TEST NUMBER 73-ROC-1

INTERNATIONAL MINERALS

AND CHEMICAL CORP.

KINGSFORD, FLORIDA

PEDCo ENVIRONMENTAL

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II. 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.

Atmospheric emissions of particulate and fluorides from the International Minerals and Chemical Corporation (IMC) phosphorus plant dryer in Kingsford, Florida were sampled to establish a guide for New Source Performance Standards as authorized by the Clean Air Act of 1970. Triplicate tests were made to determine particulate and fluoride concentrations at the inlet and outlet of the dryer scrubber. The six tests were made February 12-15, 1973.

Wet phosphate rock enters the IMC dryer where the particles are dried by hot furnace gases. Dust entrainment in the exit flue gases are scrubbed in a vertical spray chamber scrubber. Fluoride emissions in the phosphate rock are driven off to the exit gases by the heat in the dryer.

III. SUMMARY OF RESULTS

Table 3.1 presents the overall test summary of particulate and fluoride concentrations in U.S. and metric units. Three particulate and three fluoride tests were averaged to obtain values in Table 3.1. Individual test results are shown as follows in subsequent Tables:

Table	Description
3.2	Particulate Analysis Summary
3.3	Fluoride Analysis Summary
3.4	Scrubber Water Analysis Summary
3.5	Rock Material Analysis Summary

In all cases, except particulate Test 3, outlet flow rates were slightly higher than volumes sampled at the inlet. This may have been caused by a cyclonic flow distribution, but since the maximum difference is only three percent, other factors may have contributed to the variation.

Individual particulate test data (Table 3.2) do not vary appreciably for this type of process. Outlet loadings from Run 1 are approximately twice the values from the last two runs, but this could be due to the particle size distribution in the type of feed. Types of feed are discussed in Section V.

The inlet combustion gas analysis for the particulate runs indicates a rise in CO_2 and a decrease in O_2 for the three tests.

Table 3.1 OVERALL SUMMARY OF RESULTS FROM PHOSPHATE ROCK DRYER

Pollutant Measurement System	Units		PARTICULATE				FLUORIDE			
			U.S.		Metric		U.S.		Metric	
	U.S.	Metric	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Volume of Gas Sampled	DSCF[b]	DNm^3[e]	51.608	108.212	1.4614	3.0642	65.795	106.414	1.8609	3.0133
Percent Moisture by Volume	%	%	31.63	27.19	31.63	27.19	31.89	26.00	31.89	26.00
Average Stack Temperature	°F	°C	164	151	73.3	66.1	164	151	73.3	66.1
Dry Stack Volumetric Flow Rate	DSCFM[c]	DNm^3/sec[f]	65597	65040	30.958	30.696	65011	66754	30.682	31.505
Actual Stack Volumetric Flow Rate	ACFM[d]	M^3/sec[g]	110262	101562	52.038	47.932	109554	102868	51.704	48.549
Percent Isokinetic	%	%	107.6	105.0	107.6	105.0	107.8	100.5	107.6	105.0
Feed Rate	Ton/hr	Mton/hr	333	333	302	302	295	295	268	268
Partial Catch[a]										
Weight	mg	mg	5621	302	5621	302	3.8	4.7	3.8	4.7
Concentration/Dry Volume	gr/DSCF	mg/DN_m3	1.639	0.04275	3751	97.83	0.00089	0.00068	2.0	1.6
Concentration/Actual Volume	gr/ACF	$mg/_m3$	0.9783	0.02741	2239	62.72	0.00053	0.00044	1.2	1.0
Concentration/Time	lb/hr	kg/hr	923.8	23.80	419.0	10.80	0.50	0.39	0.23	0.18
Lb/ton, Feed Concentration/ Input Feed Rate	lb/ton	kg/Mton	2.741	0.076	1.371	0.0380	0.0017	0.0014	0.00085	0.00070
Total Catch										
Weight	mg	mg	5746	411	5746	411	166.2	27	166.2	27
Concentration/Dry Volume	gr/DSCF	mg/DN_m3	1.677	0.05832	3837	133.4	0.03902	0.0040	89.28	9.2
Concentration/Actual Volume	gr/ACF	mg/m^3	0.9922	0.03740	226.2	85.57	0.02319	0.0026	53.06	5.9
Concentration/Time	lb/hr	kg/hr	945.5	32.51	428.9	14.75	21.76	2.3	9.870	1.0
Lb/ton, Feed Concentration/ Input Feed Rate	lb/ton	kg/Mton	2.810	0.102	1.405	0.0510	0.0732	0.0076	0.0366	0.0038
Percent Impinger Catch	%	%	2.55	27.6	2.55	27.6				
Percent Insoluable Fluoride	%	%					97.7	63	97.7	63

a) For particulate, catch includes probe, cyclone and filter. For fluoride, catch includes the water soluble portion.

b) Dry standard cubic feet at 70°F, 29.92 in. Hg.

c) Dry standard cubic feet per minute at 70°F, 29.92 in. Hg.

d) Actual cubic feet per minute

e) Dry normal cubic meters at 21.1°C, 760 mm Hg.

f) Dry normal cubic meters per second at 21.1°C, 760 mm Hg.

g) Actual cubic meters per second

Table 3.2 PARTICULATE EMISSION DATA SUMMARY
IMC Corp., Kingsford, Florida - Rock Dryer Scrubber

