EMISSION TESTING OF CALCINER OFF-GASES AT FMC ELEMENTAL PHOSPHORUS PLANT POCATELLO, IDAHO

EMISSION TEST FINAL REPORT

VOLUME I

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GLOSSARY OF TERMS

ACFM - actual cubic feet per minute
Andersen - Andersen Cascade Impactor
CFM - cubic feet per minute
D_j - nominal jet diameter
DSCF - dry standard cubic feet
DSCFM - dry standard cubic feet per minute
DSCMM - dry standard cubic meters per minute
EADS - Environmental Assessment Data System
E_D - equivalent diameters
EERF - Eastern Environmental Radiation Facility
EPA - Environmental Protection Agency
ESED - Emission Standards and Engineering Division
FPEIS - Fine Particle Emissions Information System
GC - gas chromatograph
gr/ACF - grains per actual cubic foot
IERL - Industrial Environmental Research Laboratory
lbs/hr - pounds per hour
M5 - EPA Method 5
MRI - Midwest Research Institute
MSL - mean sea level
ORP - Office of Radiation Programs
PADRE - Particulate Data Reduction
Pb-210 - Lead-210
Po-210 - Polonium-210
QA - quality assurance
Rn - Radon
SASS - Source Assessment Sampling System
TCD - Thermal Conductivity Detection
Th-232 - Thorium-232
SCF - standard cubic feet
SCFM - standard cubic feet per minute
SCMM - standard cubic meters per minute
U-238 - Uranium-238

1. INTRODUCTION

Phosphate rock contains appreciable quantities of uranium and its decay products. The uranium concentration of phosphate rock ranges from about 20 to 200 parts per million (ppm), which is 10 to 100 times higher than the uranium concentration in most natural rocks and soil (2 ppm). The significant radionuclides present in phosphate rock are uranium-238, uranium-234, thorium-230, radium-226, radon-222, lead-210, and polonium-210. These radionuclides may be released to air in particulate form when phosphate rock is handled and processed. In addition, heating of phosphate rock to high temperatures in calciners may volatilize lead-210 and polonium-210 resulting in an enrichment of these radionuclides in the particulates in the off-gas streams.[1-3]

In April 1983, EPA proposed a radionuclide emission standard under CAA for calciners at elemental phosphorus plants (EPA83). However, because the previous emission studies were limited to stack outlet streams and did not include measurement of the radionuclide distribution by particle size, it was determined that information from additional emission testing was needed in developing the final standard. To provide the required information, EPA has conducted additional emission tests for lead-210 and polonium-210 at calciners at three elemental phosphorus plants.

One of the facilities tested as part of this program was a moving grate calciner at the FMC elemental phosphorus plant in Pocatello, Idaho. Since the particulate matter in the off-gas streams at these facilities also contains the radionuclides, the emission testing procedures involved collection of particulate matter from the off-gas streams, and analyses of these samples for their radionuclides content. This report describes the testing conducted at this facility including a detailed description of sample collection and analytical procedures. This report also presents the results of the particulate emission rates and particle size distributions. A separate report, dealing with the

radionuclide measurements and emission rates will be prepared by the Office of Radiation Programs, EPA.

This test facility was selected as one of three representative elemental phosphorus test sites based on criteria established by Midwest Research Institute (MRI) under contract to EPA (Contract No. 68-02-3177, Task No. 26).[4] The emission testing was conducted by the Environmental Operations of TRW (now part of Radian Corporation) under contract to EPA (Contract No. 68-02-3174, Task No. 131). Operating parameters of the calciner and associated control equipment were monitored by an engineer representing MRI. A pre-survey was conducted on September 30, 1983, and the emission sampling was carried out from October 24 to November 1, 1983.

The emission test program was centered around the calciner off-gases from the No. 2 calciner and its emission control equipment. The No. 1 calciner was also tested, but less extensively, in order to verify particulate matter emission rate estimates.

The emission testing program at this site involved five (5) flue gas sampling locations. The emission parameters of front half particulate matter, particle size distribution, volumetric flow rate, moisture, and gas composition were measured. The particulate matter filter catches were collected in a fashion suitable to analyze the particulate matter for associated radionuclides. The process samples collected from each of the calciners included shale feedstock, calcined product, scrubber influent, and scrubber effluent. Figure 2-1 indicates the various sampling locations around the process.

There were no major modifications to the sampling during the test period that required altering the test program as proposed in the site specific test plan (October 15, 1983).[5] There were several process related delays which required extra time on site to accomplish all of the originally planned sampling objectives. There were no weather related delays.

The following test report is divided into seven (7) sections with supplemental appendices in a separate volume. Section 2 presents the summary and conclusions of the test effort. Section 3 discusses the results of the presented data. Section 4 describes the process and associated process information. Section 5 discusses the specifics of the various test locations. Section 6 describes sampling and analytical methods. Section 7 describes the quality assurance (QA) procedures.

