

TEST NUMBER 72-MM-26

FMC CORPORATION
POCATELLO, IDAHO

PEDCo ENVIRONMENTAL



PEDCo-ENVIRONMENTAL

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I. INTRODUCTION

Under the Clean Air Act of 1970, as amended, the Environmental Protection Agency is charged with the establishment of performance standards for stationary sources which may contribute significantly to air pollution. A performance standard is based on the best emission reduction systems which have been shown to be technically and economically feasible.

In order to set realistic performance standards, accurate data on pollutant emissions must be gathered from the stationary source category under consideration.

The FMC Corporation plant in Pocatello, Idaho, was designated as a well-controlled stationary source in the elemental phosphorus reduction industry and was thereby selected by OAP for an emission testing program. This plant has two briquet calcining kilns at this location, which provide feed to four electric reduction furnaces. Each calciner is equipped with two parallel, low-energy scrubbers.

Testing was conducted before and after the No. 1 scrubber serving the No. 2 calcining kiln during the period of September 20 to October 3, 1972. Samples were collected to

determine the filterable and total particulate emissions, fluorides, P_2O_5 , and nitrogen oxides at each sampling location.

II. SUMMARY AND DISCUSSION OF RESULTS

A total of seven runs were made during the testing period for the determination of total and filterable particulates, fluorides, P_2O_5 , and nitrogen oxides. A run consisted of the simultaneous collection of an isokinetic sample at the inlet and outlet of the No. 1 scrubber serving the No. 2 calciner, and the collection of a sample in an evacuated flask at each location for the determination of nitrogen oxides. The first three runs were for the determination of particulate emissions, while the remaining four were for the determination of fluoride and P_2O_5 emissions.

The results of particulate, fluoride, and P_2O_5 sampling are summarized in Tables 1 through 4. Scrubber efficiencies are calculated in Table 5. Table 6 presents nitrogen oxide results.

There is no explanation for the large difference between inlet and outlet flows. No pattern was established such as the inlet or outlet consistently displaying the higher value. No adjoining duct was positioned between the inlet and outlet sampling sites that allowed entry or exit of an air stream.

The calciner is a continuous operation, but in some instances problems arose with the operation or testing procedure. A discussion of events is described in further detail as follows:

TABLE 1. SUMMARY OF PARTICULATE RESULTS

Inlet, Scrubber No.1, Calciner No. 2				
Run Number	<u>1</u>	<u>2</u>	<u>3</u>	<u>Avg. *</u>
Date, 1972	9/20	9/20	9/21	
Volume of Gas Sampled-DSCF ^a	57.68	56.81	49.57	
Percent Moisture by Volume	0.16	11.1	9.2	
Average Stack Temperature-°F	539	518	517	
Stack Volumetric Flow Rate-DSCFM ^b	81,500	68,600	68,200	68,500
Stack Volumetric Flow Rate-ACFM	182,000	169,000	167,000	168,000
Percent Isokinetic	105	122	107	
<u>Particulates-probe, cyclone</u> and filter catch				
mg	683.4	3213.8	2444.1	
gr/DSCF	0.183	0.873	0.76	0.816
gr/ACF	0.0817	0.355	0.311	0.333
lb/hr	128	513	445	479
lb/ton feed				
<u>Particulates-total catch</u>				
mg	884.4	3679.1	2545	
gr/DSCF	0.237	0.999	0.791	0.675
gr/ACF	0.106	0.406	0.328	0.365
lb/hr	165	588	463	526
lb/ton feed				
Percent impinger catch	23.2	12.6	3.9	8.3

^a Dry standard cubic feet at 70°F, 29.92 in. Hg.

^b Dry standard cubic feet per minute at 70°F, 29.92 in. Hg.

^c Actual cubic feet per minute.

* Results from Run #1 not included in average

TABLE 2. SUMMARY OF RESULTS, PARTICULATE EMISSIONS

Outlet, Scrubber 1, Calciner No. 2

<u>Run Number</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>Avg.</u>
Date, 1972	9/20	9/20	9/21	
Volume of Gas Sampled-DSCF ^a	45.53	56.31	60.43	
Percent Moisture by Volume	16.5	16.7	20.6	
Average Stack Temperature-°F	140	140	140	140
Stack Volumetric Flow Rate-DSCFM ^b	64,300	85,700	79,900	76,600
Stack Volumetric Flow Rate-ACFM	101,000	135,000	134,000	123,000
Percent Isokinetic	99.2	92.2	106	
<u>Particulates-probe, cyclone</u> <u>and filter catch</u>				
mg	199.9	591.6	187.4	
gr/DSCF	0.0676	0.162	0.0478	0.0925
gr/ACF	0.0428	0.102	0.0284	0.0577
lb/hr	37.3	119	32.8	63.0
lb/ton feed				
<u>Particulates-total catch</u>				
mg	415.6	1043.4	568.6	
gr/DSCF	0.141	0.285	0.145	0.190
gr/ACF	0.0893	0.181	0.0865	0.119
lb/hr	77.7	210	99.5	129
lb/ton feed				
Percent impinger catch	52.0	43.1	66.9	54.0

^a Dry standard cubic feet at 70°F, 29.92 in. Hg.