	Inlet			Outlet		
	1	2	3	1	2	3
Date	2-12-73	2-13-73	2-13-73	2-12-73	2-13-73	2-13-73
Volume of Gas Sampled-DSCF[a]	43.182	56.089	55.553	111.796	106.232	106.817
Percent Moisture by Volume	31.06	32.43	31.41	26.94	27.15	27.48
Average Stack Temperature-°F	164	164	164	151	150	152
Stack Volumetric Flow Rate-DSCFM[b]	64,172	65,578	67,040	64,707	66,569	63,843
Stack Volumetric Flow Rate-ACFM[c]	106,284	110,850	113,652	100,604	103,573	100,508
Percent Isokinetic	107.5	109.3	105.9	108.9	100.6	105.5
Percent CO_2	2.93	3.20	5.00	5.00	4.03	5.47
Percent O_2	17.93	17.50	15.70	15.43	15.13	15.57
Percent CO	0.00	0.03	0.07	0.03	0.00	0.03
Feed Rate-ton/hr	270	350	380	270	350	380
Particulates-probe, cyclone, and filter catch						
mg	3170	7566	6128	438	229	239
gr/DSCF	1.133	2.082	1.702	0.06046	0.03326	0.03452
gr/ACF	0.6840	1.231	1.020	0.03892	0.02138	0.02193
lb/hr	623.2	1170	978.2	33.53	18.98	18.90
lb/ton feed	2.307	3.343	2.574	0.124	0.054	0.049
Particulates-total catch						
mg	3315	7698	6226	537	376	320
gr/DSCF	1.185	2.118	1.729	0.07412	0.05462	0.04623
gr/ACF	0.7153	1.253	1.036	0.04772	0.03511	0.02936
lb/hr	651.7	1191	993.8	41.11	31.12	25.30
lb/ton feed	2.413	3.401	2.615	0.152	0.089	0.066
Percent impinger catch	4.37	1.71	1.57	18.4	39.1	25.3

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.

The Orsat apparatus did not have a water jacket around the gas tube which may have varied the gas temperature and, in turn, the gas analysis.

The insoluble portion of the outlet fluoride catch (Run 5, Table 3.3) is approximately an order of magnitude greater than values from the other two tests. Duplicate laboratory analysis produced the same results. A 60% pebble - 40% concentrate was being fed to the dryer during this test. The fluoride analysis of the feed (Table 3.5) confirms the rock was similar to other runs. Therefore it appears that the scrubber efficiency was lower during this run.

After each of the three fluoride runs, 100 ml of inlet and outlet sample was removed and given to IMC personnel for their analysis. This was considered when EPA analyzed the samples for fluoride concentration.

No major testing problems occurred during sampling at the Kingsford site. A pump in one of the outlet meter boxes acted up at times, so another meter box was used to complete the tests. A cyclone bypass connector on the outlet train broke after one test was complete, so the sample stayed intact. During the last fluoride test, the probe contacted the mud on the inside of the duct creating a vacuum in the sample train. The impinger water

Table 3.3 FLUORIDE EMISSION DATA SUMMARY
IMC Corp., Kingsford, Florida - Rock Dryer Scrubber

	Inlet			Outlet		
	4	5	6	4	5	6
Date	2-14-73	2-14-73	2-15-73	2-14-73	2-14-73	2-15-73
Volume of Gas Sampled-DSCF[a]	66.354	65.280	65.751	105.672	105.913	107.658
Percent Moisture by Volume	33.19	32.27	30.22	25.45	24.92	27.64
Average Stack Temperature-°F	164	164	164	151	150	151
Stack Volumetric Flow Rate-DSCFM[b]	64,884	65,641	64,528	66,251	67,700	66,312
Stack Volumetric Flow Rate-ACFM[c]	111,213	111,127	106,321	101,226	102,610	104,767
Percent Isokinetic	108.9	105.9	108.5	100.6	98.6	102.4
Feed Rate-ton/hr	270	300	315	270	300	315
Fluoride-water soluble						
mg	3.1	5.7	2.6	5.6	6.3	2.2
gr/DSCF	0.00072	0.00134	0.00061	0.00082	0.00091	0.00031
gr/ACF	0.00042	0.00079	0.00037	0.00053	0.00060	0.00019
lb/hr	0.400	0.758	0.337	0.464	0.532	0.179
lb/ton feed	0.0015	0.0025	0.0011	0.0017	0.0018	0.0006
Fluoride-total						
mg	137.2	189.6	171.9	7.9	67.4	6.9
gr/DSCF	0.03190	0.04482	0.04034	0.00115	0.00982	0.00098
gr/ACF	0.01861	0.02647	0.02448	0.00075	0.00648	0.00062
lb/hr	17.75	25.22	22.30	0.655	5.698	0.562
lb/ton feed	0.0654	0.0838	0.0705	0.0024	0.0187	0.0018
Percent insoluble fluoride	97.7	97.0	98.5	29.1	90.7	68.1

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 3.4. SCRUBBER WATER ANALYSIS SUMMARY

Item	Test Number							
	Scrubber Inlet				Scrubber Outlet			
	4	5	6	Avg.	4	5	6	Avg.
Total Fluorides, mg/l	0.4	0.4	0.4	0.4	1.3	1.2	1.1	1.2
P_2O_5, mg/l	0.1	0.1	0.1	0.1	1.4	1.2	1.4	1.3
pH	7.77	7.93	7.81	7.84	7.60	7.89	7.66	7.72

Table 3.5. ROCK MATERIAL ANALYSIS SUMMARY

Test	Total Fluoride, mg/gm		Total P_2O_5, %	
	Raw Feed	Dry Product	Raw Feed	Dry Product
1	38.9	36.2	31.1	31.8
2	37.8	39.9	30.5	32.4
3	38.5	39.2	33.0	33.1
4	38.6	38.5	31.8	30.9
5	36.9	37.1	31.5	30.7
6	38.5	39.4	32.4	33.8

Sample collection times can be found in Appendix H.

backed up and wet the filter. The sampling team replaced the filter without losing any of the sample water.

IV. PROCESS DESCRIPTION

A phosphate rock plant flow diagram, shown in Figure 4.1, illustrates the steps of the process in relation to the drying operation. After the phosphate rock is mined, a flotation process separates the impurities by chemically separating the impurities from the phosphate sand. The washed rock is then conveyed to storage bins.

The wet rock contains between 7 and 20 percent moisture while being transported to the drying site. All grades of rock are normally dried to 3 percent or less in the drying operation.

The IMC rock dryer at Kingsford, Florida is a 270 TPH fluid bed unit. The rock is dried by heat supplied by natural gas, with No. 5 residual fuel oil as a standby.

From the drier, combustion gases enter a cyclone separator where the majority of the particulate is removed and recycled back to the product conveyor belt. The gases then flow to a cyclonic scrubber using fresh water as the scrubbing media. The clean gas flow leaves the scrubber and exits through a ninety foot stack.

