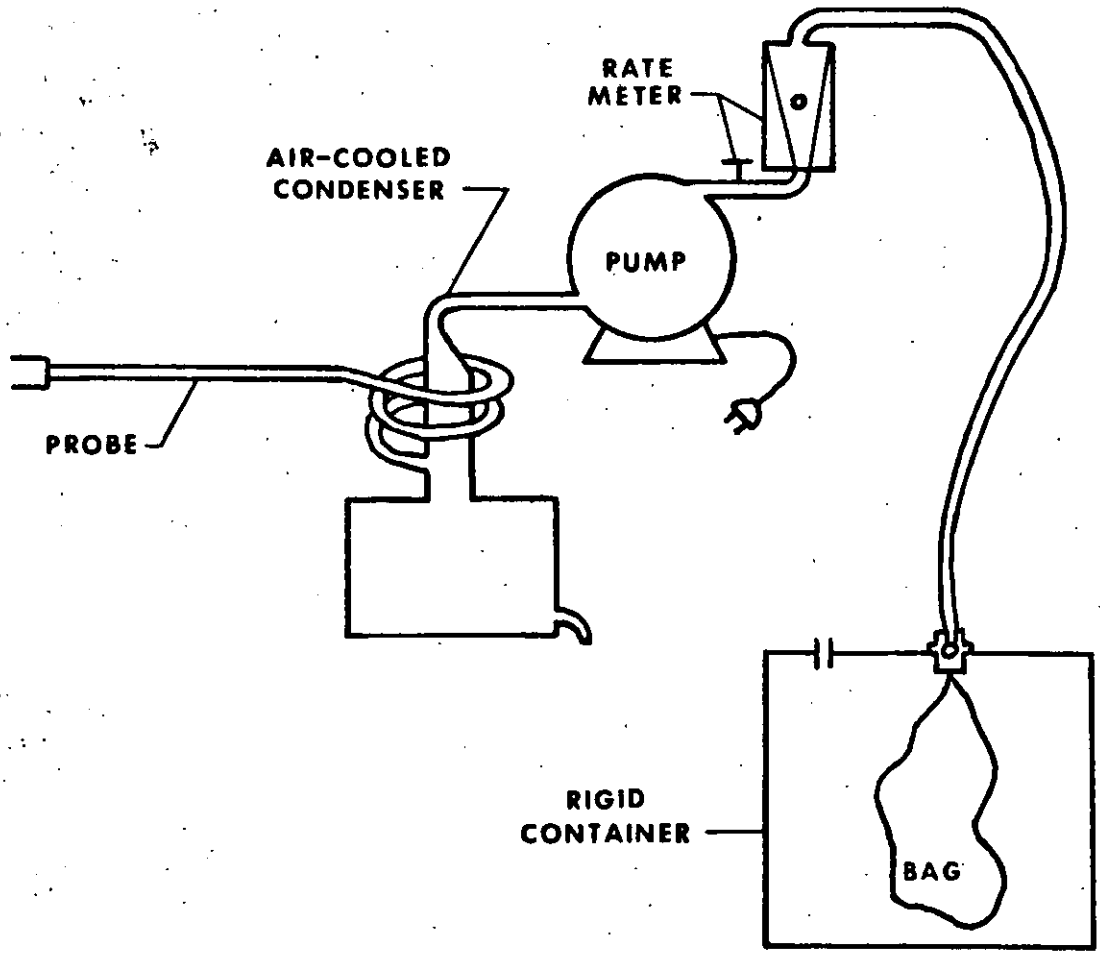


Figure 4. Integrated gas sampling train

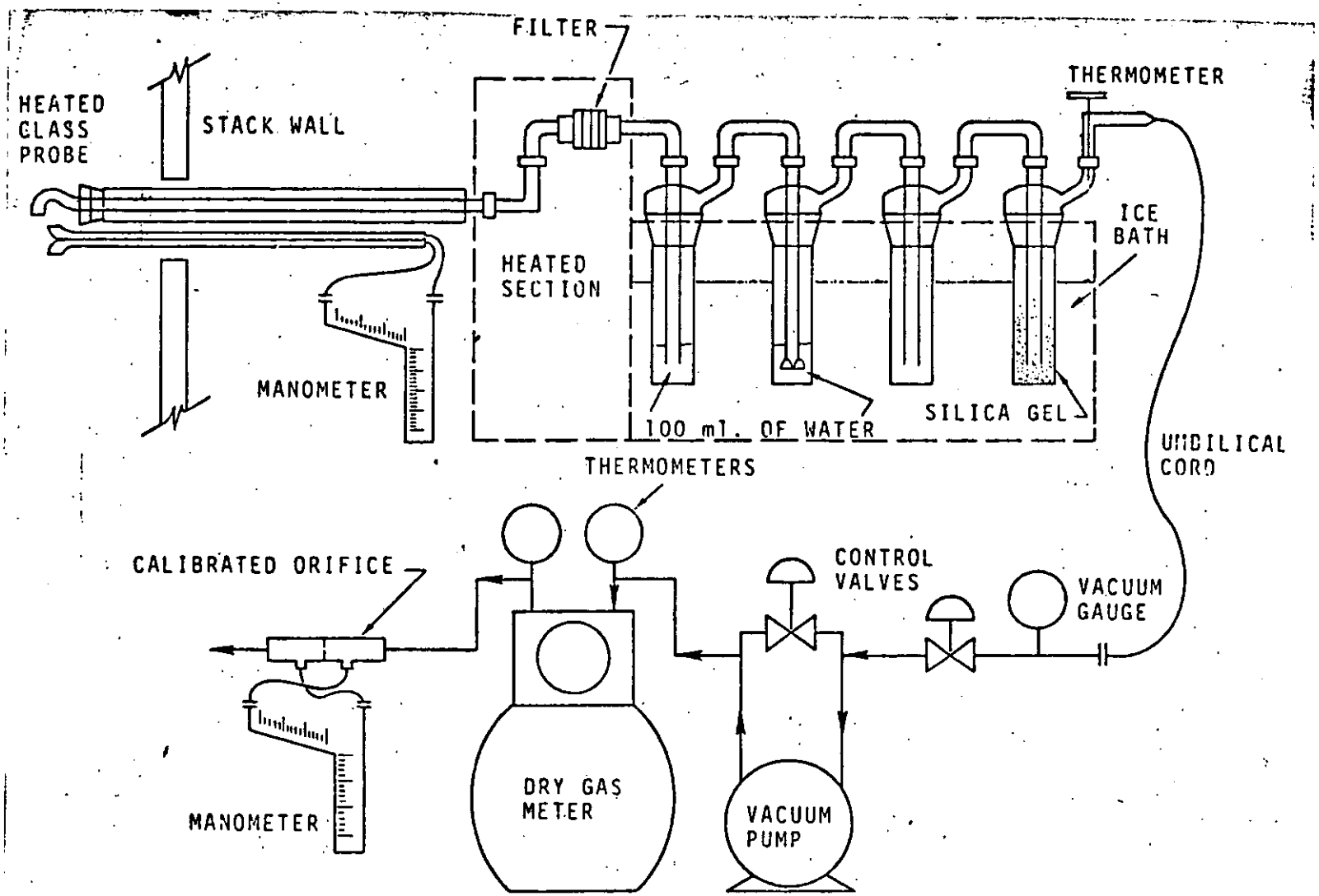


Figure 5. Particulate sample train

run) the sampling was stopped, and a 0.244 inch nozzle was installed for the remainder of the run.

This nozzle change caused no problem in the calculation of particulate grain loading; however, calculation of the isokinetic percentage was more difficult. Calculation was accomplished by treating the two parts of the run as separate runs and taking a weighted average to determine the final percentage by the following method:

$$I_0 = \frac{(I_1)(P_1) + (I_2)(P_2)}{P_1 + P_2}$$

where:

I_0 = overall isokinetic percentage
I_1 = isokinetic percentage with first nozzle
I_2 = isokinetic percentage with 2nd nozzle
P_1 = number of first nozzle points
P_2 = number of second nozzle points

During Run P-1, at approximately 150 minutes into the run, the ΔH reading dropped below its set point and the vacuum increased indicating a plug in the system. It became impossible to control the flow rate at a sufficiently high level, so the run was stopped. An inspection of the equipment revealed no noticable plug, but it was suspected that the umbilical tubing had become kinked. The run was re-started, and no additional flow stoppage occurred.

Samples from the Method 5 sampling trains were recovered as outlined in the August 17, 1971, Federal Register. After removal of the filter, all sample exposed surfaces were washed with distilled water or reagent grade acetone as specified. All sample bottles for liquid samples were obtained from Wheaton

Scientific, Catalogue No. 219630. Each of these bottles, except those used for collecting SO_2 samples, was acid-soaked with 1:1 HNO_3 for one day, rinsed with distilled water, and soaked with distilled water for one day.

The particulate sample was recovered by rinsing the nozzle, probe, cyclone by-pass, and front half of the filter holder twice with acetone into a glass container. The inside walls of the probe were brushed while rinsing. The back half of the filter holder, impingers, and connecting tubes were rinsed twice with distilled water and the washings placed in a glass container with the impinger contents. These components were then double-rinsed with acetone into another glass container. The filter was placed in a separate container. Blank samples of water and acetone were also taken.

Analytical procedures for the Method 5 samples follow the Federal Register guidelines, with one exception. Container Number 3 as indicated in the method contains water from the impingers and washing of the glassware of the train. The solution was extracted with chloroform and ether, and then the extracted portion was dried to constant weight, as specified. In addition, the water remaining after extraction was evaporated to dryness at 212°F to constant weight. Both weights were included in the total mass of particulate. Sample weights from the Method 5 samplers were reported as "front half" (probe washings and filter collection weights) and "total" (front half plus water, chloroform-ether extract and impinger acetone washing weights). Sample residues were sent to EPA for trace metal analysis.

NO_x

Nitrogen oxides were collected in evacuated 2-liter flasks containing 25 ml of a dilute sulfuric acid/hydrogen peroxide absorbing solution. The sampling and analytical procedure was described in Method 7 of the Federal Register.[1] The sampling train was assembled as shown in Figure 6. Four nitrogen oxide samples were collected at equal intervals during each of the particulate runs. Each sample was collected using a 2-foot glass-lined probe at the upper port locations. Each flask was evacuated and pressure tested for one minute, and the initial flask temperature, pressure, and barometric pressure recorded. The sampling probe was inserted into the stack and heated, and the sample flask connected. The 3-way stopcock was turned to the "purge" position and stack gas drawn through the system with a rubber squeeze bulb to check for condensation in the probe line. The 3-way stopcock was then turned to the sample position for 15 to 30 seconds. The flask valve was then closed and disconnected from the probe. The contents were shaken for at least 5 minutes.

The flasks were allowed to set for at least 16 hours. They were then shaken for at least 2 minutes, the final pressure, temperature, and barometric pressure were taken, and the sample was transferred to a storage bottle.

The samples were analyzed using the Phenoldisulfonic acid, photometric analysis described in the above mentioned Federal Register.[1] Table 3 gives the calculated amounts of nitrogen oxides given as NO_2 in both parts per million and mass per unit

[1] Federal Register, Vol. 36, No. 247, December 23, 1971.

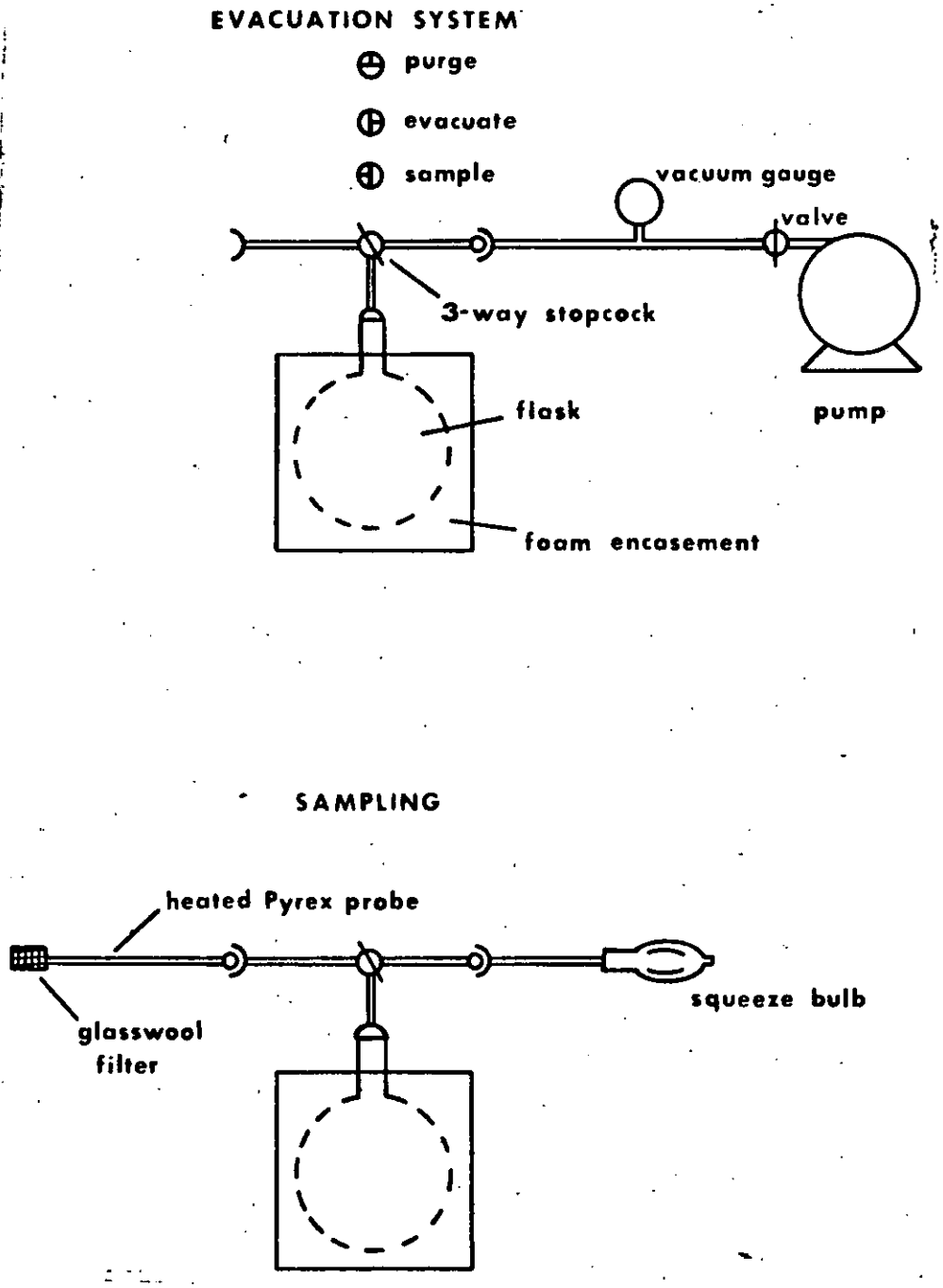


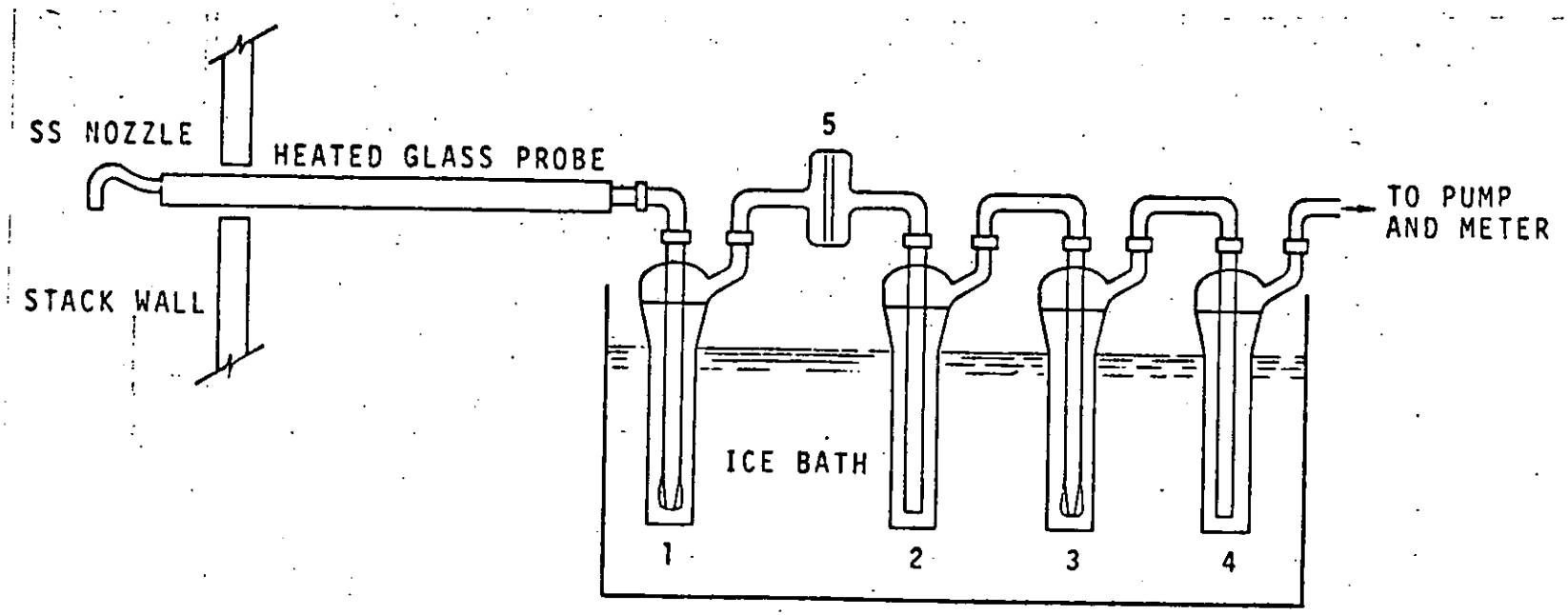
Figure 6. Nitrogen oxide sampling equipment

volume. Mass per unit time figures were calculated using the volumetric flow rate from the corresponding Method 5 run pitot traverse.

SO_2

Sulfur dioxide sampling procedures followed those described in Method 6. However, due to the low expected concentrations, the Method 8 sampling train shown in Figure 7 was used. The first impinger contained 150 ml of 80 percent isopropanol; the second and third impingers contained 100 ml each of 3 percent hydrogen peroxide/water solution. The SO_2 determination runs were performed simultaneously with each Method 5 run on the three days and are of the same general duration. The first run (S-1) was conducted using a flow rate of approximately 1 acfm and the second and third runs (S-2 and S-3) using a rate of approximately 0.5 acfm at meter conditions. On the last run (S-3), at the 164th minute, the dial shaft of the dry gas meter broke, and the run was stopped and considered to be complete at this point. For all three runs, the solution in the first impinger was replaced with fresh solution during port switching. Also during these shut downs, the silica gel was changed, if necessary. After sampling, ambient air was passed through the train for 10 to 15 minutes. The isopropanol solution was discarded, and the peroxide solution rinsed into a glass container. Samples collected from the sulfur dioxide modified Method 6 runs were analyzed by the barium perchlorate titration using thorin indicator as outlined in the December 23, 1971, Federal Register.