^b Dry standard cubic feet per minute at 70°F, 29.92 in. Hg.

^c Actual cubic feet per minute.

TABLE 3. SUMMARY OF RESULTS, FLUORIDE AND P₂O₅ LOADING

Inlet, No. 1. Scrubber, Calciner No. 2

<u>Run Number</u>	1	2	3	4	<u>Average</u>
Date, 1972	9/22	10/02	10/03	10/03	
Volume of Gas Sampled-DSCF ^a	52.21	29.16	27.438	18.31	
Percent Moisture by Volume	8.13	4.46	14.6	12.3	
Average Stack Temperature-°F	473	576	609	593	563
Stack Volumetric Flow Rate-DSCFM ^b	72,100	77,600	69,500	63,100	70,600
Stack Volumetric Flow Rate-ACFM	167,000	190,000	196,000	171,000	181,000
Percent Isokinetic	107	N/A*	133	97.9	
<u>Fluorides, Total</u>					
mg	12.22	62.9	280.2	139.9	
gr/DSCF	0.00361+	.03328	0.1576	.11793	.10294
gr/ACF	0.00156+	.01357	0.5590	.04353	.20537
lb/hour	2.23+	22.1	93.9	63.8	59.93
<u>P₂O₅, Total</u>					
mg	24.30	53.70	51.3	64.4	
gr/DSCF	0.00718+	0.0284	0.0289	0.0543	.0372
gr/ACF	0.00310+	0.0116	0.0102	0.0200	.0139
lb/hour	4.44+	18.9	17.2	29.4	21.83

^a Dry standard cubic feet at 70°F, 29.92 in. Hg.^b Dry standard cubic feet per minute at 70°F, 29.92 in. Hg.^c Actual cubic feet per minute

* Two nozzle sizes used during test.

+ Not included in average calculations, see discussion.

TABLE 4. SUMMARY OF RESULTS, FLUORIDE AND P_2O_5 EMISSIONS,
Outlet, Scrubber No. 1, Calciner No. 2

Run Number	1	2	3	4	Avg.
Date, 1972	9/22	10/02	10/03	10/03	
Volumes of Gas Sampled-DSCF ^a					
Percent Moisture by Volume	20.4	22.2	22.8	22.4	
Average Stack Temperature-°F	140	140	142	142	141
Stack Volumetric Flow Rate-DSCFM ^b	76,300	58,400	75,800	55,600	66,500
Stack Volumetric Flow Rate-ACFM	128,000	99,700	130,000	95,500	113,000
Percent Isokinetic	102	105	113	94.3	
Fluorides, Total					
mg	6.72	12.76	33.14	7.19	
gr/DSCF	0.00187	.00444	0.00893	0.00297	.00647
gr/ACF	0.00111	.00160	0.00519	0.00172	.00241
lb/hour	1.23	2.22	5.81	1.42	2.67
<u>P_2O_5, Total</u>					
mg	21.2	15.00	27.4	17.8	
gr/DSCF	0.00591	0.00522	0.00738	0.00735	.00647
gr/ACF	0.00351	0.00306	0.00429	0.00428	.00379
lb/hour	3.87	2.62	4.80	3.50	3.70

^a Dry standard cubic feet per minute at 70°F, 29.92 in. Hg.

^b Actual cubic feet per minute

TABLE 5. SCRUBBER EFFICIENCY DATA SUMMARY

<u>Constituent</u>	<u>Run No.</u>	<u>Inlet (lb/hr)</u>	<u>Outlet (lb/hr)</u>	<u>Efficiency</u>
Particulate, total catch	1	165*	77.7	
	2	588	210	64.3
	3	463	99.5	<u>78.5</u>
			Average	71.4
Fluorides, total	1	2.23*	1.23	
	2	22.1	2.22	90.0
	3	93.9	5.81	93.8
	4	63.8	1.42	<u>97.8</u>
			Average	93.9
P ₂ O ₅ , total	1	4.44*	3.87	
	2	18.9	2.62	86.1
	3	17.2	4.80	72.1
	4	29.4	3.50	<u>88.1</u>
			Average	82.1

* Not included in average, see discussion.