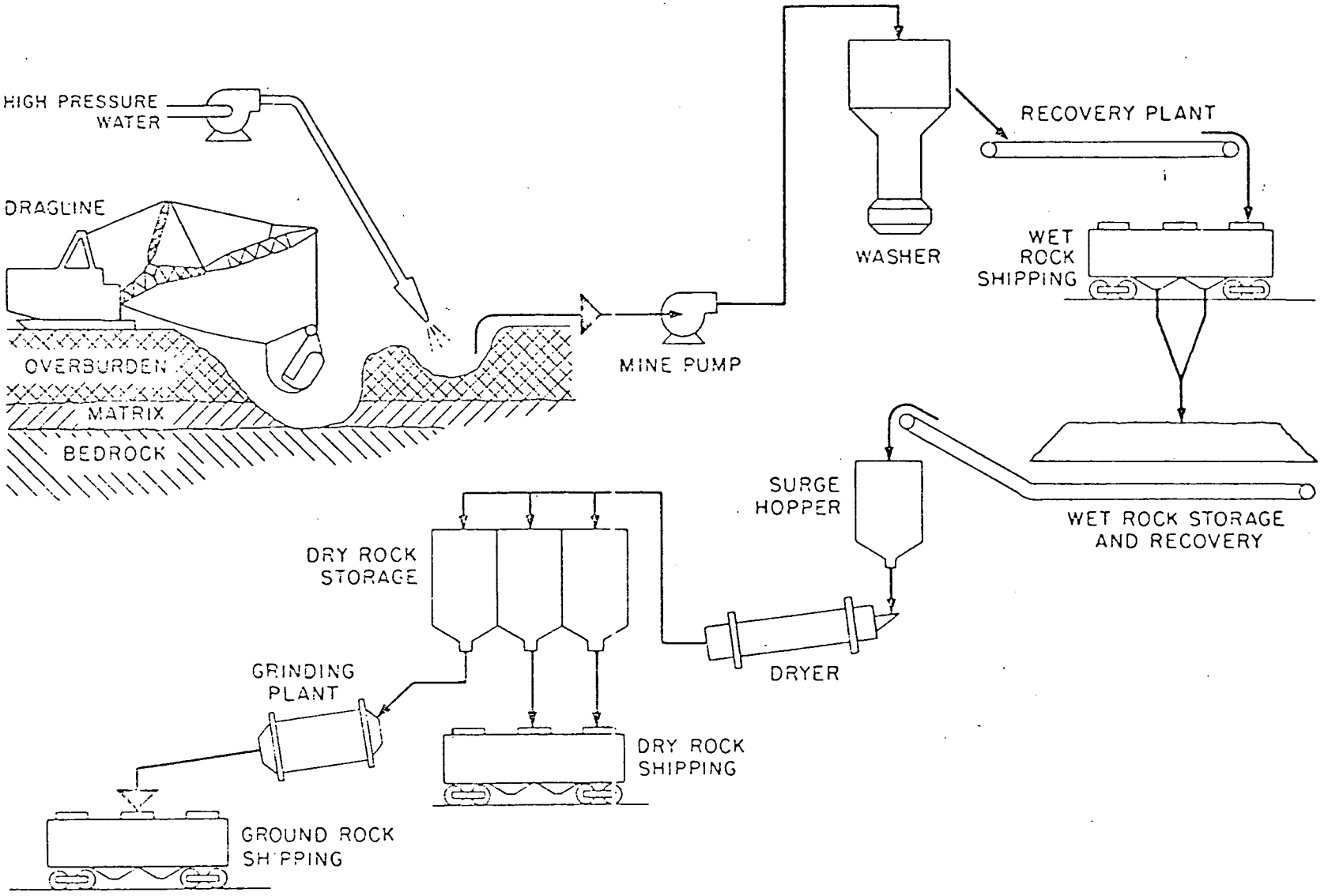

Figure 4.1. Phosphate rock flow sheet.

V. PROCESS OPERATION*

The operations at the Kingsford rock drying plant were monitored from the systems control room by Mr. J. Peoples of EPA. From this vantage point, Mr. Peoples was able to collect data and communicate any upsets or abnormalities during the testing periods to the test crew. Data recorded included recording times, feed and production rates, and other additional information related to the fluid bed dryer and scrubber cleaning operation (Appendix C tabulates this information along with related comments).

Particulate and fluoride tests were conducted at the rock drying plant during the week of February 12, 1973. On the first day, Feb. 12, 1973, no process or sampling problems were encountered while conducting Run 1. However, fuel oil to furnish heat for the dryer was substituted for natural gas due to the fuel shortage.

The raw materials fed to the dryer consisted of a mixture of 60% pebble - 40% concentrate. For the second particulate feed-in materials consisted of 70% pebble - 30% concentrate. One process shutdown occurred during the second run and delayed testing from 11:00 A.M. until 12:50 P.M. due to conveyor belt trouble. The dryer feed for the third run was 100% concentrate.

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*Written and supplied by EPA.

On Wednesday, Feb. 14, at 9:10 A.M. the fluoride tests were begun at Kingsford. A mixture of 60% pebble - 40% concentrate was being processed during runs one (1) and two (2). Test three (3) was completed on Thursday with 100% concentrate being processed under normal conditions. No process upsets interfered with these tests.

The opacity of the Kingsford dryer effluent was difficult to estimate because of the high moisture content.