2. SUMMARY

The TRW (Radian) source testing team performed particulate matter and particle sizing measurements on the emission control systems of two (2) moving grate phosphate rock calciner units at the FMC elemental phosphorus facility in Pocatello, Idaho. The No. 2 calciner was tested more extensively than the No. 1 calciner because the No. 2 unit had been previously tested for radionuclides by the host facility and the data subsequently was used in annual radionuclide emission rate estimates. Additional limited testing was performed on the scrubber outlet stacks of the No. 1 calciner unit in order to verify emission rate estimates. Figure 2-1 presents a generalized process flow diagram for the subject calciners.

Three (3) different sampling methodologies were employed to measure particulate matter emission rates and particle sizes. These included EPA Method 5, the Andersen Cascade Impactor, and the Source Assessment Sampling System (SASS). The EPA Method 5 sampling train is the reference method for the determination of particulate matter emission rate and included only the front half particulate catch. The Andersen impactor yielded principally particle sizing data. The SASS train was employed at one (1) inlet location for supplemental data and for the collection of a large volume sample for a lung clearance rate study by Pacific Northwest Laboratories under contract to EPA.

2.1 SCOPE

2.1.1 No. 2 Calciner

Figure 2-2 indicates the locations of the flue gas sampling points (A, B, and C) for the No. 2 calciner unit.

The results of the emission tests are used to determine the following items of interest for No. 2 calciner:

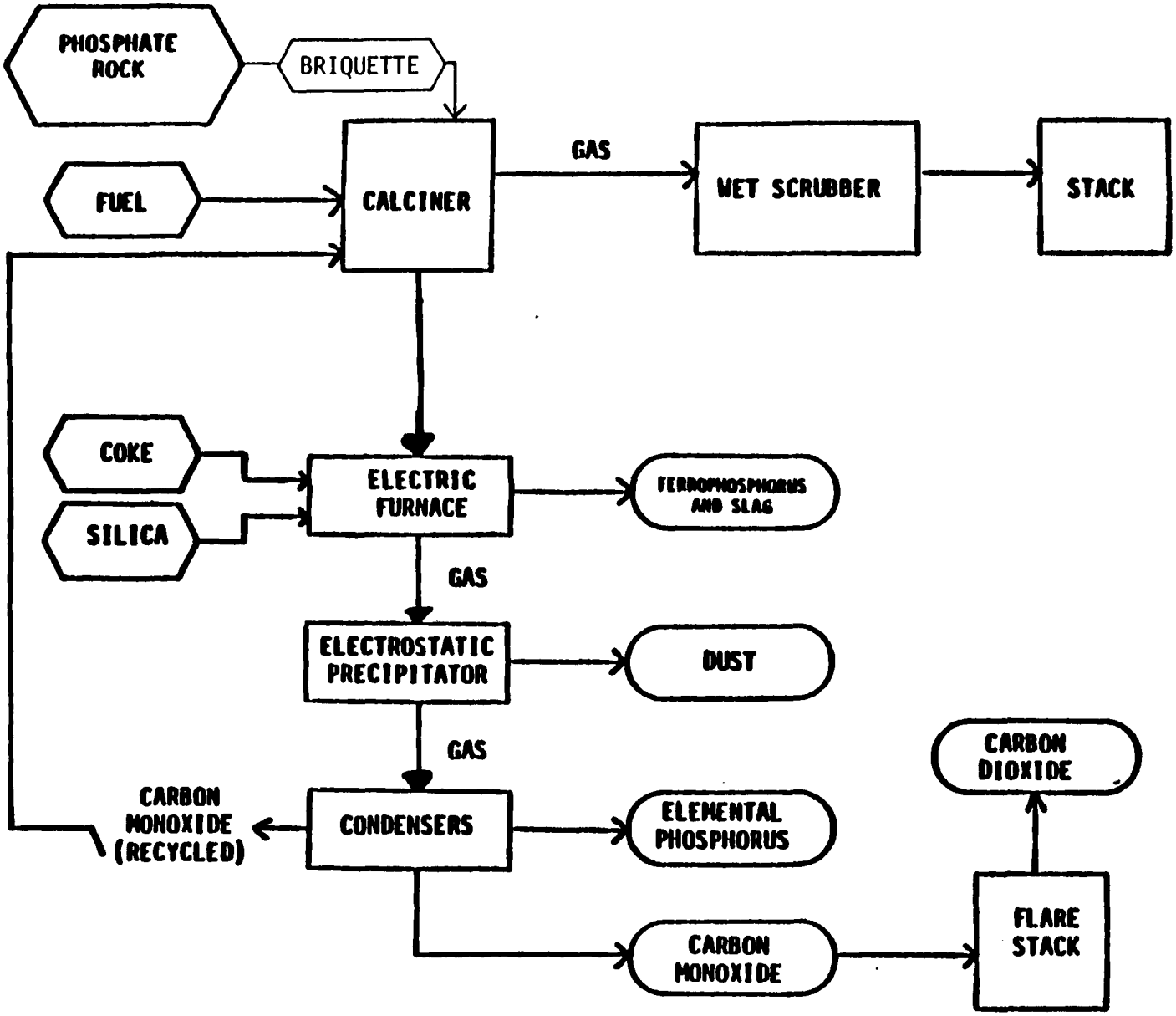

Figure 2-1. Generalized elemental phosphorus flow diagram.[4]

