

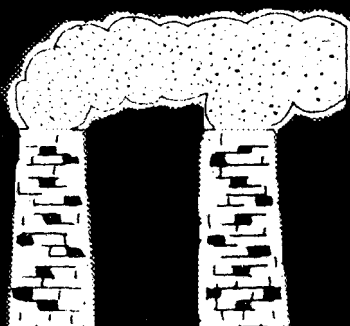
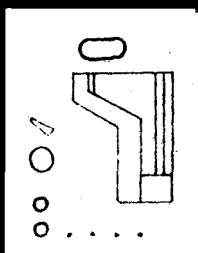
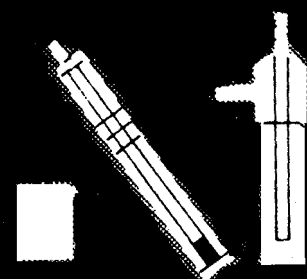
# AIR POLLUTION EMISSION TEST

FEB 18 1975

WOODVILLE LIME AND CHEMICAL COMPANY

Woodville, Ohio

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
Office of Air and Waste Management  
Office of Air Quality Planning and Standards  
Emission Measurement Branch  
Research Triangle Park, North Carolina



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  
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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 pre-heater 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<sup>1</sup> 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.<sup>2</sup>

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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<sup>1</sup>Federal Register, Vol. 36, No. 159, August 17, 1971.

<sup>2</sup>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<sub>2</sub> 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<sub>2</sub> 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 <sup>a</sup>	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 <sup>b</sup>	28986	27704	27559	28083
Stack volumetric flow rate-acfm <sup>c</sup>	65320	66488	65754	65854
Percent isokinetic	98.7	104.5	97.4	100.2
Duration of run-minutes	192	192	192	192
h. Particulates-probe, cyclone and filter catch				
mg	422.9	119.1	117.3	219.8
grains/dscf <sup>d</sup>	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 <sup>d</sup>	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

<sup>a</sup>Dry standard cubic feet @ 70°F, 29.92 in. Hg

<sup>b</sup>Dry standard cubic feet per minute @ 70°F, 29.92 in. Hg

<sup>c</sup>Actual cubic feed per minute-stack conditions

<sup>d</sup>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 <sup>3</sup> <sup>a</sup>	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 <sup>3</sup> /sec	13.68	13.08	13.01	13.26
Stack volumetric flow rate-acfm <sup>3</sup> /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 <sup>3</sup>	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 <sup>3</sup>	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

<sup>a</sup>Normal cubic meters at 21.1°C, 760 mm Hg

Table 3  
SUMMARY OF NO<sub>x</sub> RESULTS

<u>Date</u>	<u>Run #</u>	<u>Time</u>	<u>ppm<sup>a</sup></u>	<u>lb/DSCF x 10<sup>-5</sup></u>	<u>grams/Nm<sup>3</sup></u>	<u>lb/hr<sup>b</sup></u>	<u>Kg/hr<sup>b</sup></u>
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

<sup>a</sup>Parts per million, by volume

<sup>b</sup>Based on volumetric flow rate from corresponding Method 5 run (DSCFM)

Table 4  
SUMMARY OF SO<sub>2</sub> 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 <sup>3</sup> )	4.157	2.723	2.091	2.980
Run Time (min)	187	180	164	177
SO <sub>2</sub> Concentration				
(ppm, by volume)	6.08	26.6	13.1	15.3
(lb/dscf x 10 <sup>-6</sup> )	1.01	4.38	2.17	2.52
(mg/Nm <sup>3</sup> )	16.1	70.2	34.8	40.4
SO <sub>2</sub> Emission Rate <sup>a</sup>				
(lb/hr)	1.70	7.35	3.59	4.21
(Kg/hr)	0.772	3.34	1.63	1.91

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<sup>a</sup>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 <sup>a</sup>						
Start	1525	1525	1143	1143	1026	1026
End	1925	1925	1522	1522	1357	1357
Duration of observation (min) <sup>b</sup>	197	213	193	194	194	197
Total no. of readings <sup>c</sup>	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 unobserv- able	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

<sup>a</sup>24-hour clock start and end times

<sup>b</sup>Excluding the time that readings were not recorded for period of observation

<sup>c</sup>Readings recorded at 15-second intervals unless otherwise noted

Table 6  
TOTAL SULFUR CONTENT OF SILO FEED, FUEL OIL,  
PRODUCT AND ESP DUST

<u>Date</u>	<u>Sample No.</u>	<u>Material</u>	<u>% Sulfur Found</u>	<u>Avg % Sulfur</u>
8-6-74	S-74-001-788	Silo Feed	0.028, 0.028	0.028
	791	Fuel Oil <sup>a</sup>	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 <sup>a</sup>	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 <sup>a</sup>	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	

<sup>a</sup>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.