Table 4 gives the summary of results of the SO_2 testing. The SO_2 emission rates are based on the velocity determined during the Method 5 run of the same sampling period.



1. G-S Impinger - 100 ml 80% isopropanol
2. Modified G-S Impinger - 100 ml 3% H_2O_2
3. G-S Impinger - 100 ml 3% H_2O_2
4. Modified G-S Impinger - 200 g Silica Gel
5. Filter Holder w/Glass Fiber Filter

Figure 7. SO_2/SO_3 sampling train

Visible Emissions

Visible emissions were determined according to procedures presented in Method 9. Two observers were employed to record simultaneous visible emission data during the particulate testing. The observers started recording readings just prior to the start of the Method 5 run and ended just after the Method 5 run stopped. Readings were also discontinued during shut-down periods for port changing, etc.

Readings were difficult to determine at times due to trucks loading and unloading ESP dust and quarry rock in the vicinity of either the ESP unit or the observer, and the light-colored plume against an overcast and partly cloudy sky caused poor distinction. In addition, during the first run certain ESP rappers set up a visible emission condition, "puffs," that read approximately 5 to 10 percent opacity for about 2 to 3 seconds every rapping cycle. Readings were taken and recorded every 15 seconds. The summary of these readings is presented in Table 5.

Total Sulfur Analysis

Samples of the limestone feed material in the storage silo, the kiln product, the fuel oil, and the dust collected by the electrostatic precipitator (ESP) were collected near the beginning and end of each sampling run. A composite was prepared of the feed material and fuel oil on which analysis of the total sulfur content was performed along with the individual samples of product and ESP dust. The sulfur content of the fuel oil was analyzed by the Parr bomb calorimeter method.

A major problem was encountered in the analysis of the sulfur content of the limestone (silo feed), kiln product, and ESP dust. It was suggested by EPA that the materials be analyzed by the Parr bomb method (ASTM D271-46). The materials would not ignite using the standard method, and benzoic acid was added as a combustible material as suggested in the alternate method. The procedure is basically a gravimetric technique in which the sulfur is converted to sulfate and precipitated as $BaSO_4$, a white material. However, the precipitate was brown, indicating the formation of $Fe(OH)_3$ which coprecipitated and thus lead to apparently high sulfur values. An alternate procedure specific for limestone and lime analysis (ASTM C-25-72) was suggested to EPA. This Na_2CO_3 fusion method was used to obtain the sulfur content of the lime and limestone samples.

Fuel oil samples were analyzed for sulfur content by using Standard Method of Test for Sulfur in petroleum Products by the Bomb Method, D129-64. The complete sulfur results for these samples can be found in Table 6.

APPENDIX A

COMPLETE PARTICULATE RESULTS AND SAMPLE CALCULATIONS

Table A-1

ISOKINETIC CALCULATION FOR FIRST (a) AND SECOND (b) PARTS OF RUN P-1 WITH TWO DIFFERENT NOZZLE SIZES

ABBR	DESCRIPTION	UNITS	P-1A	P-1B
TT	DURATION OF RUN	MINUTES	52.0	140.0
PB	BAROMETRIC PRESSURE	IN HG	29.69	29.69
DELH	AVG ORIFICE PRESS DROP	IN H2O	2.748	0.759
VM	VOL DRY GAS(METER CON)	DCF	44.572	65.329
TM	AVG GAS METER TEMP	DEG F	86.3	87.3
VMSTD	VOL DRY GAS (STD COND)	DSCF	43.17	62.85
VW	TOTAL H2O COLLECTED	ML	96.3	141.1
VWG	VOL H2O VAPOR(STD CON)	SCF	4.565	6.688
PCNTM	PERCNT MOISTURE BY VOL		9.56	9.62
MD	MOLE FRACTION DRY GAS		0.904	0.904
CO2	PERCENT CO2		19.0	19.0
O2	PERCENT O2		8.7	8.7
N2	PERCENT N2		72.3	72.3
MWD	MOL WT OF DRY GAS		31.4	31.4
MW	MOL WT OF STACK GAS		30.1	30.1
DELP	AVG STACK VELOCITY HEAD	IN H2O	0.334	0.413
TS	STACK TEMPERATURE	DEG F	567.	627.
PM	STACK PRESSURE(STATIC)	IN H2O	-0.30	-0.30
PS	STACK PRESSURE (ABS)	IN HG	29.67	29.67
VS	AVG STACK GAS VELOCITY	FPM	2691.	3074.
DS	STACK DIAMETER	INCHES	63.50	63.50
AS	STACK AREA	SQ IN	3166.9	3166.9
QS	STACK FLOW RT(DRY STD)	DSCFM	27375.	29552.
QA	STACK FLOW RT(ACTUAL)	ACFM	59177.	67601.
DN	PROBE TIP DIAMETER	INCHES	0.374	0.244
PCTI	PERCENT ISOKINETIC		87.5	102.9

Weighted Average Calculation

$$PCTI = \frac{[PCTI(A) \times TT(A)] + [PCTI(B) \times TT(B)]}{TT(A) + TT(B)}$$

$$= \frac{(87.5)(52) + (102.9)(140)}{(140)(52)} = 98.7$$

Table A-2

PARTICULATE EMISSION DATA

ABBR	DESCRIPTION	UNITS	P-1 *	P-2	P-3
TT	DURATION OF RUN	MINUTES	192.0	192.0	192.0
PB	BAROMETRIC PRESSURE	IN HG	29.69	29.67	29.61
DELH	AVG ORIFICE PRESS DROP	IN H2O	1.298	1.724	1.529
VM	VOL DRY GAS(METER CON)	DCF	109.901	134.828	128.360
TM	AVG GAS METER TEMP	DEG F	87.0	94.8	108.4
VMSTD	VOL DRY GAS (STD COND)	DSCF	105.92	128.16	118.80
VW	TOTAL H2O COLLECTED	ML	237.5	335.8	303.8
VWG	VOL H2O VAPOR(STD CON)	SCF	11.257	15.917	14.400
PCNTM	PERCNT MOISTURE BY VOL		9.61	11.05	10.81
MD	MOLE FRACTION DRY GAS		0.904	0.890	0.892
CO2	PERCENT CO2		19.0	22.0	21.0
O2	PERCENT O2		8.7	6.1	7.0
N2	PERCENT N2		72.3	71.9	72.0
MWD	MOL WT OF DRY GAS		31.4	31.8	31.6
MW	MOL WT OF STACK GAS		30.1	30.2	30.2
DELP	AVG STACK VELOCITY HEAD	IN H2O	0.391	0.390	0.381
TS	STACK TEMPERATURE	DEG F	611.	661.	655.
PM	STACK PRESSURE(STATIC)	IN H2O	-0.30	-0.30	-0.30
PS	STACK PRESSURE (ABS)	IN HG	29.67	29.65	29.59
VS	AVG STACK GAS VELOCITY	FPM	2971.	3024.	2990.
DS	STACK DIAMETER	INCHES	63.50	63.50	63.50
AS	STACK AREA	SQ IN	3166.9	3166.9	3166.9
QS	STACK FLOW RT(DRY STD)	DSCFM	28986.	27704.	27559.
QA	STACK FLOW RT(ACTUAL)	ACFM	65320.	66488.	65754.
DN	PROBE TIP DIAMETER	INCHES	*	0.305	0.305
PCTI	PERCENT ISOKINETIC		*	104.5	97.4
MF	PARTICULATE (FRONT)	MG	422.9	119.1	117.3
MT	PARTICULATE (TOTAL)	MG	667.8	253.3	173.5
CAN	PARTICULATE (FRONT)	GR/DSCF	0.0615	0.0143	0.0152
CAO	PARTICULATE (TOTAL)	GR/DSCF	0.0971	0.0304	0.0225
CAT	PARTICULATE (FRONT)	GR/ACF	0.0273	0.0060	0.0064
CAU	PARTICULATE (TOTAL)	GR/ACF	0.0430	0.0127	0.0094
CAW	PARTICULATE (FRONT)	LB/HR	15.273	3.398	3.591
CAX	PARTICULATE (TOTAL)	LB/HR	24.118	7.226	5.312

* See Table A-1 for calculation of these data.

Table A-3

PARTICULATE CALCULATIONS

Example: Run No. P-3 (See Table A-1 for data)

1. Volume of dry gas sampled at standard conditions (dscf[a])

$$VMSTD = \frac{17.7 \times VM \left(PB + \frac{DELH}{13.6}\right)}{(TM + 460)}$$

$$= \frac{17.7 \times 128.360 \times \left(29.61 + \frac{1.53}{13.6}\right)}{108.4 + 460}$$

$$= 118.80 \text{ dscf}$$

2. Volume of water vapor at standard conditions (scf[b])

$$VWG = 0.0474 \times VW$$

$$= 0.0474 \times 303.8$$

$$= 14.400 \text{ scf}$$

3. Percent moisture in stack gas

$$PCNTM = \frac{100 \times 14.400}{118.80 + 14.400}$$

$$= 10.81$$

4. Mole fraction of dry gas

$$MD = \frac{100 - PCNTM}{100}$$

$$= \frac{100 - 10.81}{100}$$

$$= .892$$

Table A-3 (Continued)

PARTICULATE CALCULATIONS

5. Molecular weight of dry stack gas

$$MWD = (\%CO_2 \times 0.44) + (\%O_2 \times 0.32) + [(\%C) + \%N_2) \times 0.28]$$

$$= (21.0 \times 0.44) + (7.0 \times 0.32) + [(0 + 72.0) \times 0.28]$$

$$= 31.6$$

6. Molecular weight of wet stack gas

$$MW = MWD \times MD + 18\ (1 - MD)$$

$$= 31.6 \times 0.892 + 18\ (1 - 0.892)$$

$$= 30.2$$

7. Stack gas velocity at stack conditions (fpm[c])

$$VS = 4360 \times \left[\frac{\sum_{i=1}^{i=n} \sqrt{DELP \times (TS + 460)}}{n} \right] \times \left[\frac{1}{PS \times MW} \right]^{\frac{1}{2}}$$

where n = the number of data points

$$= 4360 \times 20.50 \times \left(\frac{1}{29.59 \times 30.2} \right)^{\frac{1}{2}}$$

$$= 2990 \text{ fpm}$$

8. Stack gas volumetric flow rate at standard conditions (dscfm[d])

$$QS = \frac{0.123 \times VS \times AS \times MD \times PS}{TS + 460}$$

$$= \frac{0.123 \times 2990 \times 3166.9 \times 0.892 \times 29.59}{655 + 460}$$

$$= 27{,}559 \text{ dscfm}$$

Table A-3 (Continued)

PARTICULATE CALCULATIONS

9. Stack gas volumetric flow rate at stack conditions (acfm[e])

$$QA = \frac{0.05645 \times QS\ (TS + 460)}{PS \times MD}$$

$$= \frac{0.05645 \times 27559 \times (655 + 460)}{29.59 \times 0.892}$$

$$= 65{,}754 \text{ acfm}$$

10. Area of nozzle (sq ft)

$$AN = 54.54 \times 10^{-4}(DN)^2$$

$$= 54.54 \times 10^{-4}(0.305)^2$$

$$= 5.07 \times 10^{-4} \text{ sq ft}$$

11. Percent Isokinetic

$$PCTI = \frac{100.0(TS + 460)\left[0.00267(VW) + \frac{VM}{(TM + 460)}\left(PB + \frac{DELH}{13.6}\right)\right]}{(TT)(VS)(PS)(AN)}$$

$$= \frac{100.0(655+460)\left[0.00267(303.8) + \frac{128.360}{(108.4+460)}\left(29.61+\frac{1.53}{13.6}\right)\right]}{192 \times 2940 \times 29.59 \times (4.07 \times 10^{-4})}$$

$$= 97.4$$

12. Particulate-probe, cyclone, and filter (gr/dscf)

$$CAN = 0.0154 \times \frac{MF}{VMSTD}$$

$$= 0.0154 \times \frac{117.3}{118.80}$$

$$= 0.0152 \text{ gr/dscf}$$

Table A-3 (Continued)
PARTICULATE CALCULATIONS

13. Particulate-total (gr/dscf)

$$CAO = 0.0154 \times \frac{MT}{VMSTD}$$

$$= 0.0154 \times \frac{173.5}{118.80}$$

$$= 0.0225 \text{ gr/dscf}$$

14. Particulate-probe, cyclone, and filter at stack conditions (gr/acf)

$$CAT = \frac{17.7 \times CAN \times PS \times MD}{(TS + 460)}$$

$$= \frac{17.7 \times 0.0152 \times 29.59 \times 0.892}{(655 + 460)}$$

$$= 0.0064 \text{ gr/acf}$$

15. Particulate-total at stack conditions (gr/acf)

$$CAU = \frac{17.7 \times CAO \times PS \times MD}{(TS) + 460)}$$

$$= \frac{17.7 \times 0.0225 \times 29.59 \times 0.892}{(655 + 460)}$$

$$= 0.0094 \text{ gr/acf}$$

16. Particulate-probe, cyclone and filter (lb/hr)

$$CAW = 0.00857 \times CAN \times QS$$

$$= 0.00857 \times 0.0152 \times 27559$$

$$= 3.591 \text{ lb/hr}$$

17. Particulate-total (lb/hr)

$$CAX = 0.00857 \times CAO \times QS$$

$$= 0.00857 \times 0.0225 \times 27559$$

$$= 5.312 \text{ lb/hr}$$

Table A-3 (Continued)
PARTICULATE CALCULATIONS

[a]Dry standard cubic feet @ 70°F, 29.92 in. Hg

[b]Standard cubic feet @ 70°F, 29.92 in. Hg

[c]Feet per minute @ stack conditions; n = number of data points

[d]Dry standard cubic feet per minute @ 70°F, 29.92 in. Hg

[e]Actual cubic feet per minute @ stack conditions

APPENDIX B

COMPLETE NO_x RESULTS AND SAMPLE CALCULATIONS

Table B-1

NO_x EMISSION DATA

NO_x Calculation

VSC - Sample volume at standard conditions (dry basis), ml

MASS - Mass of NO_2

LB/SCF - Concentration of NO_x as NO_2 (dry basis), lb/scf

Run #	VSC	MASS	PPM	LB/SCF
N-1A	1.82062E3	6.04710E2	1.74044E2	$2.05930E^{-5}$
N-1B	1.61576E3	6.97302E2	2.26140E2	$2.67570E^{-5}$
N-1C	1.72236E3	6.64723E2	2.02232E2	$2.39282E^{-5}$
N-1D	1.67763E3	6.40718E2	2.00125E2	$2.36789E^{-5}$
N-2A	1.64921E3	1.07453E3	3.41409E2	$4.03957E^{-5}$
N-2B	1.64054E3	1.11397E3	3.55809E2	$4.20995E^{-5}$
N-2C	1.62164E3	1.35059E3	4.36417E2	$5.16371E^{-5}$
N-2D	1.71216E3	1.04538E3	3.19936E2	$3.78550E^{-5}$
N-3A	1.55436E3	7.50457E2	2.52991E2	$2.99341E^{-5}$
N-3B	1.61657E3	8.51623E2	2.76048E2	$3.26621E^{-5}$
N-3C	1.42897E3	7.95039E2	2.91539E2	$3.44951E^{-5}$
N-3D	1.78423E3	9.40787E2	2.76294E2	$3.26913E^{-5}$

Table B-2

METHOD 7 - NO_x CALCULATIONS

Plant Woodville Lime and Chemical Company

Date 8-6-74

Test No. N-1A Example Calculation

Volume of flask and valve (ml)	= VF =	2071
Initial absolute pressure of flask (in Hg)	= PI =	2.69
Final absolute pressure of flask (in Hg)	= PF =	29.11
Initial temperature of flask (°F)	= TI =	87
Final temperature of flask (°F)	= TF =	68
Mass of NO_x as NO_2 in gas sample (μg)	= M =	604.7

Volume of sample at standard conditions, dry basis(ml) = VS

$$VS = 17.71 \times (VF-25)\left(\frac{PF}{TF+460} - \frac{PI}{TI+460}\right)$$

$$VS = 17.71 \times (2071 - 25)\left(\frac{29.11}{68+460} - \frac{2.69}{87+460}\right) = 1820 \text{ ml}$$

Concentration of NO_x as NO_2 (dry basis) (lbs/scf) = C

$$C = 6.2 \times 10^{-5} \times \left(\frac{M}{VS}\right)$$

$$C = 6.2 \times 10^{-5} \times \left(\frac{604.7}{1820}\right) = 2.059 \times 10^{-5} \frac{\text{lbs}}{\text{scf}}$$

APPENDIX C

COMPLETE SO_2 RESULTS AND SAMPLE CALCULATIONS

METHOD 6

SO_2 CALCULATION

Plant Woodville Lime Date 8-6-74

Location Woodville, Ohio Run# S-1

Barometric Pressure Absolute (in Hg)	BP =	29.69
Average Meter Temperature (°R) Absolute (°F + 460)	T_m =	539
Volume Through Dry Gas Meter (CF)	V_m =	150.434
Volume of $Ba(ClO_4)_2$ to Titrate Sample (ml)	V_t =	4.59
Volume of $Ba(ClO_4)_2$ to Titrate Blank (ml)	V_b =	0.08
Normality of $Ba(ClO_4)_2$ Solution (g-eq/l)	N =	0.0116
Total Volume of Solution (ml)	V_s =	1000
Volume of Aliquot (ml)	V_a =	25
Volume of Gas at Standard Conditions (SCF) = V_g		

$$V_g = 17.71 \frac{(V_m)(BP)}{(T_m)} = 17.71 \frac{(150.434)(29.69)}{(539)} = 146.8$$

Concentration of SO_2 (lbs/SCF) dry basis = C

$$C = 7.05 \times 10^{-5} \frac{(V_t - V_b)(N)(V_s)}{(V_g)(V_a)} = 7.05 \times 10^{-5} \frac{(4.51)(0.0116)(1000)}{(146.8)(25)}$$

C = 1.005×10^{-6} lbs/SCF

Velocity of Stack Gas (Actual)(FPS)	V =	49.5
% Moisture in Stack Gas	M =	9.61
Area of Stack (ft^2)	A =	22.0
Absolute Stack Temperature (°R)	T_s =	1103
Absolute Stack Pressure (in Hg)	P_s =	29.67

SO_2 Flow Rate (lbs/hr) = S

$$S = \frac{6.377 \times 10^{+4}(1-(M \times 10^{-2}))(V)(A)(P_s)(C)}{(T_s)}$$

$$S = \frac{6.377 \times 10^{4}(.9039)(49.5)(22.0)(29.67)(1.005 \times 10^{-6})}{(1103)}$$

S = 1.90 (lbs/hr)