VI LOCATION OF SAMPLING POINTS

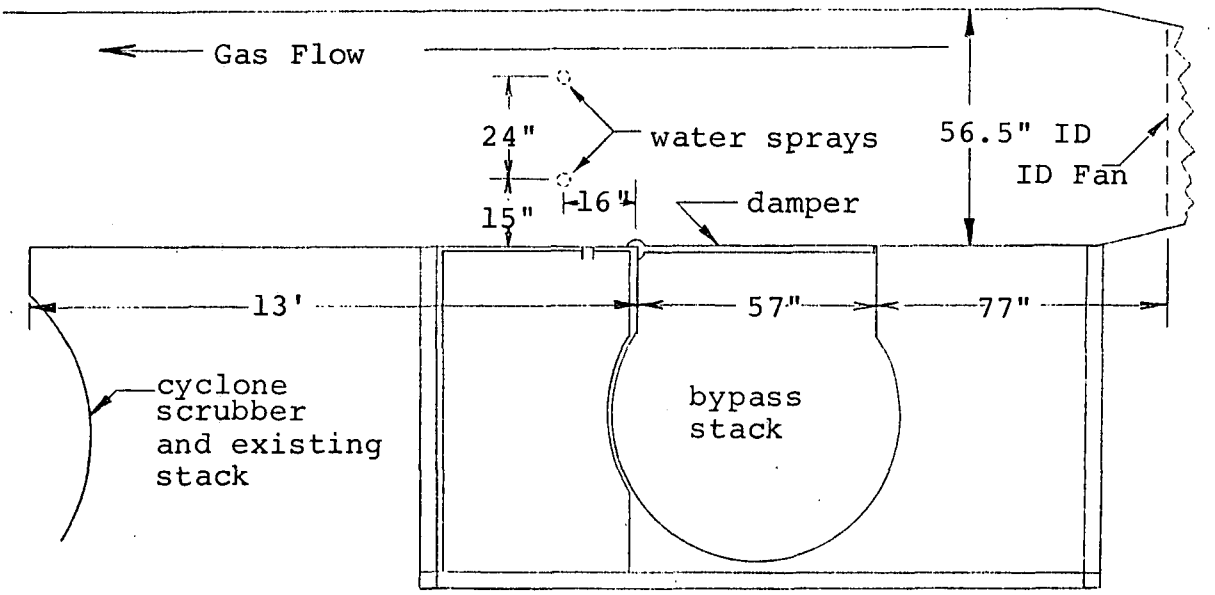
A. Inlet Location

The configuration of the ductwork downstream of the drier as shown in Figure 6.1 made it impossible to choose a section completely free of turbulent effects. The best possible sampling location was chosen at a point nine inches upstream from two vertical water sprays and approximately twelve feet downstream of an induced draft fan.

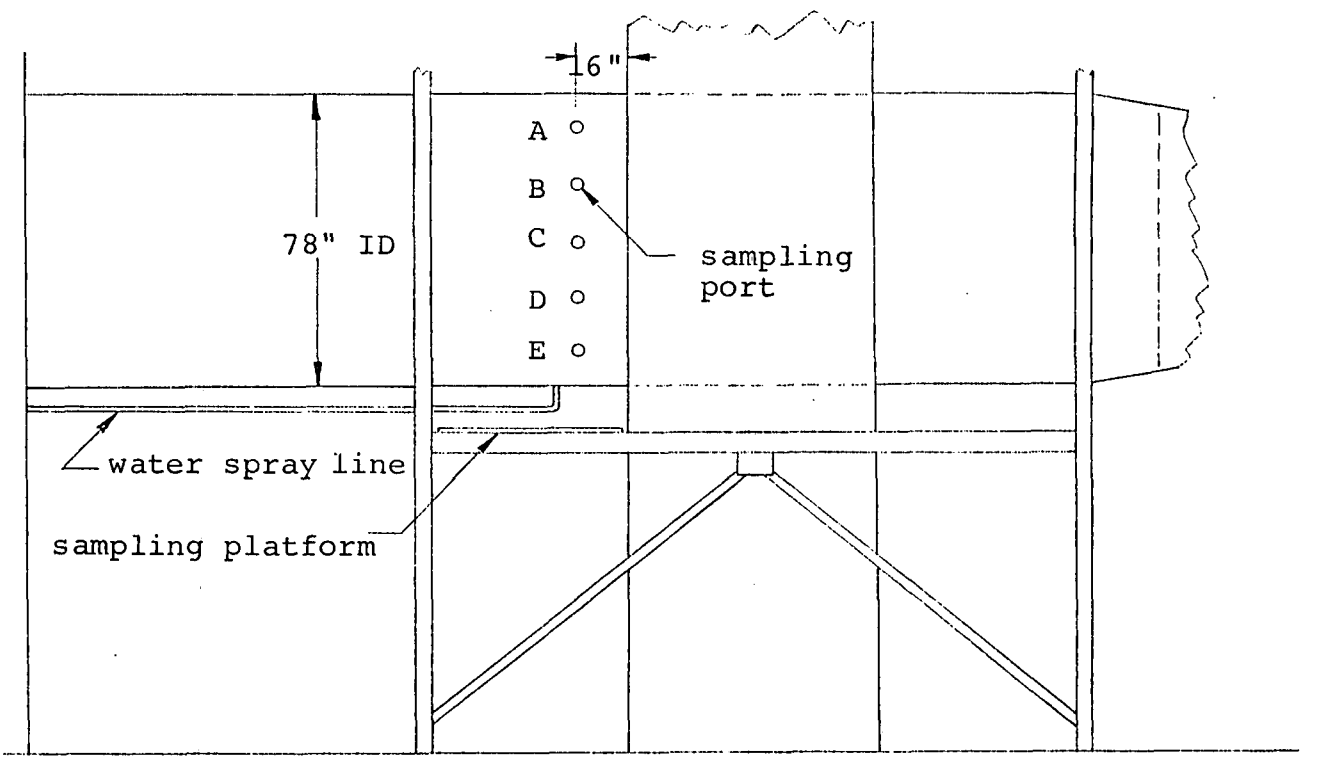
Since this chosen sampling location was less than 0.5 diameters from the water sprays, the maximum number of traverse points was chosen as required by the Federal Register, Vol. 36, No. 247. Figure 1.1, Dimensions of the duct are 79 inches high by 56.5 inches wide. Five ports were decided upon with ten traverse points per port for a total of fifty (50) points as illustrated in Figure 6.2. Each sampling area would then theoretically be 15.8 inches long by 5.65 inches wide, resulting in an aspect ratio of 2.66. Increasing the sampling ports would reduce the aspect ratio but the existing framework around the bypass stack made this impossible, since a minimum of 14 inches was needed from the existing framework to the sampling port.