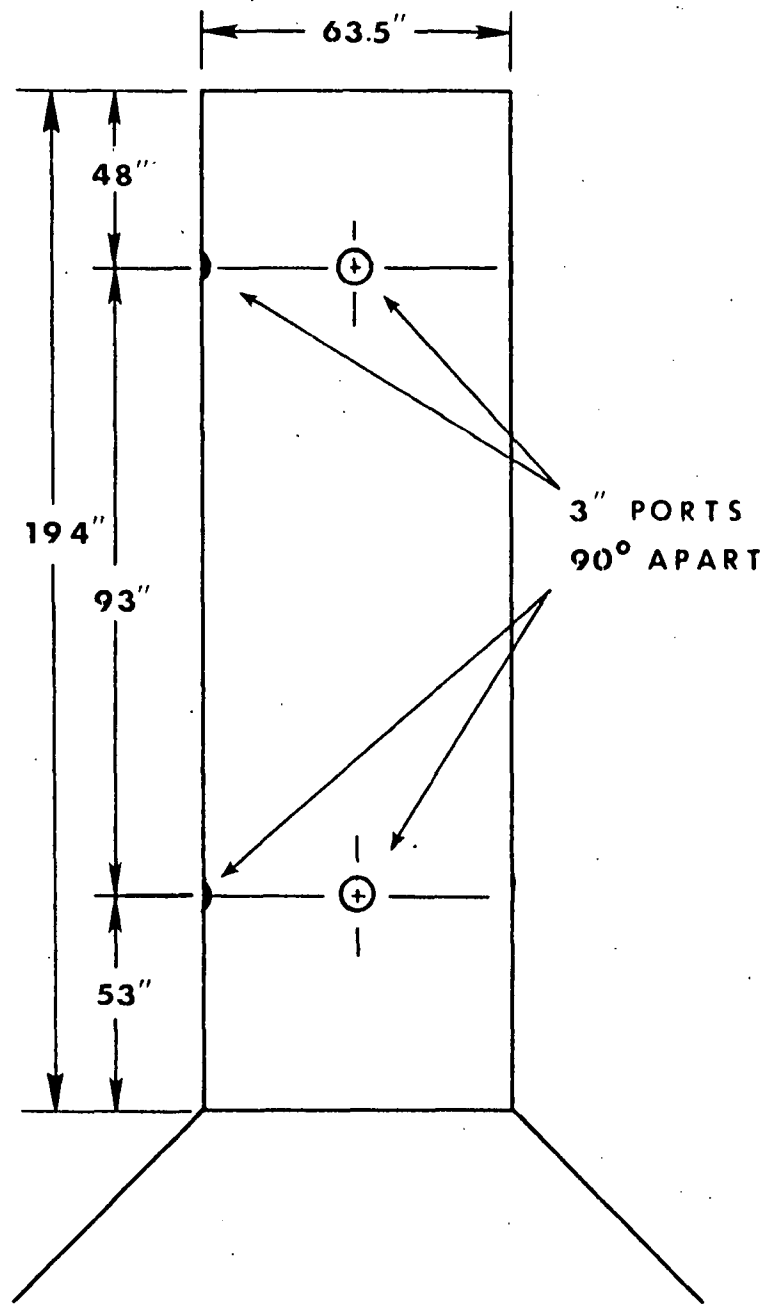


Figure 1. Stack port locations

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.