TABLE 6. SUMMARY OF NO_x RESULTS, CALCINER NO. 2

TEST NO.	DATE 1972	SITE	STACK GAS VOLUME SCFM ^a	NO _x EMISSIONS	
				PPM	LBS/HR
1A	9/22	Inlet	72,100	272	140
1B	9/22	Outlet	76,300	260	141
2A	10/2	Inlet	77,600	271	149
2B	10/2	Outlet	58,400	396	165
3A	10/3	Inlet	69,500	308	153
3B	10/3	Outlet	75,800	294	159
4A	10/3	Inlet	63,100	280	126
4B	10/3	Outlet	55,600	86	34 ^b
Average		Inlet			142
Average		Outlet			155

^a At standard conditions of 70°F and 29.92" Hg., dry basis.

^b Not included in average, process down when sample taken.

Particulate Test 1

The inlet sampling team had problems with supporting long glass probes in sample box in addition to thermal expansion of probe. Sample box clamp arrangement altered to give probe more stability. Broken probe may have allowed ambient air to enter sample stream. Results of inlet sample are questionable. When comparing moisture and particulate loadings to results from Tests 2 and 3, the values were appreciably lower. Therefore results from Test 1 are not included in the summary (Table 5).

Particulate Test 2

Calcining kiln was down for 20 minutes during test.

Particulate Test 3

This test was run without any problems.

Fluoride Test 1

The original sampling train reagent, containing 10 percent NaOH, crystallized in the outlet impingers. The filters and impingers were replaced to complete the test. Because of this difficulty, the results of this test are felt to be in error and are not included in the averages.

Fluoride Test 2

The NaOH concentration was reduced to 2.5 percent to avoid crystallization. Substitution of a smaller diameter nozzle on the inlet probe allowed reduced sampling rate and eliminated the clogging problem. The glass probe was still breaking due to duct vibration or the thermal expansion between the stainless steel and glass. It was impossible to pinpoint the time of breakage.

Fluoride Test 3

No complications arose during this test. However, sampling rate was 33% too high at the inlet location.

Fluoride Test 4

Scrubber went down at beginning of test. Flow rates in the stack increased during the test. The sampling rate on the outlet train could not be maintained. Filters were repeatedly changed in an attempt to pull an adequate flow rate. Acetone rinse portion of the outlet sample was lost in laboratory.

NO_x Tests 1, 2, 3, 4

No problems arose during the NO_x tests except for the invalid results obtained at the outlet on Test 4. The EPA process engineer inadvertently told plant operator that testing was complete and the process was shut down before the sample was taken. The scrubber did not remove any nitrogen oxides.

III. PROCESS DESCRIPTION AND OPERATION

The FMC Corporation plant at Pocatello, Idaho produces elemental phosphorus for shipment to other locations for burning or production of phosphorus trichloride. The plant has two calciners which provide feed to four electric reduction furnaces in the form of calcined briquets. In addition to phosphate ore (24% P_2O_5) the furnaces are fed bituminous coke and silica. Plant capacity is about 145,000 tons per year of P_4 .

Moist, crushed furnace feed is briquetted and fed onto a continuous grate calciner where it is dried, partially fused, and volatile components are driven off. Most of the fluorides present are driven off in this feed-preparation step. Briquets are carried at 3-5 feet per minute along the calciner. Burning CO gas (from the reduction furnaces), and supplemental LNG as needed, impinges on the 18-inch deep bed of briquets and heats them to about 2400°F. After going through the approximately 80-feet long calcining section, the briquets pass over a cooling section of about the same length. Air is pulled through the bed of hot briquets and either returned to the calcining section as preheated secondary air or exhausted.

Each of the two calciners is controlled by two low-energy scrubbers in parallel. The two scrubbers are the same except that excess secondary combustion air from the primary cooling fan is vented to the No. 1 scrubber at a point before the

calcining fan. Since the No. 1 scrubber serving the No. 2 calciner was tested, the following information applies only to it.

A 500 hp calciner fan pulls heated air and combustion products through the bed of briquets. The pressure drop across the bed is 4-6 inches H_2O , depending on the feed characteristics. Exhaust air then enters the scrubbers at a rate of 77-92,000 SCFM. The scrubber operates essentially at atmospheric pressure. Each scrubber is followed by a demister. A 250 hp scrubber booster fan follows the demister, and final exhaust is through an approximately 90 foot stack.

The scrubber is equipped with three internal rotors which throw the scrubbing solution up into the moving gas stream. The CaO scrubbing solution enters the system at the demister tower and immediately behind the No. 3 rotor. Make-up water is introduced under each rotor and mixing of water and slurry is accomplished by the action of the scrubber rotors.

The scrubber lime slurry is prepared at the No. 1 calciner which is some distance from the scrubber tested. The lime is fed from a silo into a mixing tank, mixed with water, and pumped to the scrubbers. A timer-controlled valve on the slurry line cycles the scrubber feed. Make-up water flow is

continuous. Attempts are made to hold the scrubbing liquor pH at 5 to 8. This is controlled by the speed of the slurry feed timer cycle. The control of the pH was not very well attended during testing.

In order to protect the lining in the scrubber, water sprays are provided on the inlet ductwork to the scrubbers. These sprays are activated when the temperature of the inlet gas stream gets too high. These sprays were off during all except the first test.