Actual sampling areas in Figure 6.2 are somewhat smaller than theoretical areas due to a 6 to 7 inch layer of mud on the bottom of the duct. This reduction in area was considered in

Figure 6.1 Dryer Inlet Location

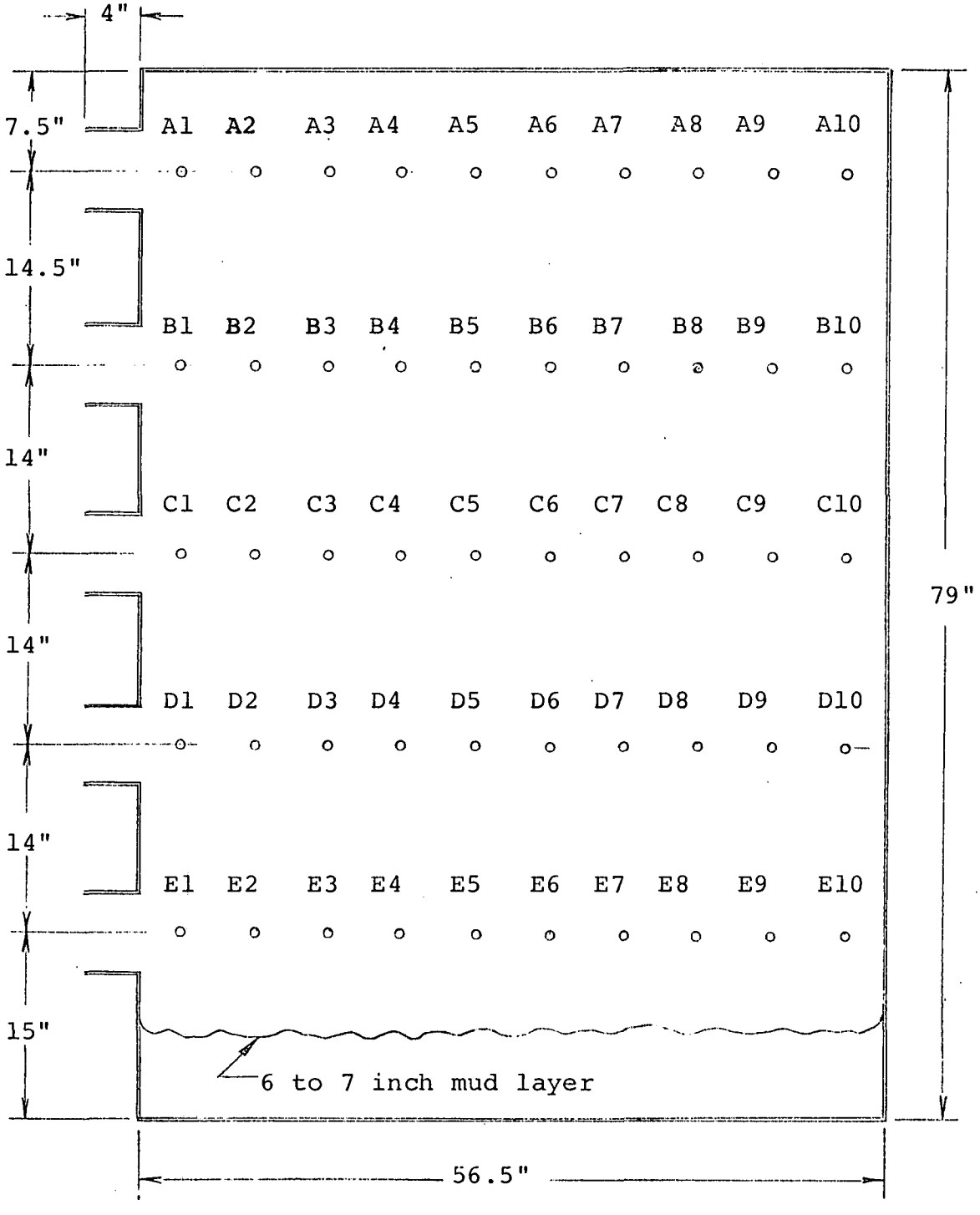


Top View



Elevation

Figure 6.2 Inlet Traverse Points



Looking Down Stream

determining air flow rates. A bypass stack without control equipment is less than one duct diameter upstream from the chosen sampling location and was blocked off by a damper which eliminated air stream turbulence.