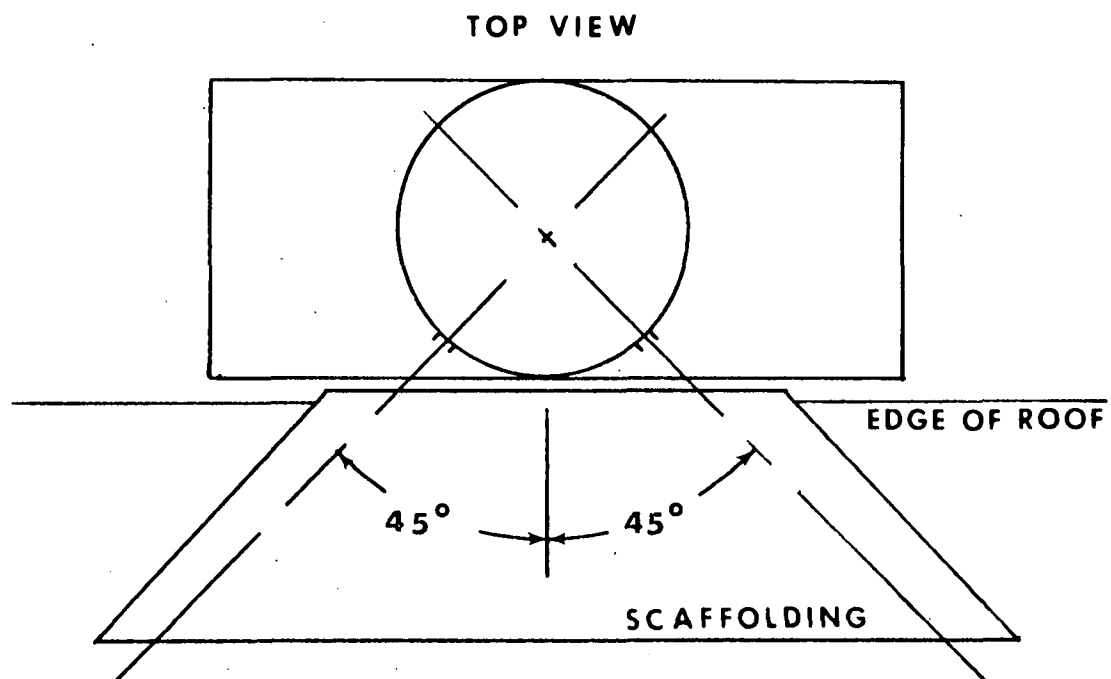


Figure 2. Test site No. 1 kiln precipitator outlet

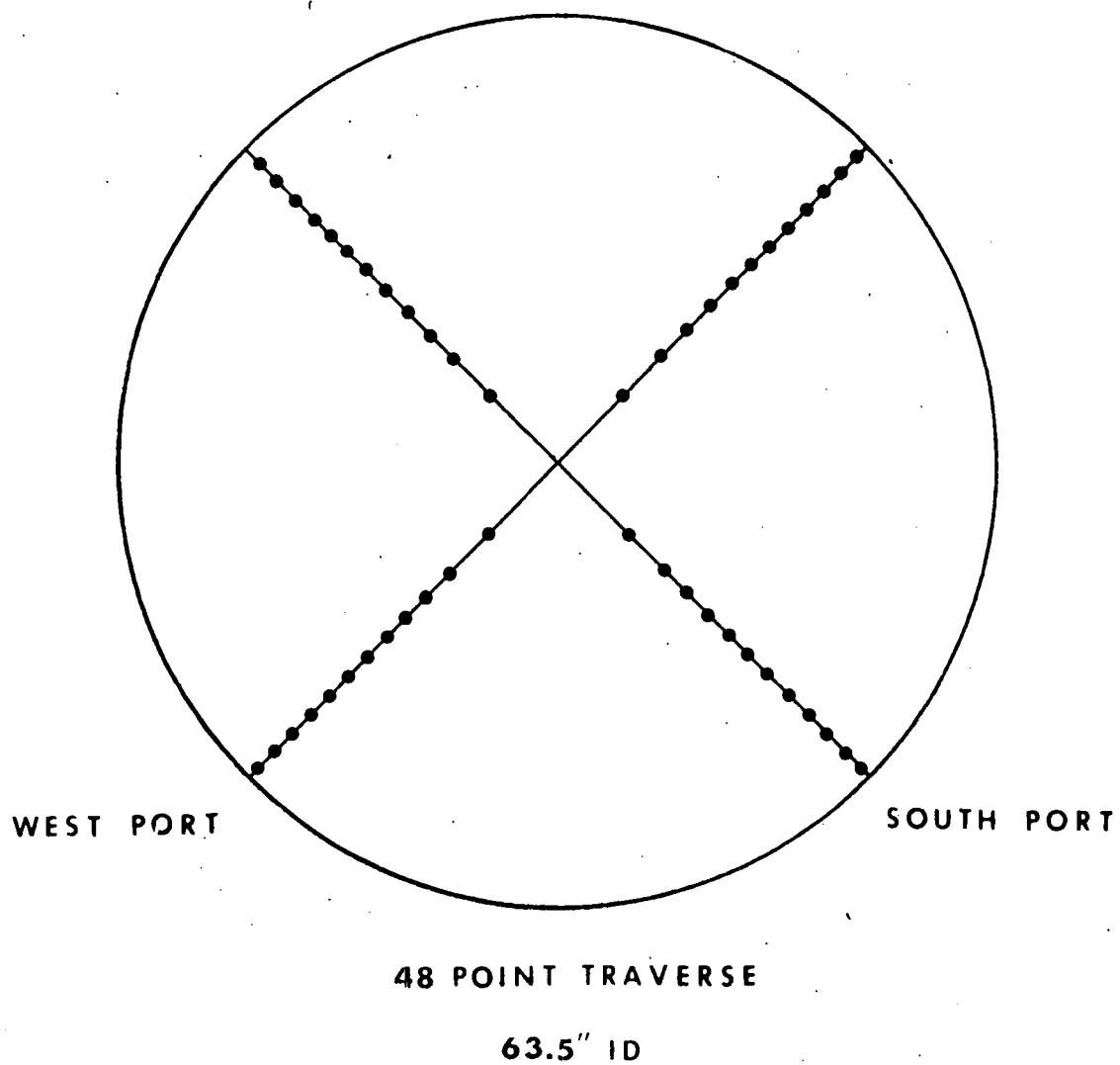


Figure 3. Particulate test traverse cross section

## 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) <sup>a</sup>	-	-	-
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

<sup>a</sup>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<sub>2</sub> and NO<sub>x</sub> 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<sup>1</sup>, Method 2. Temperatures were measured with the use of a thermocouple.

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<sup>1</sup>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<sub>2</sub>, O<sub>2</sub>, 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.<sup>1</sup> 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

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<sup>1</sup>Federal Register, Vol. 36, No. 159, August 17, 1971.