METHOD 6

SO_2 CALCULATION

Plant Woodville Lime Date 8-7-74

Location Woodville, Ohio Run# S-2

Barometric Pressure Absolute (in Hg)	BP =	29.67
Average Meter Temperature (°R) Absolute (°F + 460)	T_m =	544
Volume Through Dry Gas Meter (CF)	V_m =	99.530
Volume of $Ba(ClO_4)_2$ to Titrate Sample (ml)	V_t =	12.95
Volume of $Ba(ClO_4)_2$ to Titrate Blank (ml)	V_b =	0.08
Normality of $Ba(ClO_4)_2$ Solution (g-eq/l)	N =	0.0116
Total Volume of Solution (ml)	V_s =	1000
Volume of Aliquot (ml)	V_a =	25
Volume of Gas at Standard Conditions (SCF) = V_g		

$$V_g = 17.71 \frac{(V_m)(BP)}{(T_m)} = 17.71 \frac{(99.530)(29.67)}{(544)} = 96.14$$

Concentration of SO_2 (lbs/SCF) dry basis = C

$$C = 7.05 \times 10^{-5} \frac{(V_t - V_b)(N)(V_s)}{(V_g)(V_a)} = 7.05 \times 10^{-5} \frac{(12.87)(0.0116)(1000)}{(96.14)(25)}$$

C = 4.38×10^{-6} lbs/SCF

Velocity of Stack Gas (Actual)(FPS)	V =	50.4
% Moisture in Stack Gas	M =	11.05
Area of Stack (ft^2)	A =	22.0
Absolute Stack Temperature (°R)	T_s =	1111
Absolute Stack Pressure (in Hg)	P_s =	29.65

SO_2 Flow Rate (lbs/hr) = S

$$S = \frac{6.377 \times 10^{+4}(1-(M \times 10^{-2}))(V)(A)(P_s)(C)}{(T_s)}$$

$$S = \frac{6.377 \times 10^{4}(.8895)(50.4)(22.0)(29.65)(4.38 \times 10^{-6})}{(1111)}$$

S = 7.35 (lbs/hr)

METHOD 6

SO_2 CALCULATION

Plant Woodville Lime Date 8-8-74

Location Woodville, Ohio Run# S-3

Barometric Pressure Absolute (in Hg)	BP =	29.61
Average Meter Temperature (°R) Absolute (°F + 460)	T_m =	556
Volume Through Dry Gas Meter (CF)	V_m =	78.286
Volume of $Ba(ClO_4)_2$ to Titrate Sample (ml)	V_t =	9.88
Volume of $Ba(ClO_4)_2$ to Titrate Blank (ml)	V_b =	0.08
Normality of $Ba(ClO_4)_2$ Solution (g-eq/l)	N =	0.0116
Total Volume of Solution (ml)	V_s =	500
Volume of Aliquot (ml)	V_a =	25
Volume of Gas at Standard Conditions (SCF) = V_g		

$$V_g = 17.71 \frac{(V_m)(BP)}{(T_m)} = 17.71 \frac{(78.286)(29.61)}{(556)} = 73.84$$

Concentration of SO_2 (lbs/SCF) dry basis = C

$$C = 7.05 \times 10^{-5} \frac{(V_t - V_b)(N)(V_s)}{(V_g)(V_a)} = 7.05 \times 10^{-5} \frac{(9.80)(0.0116)(500)}{(73.84)(25)}$$

C = 2.17×10^{-6} lbs/SCF

Velocity of Stack Gas (Actual)(FPS)	V =	49.8
% Moisture in Stack Gas	M =	10.81
Area of Stack (ft^2)	A =	22.0
Absolute Stack Temperature (°R)	T_s =	1114
Absolute Stack Pressure (in Hg)	P_s =	29.59

SO_2 Flow Rate (lbs/hr) = S

$$S = \frac{6.377 \times 10^{+4}(1-(M \times 10^{-2}))(V)(A)(P_s)(C)}{(T_s)}$$

$$S = \frac{6.377 \times 10^{4}(.8919)(49.8)(22.0)(29.59)(2.17 \times 10^{-6})}{(1114)}$$

S = 3.59 (lbs/hr)

APPENDIX E

FIELD TEST DATA SHEETS

TRAVERSE POINT LOCATION FOR CIRCULAR DUCTS

PLANT Woodville Lime & Chemical
DATE 8/9/74
SAMPLING LOCATION Kiln Stack - outlet of ESP

	West	South
INSIDE OF FAR WALL TO OUTSIDE OF NIPPLE, (DISTANCE A)	62 7/8	63 5/8
INSIDE OF NEAR WALL TO OUTSIDE OF NIPPLE, (DISTANCE B)	1/8	1/8
STACK I.D., (DISTANCE A - DISTANCE B)	62 3/4	63 1/2

NEAREST UPSTREAM DISTURBANCE 146"
NEAREST DOWNSTREAM DISTURBANCE 48"
CALCULATOR D. Harris

SCHEMATIC OF SAMPLING LOCATION

TRAVERSE POINT NUMBER	FRACTION OF STACK I.D.	STACK I.D.	PRODUCT OF COLUMNS 2 AND 3 (TO NEAREST 1/8 INCH)	DISTANCE B	TRAVERSE POINT LOCATION FROM OUTSIDE OF NIPPLE (SUM OF COLUMNS 4 & 5)
1	Traverse points were used as calculated from a previous sampling job (5/20/74) at this same location. They are listed here:			1/8	1 1/8
2					2 1/8
3					3 5/8
4					5 1/8
5					6 3/4
6					8 1/2
7					10 3/8
8					12 1/2
9					14 3/4
10					17 1/2
11					20 5/8
12					25 1/2
13					38 3/8
14					43 1/8
15					46 3/8
16					49
17					51 3/8
18					53 1/2
19					55 1/4
20					56 7/8
21					58 7/8
22					60 1/8
23					61 5/8
24					62 5/8

EPA (Dur) 232
4/72

NOMOGRAPH DATA

PLANT Woodville Chemical Co.

DATE 8-6-74

SAMPLING LOCATION Lime Kiln ESP #1 Outlet

Run 1 1

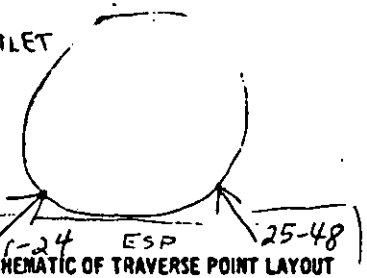
CALIBRATED PRESSURE DIFFERENTIAL ACROSS ORIFICE, in. H_2O	$\Delta H_@$	1.877
AVERAGE METER TEMPERATURE (AMBIENT + 20°F), °F	$T_{m_{avg.}}$	90
PERCENT MOISTURE IN GAS STREAM BY VOLUME	B_{wo}	12
BAROMETRIC PRESSURE AT METER, in. Hg	P_m	29.69
STATIC PRESSURE IN STACK, in. Hg ($P_m \pm 0.073$ x STACK GAUGE PRESSURE in in. H_2O)	P_s	29.72
RATIO OF STATIC PRESSURE TO METER PRESSURE	P_s/P_m	1
AVERAGE STACK TEMPERATURE, °F	$T_{s_{avg.}}$	674
AVERAGE VELOCITY HEAD, in. H_2O	$\Delta p_{avg.}$	.42
MAXIMUM VELOCITY HEAD, in. H_2O	$\Delta p_{max.}$	.52
C FACTOR	.93	
CALCULATED NOZZLE DIAMETER, in.	.31	
ACTUAL NOZZLE DIAMETER, in.	.374	
REFERENCE Δp, in H_2O	.20	

EPA (Dur) 234
4/72

FIELD DATA

E

PLANT Wordville Chemical Co.
DATE 8-6-74
SAMPLING LOCATION Lime Kiln #1 ESP OUTLET
SAMPLE TYPE EPA-5
RUN NUMBER P-1
OPERATOR Thalman, Schliesser
AMBIENT TEMPERATURE 85°F
BAROMETRIC PRESSURE 29.69
STATIC PRESSURE, (P_s) -.34 H_2O
FILTER NUMBER (s) 32-5



SCHEMATIC OF TRAVERSE POINT LAYOUT

PROBE LENGTH AND TYPE 10ft.
NOZZLE I.D. .374 Changed to .244
ASSUMED MOISTURE, % 12
SAMPLE BOX NUMBER MRC
METER BOX NUMBER MRC
METER $\Delta H_@$ 1.877
C FACTOR .93
PROBE HEATER SETTING HI
HEATER BOX SETTING HI
REFERENCE Δp .20

READ AND RECORD ALL DATA EVERY 4 MINUTES LEAK RATE .007 CFM @ 18 "Hg

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\,in}$), °F	DRY GAS METER TEMPERATURE OUTLET ($T_{m\,out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F	Probe Temp
	0	1525	489.270										
1	0	1525	489.270	.20	1.85	1.85	422	88	84	10.5	237	56	225
2	4	1529	492.120	.24	2.2	2.2	456	87	85	12.0	238	53	215
3	8	1533	495.049	.26	2.35	2.35	471	90	89	13.5	238	52	228
4	12	1537	498.344	.29	2.62	2.62	517	90	85	15.5	239	52	239
5	16	1541	501.639	.30	2.7	2.7	563	88	85	16.0	229	53	245
6	20	1545	504.959	.32	2.9	2.9	568	87	86	17.5	243	53	250
7	24	1549	508.431	.32	2.9	2.9	580	85	86	19.0	245	54	250
8	28	1553	511.934	.35	3.2	3.2	630	85	86	20.5	247	54	258
9	32	1557	515.651	.34	3.1	3.1	634	85	86	20.5	251	55	261
10	36	1601	519.215	.35	3.2	3.2	635	85	86	20.5	252	56	267
11	40	1605	523.199	.42	3.8	2.9	642	84	87	20.5	255	67	268
12	44	1609	526.791	.48	4.3	2.9	638	85	86	20.5	255	60	275
13	48	1613	530.284	.47	4.5	2.9	631	86	87	20.5	254	62	280
14	52	1656	533.842	.51	.93	1.03	632	85	86	6.5	215	55	235
15	56	1640	535.834	.51	.93	1.0[illegible]	632	87	85	7.0	206	52	279
16	60	1644	538.001	.50	.91	.91	636	90	86	6.5	212	52	271

SHUT DOWN FOR PROBE TIP CHANGE AT 1617; 52 minutes into the run

PLANT Woodville Chemical DATE 8-6-74 RUN NO. 1-P

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\ in}$), °F	OUTLET ($T_{m\ out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F	Probe Temp.
17	64	1648	540.410	.50	.91	.91	646	91	86	6.0	21	53	477
18	68	1652	542.379	.47	.86	.86	655	92	87	5.5	218	53	552
19	72	1656	544.415	.44	.80	.80	658	91	86	5.5	226	55	577
20	76	1700	546.341	.44	.80	.80	660	91	87	5.5	229	55	594
21	80	1704	548.736	.41	.77	.77	663	92	86	5.0	226	56	620
22	84	1708	550.490	.38	.69	.69	664	93	86	4.5	230	57	632
23	88	1712	552.301	.36	.66	.66	662	92	86	4.5	233	57	648
24	92	1716	554.051	.33	.61	.61	655	91	86	4.5	237	57	649
25	96	[redacted]	555.718	.19	.36	.36	384	84	85	3.5	235	67	194
26	100	1740	557.091	.30	.55	.55	418	85	85	4.0	232	65	213
27	104	1744	558.654	.33	.60	.60	534	87	85	4.0	232	63	205
28	108	1752	560.205	.33	.60	.60	584	88	84	4.0	231	61	215
29	112	1756	562.045	.40	.73	.73	610	91	89	5.0	231	63	219
30	116	1800	563.940	.35	.64	.64	655	91	85	4.5	233	62	220
31	120	1804	565.899	.40	.73	.73	662	91	85	4.5	233	64	233
32	124	1808	567.439	.40	.73	.73	668	91	85	5.0	234	56	225
33	128	1812	569.810	.46	.85	.85	670	92	85	5.0	234	53	236
34	132	1816	571.271	.48	.87	.87	669	92	85	5.5	234	53	[illegible]
35	136	1820	573.151	.48	.87	.87	668	91	85	5.5	232	55	237
36	140	1824	575.084	.48	.87	.87	655	92	85	5.5	234	55	246
37	144	1828	577.051	.51	.93	.93	651	92	85	5.5	234	56	251
38	148	1832	579.149	.52	.94	.94	645	92	85	5.5	234	57	252
39	152	[redacted]	579.926	.50	.91	.91	618	92	84	5.5	234	56	380
40	156	[redacted]	583.321	.49	.89	.89	643	87	84	5.5	234	56	548
41	160	[redacted]	585.499	.49	.89	.89	649	89	84	5.5	230	55	620
42	164	[redacted]	587.610	.47	.86	.86	644	87	84	5.5	230	55	630
43	168	[redacted]	589.847	.45	.83	.83	643	86	83	5.5	232	57	680

SHUT DOWN CHANGE PORTS AT 1720

SHUT DOWN AT 1834 or 150 min. into the run. Drop in ΔH, increase in pressure beyond a point where we could run STOPPED A [illegible] inserted for play in line

SUBTRACT DIFF-

Start at 1842

CU. FT. READS 589,270 - start

SUBTRACT DIFFERENCE

PLANT Woodville Chemical Co. DATE 8-6 74 RUN NO. 1-P

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\ in}$), °F	OUTLET ($T_{m\ out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F	
44	172	1904	591.369	.40	.73	.73	64	87	83	5.0	218	60	693
45	176	1908	593.414	.37	.68	.68	33	87	83	4.5	219	62	704
46	180	1912	595.344	.34	.62	.62	25	87	83	4.0	221	64	658
47	184	1916	596.64	.24	.44	.44	22	87	83	3.5	225	62	641
48	188	1920	598.439	.21	.39	.39	577	89	83	3.0	220	61	642
end	192	1924	599.515										
			109.901				611	86.97					
								87°					
				$\sqrt{\Delta P}$ = .6359									

CLEAN-UP DATA

Plant Woodville

Date 8-6-74

Sampling Location ESP OUTLET

Sample Type EPA-5

Run Number 1-P

Sample Box Number 1

Clean-up Man THALMAN

Comments:

IMPINGERS

	#1	#2	#3	#4	#5
Final Vol.	400 ml	ml	ml	ml	ml
Initial Vol.	200 ml	ml	ml	ml	ml
Net Vol.	200 ml	ml	ml	ml	ml

Total Net Volume in Impingers 200 ml

(2) SILICA GEL

Final Weight	~~377~~ 322.5 g	315 g	g
Initial Weight	300 g	300 g	g
Net Weight	22.5 g	15 g	g

Total Net Weight in Silica Gel 37.5 g

Total Moisture 237.5 g

Filter Number(s) 32-5

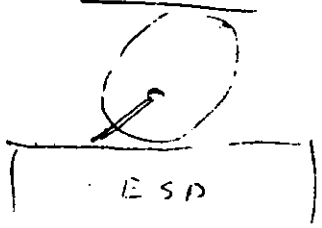
MRC 7/73

(261)

FIELD DATA

single point

PLANT Woodville Lime
DATE 8-6-74
SAMPLING LOCATION ~~Stk~~
SAMPLE TYPE SO_2
RUN NUMBER S-1
OPERATOR T. Stewart
AMBIENT TEMPERATURE 86
BAROMETRIC PRESSURE 29.69
STATIC PRESSURE, (P_s) -.3" H_2O
FILTER NUMBER (s) 32-4 (Petrian 1st & 2nd depending on)



PROBE LENGTH AND TYPE 3' glass
NOZZLE I.D. .375
ASSUMED MOISTURE, % 12%
SAMPLE BOX NUMBER MRC #2
METER BOX NUMBER " "
METER $\Delta H_@$ 1.767
C FACTOR none
PROBE HEATER SETTING Low
HEATER BOX SETTING 5-6
REFERENCE Δp (.2) — zero at 2.5" H_2O

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY 12 MINUTES LEAK RATE .0025 CFM @ 15" Hg

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\ in}$), °F	DRY GAS METER TEMPERATURE OUTLET ($T_{m\ out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F	PROBE
		15:25	656.280										
	0		656.280	.03	2.0	2.0	637	76	80	14.0	80	67	762
	12		665.621	.03	2.0	2.0	637	82	77	13.0	92	69	755
	24		674.865	.03	2.0	2.0	636	82	78	13.5	99	84	751
	36		683.135	.03	2.0	2.0	633	82	78	13.5	105	90	743
*	48	16:13 stopped 16:36 started	693.482	.03	2.0	2.0	636	81	79	13.5	108	89	748
	60		702.928	*.15	2.0	2.0	635	82	78	13.5	105	73	753
	72		712.422	.20	2.0	2.0	644	81	78	13.5	110	77	755
	84		721.926	.26	2.0	2.0	644	81	79	13.5	114	80	759
	96	stopped 17:14 started	731.329	.20	2.0	2.0	643	81	79	13.5	117	76	757
	108		741.367	.31	2.0	2.0	648	78	77	13.0	108	67	754
	120		751.942	.30	2.0	2.0	649	79	78	13.0	112	68	752
	132		761.170	.32	2.0	2.0	647	79	78	13.0	116	73	751
	144	18:14 stopped	771.035	.32	2.0	2.0	651	80	78	13.0	118	23	750
	156	18:42 started	780.937	.34	2.0	2.0	649	78	77	13.0	113	21	755
	168		790.070	.40	2.0	2.0	645	76	76	13.0	115	72	753
	180		800.724	.42	2.0	2.0	649	76	76	13.0	117	77	753

187 - ~~[illegible]~~ top short datum 5 min - 806.714 - 814 early to purge system [illegible] 15 min. (T.S.) (stopped) 816.527

* stopped to change filter tips - on P-1 unit - (T.S)

CLEAN-UP DATA

Plant Woodville Lime Comments:

Date 8-6-74

Sampling Location ESP. OUTLET

Sample Type EPA - 8

Run Number ~~Test~~ S-1

Sample Box Number 2

Clean-up Man THALMAN

IMPINGERS

	Isopropanol #1	H_2O_2 #2	#3	~~#4~~	Isopropanol ~~[illegible]~~ other impinger #1 ~~#5~~
Final Vol.	120.5 ml	344 ml	ml	ml	117 ml
Initial Vol.	100.0 ml	200 ml	ml	ml	100 ml
Net Vol.	20.5 ml	144 ml	~~334~~ ml	ml	17 ml

Total Net Volume in Impingers 181.5 ml

SILICA GEL

	1st time	2nd time	
Final Weight	351.5 g	357.5 g	g
Initial Weight	300.0 g	300.0 g	g
Net Weight	51.5 g	57.5 g	g

Total Net Weight in Silica Gel 109.0 g

Total Moisture 290.5 g

Filter Number(s) 32-4

MRC 7/73

DRY MOLECULAR WEIGHT DETERMINATION

PLANT Woodville Lime & Chem.
DATE 8-6-'74
SAMPLING TIME (24-hr CLOCK) Runs P-1 and S-1
SAMPLING LOCATION Lime kiln Stack
SAMPLE TYPE (BAG, INTEGRATED, CONTINUOUS) Integrated
ANALYTICAL METHOD Orsat
AMBIENT TEMPERATURE 86°F
OPERATOR Davis & D'Hurin