A total of 7 sampling runs were made. A run consisted of the simultaneous collection of a sample at the inlet and outlet location. Three runs were made to determine particulate emissions and four were made to determine fluoride emissions. One of these four runs was not completed and was discarded. Nitrogen oxide samples were collected at each location during each run. The following discussion covers the process operation during each run.

Run 1. September 20, 1972

Just prior to the run, the No. 2 burner fan was taken down for repair. This unit was back in operation by the time testing began. From the beginning of the run at about 1100 hours until 1500 hours, the emergency water sprays in the inlet scrubber duct were on. Estimated flow rate was about 10 gpm. From 1500 to 1530 the sprays were off. The scrubber pH was 2.3 until about 1430 when the slurry feed equipment was repaired. The scrubber pH then rose to above 7.

Run 2, September 20, 1972

At 1955 the grate drive motor overloaded and kicked out for about 15 minutes. Sampling was discontinued for about 5 minutes of this period. During this minor upset, the sampling crew on the inlet side noticed a noisy passage of coarse particles. Also particulate collected on the sampling train filter at this time appeared to be larger. In such upsets, the calciner is not shut down. Only the burners are cut back to prevent overheating. Consequently, there is only a short lead time in returning to normal when the grate is again put into operation.

Run 3, September 21, 1972

The pH when checked this morning was low. Although the operator's log did not reflect this, it had evidently been low for some time. Discussing this with plant personnel, it seems that the lime slurry circuit is prone to plugging and that the plug is not discovered quickly. After the low pH was brought to the plant's attention, it was brought up to 6.0.

The calciner was shut down for repairs to the feeder at 1230. The calciner was back in service at 1530. Sampling was begun at 1630 when feed rate and operating temperatures were back to normal.

During the test, the scrubber pH fluctuated from 2.3 to 5.5+. During most of the run, the pH was 5.5+. Litmus paper was used for these pH measurements.

Fluoride Run 1, September 22, 1972

Scrubber pH was spot checked at 0830 and found to be 5.5+. Operator's log showed the pH was 3-4.5 during the preceding night shift. Calciner was down due to malfunction from 1030-1100. The system was down again from 1130-1300 when a pallet jumped the track. At 1310, the calciner was back in operation. Testing began at 1330.

Fluoride Run 2 (discarded), September 26, 1972

At 1555, testing was begun. Although the process appeared to be operating normally, there were constant problems with the sampling trains. Apparent clogging of the sampling lines and erratic velocity profiles were unexplained. Because of these difficulties and inclement weather, the tests were terminated and the run was discarded.

Fluorides, Run 2 (Repeated), October 2, 1972

Scrubber water make-up had to be increased part way through the test because no overflow was occurring. Loss of water around the No. 3 rotor shaft leaked enough make-up water such that the rate was increased from 100 gpm to 140 gpm. This rotor also appeared to be turning slower.

A small amount (less than 1%) of natural gas was fired during this test.

Litmus paper was used to periodically check the scrubber water pH during testing. While this indicated a comfortable pH of 4, a check with a pH meter indicated it to be only 1.8-2.2.

Fluorides Run 3, October 3, 1972

The pH was checked with litmus paper and it again appeared to be high. The No. 3 rotor was not working due to either a frozen-up bearing or slipping belt. In addition water was being fed into the scrubber under the No. 2 rotor. Normally it was fed in through the demister and a lower flow is put in under the rotors. The water line under the No. 1 rotor was cracked and useless. These flow changes should not significantly affect the scrubber performance. Testing began at 0945. The pH was found to be low again. Problem was traced to the lime feed being shut off. Feed was restored at 1120. A belt for No. 3 rotor was installed half-way through the test, but the rotor was now buried in sludge and would not turn. The scrubber was taken down to free the No. 3 rotor after testing was concluded. At this time a bearing was replaced.

The only process upset occurred at 1145 when the grate speed and heating was cut back 15 minutes due to a feed loss.

Run 4, October 3, 1972

All rotors were working and the leakage around the No. 3 rotor had been reduced. Scrubber water pH was still difficult to maintain. First, the lime slurry pump went out. When this was repaired, the pH on the tested scrubber did not increase. In contrast, the pH in the No. 2 scrubber was 7.6. Having confined the problem to the scrubber, the operator spent over an hour trying to get the pH up. Success was limited to keeping it at a barely acceptable level. Towards the end of the run, the pH was being maintained above 5. the scrubber pH for the last three fluoride runs are summarized below:

TABLE 7. SCRUBBER SOLUTION pH

<u>Run No.</u>	<u>Minutes into Run</u>			
	<u>30</u>	<u>60</u>	<u>90</u>	<u>Composite</u>
2	3.40	2.25	2.35	2.20
3	2.90	3.50	2.25	2.20
4	5.25	4.50	3.25	4.90

IV. LOCATION OF SAMPLING POINTS

Figure 1 shows the sampling locations at the calcining kiln scrubber inlet and outlet. Sampling points are noted in Figure 2. As shown, at the inlet of the calcining kiln scrubber, the inlet samples were collected at 40 points, 10 points horizontally along each of 4 ports in the rectangular horizontal duct. The outlet samples were collected simultaneously at 32 points (16 along each diameter) in the 79.5 inch diameter vertical exhaust stack.