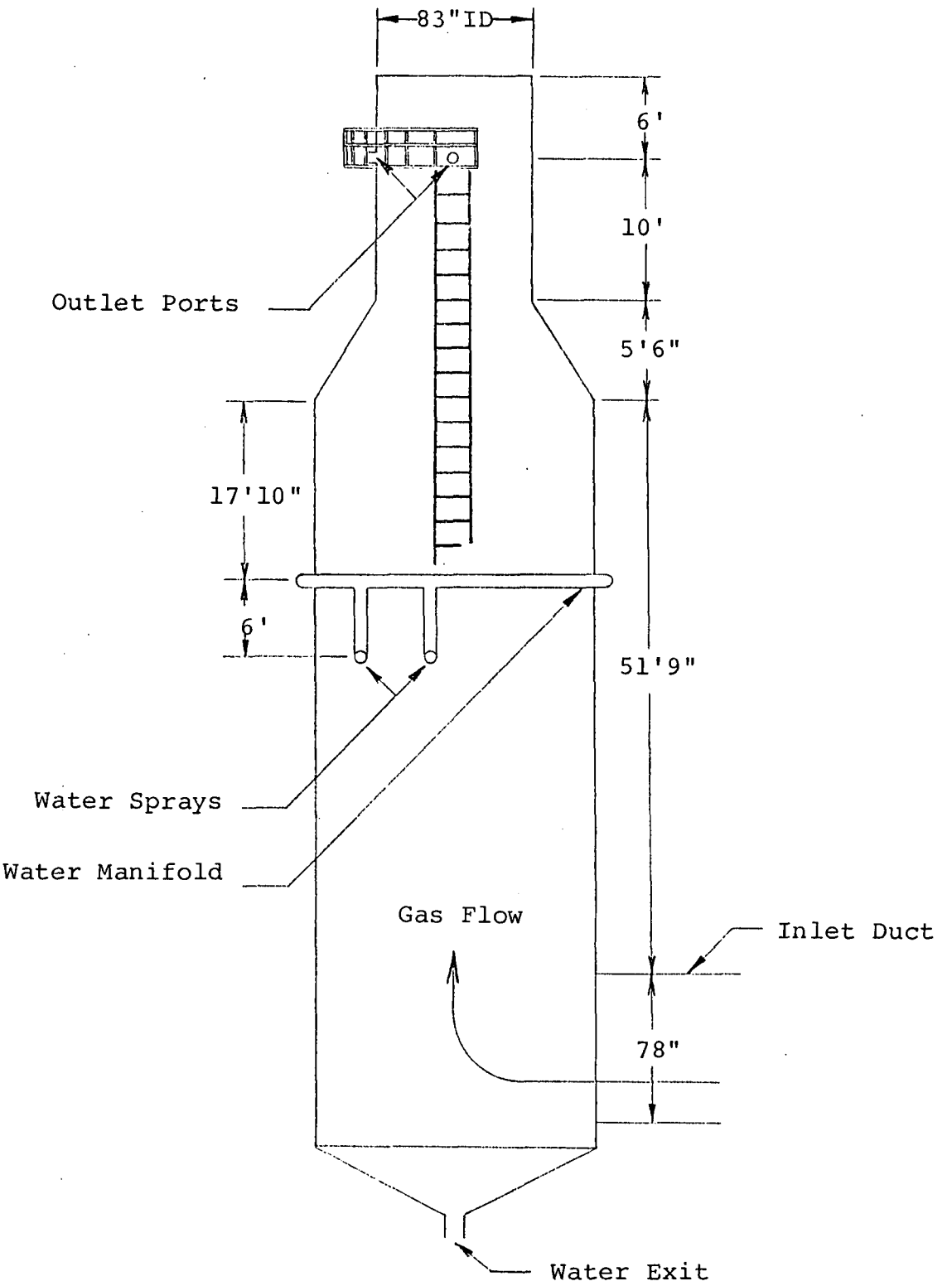
B. Outlet Location

The rock drying operation is equipped with a stack approximately 90 feet high. Two existing ports were found to be satisfactory for emission testing. A diagram of the stack is shown in Figure 6.3 with sampling port and water spray locations. Two sampling locations, 90 degrees apart are needed for vertical stack sampling (Federal Register, Vol. 36, No. 247, Page 24882). When dimensions of the stack were taken, it was noticed the ports were only 80 to 85 degrees apart, which was assumed to be satisfactory.

The stack is 83 inches in diameter at the sampling location. Ports are located 6 ft. below the top of the stack, and 10 ft. above the closest obstruction. Forty-eight sampling points (24 along each diameter) were chosen to satisfy traverse point requirements as specified by the Federal Register. A cross section of the stack is shown in Figure 6.4 with the chosen sampling points.

A portion of the existing railing had to be removed to allow for the sample box traverse. A support platform was designed so the sample box could be overhung over the walk, since the needed sampling width was much greater than the catwalk width of 30 inches.

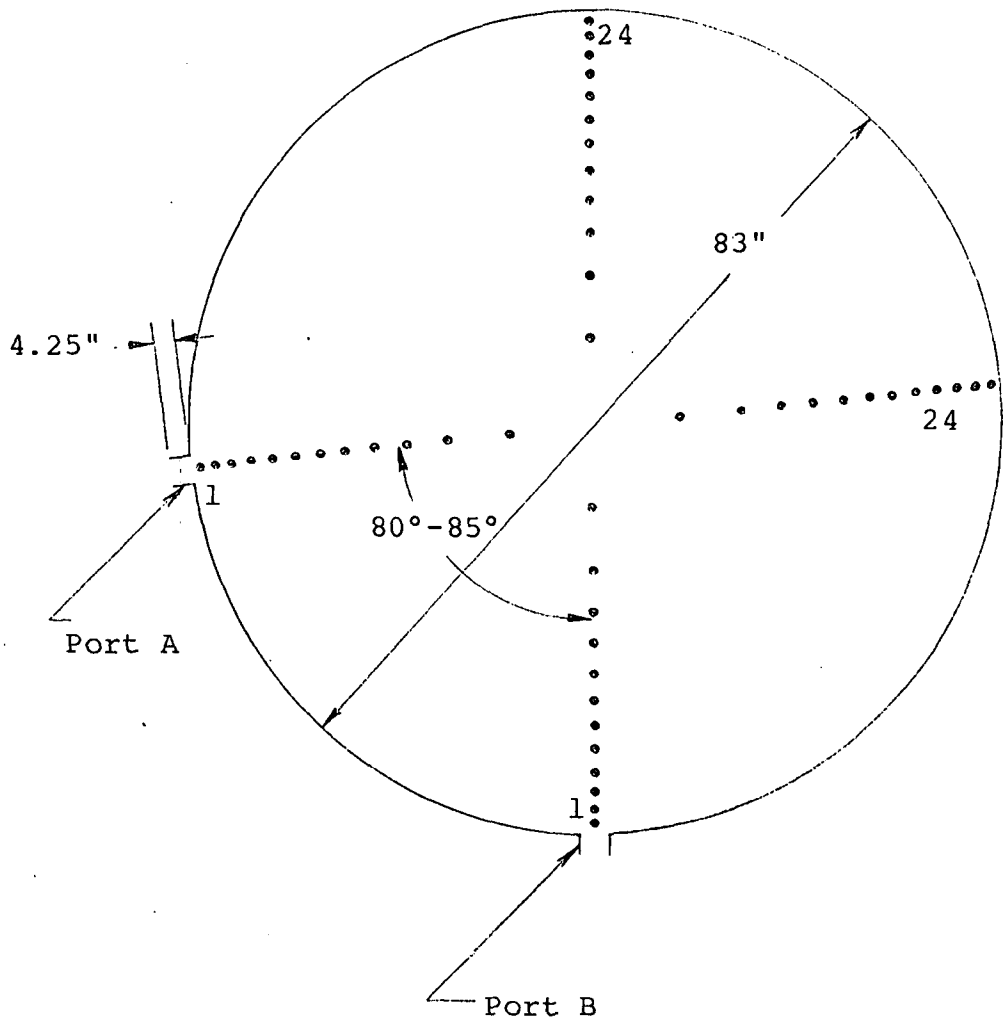
Figure 6.3 Dryer Outlet Location



Elevation

Figure 6.4 Outlet Traverse Points

Traverse Point Locations are Tabulated in Appendix D



Looking Down

VII. SAMPLING PROCEDURES

All particulate and fluoride sampling procedures were selected and approved by EPA prior to field sampling. The contractor performed all testing and sample recovery operations; collected samples were analyzed by EPA personnel. The inlet and outlet sampling crew consisted of two man teams which included a meter and probe technician.