COMMENTS: Meter seal in Orsat 8/5/74

GAS \ RUN	1		2		3		AVERAGE NET VOLUME	MULTIPLIER	MOLECULAR WEIGHT OF STACK GAS (DRY BASIS) M_d, lb/lb-mole
	ACTUAL READING	NET	ACTUAL READING	NET	ACTUAL READING	NET			
CO_2	19	19	19	19	19	19	19	44/100	8.36
O_2 (NET IS ACTUAL O_2 READING MINUS ACTUAL CO_2 READING)	27.8	8.8	27.8	8.8	27.4	8.4	8.7	32/100	2.784
CO (NET IS ACTUAL CO READING MINUS ACTUAL O_2 READING)	27.8	0	27.8	0	27.4	0	0	28/100	20.244
N_2 (NET IS 100 MINUS ACTUAL CO READING)							72.3	28/100	31.388
								TOTAL	31.37

EPA (Dur) 230
4/72

NOMOGRAPH DATA

PLANT Woodville, Ohio

DATE 8-7-74

SAMPLING LOCATION Kenel Stack

Run # P-2

CALIBRATED PRESSURE DIFFERENTIAL ACROSS ORIFICE, in. H_2O	$\Delta H_@$	1.877 ~~4.658~~
AVERAGE METER TEMPERATURE (AMBIENT + 20°F), °F	$T_{m_{avg.}}$	85
PERCENT MOISTURE IN GAS STREAM BY VOLUME	B_{wo}	10
BAROMETRIC PRESSURE AT METER, in. Hg	P_m	29.67
STATIC PRESSURE IN STACK, in. Hg ($P_m \pm 0.073$ x STACK GAUGE PRESSURE in in. H_2O)	P_s	-.3
RATIO OF STATIC PRESSURE TO METER PRESSURE	P_s/P_m	1
AVERAGE STACK TEMPERATURE, °F	$T_{s_{avg.}}$	610
AVERAGE VELOCITY HEAD, in. H_2O	$\Delta p_{avg.}$	.4
MAXIMUM VELOCITY HEAD, in. H_2O	$\Delta p_{max.}$	.5
C FACTOR		.96 8-8-74 .94
CALCULATED NOZZLE DIAMETER, in.		.300
ACTUAL NOZZLE DIAMETER, in.		.305
REFERENCE Δp, in. H_2O		.42 .42

EPA (Dur) 234
4/72

FIELD DATA

PLANT WOODVILLE - Lime Stone
DATE 8-7-74
SAMPLING LOCATION ~~Kraft~~ S ~~Scrubber~~ Outlet
SAMPLE TYPE METHOD 5
RUN NUMBER P-2
OPERATOR T. STEWART + C. OLNCAN
AMBIENT TEMPERATURE 85
BAROMETRIC PRESSURE 29.67
STATIC PRESSURE, (P_s) ~~-8~~ -3
FILTER NUMBER (s) 32-1

PROBE LENGTH AND TYPE 7' GLASS
NOZZLE I.D. .305
ASSUMED MOISTURE, % 10
SAMPLE BOX NUMBER MRC 1
METER BOX NUMBER " "
METER $\Delta H_@$ 1.877
C FACTOR .96
PROBE HEATER SETTING HIGH
HEATER BOX SETTING "
REFERENCE Δp .42

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY 4 MINUTES LEAK RATE .001 CFM @ 15 "Hg

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\ in}$), °F	OUTLET ($T_{m\ out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F	PROBE
	~~10:44~~ 11:44	~~10:44~~ 44	599.634										
1	~~0~~	0	599.634	.19	.86	.84	525	85	82	3.5	232	46	209
2		4	601.828	.30	1.35	1.35	620	88	82	4.0	250	46	221
3		8	604.034	.32	1.42	1.42	646	92	82	4.5	265	45	234
4		12	606.569	.39	1.7	1.7	675	93	83	5.0	256	43	238
5		16	609.278	.35	1.5	1.5	689	94	84	4.8	256	44	230
6		20	611.934	.40	1.8	1.8	693	95	85	5.5	258	44	248
7		24	614.785	.38	1.7	1.7	694	96	85	5.2	259	46	248
8		28	617.605	.40	1.8	1.8	694	96	86	5.5	261	45	253
9		32	620.405	.40	1.8	1.8	689	97	86	5.5	263	45	260
10		36		.40	1.8	1.8	688	97	87	5.5	265	46	262
11		~~40~~	626.184	.48	2.1	2.1	684	98	88	6.3	268	46	248
12		44	629.262	.48	2.1	2.1	673	99	88	6.3	270	46	248
13		~~48~~ 48	632.302	.56	2.4	2.4	662	99	89	7.1	272	47	249
14		~~50~~ 52	635.592	.56	2.4	2.4	663	99	89	7.2	272	46	274
15		56	638.832	.56	2.4	2.4	664	98	90	7.2	274	47	312
16		60	642.089	.54	2.3	2.3	662	98	90		275	49	406

(2.)

PLANT Toledo Lime Stone DATE 8-7-74 RUN NO. P-2

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F	probe
17		64	645.321	.51	2.2	2.2	666	98	91	7.0	277	48	574
18		68	648.504	.50	2.2	2.2	667	99	91	7.0	267	50	655
19		72	651.675	.45	2.0	2.0	665	99	91	6.5	263	52	680
20		76	654.758	.45	2.0	2.0	663	99	92	6.5	257	52	679
21		80	657.942	.38	1.7	1.7	657	100	91	6.0	254	52	680
22		84	660.788	.34	1.5	1.5	651	100	91	5.2	251	52	682
23		88	663.498	.28	1.25	1.25	632	99	92	4.5	250	53	686
24		92	665.352	.15	.70	.70	583	101	92	3.0	250	54	674
	Stop 13:20	96	Stop 667.942										
Changed Points													
25	Start 13:45	96	667.942	.27	1.19	1.19	611	91	92	4.2	257	55	243
26		100	670.235	.25	1.14	1.14	601	92	92	4.2	249	47	247
27		104	672.622	.27	1.2	1.2	624	92	92	4.2	249	47	256
28		108	675.965	.30	1.32	1.32	647	100	91	4.5	256	48	268
29		112	677.448	.31	1.4	1.4	657	101	92	4.8	249	50	278
30		116	680.020	.34	1.5	1.5	665	102	92	5.0	249	49	276
31		120	682.652	.34	1.5	1.5	676	102	92	5.0	255	53	273
32		124	685.315	.35	1.55	1.55	674	100	92	5.1	259	53	270
33		128	—	.38	1.55	1.55	683	102	93	5.1	263	54	275
34		132	693.642	.35	1.55	1.55	681	101	93	5.1	265	55	277
35		136	693.322	.41	1.81	1.81	680	100	93	5.9	267	51	281
36		140	696.185	.44	1.95	1.95	665	102	93	6.5	269	53	294
37		144	—	.50	2.2	2.2	660	104	94	7.0	271	53	314
38		148	702.272	.56	2.25	2.25	681	104	95	7.3	274	54	326
39		152	705.412	.52	2.3	2.3	665	105	95	7.5	276	54	319
40		156	708.656	.50	2.2	2.2	675	104	94	7.5	278	54	395
41		160	711.842	.47	2.0	2.0	682	105	95	7.2	279	55	531

24 5/5 (3.)

PLANT Toledo Limestone DATE 8-7-74 RUN NO P-2

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\ in}$), °F	OUTLET ($T_{m\ out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F	Probe
42		164	715.089	.45	2.0	2.0	684	105	95	6.8	279	56	569
43		168	717.025	.40	1.8	1.8	683	105	95	6.2	281	56	599
44		172	720.352	.38	1.7	1.7	684	105	95	6.3	281	56	624
45		176	723.040	.37	1.6	1.6	683	105	95	6.0	281	55	644
46		180	726.605	.35	1.55	1.55	677	103	95	5.5	281	56	653
47		184	729.328	.32	1.45	1.45	672	101	95	5.5	282	57	654
48		188	732.262	.23	1.0	1.0	650	101	95	4.5	283	57	653
	stop 1821	192	stop 734.462										
				29.7621			31739						
				$\sqrt{.6200}$			661.23						

CLEAN-UP DATA

Plant Woodville Lime Comments:
Date 8/7/74
Sampling Location Kiln Stack
Sample Type Particulate
Run Number P-2
Sample Box Number MRC - 2
Clean-up Man Stewart, Duncan, Harris

IMPINGERS

	#1	#2	#3	#4	#5
Final Vol.	354 ml	140 ml	4 ml	ml	ml
Initial Vol.	100 ml	100 ml	0 ml	ml	ml
Net Vol.	254 ml	40 ml	4 ml	ml	ml

Total Net Volume in Impingers 298 ml

SILICA GEL

Final Weight	337.8 g	g	g
Initial Weight	300.0 g	g	g
Net Weight	37.8 g	g	g

Total Net Weight in Silica Gel _____ g

Total Moisture _____ g

Filter Number(s) 32-1 _____

MRC 7/73

1-16-72-04

FIELD DATA

PLANT Mecklenburg Lime
DATE 8-7-74
SAMPLING LOCATION [illegible] OUTLET
SAMPLE TYPE [illegible] 8
RUN NUMBER 2-S S-2
OPERATOR [illegible]
AMBIENT TEMPERATURE 80
BAROMETRIC PRESSURE
STATIC PRESSURE, (P_s) -.30
FILTER NUMBER (s) 34-17 INSIDE 3½"
32-15 BETWEEN IMPINGERS 1 & 2
4½"

PROBE LENGTH AND TYPE 3' glass
NOZZLE I.D.
ASSUMED MOISTURE, % 10
SAMPLE BOX NUMBER MRC-2
METER BOX NUMBER MRC-2
METER $\Delta H_@$ 1.762
C FACTOR —
PROBE HEATER SETTING off
HEATER BOX SETTING off
REFERENCE Δp Run at ΔH of 1.0

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY 12 MINUTES LEAK RATE .008 CFM @ 19.5" Hg

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\ in}$), °F	DRY GAS METER TEMPERATURE OUTLET ($T_{m\ out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F	PROBE TEMP °F
	0	11:44	817.212	.18	1.0	1.0	654	81	79	6.5	77	66	611
	12	11:56	—	.20	1.0	1.0	651	83	80	6.3	80	67	622
	24	12:08	830.832	.22	1.0	1.0	653	83	80	6.5	82	69	624
	36	12:20	837.727	.22	1.0	1.0	651	84	81	6.5	85	71	620
	48	12:32	—	.24	1.0	1.0	652	85	82	6.5	88	67	624
	60	12:44	851.165	.20	1.0	1.0	653	85	82	6.5	89	68	625
	72	12:56	857.215	.20	1.0	1.0	653	85	82	6.6	91	68	630
	84	1:08	864.595	.25	1.0	1.0	654	87	83	6.6	92	73	629
RESTARTED	96	1:20	871.625	.23	1.0	1.0	654	86	84	6.6	93	86	625
AT 1:46	108	1:58	878.061	.24	1.0	1.0	651	88	83	6.5	87	66	620
	120	2:10	884.951	.23	1.0	1.0	648	87	83	6.5	89	70	618
	132	2:22	—	.18	1.0	1.0	650	86	83	6.5	92	71	615
	144	2:34	898.612	.20	1.0	1.0	648	87	84	6.6	94	70	621
	156	2:46	904.886	.22	1.0	1.0	650	90	84	6.6	94	71	621
	168	2:58	910.910	.20	1.0	1.0	650	90	85	6.6	95	74	620
STOPPED	180 ~~192~~	3:10 ~~3:22~~	916.742										

* PURGED @ ΔH=1.0 FOR 10 MIN. V_m: 921.514

CLEAN-UP DATA

Plant Woodville Lime-Chemical Comments:
Date 8-7-74
Sampling Location ESP #1
Sample Type EPA-8
Run Number 2-S
Sample Box Number MRC
Clean-up Man Thalman, Schliesser

IMPINGERS

	#1 (Isopropanol)	#2 (H_2O_2)	#3 (H_2O_2)	#4	#5
Final Vol.	252 ml	320 ml	ml	ml	ml
Initial Vol.	200 ml	200 ml	ml	ml	ml
Net Vol.	152 ml	120 ml	ml	ml	ml

Total Net Volume in Impingers ~~272~~ 172 ml

SILICA GEL

Final Weight	350.5 g	g	g
Initial Weight	300 g	g	g
Net Weight	50.5 g	g	g

Total Net Weight in Silica Gel 50.5 g

Total Moisture 222.5 g

Filter Number(s) oven — 34-17
first + 2nd → 32-15

MRC 7/73

DRY MOLECULAR WEIGHT DETERMINATION

PLANT Hydrite Lime & Chemical Co.
DATE 8-7-74
SAMPLING TIME (24-hr CLOCK) 1145 – 1522
SAMPLING LOCATION Kiln Stack
SAMPLE TYPE (BAG, INTEGRATED, CONTINUOUS) integrated
ANALYTICAL METHOD Orsat
AMBIENT TEMPERATURE ~85
OPERATOR [illegible]

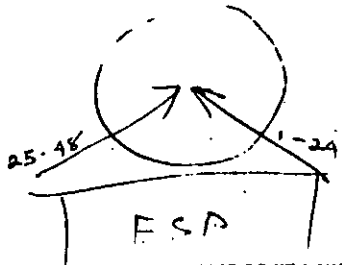
COMMENTS: first series was contaminated with air & was not included in average. (see run #3 below)

RUN / GAS	1		2		3		AVERAGE NET VOLUME	MULTIPLIER	MOLECULAR WEIGHT OF STACK GAS (DRY BASIS) M_d, lb/lb-mole
	ACTUAL READING	NET	ACTUAL READING	NET	ACTUAL READING	NET			
CO_2	22.0	22	22.0	22	19.8	19.8	22	44/100	
O_2 (NET IS ACTUAL O_2 READING MINUS ACTUAL CO_2 READING)	28.0	6	28.2	6.2	27.6		6.1	32/100	
CO (NET IS ACTUAL CO READING MINUS ACTUAL O_2 READING)	28.0	0	28.2	0	27.6		0	28/100	
N_2 (NET IS 100 MINUS ACTUAL CO READING)		72		71.8			71.9	28/100	
								TOTAL	

EPA (Dur) 230
4/72

FIELD DATA

PLANT Woodville Lime
DATE 8-8-74
SAMPLING LOCATION @ ESP OUTLET
SAMPLE TYPE ~~ESP~~ METHOD 5
RUN NUMBER R-7
OPERATOR Steve & Duncan
AMBIENT TEMPERATURE 85
BAROMETRIC PRESSURE 29.61
STATIC PRESSURE, (P_s) -0.3 "H_2O
FILTER NUMBER (s) 32-25



SCHEMATIC OF TRAVERSE POINT LAYOUT

PROBE LENGTH AND TYPE 7' Glass
NOZZLE I.D. .305
ASSUMED MOISTURE % 16
SAMPLE BOX NUMBER MRC #1
METER BOX NUMBER "
METER $\Delta H_@$ 1.877
C FACTOR .96
PROBE HEATER SETTING High
HEATER BOX SETTING "
REFERENCE Δp .42

READ AND RECORD ALL DATA EVERY 4 MINUTES LEAK RATE .026 CFM @ 15 "Hg

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\ in}$), °F	DRY GAS METER TEMPERATURE OUTLET ($T_{m\ out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F	PROBE TEMP
1	10:27	0	734.646	.12	.46	.46	406	95	91	3.8	287	55	240
2	10:31	4	736.441	.22	.80	.80	490	100	92	4.6	285	50	232
3	10:35	8	–	.32	1.45	1.45	595	104	92	6.6	290	48	226
4	10:39	12	740.820	.36	1.35	1.35	658	107	92	7.0	295	47	234
* 5	10:43	16	743.341	.41	1.51	1.51	679	108	93	7.5	296	47	239
6	10:47	20	–	.39	1.60	1.60	691	110	94	7.8	302	48	252
7	10:51	24	748.646	.39	1.55	1.55	685	110	94	7.9	302	51	255
8	10:55	28	751.395	.42	1.69	1.69	686	112	95	8.2	300	52	259
9	10:59	32	754.042	.45	1.80	1.80	687	112	95	8.9	308	52	269
10	11:03	36	756.902	.475	1.87	1.87	682	114	97	8.5	316	52	279
11	11:07	40	759.871	.475	1.87	1.87	679	114	98	9.2	312	52	280
12	11:11	44	–	.44	1.75	1.75	665	115	99	9.2	320	52	288
13	11:15	48	765.521	.50	1.98	1.98	656	116	98	9.8	325	51	304
14	11:19	52	768.532	.49	1.95	1.95	659	117	100		329	51	338
15	11:23	56	771.521	.47	1.87	1.87	661	116	101	10.0	340	54	454
16	11:27	60	774.482	.46	.46	1.81	660	116	102	9.8	342	56	450

* - Reset manometer to Ref .48 "H_2O Stack temp ≈ 675°

PLANT Wilsonville DATE 8-8-74 RUN NO P-3

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\,in}$), °F	DRY GAS METER TEMPERATURE OUTLET ($T_{m\,out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE °F	
17	64	11:31	—	.44	1.75	1.75	660	118	103	9.5	353	57	549
18	68	11:35	780.311	.42	1.66	1.66	658	119	103	9.0	358	58	635
19	72	11:39	783.121	.40	1.60	1.60	656	119	104	8.8	364	57	657
20	76	11:43	785.932	.39	1.55	1.55	655	119	104	8.7	370	58	664
21	80	11:47	788.684	.35	1.41	1.41	648	120	105	8.0	374	58	665
22	84	11:51	791.388	.33	1.32	1.32	638	120	105	7.6	379	60	663
23	88	11:55	793.871	[redacted].29	1.16	1.16	629	120	106	7.0	384	62	661
24	92	11:59	796.345	.23	.93	.93	602	119	106	5.7	389	64	658
	96.[redacted]	12:03	798.574										
25	96.[redacted]	12:22	798.574	.17	.70	.70	522	105	105	4.3	238	49	193
26	100	12:26	800.304	.24	.96	.96	575	107	105	5.4	272	48	222
27	104	12:30	802.425	.31	1.23	1.23	625	111	105	6.5	295	46	249
28	108	12:34	804.782	.32	1.29	1.29	655	113	105	6.6	302	[redacted]49	261
29	112	12:38	807.208	.33	1.31	1.31	668	113	104	7.0	310	45	258
30	116	12:42	809.692	.33	1.31	[redacted]1.31	672	113	104	7.0	316	46	258
31	120	12:46	812.[redacted]210	.35	1.40	1.40	679	113	104	7.4	323	47	263
32	124	12:50	814.781	.35	1.40	1.40	682	114	105	7.4	322	50	268
33	128	12:54	817.327	.37	1.50	1.50	686	115	106	7.9	335	49	283
34	132	12:58	820.000	.39	1.55	1.55	692	116	104	8.3	337	53	302
35	136	1:02	—	.43	1.72	1.72	686	116	106	9.0	333	51	311
36	140	1:06	—	.47	1.86	1.86	678	117	107	9.8	319	51	328
37	144	1:10	828.324	.51	2.0	2.0	672	117	107	10.7	319	51	330
38	148	1:14	831.365	.5	1.85	1.85	676	120	107	10.7	319	55	352
39	152	1:18	834.360	.5	1.85	1.85	685	120	108	10.7	320	54	319
40	156	1:22	836.632	.48	1.80	1.80	692	120	108	10.7	318	56	518
41	160	1:26	838.618	.46	2.0	2.0	695	127	108	10.7	322	56	602