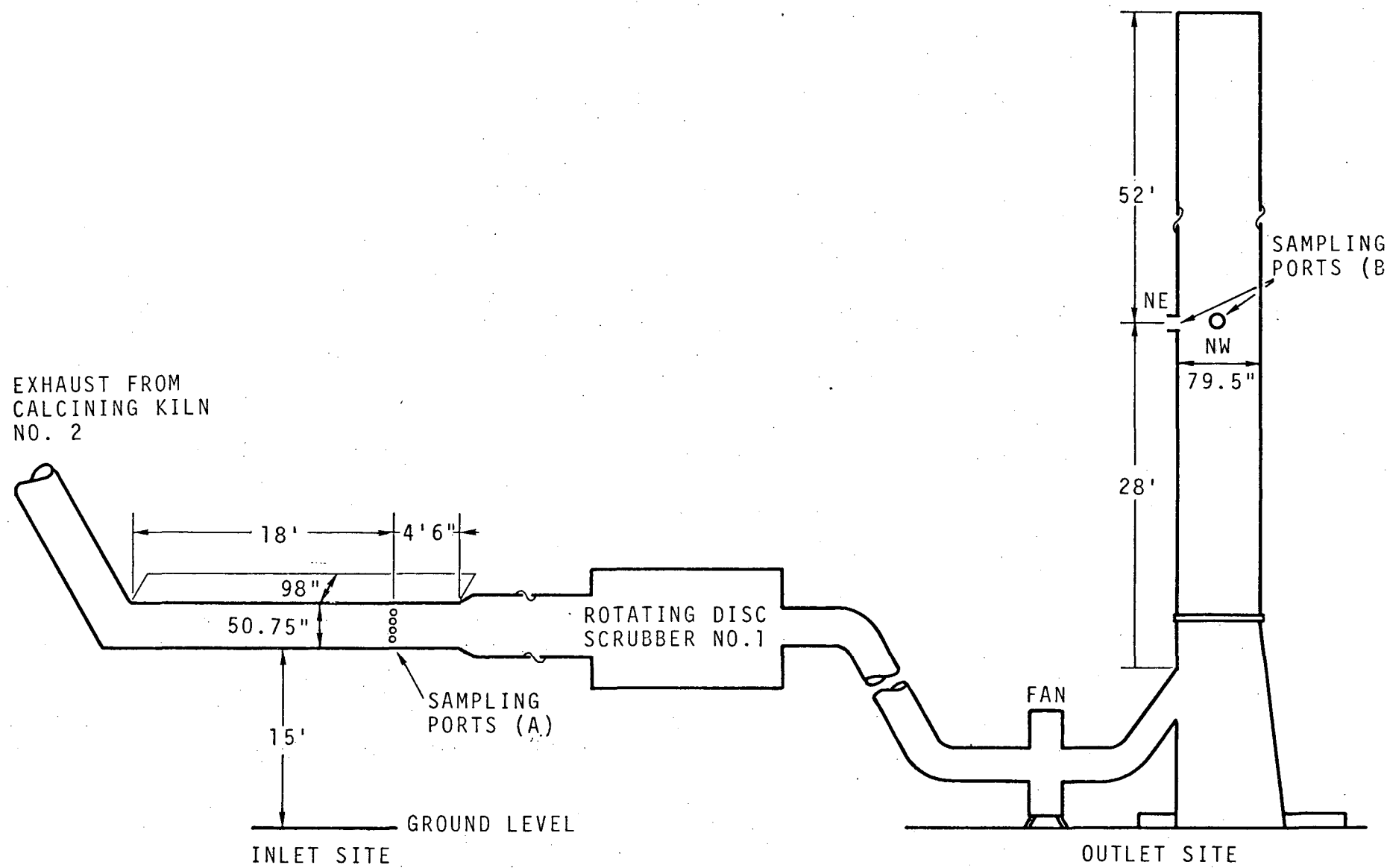


Figure 1. Calcining kiln scrubber sampling sites, F.M.C. Corporation, Pocatello, Idaho.