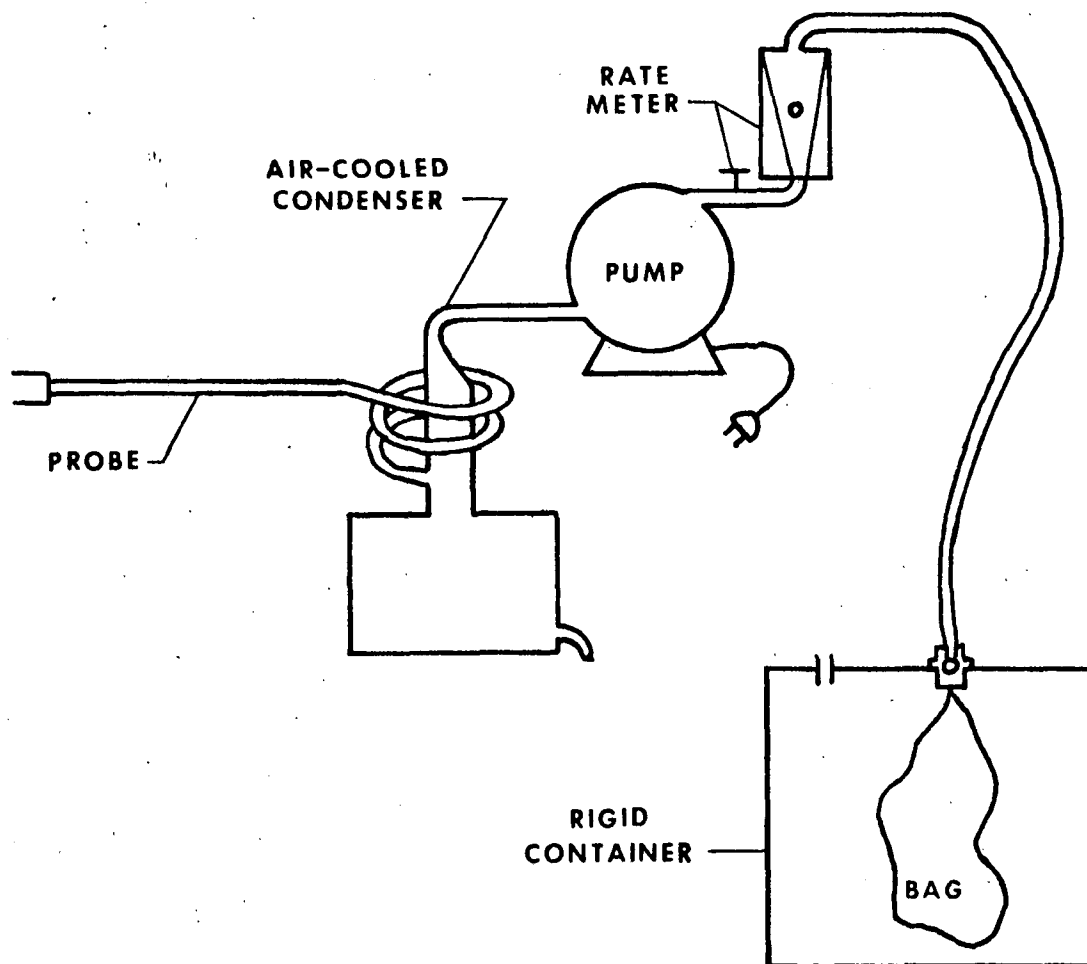


Figure 4. Integrated gas sampling train

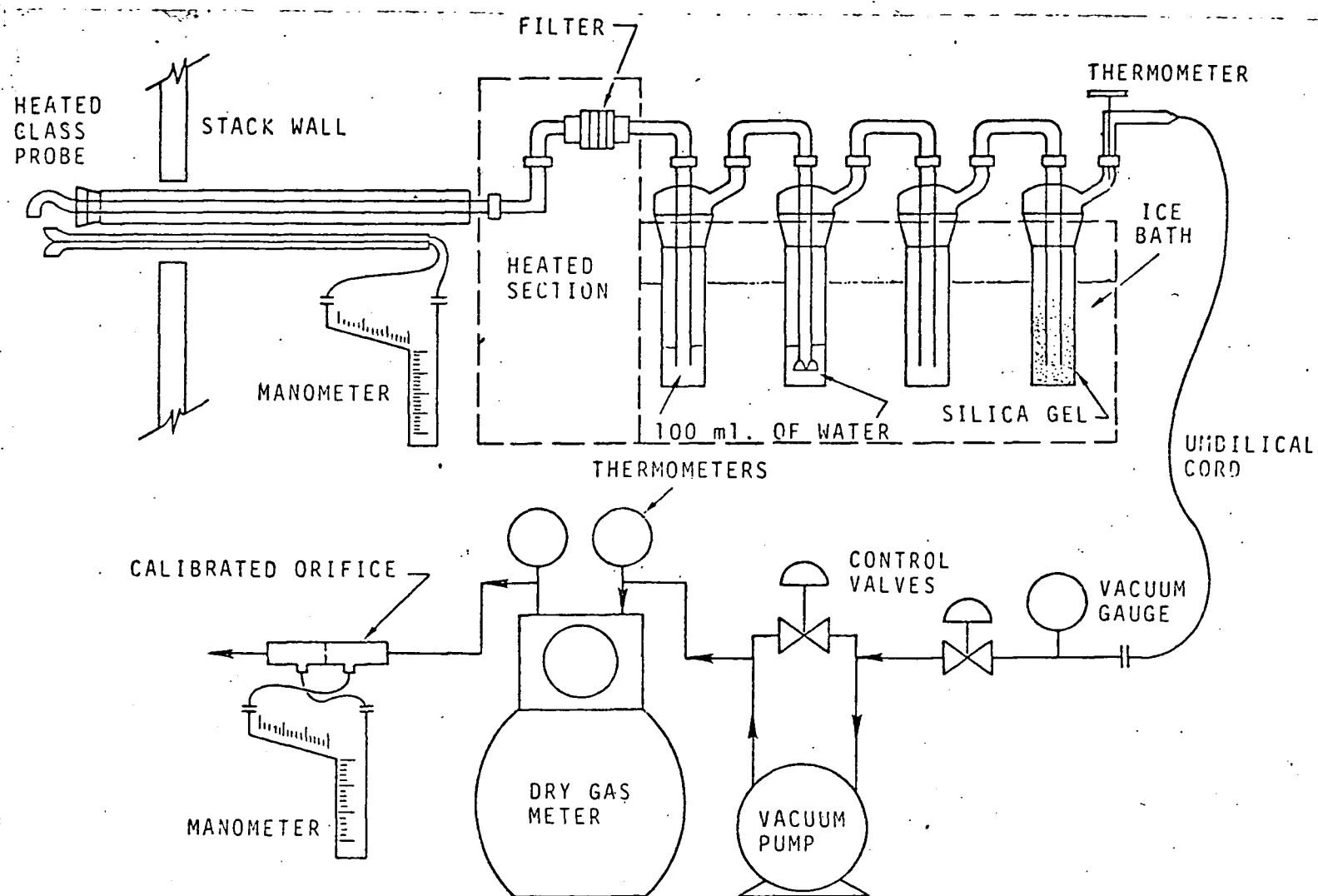


Figure 5. Particulate sample train

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  $\Delta H$  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<sub>2</sub> samples, was acid-soaked with 1:1 HNO<sub>3</sub> 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<sub>x</sub>

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.<sup>1</sup> 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.

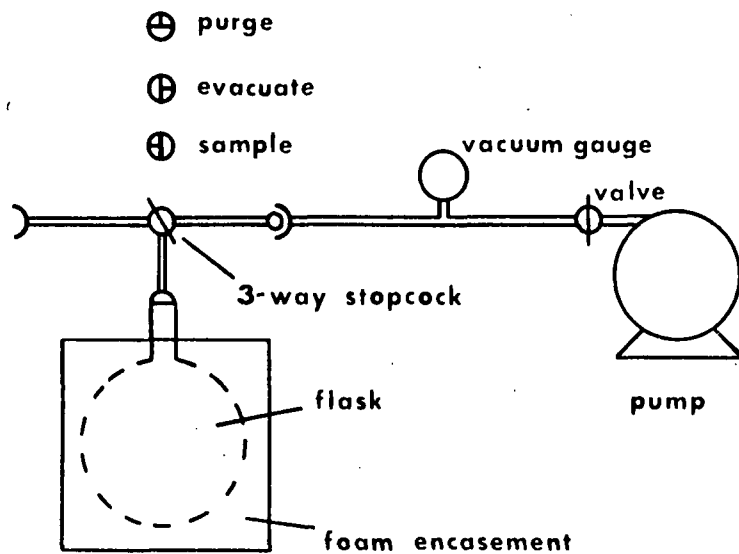
The flasks were allowed to set for at least 16 hours. They were then shaken for at least 2 minutes, the final pressure, temperature, and barometric pressure were taken, and the sample was transferred to a storage bottle.

The samples were analyzed using the Phenoldisulfonic acid, photometric analysis described in the above mentioned Federal Register.<sup>1</sup> Table 3 gives the calculated amounts of nitrogen oxides given as NO<sub>2</sub> in both parts per million and mass per unit

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<sup>1</sup>Federal Register, Vol. 36, No. 247, December 23, 1971.