PLANT Woodville DATE 8-8-74 RUN NO P-3

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\ in}$), °F	DRY GAS METER TEMPERATURE OUTLET ($T_{m\ out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F	
42	164	1:30	843.360	.42	1.8	1.8	698	120	109	10.7	319	56	658
43	168	1:34	846.346	.42	1.8	1.8	698	117	109	10.6	326	54	676
44	172	1:38	849.318	.38	1.65	1.65	695	115	109	10.0	323	54	693
45	176	1:42	852.270	.38	1.65	1.65	693	115	108	10.0	318	55	703
46	180	1:46	855.120	.35	1.52	1.52	686	113	108	9.3	309	58	712
47	184	1:50	857.901	.30	1.32	1.32	680	114	108	8.2	311	58	718
48	188	1:54	860.540	.28	1.22	1.22	674	111	108	8.0	316	57	720
	192	1:58	863.006										
						1.53	674 / 110						
			128.360										
				$\sqrt{\Delta p}$ = .42			110 R						
								108.5 / 568.5 R					

24

CLEAN-UP DATA

Plant Woodville Lime Comments:
Date 8/8/74
Sampling Location Kiln ESP Stack
Sample Type Particulate
Run Number P-3
Sample Box Number MRC-1
Clean-up Man Duncan, Harris, + Stewart

IMPINGERS

	#1	#2	#3	#4	#5
Final Vol.	324 ml	142 ml	6 ml	ml	ml
Initial Vol.	100 ml	100 ml	ml	ml	ml
Net Vol.	224 ml	42 ml	6 ml	ml	ml

Total Net Volume in Impingers 272 ml

SILICA GEL

Final Weight	331.8 g	g	g
Initial Weight	300 g	g	g
Net Weight	31.8 g	g	g

Total Net Weight in Silica Gel 31.8 g

Total Moisture 303.8 g

Filter Number(s) 3225

MRC 7/73

DRY MOLECULAR WEIGHT DETERMINATION

PLANT Woodville Lime Eliminator
DATE 8-8-74
SAMPLING TIME (24-hr CLOCK) 10:30 to 1:56
SAMPLING LOCATION EST #2
SAMPLE TYPE (BAG, INTEGRATED, CONTINUOUS) Integrated
ANALYTICAL METHOD Orsat
AMBIENT TEMPERATURE
OPERATOR

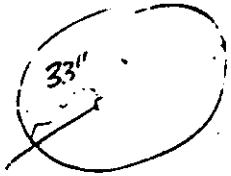
COMMENTS:

RUN / GAS	1		2		3		AVERAGE NET VOLUME	MULTIPLIER	MOLECULAR WEIGHT OF STACK GAS (DRY BASIS) M_d, lb/lb-mole
	ACTUAL READING	NET	ACTUAL READING	NET	ACTUAL READING	NET			
CO_2	21.0	21.0	20.8	20.8	20.8	20.8	20.86	44/100	9.1784
O_2 (NET IS ACTUAL O_2 READING MINUS ACTUAL CO_2 READING)	28.0	7	27.8	7	27.8	7	7	32/100	2.24
CO (NET IS ACTUAL CO READING MINUS ACTUAL O_2 READING)	28.0	0	27.6	.2	27.6	.2	.2	28/100	0.056
N_2 (NET IS 100 MINUS ACTUAL CO READING)	—	72	—	72.4	—	72.4	72.26	28/100	20.2328
								TOTAL	31.7072

EPA (Dur) 230
4/72

FIELD DATA

PLANT W.C. Ville Lime
DATE 8-8-74
SAMPLING LOCATION ESP - Outlet
SAMPLE TYPE EPA 8
RUN NUMBER 3-5 (S-3)
OPERATOR T. Stewart
AMBIENT TEMPERATURE 85
BAROMETRIC PRESSURE 29.61
STATIC PRESSURE, (P_s) -.36
FILTER NUMBER (s) 32-24 (outside)
34-16 (oven)



999,944

SCHEMATIC OF TRAVERSE POINT LAYOUT

PROBE LENGTH AND TYPE 3' Glass
NOZZLE I.D.
ASSUMED MOISTURE, % 10
SAMPLE BOX NUMBER MRC 2
METER BOX NUMBER 7
METER $\Delta H_@$ 1.767
C FACTOR
PROBE HEATER SETTING Off
HEATER BOX SETTING ~50%
REFERENCE Δp Flow - zero at ΔH = 1.0

READ AND RECORD ALL DATA EVERY 12 MINUTES LEAK RATE .005 CFM @ 15 "Hg

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
			921.658									probe off
	0	Started 1028	921.658	.30	1.0	1.0	648	91	91	6.0	246	70
	12		928.216	.29	1.0	1.0	648	91	91	5.0	253	72
	24		933.005	.30	1.0	1.0	647	92	92	5.0	261	64
	36		939.032	.35	1.0	1.0	648	95	92	5.0	278	66
	48		945.496	.35	1.0	1.0	649	97	92	5.0	284	62
	60		951.124	.40	1.0	1.0	648	99	94	5.0	293	70
	72		955.828	.45	1.0	1.0	650	102	95	5.0	301	77
	84		962.248	.48	1.0	1.0	652	103	96	5.0	307	79
	96	Stopped 1209 Started 1212	967.944	.34	1.0	1.0	657	98	98	5.0	214	63
	108		974.078	.36	1.0	1.0	659	98	97	5.0	259	61
	120		—	.38	1.0	1.0	658	96	95	5.1	251	58
	132		—	.38	1.0	1.0	662	97	97	5.1	279	62
	144		991.462	.40	1.0	1.0	663	101	99	5.1	283	65
	156		996.915	.45	1.0	1.0	663	103	98	5.0	299	73
			999.944									
	192											

Total 164 O.

* 12:03 stopped to change parts - per EPA 5 train - & to change Si Gel desiccant cartridge.
meter hung at 999.944 - 164 min into run - shut down + purged for 19.5 minutes at ΔH of 1.0 - meter malfunctioning

CLEAN-UP DATA

Plant WOODVILLE, LIME Comments:

Date 8-8-74

Sampling Location ESP OUTLET

Sample Type EPA-8

Run Number 3-S

Sample Box Number #1 MRC

Clean-up Man THALMAN, SCHLIESSER

IMPINGERS

	#1	#2 (2 & 3 H_2O_2)	#3	#4	#5 (other impinger)
Final Vol.	97 ml	320 ml	ml	ml	130 ml
Initial Vol.	100 ml	200 ml	ml	ml	100 ml
Net Vol.	-3 ml	120 ml	ml	ml	30 ml

Total Net Volume in Impingers 147 ml

SILICA GEL

Final Weight	345 g	g	g
Initial Weight	300 g	g	g
Net Weight	45 g	g	g

Total Net Weight in Silica Gel 45 g

Total Moisture 192 g

Filter Number(s) 32-24

34-16

MRC 7/73

NO_x samples PB 29.69

Run #N-1 8/6/79	initial Press "Hg	initial Temp °F	Flask #	Time
A	27.0	87°F	19	3:40PM
B	27.1	81°F	22	4:30PM
C	27.0	86°F	20	6:20PM
D	27.0	86	18	7:20PM
Run# N-2 8/7/79 A	27.0	80	9	1200
B	27.1	81	11	12:40
C	27.0	82	14	2:15
D	26.9	83	7	3:10

NCx Run N-3

sample	flask	Press	Temp	time
A	#18	27.2	83°F	1045
		23.5	85°F	11:30
B	#19			
C	#20	26.0	90°F	12:30
D	#22	25.9	98F	1:35

* #20 ~~[illegible]~~ stop cock come off - was put back on before being evacuated. (T.S)

APPENDIX F

PROCESS OPERATION FIELD DATA SHEETS

Company Woodville Lime & Chemical | Location Woodville, Ohio

Facility Lime Kiln | Unit No. No. 1

Operators C. E. Bley (data) + D. Mannis (data) | Date 8-6-74

Data Set No. | Page

		Units		Time (24 hour clock) 1500	1600	1700	1800	1900	2000	Sampling Time 1500 2000 Average
1	Stone Totalizer	N.A.		79839	836.86	87546	91425	95284	99046	1925
2	Stone Rate	Tons/hr.		38.52	38.55	38.74	38.65	38.64	37.57	38.43
3	Oil Totalizer	Gals.		90233	90493	90798	91105	91395	91705	
4	Oil Rate	Gals/hr.		318	260	305	307	290	310	294
				—	—	—	—	—		
	Gas Rate	Ft^3/hr		—	—	—	—	—		
	% Oil to % Gas	% Oil / % Gas								
5	Firing Zone Tempt	°F		2650	2675	2640	2650	2640	2640	2649
6	Middle Kiln Tempt	°F		1350	1350	1370	1360	1340	1340	1352
7	Feed End Kiln Tempt	°F		1025	1025	1050	1030	1000	1020	1025
8	ESP Inlet Tempt	°F		698	690	710	709	700	703	702
9	ESP Outlet Tempt	°F		650	660	665	670	670	665	663
10	ESP "A" Field									
	Primary Current	amps		70	74	71	75	77	76	74
	Primary Voltage	volts		255	250	250	250	250	250	251
	Precipitator Current	amps		0.42	.44	0.43	.46	.46	.46	.45
	ESP "B" Field									
	Primary Current	amps		68	64	69	78	78	78	72
	Primary Voltage	volts		240	235	235	240	235	240	238
	Precipitator Current	amps		0.40	.38	0.42	.48	.50	.46	.44
	Mode of Operation	Auto manual		auto	auto	auto	auto	auto	auto	

COMMENTS: 14:30 - Oil feed rate increased due to ESP inlet tempt dropping down to 685°F. Stack opacity ranged between 5 and 15 percent. Reason for tempt dropping was due to another kiln starting up which reduced the overall gas pressure.

Company: Woodville Lime & Chemical | Location: Woodville, Ohio

Facility: Lime Kiln | Unit No.: No. 1

Operators: C. E. Riley + P. L. Harris | Date: 8-7-74

Data Set No. | Page

		Units		Time (24 hour clock) 1100	1200	1300	1400	1500	1600	Sampling Period 1100 1600 Average
1	Stone Totalizer	N.A.		151963	160810	164669	168539	172906	176286	19323
2	Stone Rate	Tons/hr.		38.42	38.47	38.59	38.65	38.72	38.80	38.65
3	Oil Totalizer	Gals.		96452	96778	97118	97397	97703	98009	1557
4	Oil Rate	Gals/hr.		313	326	340	279	306	306	311
			—	—	—					
	Gas Rate	Ft^3/hr	—	—	—					
	% Oil to % Gas	% Oil / % Gas								
5	Firing Zone Tempt	°F		2650	2700	2670	2650	2650	2660	2662
6	Middle Kiln Tempt	°F		1370	1375	1410	1360	1360	1360	1373
7	Feed End Kiln Tempt	°F		1025	1015	1090	1025	1090	1040	1031
8	ESP Inlet Tempt	°F		710	710	720	710	710	705	711
9	ESP Outlet Tempt	°F		675	670	680	670	670	670	673
	ESP "A" Field									
10	Primary Current	amps		68	68	65	69	69	69	68
11	Primary Voltage	volts		255	258	260	257	255	257	257
12	Precipitator Current	amps		.39	.39	.37	.40	.39	.40	.39
	ESP "B" Field									
13	Primary Current	amps		70	72	66	71	71	71	70
14	Primary Voltage	volts		248	245	250	245	250	250	248
15	Precipitator Current	amps		.44	.45	.41	.44	.44	.44	.44
	Mode of Operation	Auto / manual		auto / auto	auto / auto	auto / auto	auto / auto	auto / auto	auto / auto	

COMMENTS:

Company Woodville Lime & Chemical | Location Woodville, Ohio

Facility Lime Kiln | Unit No. No. 1

Operators C. E. Riley & D. Harris

Date 8-8-74

Data Set No. | Page

	Units		Time (24 hour clock) 1100	1100	1200	1300	1400		Sampling Interval 1000 1400 Average
Stone Totalizer	N.A.		246044	250922	253789	257710	261622		15558
Stone Rate	Tons/hr.		38.52	38.58	38.67	39.21	39.12		38.90
Oil Totalizer	Gals.		103411	103736	104033	104397	104676		1265
Oil Rate	Gals/hr.		326	325	297	314	329		316
Gas Totalizer	Ft^3	—	—	—	—	—	—		
Gas Rate	Ft^3/hr	—	—	—	—	—	—		
% Oil to % Gas	% Oil / % Gas								
Firing Zone Tempt	°F		2600	2590	2600	2600	2610		2600
Middle Kiln Tempt	°F		1350	1370	1360	1360	1360		1360
Feed End Kiln Tempt	°F		1005	1000	1030	1090	1040		1023
ESP Inlet Tempt	°F		702	702	720	720	718		712
ESP Outlet Tempt	°F		660	660	670	680	675		669
ESP "A" Field									
Primary Current	amps		70	70	70	68	66		69
Primary Voltage	volts		255	255	255	257	259		256
Precipitator Current	amps		.40	.41	.40	.40	.38		.40
ESP "B" Field									
Primary Current	amps		72	71	71	69	69		70
Primary Voltage	volts		245	243	245	245	249		245
Precipitator Current	amps		.45	.45	.44	.43	.43		.44
Mode of Operation	Auto / manual		Auto / Auto	Auto / Auto	Auto / Auto	Auto / Auto	Auto / Auto		

COMMENTS.

APPENDIX G

SAMPLE IDENTIFICATION LOG

Particulate
Woodrill

SAMPLE IDENTIFICATION LOG

EPA Number	Sample Location	Run	Contents	Comments
574-001-777	ESP outlet	P1 R	Filter 32-5	8-6-74
-778	" "	P1 R	Front half acetone wash	"
-779	" "	P1 R	Back half acetone wash	"
-780	" "	P1 R	Impg H_2O + wash	"
-781	" "	P1 R	Impg E/C extraction	"
574-001-846	ESP outlet	P2	Filter 32-1	8-7-74
-847	" "	P2	Front half acetone wash	"
-848	" "	P2	Back half acetone wash	"
-849	" "	P2	Impg H_2O + wash	"
-850	" "	P2	Impg E/C extraction	"
574-001-851	ESP outlet	P3	Filter 32-25	8-8-74
-852	" "	P3	Front half acetone wash	"
-853	" "	P3	Back half acetone wash	"
-854	" "	P3	Impg H_2O + wash	"
-855	" "	P3	Impg H_2O E/C extraction	"
574-001-815	Acetone blank		Acetone blank for P1 run conducted on 8-6-74	
574-001-856	Acetone blank		Acetone blank for P2 run on 8-7-74	
-857	Acetone blank		Acetone blank for P3 run on 8-8-74	
-858	Dist H_2O blank		Dist. H_2O blank for P1, P2, + P3	
-859	Dist H_2O blank		E/C extraction blank for P1, P2, + P3	
-860	Filters for		trace metal blanks (need 6-3" size not prewighed).	

NOx
Woodville

SAMPLE IDENTIFICATION LOG

EPA Number	Sample Location	Run	Contents	Flask No.	Comments
S74-001-801	ESP outlet	N1	NOx sample + H_2O wash	8A - 1540	8-6-74
-802	" "	N1	" " " " "	22B - 1630	"
-803	" "	N1	" " " " "	20C - 1820	"
-804	" "	N1	" " " " "	18D - 1920	"
S74-001-805	ESP outlet	N2	NOx sample + H_2O wash	9 - 1200	8-7-74
-806	" "	N2	" " " " "	11 - 1240	"
-807	" "	N2	" " " " "	14 - 1415	"
-808	" "	N2	" " " " "	7 - 1510	"
S74-001-809	ESP outlet	N3	NOx sample + H_2O wash	18 - 1045	8-8-74
-810	" "	N3	" " " " "	19 - 1130	8-8-74
-811	" "	N3	" " " " "	20 - 1230	8-8-74
-812	" "	N3	" " " " "	22 - 1335	8-8-74
S74-001-813	Blank NOx	—	NO_2 collection reagent		
S74-001-814	Blank Dist H_2O	—	NO_2 wash water Blank		

Samples 801 through 804 cleanup performed at Woodville plant due to limited No. of collection flask CER

SO_2
Woodhill

SAMPLE IDENTIFICATION LOG

EPA Number	Sample Location	Run	Contents	Comments
S74-001-816	ESP outlet	S1	SO_2 impg contents + wash	(method 6) 8-6-74
S74-001-817	ESP outlet	S2	SO_2 impg contents + wash	(method 6) 8-7-74
S74-001-818	ESP outlet	S3	SO_2 impg contents + wash	(method 6) 8-8-74
S74-001-819	Woodhill Lim	Blank	SO_2 collection reagent	(3% H_2O_2) Blank
-820	" "	Blank	Dist. used H_2O Blank	
-821	" "	Blank	80% Isopropyl reagent Blank	

Impg contents - 3% H_2O_2 plus collected H_2O /

Raw materials

1 of

SAMPLE IDENTIFICATION LOG

EPA Number	Sample Location	Run	Contents	Comments
S-74-001-786	Feed-in silo	P1	Rock feed-in to kiln	8-6-74 – 1615
787	Feed-in silo	P1	" " " " "	8-6-74 – 1930
788	Composite	P1	Rock feed-in composite	of -786 & 787
S-74-001-789	No.6 fuel oil line	P1	No.6 fuel oil for kiln	8-6-74 – 1615
-790	" " " "	P1	" " " " " "	8-6-74 – 1930
-791	Composite	P1	No.6 fuel oil composite	of 789 & 790
S-74-001-792	Kiln discharge	P1	Kiln product (lime)	8-6-74 1600
-793	" "	P1	" " "	8-6-74 1945
-794	Composite	P1	Kiln product composite	of -792 & 793
S-74-001-795	ESP silo	P1	ESP collector dust	8-6-74 1600
-796	" "	P1	" " "	8-6-74 1950
-797	Composite	P1	ESP collector dust composite	of -795 & 796
S74-001-834	Feed-in silo	P2	Rock feed-in to kiln	8-7-74 1100
-835	" " "	P2	" " " " "	8-7-74 1530
-836	Composite	P2	Rock feed-in composite	of -834 & 835
S74-001-837	No.6 Fuel Oil line	P2	No.6 fuel oil for kiln	8-7-74 1100
-838	" " " "	P2	" " " " "	8-7-74 1530
-839	Composite	P2	No.6 fuel oil for composite	of 837 & 838
S-74-001-840	Kiln discharge	P2	Kiln product (lime)	8-7-74 1100
-841	" "	P2	" " "	8-7-74 1530
-842	Composite	P2	Kiln product composite	of 840 & 841
S-74-001-843	ESP silo	P2	ESP collector dust	1230 8-7-74
-844	" "	P2	" " "	1600 8-7-74
-845	Composite	P2	ESP collector dust composite	of -843 & 844
S74-001-871	Feed-in silo	P3	Rock feed-in to kiln	10:30 8-8-74
-872	" " "	P3	" " " " "	14:00 8-8-74
-873	Composite	P3	Rock feed-in composite	of 871 & 872

Raw materials

2 of 2

SAMPLE IDENTIFICATION LOG

EPA Number	Sample Location	Run	Contents	Comments
S74-001-874	No.6 fuel oil line	~~P3~~ P3	No.6 fuel oil for kiln	8-8-74 10:30
-875	" " " " "	P3	" " " " "	8-8-74 14:00
-876	Composite	P3	No.6 fuel oil composite of 874 & 875	
S74-001-877	Kiln discharge	P3	Kiln product (lime)	8-8-74 1100
-878	" "	P3	" " "	8-8-74 14:00
-879	Composite	P3	Kiln product composite of 877 & 878	
S74-001-880	ESP side	P3	ESP collector dust	8-8-74 1200
-881	" "	P3	" " "	8-8-74 1430
-882	Composite	P3	ESP collector dust composite of 880 + 881	

EPA SO_2 Research

SAMPLE IDENTIFICATION LOG

EPA Number	Sample Location	Run	Contents	Comments
S74-001-822	ESP outlet	S1	Filter from between IPA + H_2O_2 in	
	Method	6 —	SO_2 sampling train.	(32-4)
S74-001-823	ESP outlet	S1	Front half contents + IPA wash of	
			SO_2 method 6 train	
S74-001-824	ESP outlet	S2	Filter in front of IPA (in oven chamber	
			but oven + probe were not heated)	34-17
S74-001-825	ESP outlet	S2	Probe wash (80% Isopropanol used)	
S74-001-826	ESP outlet	S2	1st IPA imp contents + 80% IPA wash	
S74-001-827	ESP outlet	S2	Filter between IPA + H_2O_2 impingers	32-15
S74-001-828	ESP outlet	S3	Filter in front of IPA (in heated oven chamber	
			probe was heated)	34-16
S74-001-829	ESP outlet	S3	Probe wash (80% Isopropanol used)	
S74-001-830	ESP outlet	S3	1st IPA imp contents + 80% IPA wash.	
S74-001-831	ESP outlet	S3	Filter between IPA + H_2O_2 impingers	32-24
~~S74-001-832~~	~~Woodchip inc.~~	~~Blank~~	~~Dist. H_2O wash blank~~	~~Voided (washed probe with~~ no H_2O
S74-001-833	IPA blank	SO_2	Isopropanol blank	80%

Note — These samples are to be sent to EPA for research analysis.