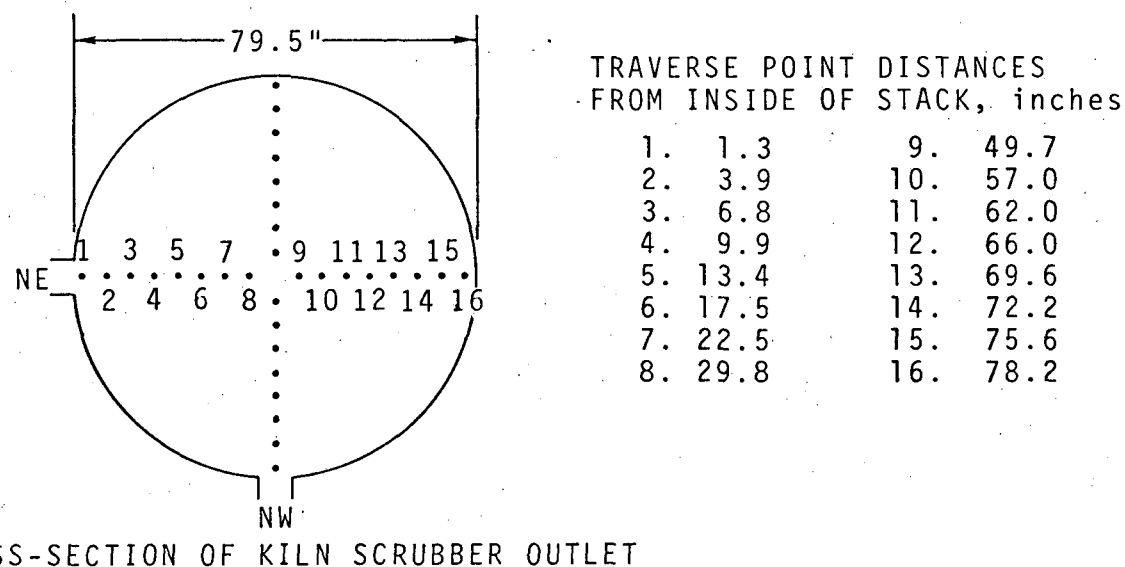
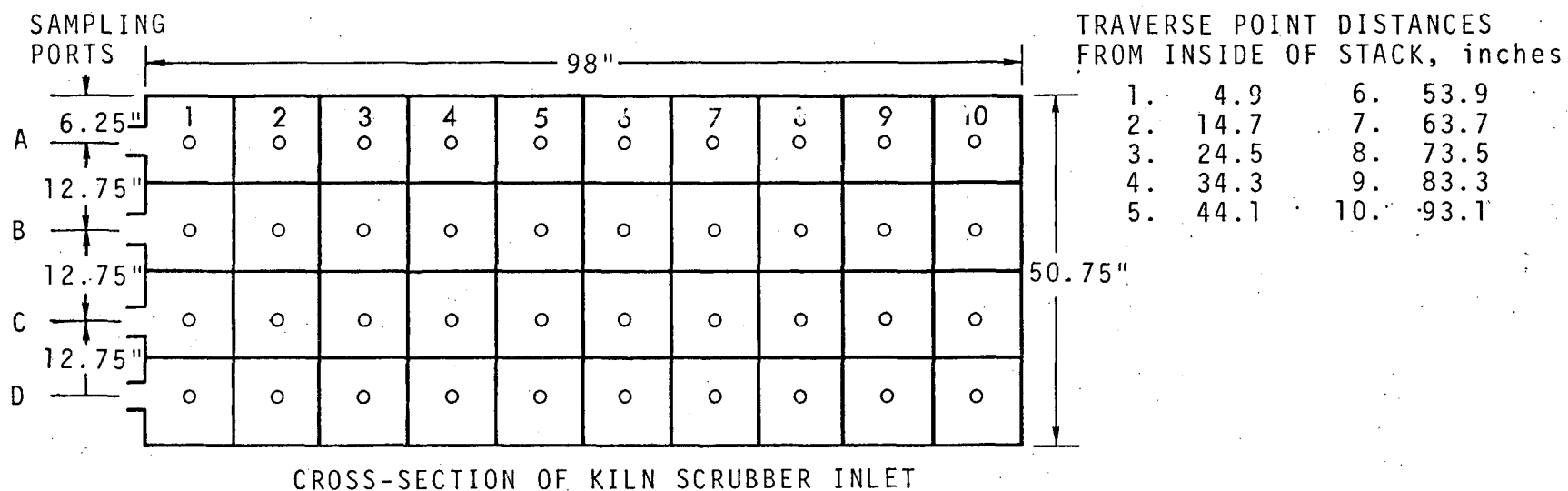


Figure 2. Calcining kiln scrubber sampling sites with location of sampling points -- F.M.C. Corporation.

V. SAMPLING AND ANALYTICAL PROCEDURES

Particulate sampling and analysis was conducted according to Method 5, Federal Register, V. 36, N. 247, December 23, 1971. In addition, the particulates collected in the impinger portion of the train were recovered and analyzed for mass. As specified in this procedure, the stack gas velocity was determined by Method 1, Federal Register, V. 36, N. 247, December 23, 1971, and the average gas molecular weight was determined by Method 3 using an Orsat apparatus.