### EVACUATION SYSTEM



### SAMPLING

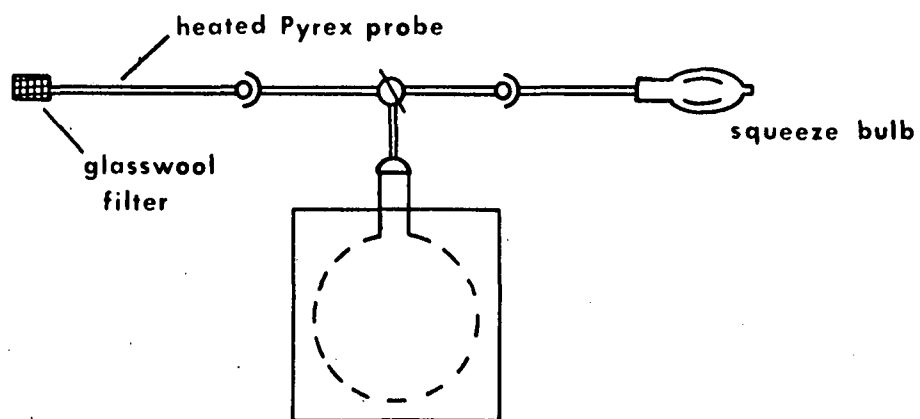


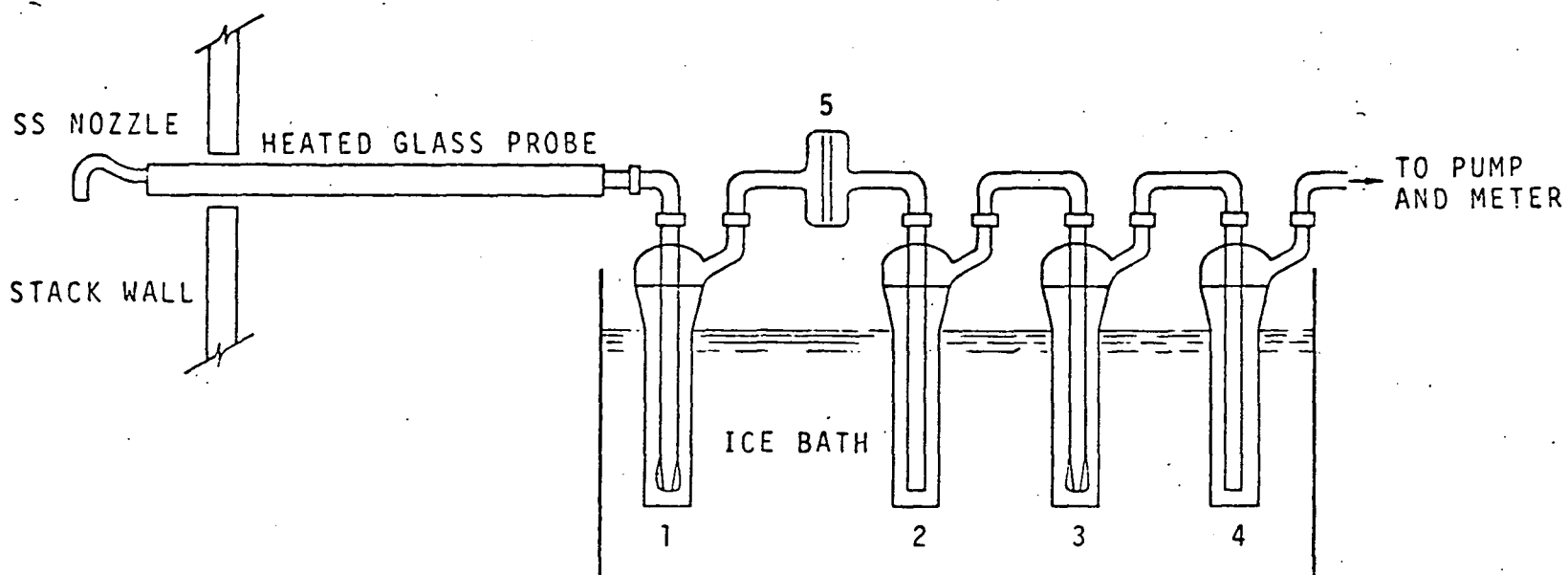
Figure 6. Nitrogen oxide sampling equipment

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

## SO<sub>2</sub>

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<sub>2</sub> 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<sub>2</sub> testing. The SO<sub>2</sub> 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<sub>2</sub>O<sub>2</sub>
3. G-S Impinger - 100 ml 3% H<sub>2</sub>O<sub>2</sub>
4. Modified G-S Impinger - 200 g Silica Gel
5. Filter Holder w/Glass Fiber Filter

Figure 7. SO<sub>2</sub>/SO<sub>3</sub> 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  $\text{BaSO}_4$ , a white material. However, the precipitate was brown, indicating the formation of  $\text{Fe}(\text{OH})_3$  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  $\text{Na}_2\text{CO}_3$  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.