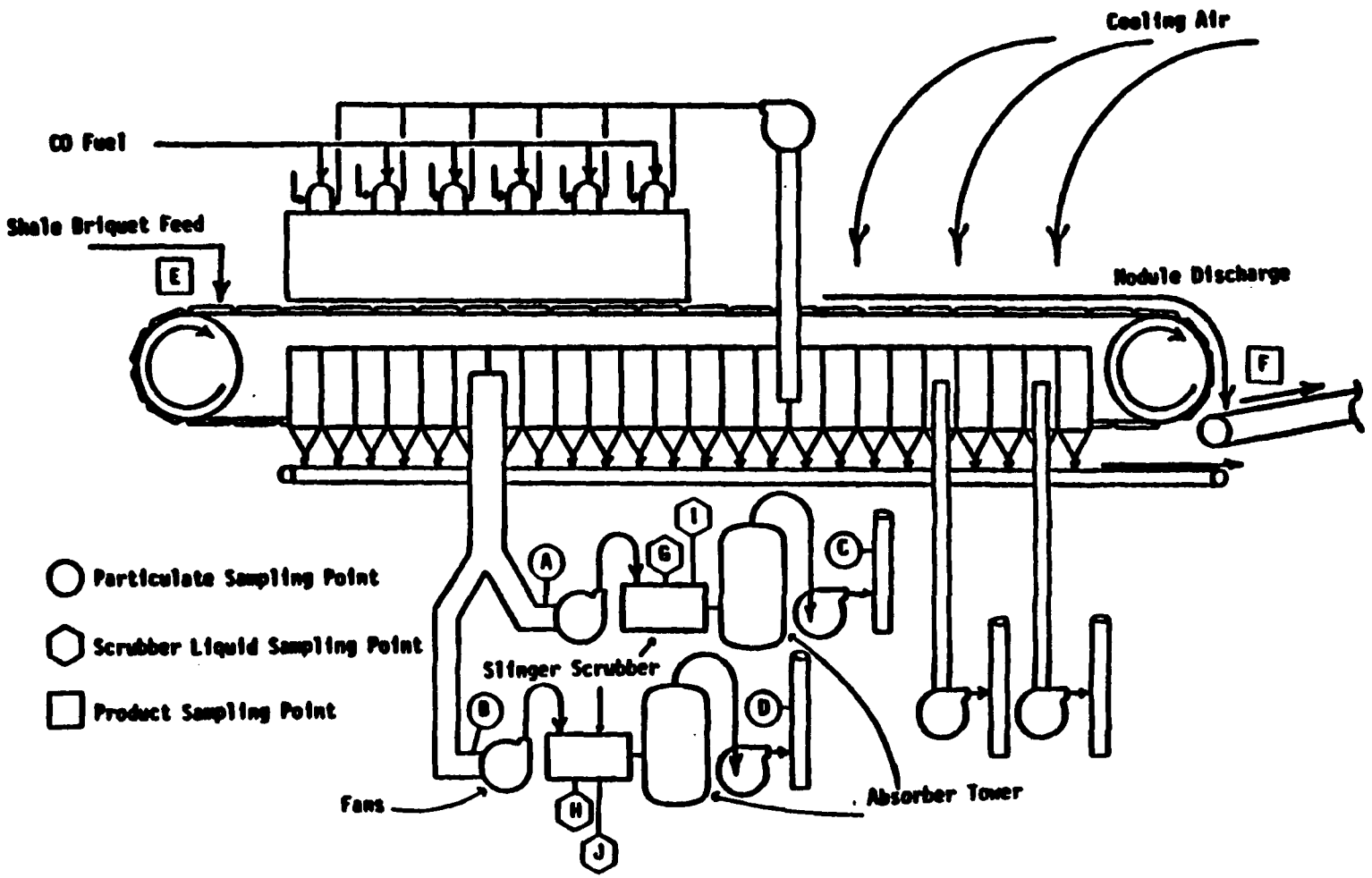

Figure 2-2. Process diagram with emission control points - No. 2 moving grate calciner, FMC Elemental Phosphorus Plant - Pocatello, Idaho.