Because of poor sampling accessibility at the outlet location, a flexible unheated Teflon-connector, approximately 9 feet long, was used to connect the sampling probe to the filter holder. The connector was cleaned with the same brush used to clean the heated glass probes at the outlet.

The EPA project officer requested a water and acetone wash on the front and back half of the sampling train. This added a probe water wash to the conventional method of recovering the sample.

Nitrogen oxides were determined by Method 7, F.R. December 23, 1971. Fluorides and P_2O_5 were determined by isokinetically extracting a sample of the stack gas using the basic train and procedure as described in Method 5 for particulates. However,

this train was modified such that the probe was followed by three Greenburg-Smith impingers containing 100 ml of 10% NaOH,* an empty straight-tip impinger, an unheated three-inch Whatman #1 filter paper, and finally an impinger containing approximately 200 gm of preweighed silica gel. The impinger train was placed in an ice-water bath. As was the case during the particulate sampling, a flexible Teflon connector was used to connect the probe to the first impinger at the outlet sampling location. A diagram of this train is shown in Figure 3.

Sample recovery consisted of measuring the volume increase of the impinger contents and the silica gel weight gain, a triple water rinse of all components from nozzle tip to filter holder, and a triple acetone rinse of all components. The impinger contents, filter, and the water rinse were combined in one glass sample jar, and the acetone rinse was placed in another sample jar.

In some instances the pump used to obtain an Orsat sample was not properly working. The EPA project officer agreed to assume a molecular weight based on previous samples.

Fluorides in the sample were determined by the Spadns-Zirconium-Lake Method, and the P_2O_5 content was determined by the ammonium molybdate procedure.

*100 grams NaOH mixed with a liter of water.