At the inlet site, there was a relatively high positive pressure combined with high humidity air at approximately 165°F. Water condensed in the first two impingers which had to be replaced midway through the test. After each inlet test the total water volume was determined and properly stored for analysis.

The outlet meter box was positioned approximately fifty feet below the sampling site. Since the diameter of the stack was much greater than the width of the catwalk, two different probe lengths reduced the sample box overhang.

Preliminary Traverse and Moisture

A preliminary velocity traverse at the inlet and outlet location determined approximate nozzle sizes and isokinetic sampling conditions. A 0.180 inch I.D. nozzle was used for subsequent inlet testing while the outlet was sampled with a 0.277 inch I.D. nozzle.

The inlet stack gas moisture was determined by a train similar to Figure 4.2, Federal Register, Vol. 36, No. 247, Page 24887. Tests on inlet flue gas indicated values were close to saturation; the outlet gas was assumed to be saturated.

Gas Velocity and Temperature

Velocities were measured at each sampling point across the stack diameter to determine an average value according to procedures described in the Federal Register. Flow rates were calculated from velocities at inlet and outlet stations from which flow continuity was checked. Gas flow temperatures were measured by long stem dial thermometers.

Molecular Weight and Gas Analysis

An integrated sample of the stack gases was collected at the inlet and outlet during each particulate run by pumping flue gas into a Mylar bag at the rate of approximately 0.5 liter per minute. This bag sample was then analyzed on-site with an Orsat apparatus for CO_2, O_2, and CO. Prior to the first test all chemical reagents were changed to assure accurate readings. The molecular weights of the particulate combustion gas ranged from 29.19 to 29.50. A molecular weight of 29.00 was assumed for all fluoride test calculations.

Particulate Sampling

Particulate matter was isokinetically sampled from the drying operation with a train shown in Figure 7.1 Although the Federal Register stipulates a minimum sampling time of 5 minutes for each sampling point, shorter times were used due to the number of traverse points selected.

The train consisted of a stainless steel nozzle, a heated glass probe (a 6 ft. probe was used at the inlet while testing at the outlet was done with 4 ft. and 8 ft. lengths), a heated glass fiber filter, and four impingers connected in series with glass ball joint fittings. The first two impingers contained 100 ml of water each, the third impinger was left empty and approximately 200 grams of preweighed silica gel were placed in the fourth impinger.

In all cases sampling was conducted under isokinetic conditions by continually monitoring the velocity with a pitot tube and adjusting the sampling rate accordingly.

At the inlet site, the first impinger initially containing 100 ml. of water had to be replaced midway during the tests due to the large volume of water which condensed in the impinger. The stack team was very careful so as not to contact the nozzle with the muddy ports and stack walls when changing ports.

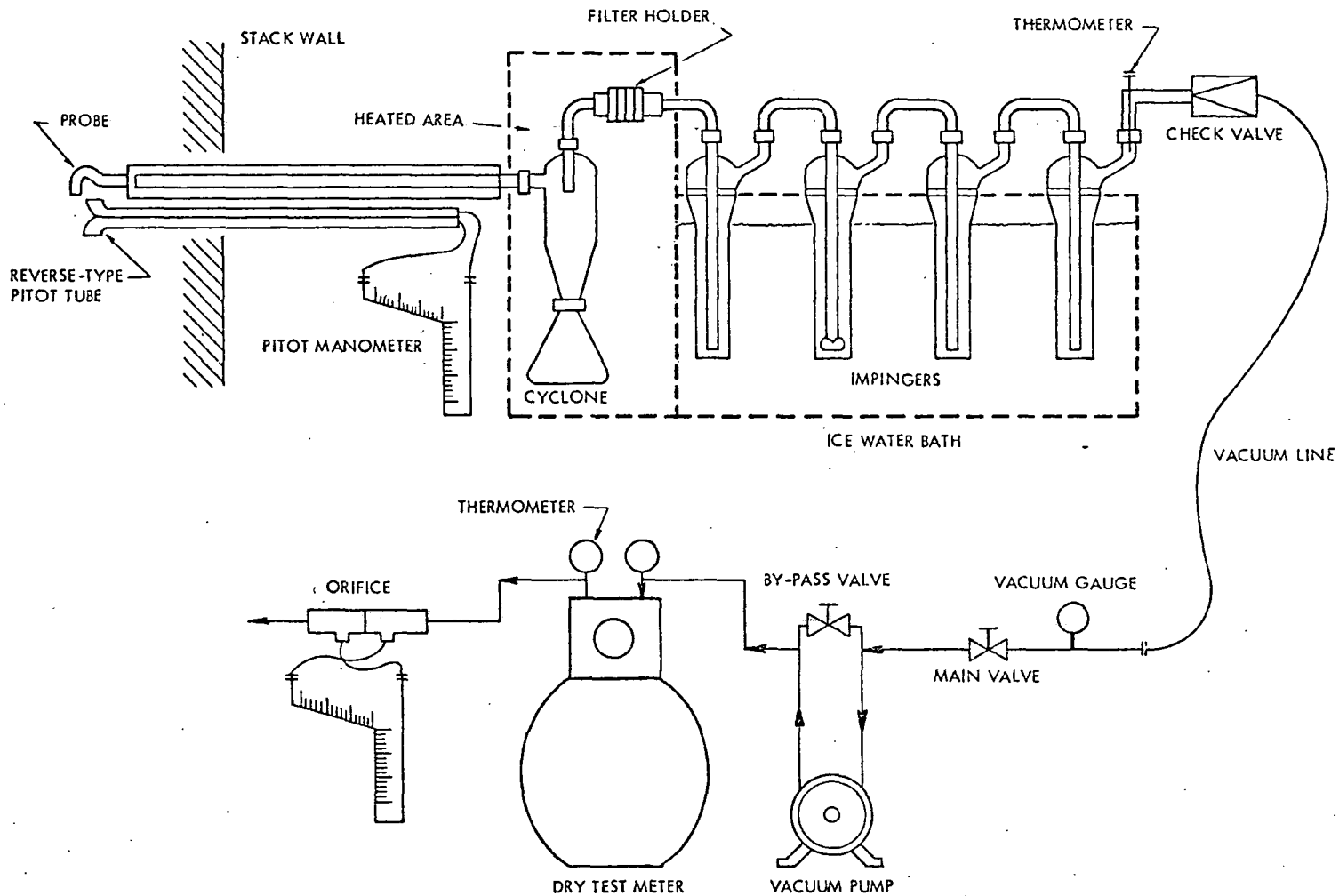


Figure 7.1. Particulate sampling train.