APPENDIX H

SAMPLE HANDLING LOG

SAMPLE HANDLING LOG

Task or Contract Number 68-02-1404 Task 2

Source Identification 75-LIM-8

Test Run Number P-1

Pollutant(s) in Sample Particulate

Task	Date	Time	Personnel	Remarks
Sampling	8/6/74			
Probe Technician	"		Steve Schlosser	
Meter Technician	"		Tim Thalman	
Other	"		Darell Harris	
Transport of Train to Cleanup Site	"	1930	Crew in wagon	
Train Disassembly	"	1945	Thalman + Harris	
Train Cleanout	"	"	"	
Packing and Storage	"	"	"	
Shipment to Analysis Site	8/9/74	1300–1600	Stewart + Duncan	
Lab Analysis	8/12-23/74	—	Kulick + McDonald	
Write Up				

SAMPLE HANDLING LOG

Task or Contract Number 68-02-1404 task 2

Source Identification 75-LIM-8

Test Run Number P-2

Pollutant(s) in Sample particulate

Task	Date	Time	Personnel	Remarks
Sampling	8/7/74			
Probe Technician			Duncan	
Meter Technician			Stewart	
Other			C Harris	
Transport of Train to Cleanup Site		1600 lunch	crew	
Train Disassembly	8/7/74	1645	Harris Duncan + Stewart	
Train Cleanout		"	"	
Packing and Storage				
Shipment to Analysis Site	8/9/74	1300 - 1600	Stewart + Duncan	
Lab Analysis	8/12-23/74	—	Kulick + McDonald	
Write Up				

SAMPLE HANDLING LOG

Task or Contract Number 68-02-1404 Task #2

Source Identification 75-LIM-8

Test Run Number P-3

Pollutant(s) in Sample Particulate

Task	Date	Time	Personnel	Remarks
Sampling	8/8/79			
Probe Technician	"	1030-1400	Duncan	
Meter Technician	"	" "	Schlissier	
Other	"			
Transport of Train to Cleanup Site	"	1430	Crew	
Train Disassembly	"	1430	Duncan, Stewart, Hani	
Train Cleanout	"	"	" " "	
Packing and Storage	"	"	" " "	
Shipment to Analysis Site	8/9/79	1300-1600	Duncan & Stewart	
Lab Analysis	8/12-23/79	—	Kulick & McDonald	
Write Up				

APPENDIX I

ANALYTICAL DATA SHEETS

ANALYTICAL DATA

PLANT Woodville Chemical Co. COMMENTS:

DATE 8/6/74

SAMPLING LOCATION Lime Kiln # 1 ESP outlet

SAMPLE TYPE EPA-5

RUN NUMBER P-1

SAMPLE BOX NUMBER MCC-1

CLEAN-UP MEN Hofman

ANALYST L. Kulch

FRONT HALF

LABORATORY RESULTS

ACETONE WASH OF NOZZLE, PROBE, CYCLONE (BYPASS), FLASK, FRONT HALF OF FILTER HOLDER — CONTAINER 574-001-778 286.5 mg

FILTER NUMBER 32-5 0.5642 g Final CONTAINER 574-001-777 136.4 mg
0.4278 g Tare wt.

FRONT HALF SUBTOTAL 422.9 mg

BACK HALF

IMPINGER CONTENTS AND WATER WASH OF IMPINGERS, CONNECTORS, AND BACK HALF OF FILTER HOLDER — CONTAINER 574-001-780 46.4 mg

ETHER-CHLOROFORM EXTRACTION 574-001-781 127.8 mg

ACETONE WASH OF IMPINGERS, CONNECTORS, AND BACK HALF OF FILTER HOLDER — CONTAINER 574-001-779 70.7 mg

BACK HALF SUBTOTAL 244.9 mg

TOTAL WEIGHT 667.8 mg

MOISTURE

IMPINGERS

FINAL VOLUME	400	ml
INITIAL VOLUME	200	ml
NET VOLUME	200	ml

SILICA GEL

FINAL WEIGHT	322.5 g	315 g	g
INITIAL WEIGHT	300 g	300 g	g
NET WEIGHT	22.5 g	15 g	g

TOTAL MOISTURE 237.5 g

EPA (Dur) 231
4/72

ANALYTICAL DATA

PLANT Woodville Lime + Chemical Co. COMMENTS:
DATE 8/7/74
SAMPLING LOCATION ESP Outlet
SAMPLE TYPE EPA-5
RUN NUMBER P-2
SAMPLE BOX NUMBER MRC 1
CLEAN-UP MEN Stewart, Duncan, Harris
ANALYST F. Kulik

FRONT HALF

		LABORATORY RESULTS
ACETONE WASH OF NOZZLE, PROBE, CYCLONE (BYPASS), FLASK, FRONT HALF OF FILTER HOLDER	CONTAINER S74-001-847	53.7 mg
FILTER NUMBER 32-1 0.4910 g Tared 0.4256 g Tared	CONTAINER S74-001-846	65.4 mg
	FRONT HALF SUBTOTAL	119.1 mg

BACK HALF

IMPINGER CONTENTS AND WATER WASH OF IMPINGERS, CONNECTORS, AND BACK HALF OF FILTER HOLDER	CONTAINER S74-001-849	35.2 mg
	ETHER-CHLOROFORM EXTRACTION S74-001-850	51.2 mg
ACETONE WASH OF IMPINGERS, CONNECTORS, AND BACK HALF OF FILTER HOLDER	CONTAINER S74-001-848	27.8 mg
	BACK HALF SUBTOTAL	134.2 mg
	TOTAL WEIGHT	253.3 mg

MOISTURE

IMPINGERS

FINAL VOLUME	354 ml	140	4
INITIAL VOLUME	100 ml	100	0
NET VOLUME	254 ml	40	4

SILICA GEL

FINAL WEIGHT	337.8 g	g	g
INITIAL WEIGHT	300 g	g	g
NET WEIGHT	37.8 g	g	g

TOTAL MOISTURE 335.8 g

EPA (Dur) 231
4/72

ANALYTICAL DATA

PLANT Woodville Fine Chemicals COMMENTS:

DATE 8/8/74

SAMPLING LOCATION ESP Outlet

SAMPLE TYPE EPA 5

RUN NUMBER P-3

SAMPLE BOX NUMBER MRC1

CLEAN-UP MEN Duncan, Harris, Stewart

ANALYST [illegible]

FRONT HALF

LABORATORY RESULTS

ACETONE WASH OF NOZZLE, PROBE, CYCLONE (BYPASS), FLASK, FRONT HALF OF FILTER HOLDER — CONTAINER S74-001-852 — 47.5 mg

FILTER NUMBER 32-25 — 0.5004 g Initial — CONTAINER S74-001-851 — 69.8 mg

0.4306 g Tare wt.

FRONT HALF SUBTOTAL 117.3 mg

BACK HALF

IMPINGER CONTENTS AND WATER WASH OF IMPINGERS, CONNECTORS, AND BACK HALF OF FILTER HOLDER — CONTAINER S74-001-854 — 33.2 mg

ETHER-CHLOROFORM EXTRACTION S74-001-855 — 11.8 mg

ACETONE WASH OF IMPINGERS, CONNECTORS, AND BACK HALF OF FILTER HOLDER — CONTAINER S74-001-853 — 11.2 mg

BACK HALF SUBTOTAL 56.2 mg

TOTAL WEIGHT 173.5 mg

MOISTURE

IMPINGERS

FINAL VOLUME	324 ml	142	6
INITIAL VOLUME	100 ml	100	0
NET VOLUME	224 ml	42	6

SILICA GEL

FINAL WEIGHT	331.8 g	g	g
INITIAL WEIGHT	300 g	g	g
NET WEIGHT	31.8 g	g	g

TOTAL MOISTURE 303.8 g

EPA (Dur) 231
4/72

Tare Weights for Method 5 Samples

Run	Number	Description	Vol (ml)	Final wt (gm)	Tare wt (gm)	Particulate (mg)	Blank Correction (mg)	Corrected Particulate (mg)
P-1	S74-001-778	Front 1/2 Acetone	675	78.2479	77.9573	290.6	4.1	286.5
P-1	S74-001-779	Back 1/2 Acetone	530	94.1359	94.0620	73.9	3.2	70.7
P-1	S74-001-780	Impinger H_2O Wash	735	89.3082	89.2581	50.1	3.7	46.4
P-1	S74-001-781	Impinger E/C Extract	735	89.5569	89.4247	132.2	4.4	127.8
P-2	S74-001-847	Front 1/2 Acetone	625	89.1338	89.0747	59.1	5.4	53.7
P-2	S74-001-848	Back 1/2 Acetone	530	87.8668	87.8344	32.4	4.6	27.8
P-2	S74-001-849	Impinger H_2O Wash	1000	87.5273	87.4671	60.2	5.0	55.2
P-2	S74-001-850	Impinger E/C Extract	1000	84.9433	84.8861	57.2	6.0	51.2
P-3	S74-001-852	Front 1/2 Acetone	905	84.5070	84.4561	50.9	3.4	47.5
P-3	S74-001-853	Back 1/2 Acetone	485	92.2971	92.2841	13.0	1.8	11.2
P-3	S74-001-854	Impinger H_2O Wash	745	80.0283	79.9914	36.9	3.7	33.2
P-3	S74-001-855	Impinger E/C Extract	745	87.6146	87.5983	16.3	4.5	11.8
P-1	S74-001-815	Acetone Blank	245	90.7818	90.7803	1.5	.006/ml	
P-2	S74-001-856	Acetone Blank	360	88.7512	88.7481	3.1	.0086/ml	
P-3	S74-001-857	Acetone Blank	425	92.6786	92.6770	1.6	.0038/ml	
P-1,P-2, P-3	S74-001-858	H_2O Blank	350	89.7784	89.7768	1.6	.005/ml	
P-1,P-2, P-3	S74-001-859	H_2O E/C Blank	350	89.3314	89.3293	2.1	.006/ml	
	Filter Weights							
P-1	S74-001-777			0.5642	0.4278	136.4		136.4
P-2	S74-001-846			0.4910	0.4256	65.4		65.4
P-3	S74-001-851			0.5004	0.4306	59.8		69.8

2

SULFUR CONTENT ANALYSIS

HEAT OF COMBUSTION DATA SHEET

Sample No. 574-001-791 #6 Fuel Oil 8/14/74 Date 10/25/74 Observer LAK

Sample + Capsule 10.1320

Tare Wt. Capsule 9.6178

Net Wt. Sample .5242

Average Room Temp. 23 °C

OBSERVATIONS

Time Min.	Time Sec.	Temperature °C
5		20.794
10		20.815
11	00	21.55
11	15	21.90
11	30	22.15
11	45	22.34
16		23.014
17		23.021
18		23.031
19		23.032
20		23.032
21		23.031
22		23.030
23		23.030
24		23.030
25		
26		

Corrected Temperature Rise:

Final Temp. 23.032

Scale Corr. .002

True Final = 23.034

R_2(c-b) = .003

Corrected Final = 23.037

Initial Temp. 20.815

Scale Corr. .005

True Initial = 20.820

r_1(b-a) = .006

Corrected Initial = 20.826

Net Temp. Rise = 2.211

1.8 x 2418.6 (Water Equiv.) x 2.211 (Net Temp. Rise) = 9621.6 Total BTU

Interpolation to Find "Six-Tenths Point:"

0.6R = .6(23.032 (Final Temp.) - 20.815 (Initial Temp.)) = 1.330 °C

Temp. at .6R = 20.815 (Init. Temp.) + 1.330 (0.6R) = 22.145 °C

Time (b) at .6R = 11:15 (Prior Time) + :15 (Time Gain) = 11:30 Min. + Sec.

(b-a) = 11:30 (Time (b)) - 10:00 (Time Fired) = 1:30 (Min. + Sec.) = 1.5 (+ 100 Ths.) Min.

(c-b) = 19:00 (Final Time) - 11:30 (Time (b)) = 7:30 (Min. + Sec.) = 7.5 (+ 100 Ths.) Min.

Radiation Rates:

Initial Rate (r_1) = .021 (Prelim. Rise) ÷ 5 (Minutes) = .004 (per Min.) °C

Final Rate (r_2) = .002 (Final Fall) ÷ 5 (Minutes) = .0004 (per Min.) °C

Corrections for Total BTU:

Fuse Wire = 4.14 x ~~4.85~~ ~~5.15~~ 5.15 ~~4.46 cm~~ = 21.3 BTU

Sulfur = 23 x 0.5242 (Wt. Sample) x 1.52% (% S) = 18.3 BTU

Acid Titration = 9.45 ml x 0.0999 N x 24.8 = 22.9 BTU

Total Correction = 62.5 BTU

Net Heat Liberated = ~~9547.8~~ 9557.1 BTU

Heat of Combustion = ~~9547.8~~ 9557.1 (Net Heat Liberated) ÷ 0.5242 (Wt. Sample) = ~~18232.2~~ 18235.6 BTU/lb

(4)

HEAT OF COMBUSTION DATA SHEET

Sample No. 574-01-835 #6 Fuel oil 8th/74 Date 10/25/74 Observer F.A.K.

Sample + Capsule 10.2041

Tare Wt. Capsule 9.6183

Net Wt. Sample 0.5858

Average Room Temp. 23 °C

OBSERVATIONS

Time Min.	Time Sec.	Temperature °C
5		20.660
10		20.690
11	00	21.56
11	15	21.96
11	30	22.24
11	45	22.44
16		23.178
17		23.186
18		23.190
19		23.193
20		23.195
21		23.195
22		23.194
23		23.194
24		23.190
25		23.188
26		

Corrected Temperature Rise:

Final Temp. 23.195

Scale Corr. .002

True Final = 23.197

r_2(c-b) = .009 subtract

Corrected Final = 23.188

Initial Temp. 20.690

Scale Corr. .007

True Initial = 20.697

r_1(b-a) = .009

Corrected Initial = 20.706

Net Temp. Rise = 2.482

1.8 x 2417.6 (Water Equiv.) x 2.482 (Net Temp. Rise) = 10800.9 Total BTU

Interpolation to Find "Six-Tenths Point:"

0.6R = .6(23.195 (Final Temp.) - 20.690 (Initial Temp.)) = 1.503 °C

Temp. at .6R = 20.690 (Init. Temp.) + 1.503 (0.6R) = 22.193 °C

Time (b) at .6R = 11:15 (Prior Time) + :12 (Time Gain) = 11:27 Min. + Sec.

(b-a) = 11:27 (Time (b)) - 10:00 (Time Fired) = 1:07 (Min. + Sec.) = 1.45 Min. + 100 Ths.

(c-b) = 20:00 (Final Time) - 11:27 (Time (b)) = 7:33 (Min. + Sec.) = 8.55 Min. + 100 Ths.

Radiation Rates:

Initial Rate (r_1) = .030 (Prelim. Rise) ÷ 5 (Minutes) = .006 °C per Min.

Final Rate (r_2) = .005 (Final Fall) ÷ 5 (Minutes) = .0011 °C per Min.

Corrections for Total BTU:

Fuse Wire = 4.14 x 3.3 ~~9.7745~~ = 13.7 BTU

Sulfur = 23 x 0.5858 (Wt. Sample) x 1.36 (% S) = 18.3 BTU

Acid Titration = 9.77 ml x .0979 N x 24.8 = 23.7 BTU

Total Correction = 55.7 BTU

Net Heat Liberated = 10745.2 BTU

Heat of Combustion = 10745.2 (Net Heat Liberated) ÷ 0.5858 (Wt. Sample) = 18342.8 BTU/lb

HEAT OF COMBUSTION DATA SHEET

Sample No. 574-001-876 #6 Fuel Oil 8/1/74 Date 10/5/74 Observer LAK

Sample + Capsule 10.2202

Tare Wt. Capsule 9.6172

Net Wt. Sample 0.6030

Average Room Temp. 23.5 °C

OBSERVATIONS

Time Min.	Time Sec.	Temperature °C
5		21.000
10		21.030
11	00	22.260
11	15	23.54
11	30	22.64
11	45	22.88
16		23.580
17		23.597
18		23.599
19		23.600
20		23.602 ←
21		23.601
22		23.601
23		23.600
24		23.600
25		23.600
26		

Corrected Temperature Rise:

Final Temp. 23.602

Scale Corr. .003

True Final = 23.605

R_2(c-b) = .003

Corrected Final = 23.608

Initial Temp. 21.030

Scale Corr. .004

True Initial = 21.034

r_1(b-a) = .008

Corrected Initial = 21.042

Net Temp. Rise = 2.566

1.8 x 2417.6 (Water Equiv.) x 2.566 (Net Temp. Rise) = 11166.4 Total BTU

Interpolation to Find "Six-Tenths Point:"

0.6R = .6(23.602 (Final Temp.) - 21.030 (Initial Temp.)) = 1.543 °C

Temp. at .6R = 21.030 (Init. Temp.) + 1.543 (0.6R) = 22.573 °C

Time (b) at .6R = 11:15 (Prior Time) + .05 (Time Gain) = 11:20 Min. + Sec.