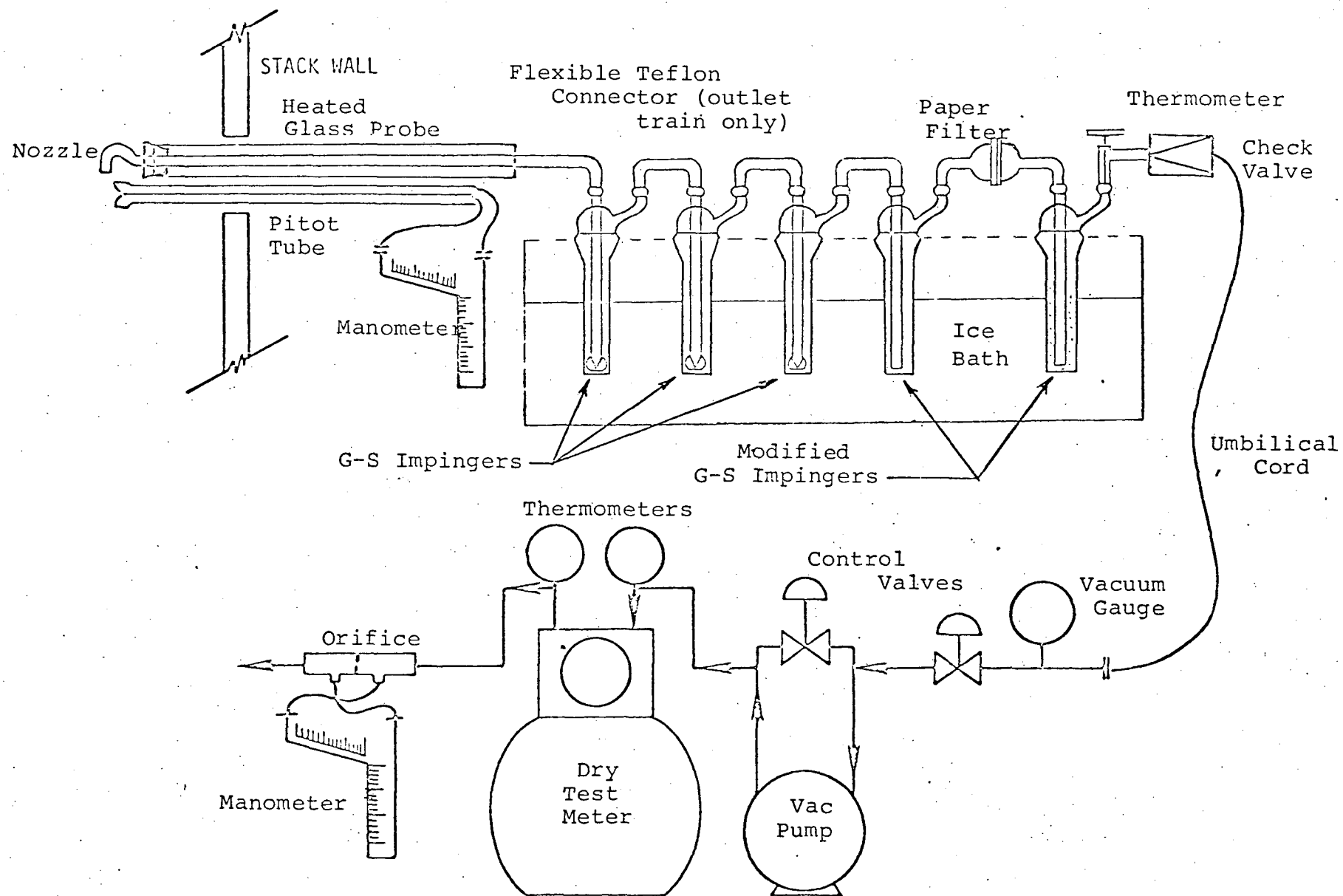


Figure 3. Fluoride and P_2O_5 Sampling train.

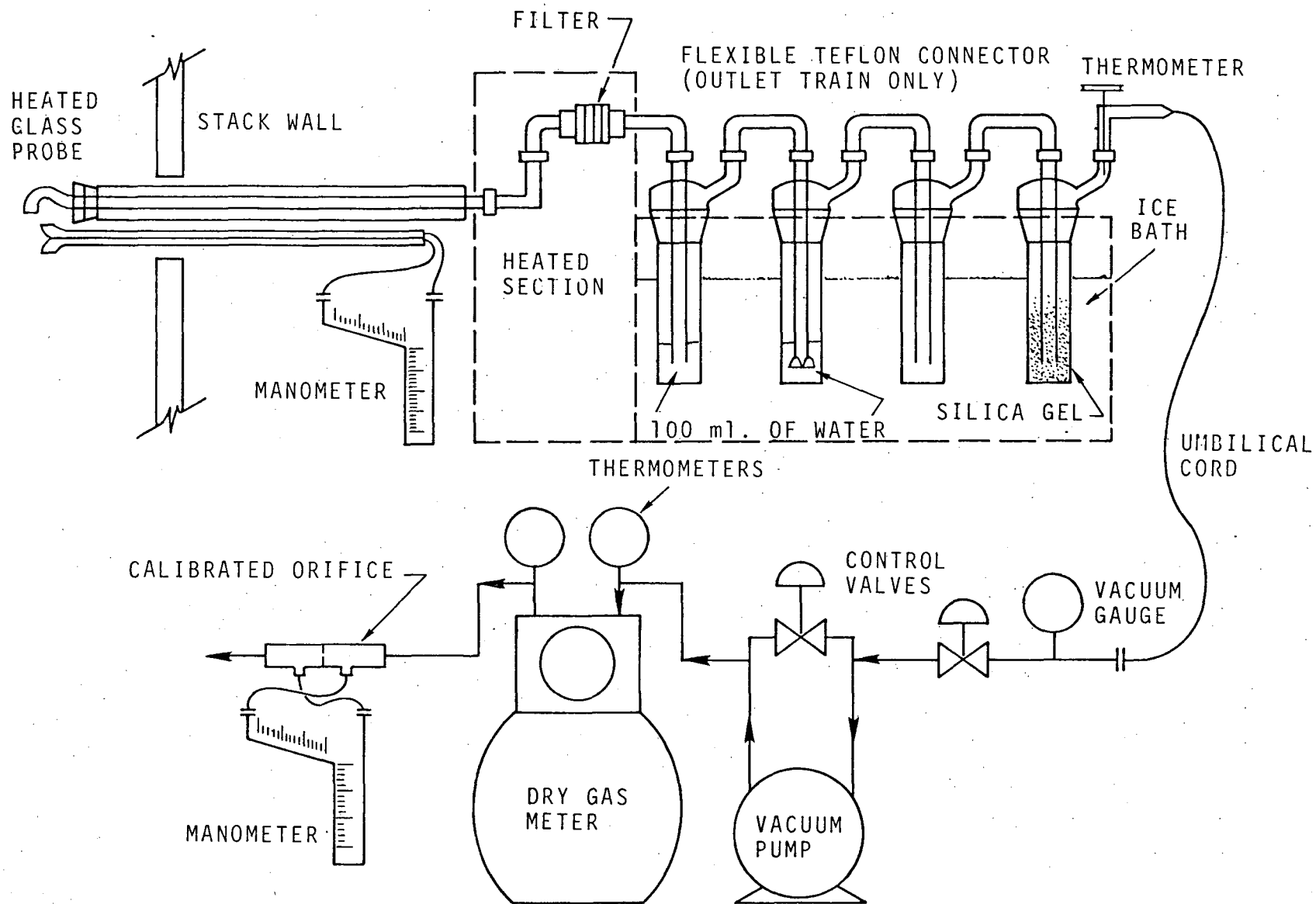


Figure 4. Particulate sampling train.