The train cleanup procedure consisted of measuring the water collected and weighing the silica gel to determine moisture content. The water was then poured into a glass jar. The filter was removed and placed in a marked container. The probe and front half of the filter holder were then rinsed with analytical reagent grade acetone and the washings placed in a glass container. The rear half of the train consisting of filter holder, impingers, and connectors was rinsed with distilled water and this water added to the impinger contents. The rear half of the train was then rinsed with acetone and placed in a third sample jar. A portion of the acetone and distilled water used in the sample recovery were set aside and used as blanks for analysis.

Fluoride Sampling

Particulate and gaseous fluorides were withdrawn isokinetically from the phosphate drying operation with the train shown in Figure 7.2.

The design and contents of the four impingers are identical to the particulate train. An unheated filter holder containing a Whatman No. 41 paper filter was placed between the third and fourth impingers to trap particulate fluorides. Contents of the first three impingers, water wash of probe, nozzle, and filter holder were placed in the same container. No acetone wash was required for the fluoride clean up.

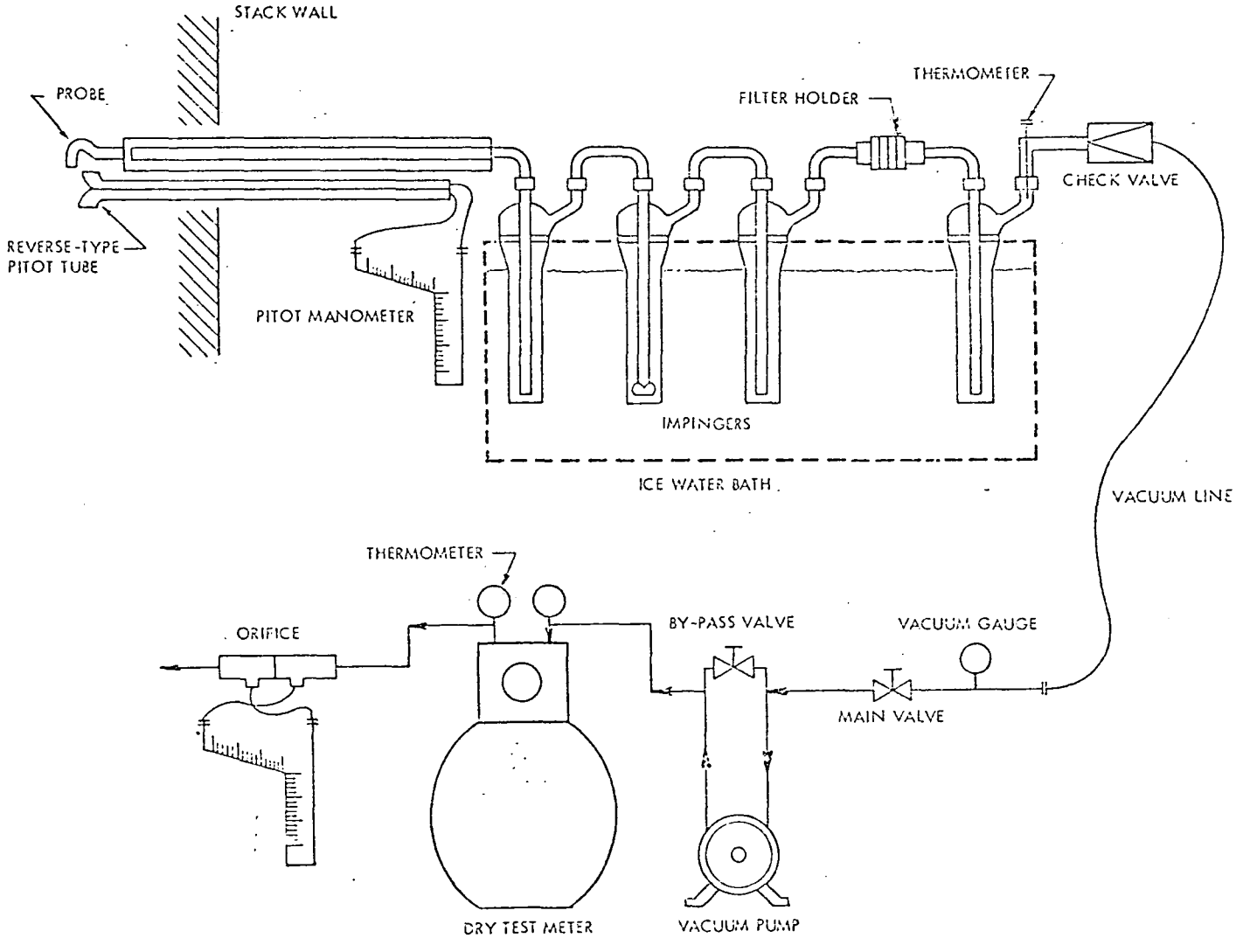


Figure 7.2 Fluoride Sampling Train

During the first fluoride test, the filter exhibited a darkish brown color. Since the Whatman filter was unheated, the exhaust gas constituents must have accounted for the color change. As explained earlier in Section 3, the original pump used at the outlet site sounded as if the vanes were sticking. On the first outlet fluoride test, the flow rate was difficult to maintain for isokinetic sampling (see Appendix D). At high velocities either the filter was starting to plug up or the pump connections could have been loose. In either case, another pump was used for subsequent outlet testing.

Scrubber water along with raw and finished product rock samples were taken during each test. Three scrubber water samples during each test were measured for pH and temperature and identified on the individual bottles.

Sample Storage

All samples were placed in 1000 ml glass and polyethylene containers and marked with EPA identification tags (see Appendix G). The bottles were then put in wooden boxes with styrofoam separators and hand delivered to EPA, North Carolina, after all tests were completed.