(b-a) = 11:20 (Time (b)) - 10:00 (Time Fired) = 1:20 (Min. + Sec.) = 1.33 Min. + 100 Ths.

(c-b) = 20:00 (Final Time) - 11:20 (Time (b)) = 8:40 (Min. + Sec.) = 8.67 Min. + 100 Ths.

Radiation Rates:

Initial Rate (r_1) = 0.030 (Prelim. Rise) ÷ 5 (Minutes) = 0.006 °C per Min.

Final Rate (r_2) = .002 (Final Fall) ÷ 5 (Minutes) = .0004 °C per Min.

Corrections for Total BTU:

Fuse Wire = 4.14 x 6.3 = 26.1 BTU

Sulfur = 23 x 0.6030 (Wt. Sample) x 1.37 (% S) = 19.0 BTU

Acid Titration = 9.70 ml x .0979 N x 24.8 = 23.6 BTU

Total Correction = 68.7 BTU

Net Heat Liberated = 11097.7 BTU

Heat of Combustion = 11097.7 (Net Heat Liberated) ÷ .6030 (Wt. Sample) = 18404.1 BTU/lb

APPENDIX J

SAMPLING AND ANALYTICAL PROCEDURES

Determination of Particulate Emissions

The following method was used in this test program. Sampling procedures followed those described in the Federal Register.[1]

Sampling Apparatus

The particulate sampling train used in these tests met design specifications established by the Federal EPA and was assembled by MRC personnel. It consisted of:

Nozzle - stainless steel (316) with sharp, tapered leading edge and accurately measured round opening

Probe - Pyrex glass with a heating system capable of maintaining a minimum gas temperature of 250°F at the exit end during sampling

Pitot Tube - calibrated type S attached to probe to monitor stack gas velocity

Filter Holder - Pyrex glass with heating system capable of maintaining minimum temperature of 225°F

Draft Gage - an inclined manometer made by Dwyer with a readability of 0.01 inches H_2O in the 0-1 inch range was used

Impingers - four impingers connected in series with glass ball joints. The first, third, and fourth impingers were of the Greenburg-Smith design, modified by replacing the tip with a 1/2 inch I.D. glass tube extending to 1/2 inch from the bottom of the flask. The second impinger was of the Greenburg-Smith design with a standard tip.

Thermometer - Type K (chromel/alumel) thermocouples with a Digitec Model 590 digital thermometer (range of -310° to 2500°F)

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[1]Federal Register, Vol. 36, No. 247, Part II, December 23, 1971.

Metering System - vacuum gauge, leak-free pump, thermocouples capable of measuring temperature to within 5°F, dry gas meter with 2% accuracy, and related equipment to maintain an isokinetic sampling rate and to determine sample volume. The dry gas meter is by Rockwell and the fiber vane pump is by Gast.

Barometer - Bourden tube type to measure atmospheric pressures to ±0.1 inches Hg

Sampling Procedure

After selecting the sampling site and the minimum number of traverse points, the stack pressure, temperature, moisture, and range of velocity head was measured according to procedures described in Method 1 of the Federal Register (December 23, 1971).

Approximately 200 grams of silica gel were weighed in a sealed impinger prior to each test. Glass fiber filters* (3" diameter) were desiccated for at least 24 hours and weighed to the nearest 0.1 milligram on an analytical balance. One hundred ml of distilled water was placed in each of the first two impingers, the third impinger was initially empty, and the impinger containing the silica gel was placed next in series. The train was set up without the probe as shown in Figure J-1. The sampling train was leak checked at the sampling site by plugging the inlet to the filter holder and pulling at least a 15-inch Hg vacuum. Leakage rates of less than 0.02 cfm at a vacuum of 15 in. Hg were recorded in all cases. The probe assembly was then attached, and crushed ice placed around the impingers. More ice was added during the run to keep the temperature of the gases leaving the last impinger at approximately 70°F.

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* MSA 1106 BH or equivalent

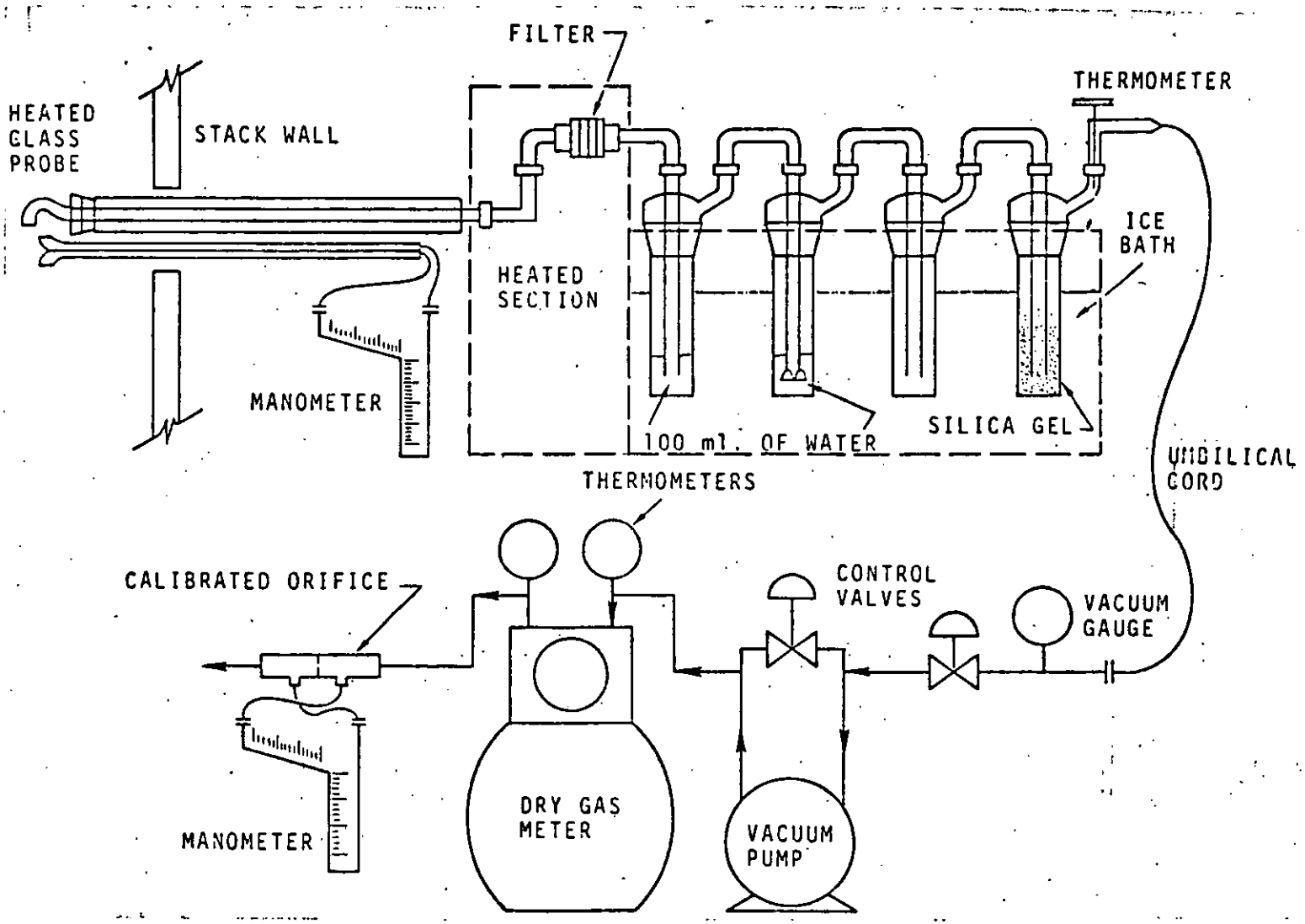

Figure J-1. Particulate sample train

During sampling, stack gas and sampling train data were recorded at each sampling point and when significant changes in stack flow conditions occurred. Isokinetic sampling rates were set throughout the sampling period with the aid of a nomograph.

Sample Recovery Procedure

The sampling train was moved carefully from the test site to the clean up area. Samples of the acetone and water used in the sample recovery were taken for use as blanks. The sample fractions were recovered as follows:

Container No. 1 - the filter was removed from its holder and placed in a petri dish and sealed.

Container No. 2 - Loose particulate and acetone washings from all sample-exposed surfaces prior to the filter were placed in a glass jar and sealed. Particulate was removed from the probe with the aid of a brush and acetone rinsing.

Container No. 3 - The condensate from the first three Greenburg-Smith impingers was measured within ±1 ml and placed into a glass jar. Water rinsings from the back half of the filter holder, all connectors, and the first three Greenburg-Smith impingers were placed in this container and the container sealed.

Container No. 4 - Acetone rinsings from the back half of the filter holder, support, all connectors, and the first three Greenburg-Smith impingers were placed in this container and sealed.

Container No. 5 - A minimum of 300 ml of acetone was taken for the blank analysis.

Container No. 6 - A minimum of 300 ml of distilled water was taken for a blank.

The silica gel from the fourth impinger was weighed and recorded. The used silica gel was discarded.

Detailed procedures follow.

Method 5 - MRC Clean Up Procedure

1. Probe Clean Up

Remove any material from the outside of the probe and nozzle that may contaminate the sample by falling or being washed into the sample bottle. Remove the nozzle and rinse the inside with acetone into the front 1/2 acetone sample bottle. Brush the inside with a clean test tube brush while flushing with acetone into the sample bottle. Wash the brush with acetone into the bottle. Rinse the inside of the nozzle a final time with acetone.

Elevate one end (the nozzle end) of the probe and rinse through with acetone into the front 1/2 sample bottle while slowly rotating through 720°. Run a clean probe brush through the probe while flushing with acetone and slowly rotating the brush. When the brush is exposed on the down stream end of the probe, rinse it into the sample bottle with acetone. Do not pull the brush back through the probe. Repeat the brushing step. Rinse the probe a final time by flushing with acetone while rotating it 360°. Wash the funnel into the sample bottle. Observe the probe inside and repeat the clean up procedure if sample particles are obvious.

2. Cyclone and Filter Clean Up

Remove the cyclone and connecting glassware. Rinse the cyclone with acetone into the collection flask and pour into the front 1/2 sample bottle. Repeat until all loose material is collected. If sample material adheres to the

inside surfaces, a small test tube brush can be used to free it, and it should be rinsed into the sample bottle. If a brush or spatula is used to clean the cyclone and catch bottle, it must be cleaned into the sample bottle. All connecting glassware between the cyclone system and filter must be treated in the same manner, i.e., flushing and/or brushing the sample exposed surfaces into the sample container with acetone until no sample material is visible.

The filter assembly must be disassembled in a dust-free area. The filter membrane should be loosened from the silicon rubber gasket by running a knife blade or spatula between them being careful not to scrape or loosen particles of glass from the glass frit. Lift the filter, using a spatula, and place into its storage container. Carefully scrape all particles of filter material from both mating surfaces (i.e., the silicon rubber gasket and the front-half glass holder) and place them in the storage container. Rinse and brush the front-half glass holder's sample exposed surfaces into the sample bottle with acetone. Do not wash or rinse the frit. Wash brushes, spatulas and funnels into the sample and store.

3. Back 1/2 Clean Up

Rinse the back half filter holder, connecting glass to the first impinger, and impinger "U" bends with distilled water into the back half water wash sample bottle by filling or flushing twice. Remove each of the first three impingers and pour the content into a graduated cylinder for measurement. Transfer these contents into the sample bottle. Wash each of the three impingers two times by squirting about 100 ml of distilled water into each, covering the

inlet and outlet openings with clean caps or clean fingers, and shaking to cause the wash water to contact all of the inside surfaces. Pour this wash water into the sample bottle and rinse the graduated cylinder and funnel into sample bottle with distilled water. Seal the bottle and store.

Rinse the back half filter holder, the filter to impinger connectors, and the impinger "U" bends with acetone into the back half acetone wash sample bottle by filling or flushing two times. Wash each of the three impingers two times by adding about 100 ml of acetone and shaking as with the water wash. Transfer these washings to the sample bottle. Rinse the graduated cylinder used to measure the water and the funnel with acetone into the sample bottle. Cap the bottle and store.

Analytical Procedures

The following procedures were used and follow the methods described in the Federal Register[1]:

Container No. 1 - the filter and any loose particulate matter from this sample container were placed into a tared glass weighing dish, desiccated to a constant weight, and weighed to the nearest 0.1 mg.

Container No. 2 - the acetone washings were transferred to a tared beaker and evaporated to dryness at ambient temperature and pressure, desiccated to a constant weight, and weighed to the nearest 0.1 mg.

Container No. 3 - organic matter from the impinger solution was extracted with three 25 ml portions each of ethyl ether and chloroform. The extracts were combined into a tared beaker, and evaporated until no solvent remained at about 70°F. The sample was then desiccated to a constant weight and weighed to the nearest 0.1 mg. The remaining water was evaporated by boiling in a tared beaker, and the residue weighed.

Container No. 4 - the acetone washings from the back half of the filter holder, connectors, and first three Greenburg-Smith impingers were transferred to a tared beaker, evaporated to dryness at ambient temperature and pressure, desiccated to a constant weight, and weighed to the nearest 0.1 mg.

Container No. 5 - the acetone blank was transferred to a tared beaker, and evaporated to dryness at ambient temperature and pressure. The blank was then desiccated to a constant weight and weighed to the nearest 0.1 mg.

Container No. 6 - the water blank was evaporated by boiling in a tared beaker, desiccated to a constant weight, and weighed to the nearest 0.1 mg. Blank corrections were made in proportion to the amount of reagent used.

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[1]Federal Register, Vol. 36, No. 159, Part II, August 17, 1971.

Determination of Nitrogen Oxides in Stack Gas: Phenoldisulfonic Acid Method

Introduction

The following method was used to determine total oxides of nitrogen. Samples were collected in evacuated flasks containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxides, were measured spectrophotometrically at 410 nm.

Reagents

The following reagents were used in this sampling program (all chemicals were ACS analytical-reagent grade):

Water - distilled, deionized water

Hydrogen peroxide (3%) - 10 ml of 30% H_2O_2 was diluted to 100 ml in a 100 ml volumetric flask with water.

Absorbing reagent - 2.8 ml of concentrated H_2SO_4 was diluted to one liter with distilled water. After mixing well, 6 ml of 3% hydrogen peroxide was added. The solution is prepared fresh every two or three days.

Sodium hydroxide (1 N) - 40 gm of NaOH were dissolved in distilled water and diluted to one liter.

Ammonium hydroxide (concentrated)

Sulfuric acid (fuming) - 15-18% SO_3

Phenoldisulfonic acid solution - 25 grams of pure white phenol were dissolved in 150 ml of concentrated H_2SO_4 on a steam bath. After the solution cooled, 75 ml fuming sulfuric acid were added. It was then heated to 100°C for two hours. The reagent was stored in a dark stoppered reagent bottle.

Potassium nitrate solution (standard) - 0.5495 grams of KNO_3 were dissolved in one liter of water in a volumetric flask. One hundred ml of this solution were diluted to one liter in a volumetric flask. One ml of the final solution was equivalent to 0.025 mg NO_2.

Sampling Apparatus

Flasks - two-liter, Pyrex, round-bottom flasks were encased in a simple container with a sleeve and accompanying stopcock valve as shown in Figure J-2.

Vacuum system - the vacuum system consisted of a vacuum pump, capable of producing a vacuum of 3 inches Hg absolute pressure, connected to a 0-30" Hg gauge.

Thermometer - dial thermometer, range 25 to 125°F, 5-inch stem.

Probe - Pyrex glass, heated, with a filter to remove particulate matter.

Spectrophotometer - a Bausch & Lomb Spectronic 70, capable of measuring optical density at 410 nm in 0.5 inch absorbance cells was used.

Sampling Procedures

Twenty-five ml of absorbing solution were pipetted into a sample flask. The flask valve stopper was inserted into the flask with the valve in the "closed" position. The sampling train was assembled as shown in Figure J-2. Each sample was collected using a 2-foot glass-lined probe at the upper port locations. Each flask was evacuated and pressure tested for one minute, and the initial flask temperature, pressure, and barometric pressure were recorded. The sampling probe was inserted into the stack and heated, and the sample flask connected. The 3-way stopcock was turned to the "purge" position, and stack gas was drawn through the system with a rubber squeeze bulb to check for condensation in the probe line. The 3-way stopcock was then turned to the sample position for 15 to 30 seconds. The flask valve was then closed and disconnected from the probe. The contents were shaken for at least 5 minutes.

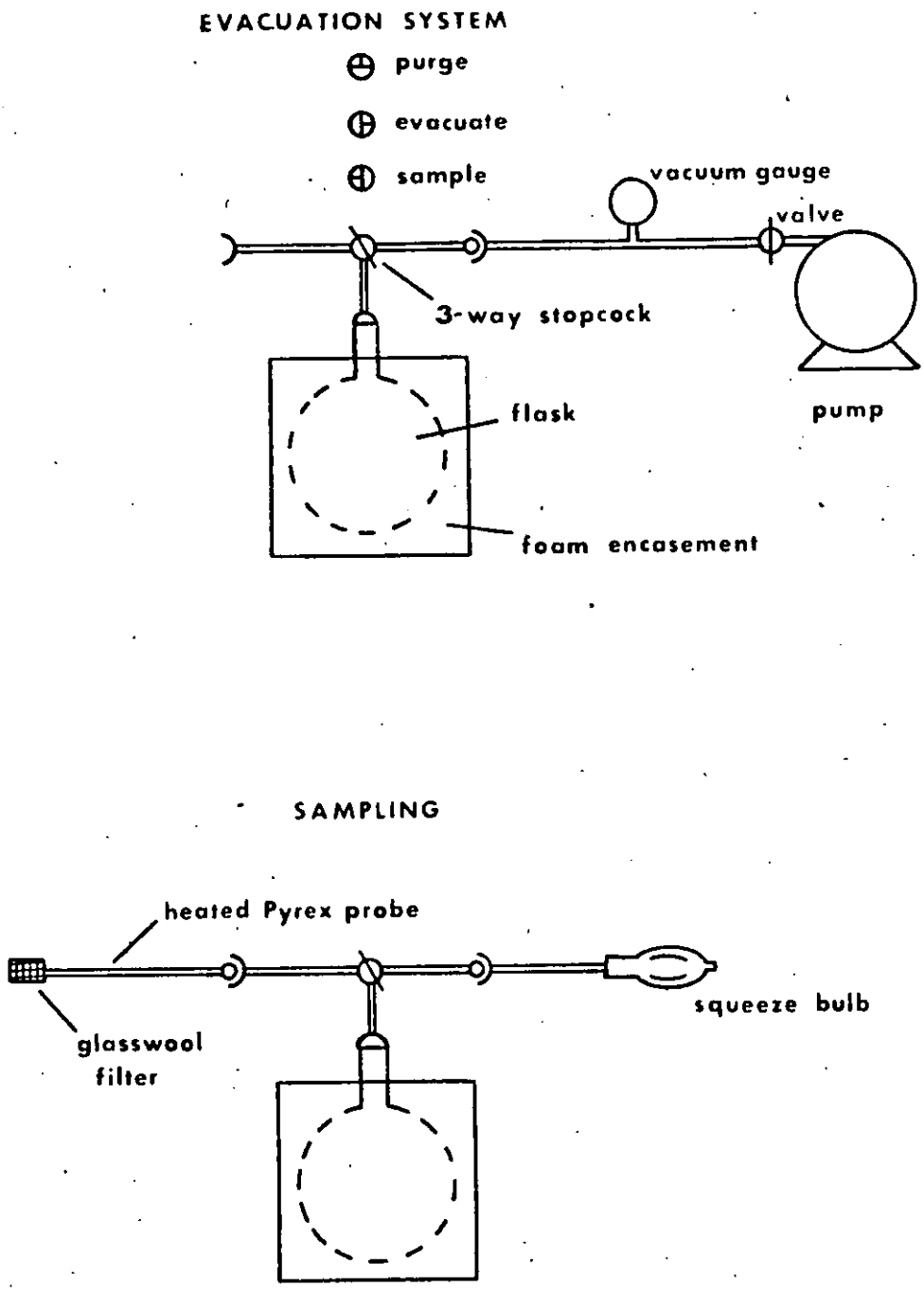

Figure J-2. Nitrogen Oxides Sampling Train

Sample Recovery

The flasks were allowed to set for at least 16 hours. They were then shaken for at least 2 minutes, the final pressure, temperature, and barometric pressure were taken, and the sample was transferred to a storage bottle using a small amount of water wash. The solution was then treated with sodium hydroxide (1 N) until a pH of 9 to 12 was reached.

Analysis

The sample was evaporated to dryness on a steam bath and then cooled. Two ml of phenoldisulfonic acid solution were added to the dried residue and triturated thoroughly with a glass rod. One ml of water and 4 drops of concentrated sulfuric acid were then added. The solution was heated on a steam bath for three minutes with occasional stirring. After cooling, 20 ml of water were added and mixed well by stirring. Concentrated ammonium hydroxide was added dropwise with constant stirring until alkaline to litmus paper. The solution was transferred to an appropriate volumetric flask and the beaker washed three times with 4 to 5 ml portions of water. The solution was diluted to the mark and mixed thoroughly. The absorbance of each sample was measured at 410 nm using the blank solution as a zero. A half-inch absorbance cell was used.

Calibration Procedures

Flask volume - the flask and flask valve were assembled and filled with water to the stopcock. The volume of water was measured to ±1 ml. The flasks were numbered and the volume recorded on each.

Spectrophotometer - aliquots of 0.0, 1.0, 2.0, 3.0, and 4.0 ml nitrate solution (standard) were added to a series of

beakers, and 25 ml of absorbing solution added to each. Sodium hydroxide (1 N) was added dropwise until alkaline to litmus paper (about 25 to 35 drops). These solutions were treated exactly like the samples by evaporating, adding reagents, and they were then transferred to 100 ml volumetric flasks and the absorbance read at 410 nm. The calibration curve was checked prior to each set of samples.

Determination of Sulfur Dioxide Emissions in Stack Gases

This method was used to measure sulfur dioxide in stack gases and follows Method 6 of the Federal Register[1], with modifications as noted below.

Sampling Apparatus

Nozzle - stainless steel (316) with sharp, tapered leading edge.

Probe - Pyrex glass with a heating system capable of maintaining a minimum gas temperature of 225°F at the exit end during sampling and prevent condensation from occurring.

Pitot tube - type S, attached to probe to monitor stack gas velocity

Filter holder - Pyrex glass

Impingers - four as shown in Figure J-3. The first and third are of the Greenburg-Smith design with standard tip. The second and fourth were modified by replacing the standard tip with a 1/2-inch ID glass tube extending to within one-half inch of the bottom of the impinger flask.

Metering system - vacuum gauge, leak-free pump, thermocouples capable of measuring temperature to within 5°F, dry gas meter with 2% accuracy, and related equipment, to maintain an isokinetic sampling rate and to determine sample volume.

Barometer - Bourden tube type to measure atmospheric pressure to ±0.1 inches Hg.

Sampling Reagents

Filters - glass fiber, MSA type 1106 BH or equivalent of a suitable size to fit in the filterholder.

Silica Gel - indicating type, 6-16 mesh.

Water - deionized, distilled.

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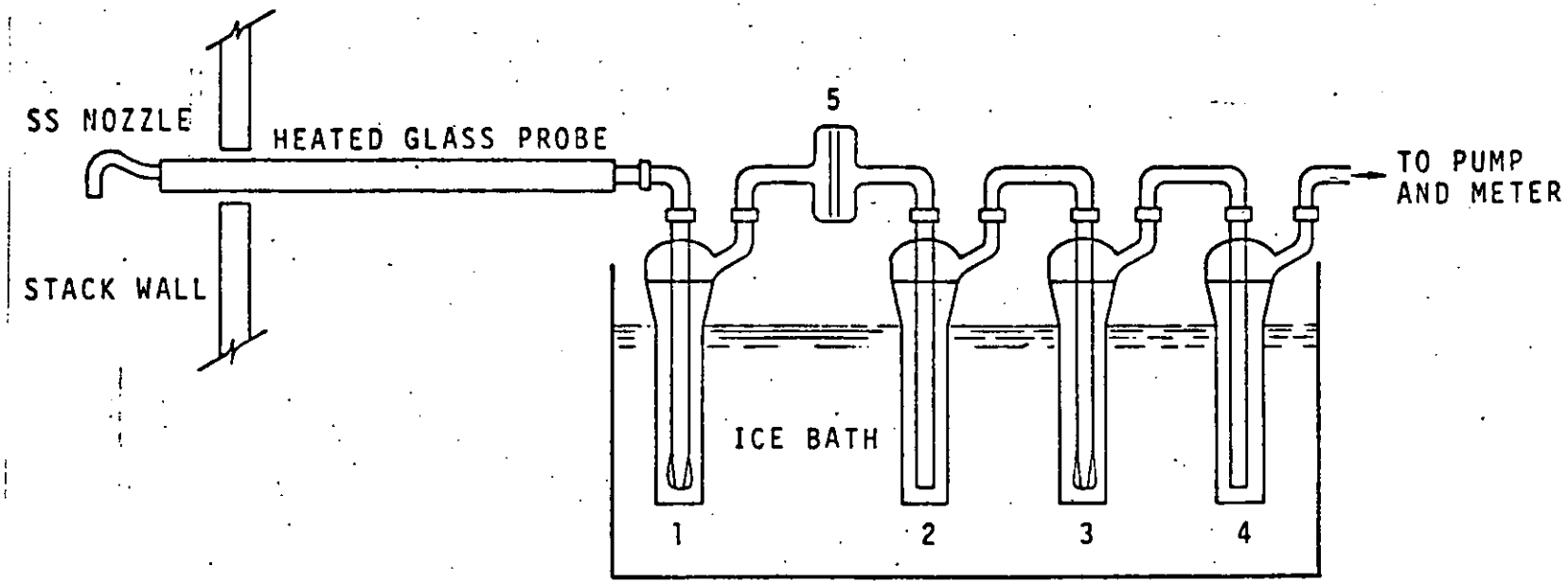
[1]Federal Register, Vol. 36, No. 247, December 23, 1971.



1. G-S IMPINGER - 100 ml. 80% ISOPROPANOL
2. MODIFIED G-S IMPINGER - 100 ml. 3% H_2O_2
3. G-S IMPINGER - 100 ml. 3% H_2O_2
4. MODIFIED G-S IMPINGER - 200 g. SILICA GEL
5. FILTER HOLDER w/GLASS FIBER FILTER

Figure J-3. SO_2/SO_3 sampling train

Isopropanol, 80% - mix 800 ml of isopropanol with 200 ml of deionized, distilled water.

Hydrogen peroxide, 3% - dilute 100 ml of 30% hydrogen peroxide to 1 liter with deionized, distilled water.

Analytical Reagents

Water - deionized, distilled

Isopropanol

Thorin indicator - 1-(0-arsonophenylazo)-2-naphthol-3, 6-disulfonic acid, disodium salt. Dissolve 0.20 gram in 100 ml distilled water.

Barium chloride (0.0200 N) - dissolve 2.4431 grams of barium chloride ($BaCl_2$) in 200 ml distilled water and dilute to 1 liter with isopropanol. Standardize with sulfuric acid.

Sulfuric acid standard (0.02 N) - standardize to ±0.0002 N against 0.01 N NaOH which has previously been standardized against primary standard potassium acid phthalate.

Sampling Procedure

One sampling point of average velocity was chosen which was 33 inches from the inner wall of the stack. The train was assembled as follows: 150 ml of 80% isopropanol in the first impinger, 100 ml of 3% hydrogen peroxide in both the second and third impingers, and about 200 grams of weighed silica gel in the fourth impinger. A portion of the reagents were retained for use as a blank. The train was assembled without the probe and was leak checked at the sampling site by plugging the inlet of the first impinger and pulling a 15-inch Hg vacuum. A leakage rate not in excess of 0.02 cfm at a vacuum of 15 inches Hg was recorded in all cases. The probe was then attached and the probe heating system turned on. The probe heater setting was adjusted during sampling to prevent any visible condensation. Crushed ice was placed around the impingers and more ice was added during the run to keep the temperature of the gases leaving the last impinger to 70°F or less.

For each run, the data were recorded at startup, on completion of the test, and at 12-minute intervals. Sampling was begun by positioning the probe at the sample point. The pump was then started and sampling proceeded at a rate of approximately 0.8 cfm for essentially the same time period as the Method 5 sampling.

At the completion of the test, the probe was removed from the stack and the train was purged for 10-15 minutes by drawing ambient air through the system.

The Method 6 samples were collected using the Method 8 train modified for each run as follows:

Run S-1 - this run used a standard Method 8 train with a filter between No. 1 and No. 2 impingers and no heat applied in the oven.

Run S-2 - this run used the Method 8 train with an additional filter between the probe and first impinger. The added filter was not heated.

Run S-3 - this run used the same train as Run S-2 but the additional filter was heated to approximately 260°F. Samples of the probe washing, isopropol solution in the first impinger, first impinger wash, and filters were collected and forwarded to EPA for further analysis.

Sample Recovery

The solutions from the second and third impingers were transferred to a Wheaton bottle. These impingers and their connecting glassware were rinsed with deionized distilled water which was added to the same container.

Analytical Procedures

The volume of the sample was recorded. A 10 ml aliquot of sample was pippetted into a 100 ml Erlenmeyer flask. Forty ml

of isopropanol and 2-4 drops of thorin indicator were added. The sample was then titrated with barium chloride to a pink end point. Each titration was repeated with a second aliquot of sample. The blanks were titrated in the same manner as the samples.

Visual Determination of the Opacity of Emissions for Stationary Sources

This method was used to measure visual emissions for stack gases and follows Method 9 of the Federal Register.[1]

Applicability - This method is applicable for the determination of the opacity of emissions from stationary sources.

Procedures - The observer qualified in accordance with this method shall use the following procedures for visually determining the opacity of emissions.

Position - The qualified observer shall stand at a distance sufficient to provide a clear view of the stack emissions with the sun oriented in the quadrant to his back. Consistent with maintaining the above requirement, the observer shall, as much as possible, stand at a position such that his line of vision is approximately perpendicular to the plume direction; and when observing opacity of emissions from rectangular openings with length-to-width dimensional ratios greater than 2:1, his position should be approximately perpendicular to the longer dimension.

Field records - The observer shall record the name of the plant, stack location, type facility, observer's name and affiliation, and the date on a field data sheet. The time, estimated distance to the stack, approximate wind direction, estimated wind speed, description of the sky condition (presence and color of clouds), and plume background are recorded on a field data sheet at the time opacity readings are initiated and completed.

[1]Federal Register, Vol. 36, No. 247, December 23, 1971.

Observations - Opacity observations shall be made at the point of greatest opacity in that portion of the plume where condensed water vapor is not present. The observer shall not look continuously at the plume, but instead shall observe the plume momentarily at 15-second intervals.

Recording observations - Opacity observations shall be recorded to the nearest 5% at 15-second intervals on an observational record sheet. Each momentary observation recorded shall represent the opacity of emissions for that 15-second time period.

Data Reduction - The data have been reduced by summing the number of like readings taken during any run period and calculating the percentage of readings at 0, 5, 10, 15, and >20% levels. This calculation is made by obtaining the fractional part of the total readings at each level (number of readings at this level/total number of readings) and making this value a percentage by multiplying by 100.

Determination of Total Sulfur in Material Samples

The solid samples collected at the site were analyzed for total sulfur content. The fuel oil samples were analyzed by the Parr oxygen bomb calorimeter to provide both the Btu content and the percent sulfur by gravimetric analysis.

The EPA suggested that the method for the analysis of the sulfur content of the silo feed, kiln product, and ESP dust was the sodium peroxide bomb procedure as outlined in Parr Manual No. 121 and based on the ASTM D 271-46. Due to the nature of the materials, it was necessary to add benzoic acid in order to get the material to burn. In this method, potassium perchlorate, the sample, benzoic acid and sodium peroxide are placed in the bomb and the mixture ignited. After ignition the mixture is washed into hot water, neutralized with hydrochloric acid, filtered, bromine water added, boiled to expell excess bromine, neutralized and the sulfate formed precipitated with barium chloride. Four samples were treated as described in the method. The precipitate formed, instead of being the expected white $BaSO_4$, was brown in color indicating that $Fe(OH)_3$ had co-precipitated with the $BaSO_4$. It was not established if the iron was from the sample or from the bomb. In addition to this problem, it was found that for several samples the ignition did not take place smoothly.

Alternative procedures were investigated and after review of these methods, it was suggested to EPA that ASTM C-25-72 would be preferrable. This ASTM procedure contains a method for sulfur in limestone and lime. In brief, the method is based on a sodium carbonate fusion and gravimetric sulfur determination using adjustment of the pH of the solution to

eliminate $Fe(OH)_3$ precipitation. In addition to the samples obtained from the plant, several standards with known amounts of sodium sulfate in calcium carbonate or calcium oxide were also analyzed as a check on the analytical method.

The results of the analysis of these standards are as follows:

Na_2SO_4 (g)	CaO (g)	$CaCO_3$ (g)	% Sulfur in Sample	% Sulfur Found	% Recovery
0.03982	1.00367	-	0.862	0.862	100
0.04443	-	1.02216	0.940	0.912	97
0.03483	-	1.10619	0.689	0.622	90

These results indicate that the method gives values within 90% of the true value.

APPENDIX K

SAMPLING LOG

SAMPLING TASK LOG

Plant Woodville Lime & Chemical
Date 8-6-74

Plant Location Woodville, Ohio
Recorded by C. E. Riley

8-6-74 Comments	Run	Sampling location (Port)	Pollutant	Clock Time Began	Clock Time Ended	Elapsed Time (min)	Sample Nos.
Monsanto sampling crew arrived				8:15			
Kiln and ESP appear to be operating satisfactory with normal production and operating parameters							
Draft fan down for removal of adhering dust				8:30			
Draft fan back on line				8:41			
Monsanto setting up equipment for test				8:30 to 1500			
Leak-checked sampling train; Part. & SO_2 train within EPA specifications							
Started particulate test	P1	West	Particulate	1525	1617	52	
Wrong probe tip size was used in setting up the train. In order to maintain isokinetic sampling conditions a smaller nozzle size was used - 0.374 was replaced with 0.244							
Replaced nozzle				1617	1636		
Continued test	P1	West	Part.	1636	1720	44	
Switched ports	P1	South	Part.	1720	1740		
Continued test	P1	South	Part.	1740	1834	54	
Stopped test due to pump operational problems				1834	1842		
Continued test	P1	South	Part.	1842	1924	42	
Completed part. test	P1			1924		192 min total	

Visible emissions observation are ranging between 0 and 5 percent opacity. However several readings at 10 percent were recorded.
The oil feed ~~was~~ ratio was increased @ 14:30 due to a drop in the natural gas pressure. Collected samples of feed and product during test.
SO_2 ~~and~~ an integrated gas sample were collected during the Part. testing.
For NOx sampling times see the Sample Identification log.
Carried out cleaning and sample recovery also conducted Orsat analysis 1930—2130

SAMPLING TASK LOG

Plant Woodhill Lime
Date 8-7-74

Plant Location Woodhill, Ohio
Recorded by C. E. Riley

8-7-74 Comments	Run	Sampling location (Port)	Pollutant	Clock Time Began	Clock Time Ended	Elapsed Time (min)	Sample Nos.
Arrived plant				0800			
Stack opacity is almost 0. Process control room operator stated that the ESP inlet temperature had not dropped below 700°F for the last 16 hours. This with other operating parameters seem to have a definite effect on the ESP collection efficiency.							
Started Part. test	P2	West	Part.	1144	1320	96	
Stack opacity still reading almost 0 with several puffs of 5 percent every so often.							
Switched ports				1320	1345		
Continued sampling	P2	South	Part.	1345	1521	96	
Completed part. test	P2			1521		192 min total	
Stack opacity still reading mostly 0 percent.							
SO_2 and NO_x sampling times taken integrated gas samples were collected during the particulate test times. Collected feed, ESP dust, fuel oil, and product samples during particulate testing. Inlet temperature did not drop below 700°F during the entire testing period. Normal production rates with normal process operations were adhered to during the testing							
Carried out cleanup and sample recovery; performed orsat analysis 1530-1700							

SAMPLING TASK LOG

Plant Woodville Lime
Date 8-8-94

Plant Location Woodville, Ohio
Recorded by C. E. Riley

8-8-74 Comments	Run	Sampling location (Port)	Pollutant	Clock Time Began	Clock Time Ended	Elapsed Time (min)	Sample Nos.
Arrived plant				0800			
Stack plume is almost clear - 0 opacity							
ESP inlet temperature is 700°F							
Started part. test	P3	West	Part.	10:27	1203	96	
Stack opacity is 0 percent							
Switched ports				1203	1222		
~~Started~~ Continued part. test	P3	South	Part.	1222	1358	96	
Completed part. test	P3			1358		192 min total	
Collected SO_2 NO_x and integrated gas samples during the part. test.							
Also collected feed, product, oil, and duct samples during the part. test.							
Stack opacity looks very good; 0 opacity - ESP temp still above 700°F.							
Carried out cleanup and sample recovery; performed orsat analysis 1400-1700							

APPENDIX L

RELATED REPORTS

Two additional test series were performed at the Woodville Lime and Chemical Company. The first test was conducted on May 21, 1974, and is discussed in EPA Report No. 74-LIM-3A. The second test series was conducted during the week of July 7, 1974, and is presented in EPA's Report No. 74-LIM-3B.