- the calciner particulate mass rates to the slinger scrubbers;
- the calciner particle size distributions to the slinger scrubbers;
- the controlled particulate emission rates from the outlet of the wet scrubber;
- the controlled particle size distributions at the outlet of the wet scrubber; and
- the radionuclide activity of particulate and particle size samples in order to determine a radionuclide emission rate.

Three (3) series of tests were performed at one of two stack outlets (location C) from the dual scrubber systems serving the No. 2 calciner unit. Location D was not tested due to deteriorated duct conditions. A test series includes one (1) EPA Method 5 test run and a set of Andersen impactor test runs performed simultaneously. Three (3) series of tests were also performed at each of the inlets (A and B in Figure 2-2) to the above slinger scrubbers. An additional fourth EPA Method 5 test run was necessary at these two (2) inlet locations as described in Section 3.

2.1.2 No. 1 Calciner

As indicated in the site specific test plan, the goals of the emission tests on this calciner unit were limited to the following major items:

- the controlled particulate mass emission rates from the slinger scrubbers;
- verification of projected emission rate estimates based upon the No. 2 calciner; and
- verification of controlled particle size emissions.

Three (3) particulate matter tests were performed on both slinger scrubber outlet stacks on the No. 1 calciner (locations K and L). A single Andersen particle sizing test was performed at each of these sampling locations. More extensive emission testing was not performed on the No. 1 calciner due to budget and time constraints of the test program.

2.2 GENERAL SUMMARY OF PARTICULATE MATTER AND PARTICLE SIZE MEASUREMENTS

Tables 2-1 and 2-2 present summaries of the particulate matter and particle size emission test results. The data presented are the averages of the most representative test runs performed at each flue gas sampling location. Certain test runs were omitted from these reported averages because of associated operational difficulties discussed in Section 3.

The following subsections summarizes the particulate matter and particle size results obtained at the five (5) flue gas sampling locations. The EPA Method 5 sampling train data are the primary source of the particulate mass rate data presented. Supplemental SASS data are also presented for location A. The particulate matter size distribution data presented are derived from the Andersen impactor tests with supplemental SASS data at location A. The percentage of sample mass less than or equal to 10 microns (μm) is indicated for the various flue gas sampling locations.

2.2.1 Scrubber Inlet 2-2 (Location A)

The mass rate of particulate matter to the #2-2 slinger scrubber averaged 1,840 lbs/hr for three (3) EPA Method 5 test runs. The range extended from 525 lbs/hr to 3,000 lbs/hr with an increase in mass rate prior to test series no. 3. During this time period, fuel gas was diverted from the No. 1 calciner unit to supplement the No. 2 calciner. The process feed rate was simultaneously increased by a 40 percent factor. The SASS train determination of particulate matter mass rate was 999 lbs/hr prior to the change in process feed rate. The average particulate matter mass rate including the three valid EPA Method 5 and SASS runs was 1,629 lbs/hr.

The Andersen impactor results indicate that the percent of particulate matter less than 10 (μm) may vary in the range of 7.1 to 16 percent at location A depending on process variations. The reported average was 11.7 percent less than or equal to 10 μm.

2.2.2 Scrubber Inlet 2-1 (Location B)

The mass rate of particulate matter to the #2-1 slinger scrubber averaged 217 lbs/hr for three (3) EPA Method 5 test runs. .The range extended from 135 to 313 lbs/hr. The mass rate increased proportionally when the process feed rate was increased prior to test B-3.

Table 2-1. SUMMARY OF PARTICULATE MATTER AND PARTICLE SIZE DISTRIBUTION NO. 2 CALCINER OFF-GASES AT FMC - POCATELLO, IDAHO (10/83)

Location	A	A	A	B	B	C	C
Sample Train	M5	Andersen	SASS	M5	Andersen	M5[b]	Andersen
1. Average emission rate of total particulate (lb/hr)	1,840[c]	477	998[a]	217	100	23.1	20.4
2. Average concentration of particulate (gr/DSCF)	2.98	0.649	1.141	0.454	0.185	0.039	0.024
3. Average particulate sizing data as cumulative percent of total mass at or less than:							
Diameter (micron)[d]							
.63		0.9			1.7		66.4
1.00		1.3	0.6		4.0		74.5
1.25		1.6			4.7		78.7
2.5		2.64			7.1		85.4
3.00		—	1.4		—		—
6.00		6.2			13.1		87.5
10.00		11.7	6.7		22.6		92.9
15.00		19.8			32.5		97.2
20.00		30.4			41.3		97.8

[a]One SASS run.

[b]Excluding first test run from reported averages.

[c]Excludes the first test due to low isokinetics.

[d]Andersen data calculated by PADRE; SASS data calculated manually.

Table 2-2. SUMMARY OF PARTICULATE MATTER AND PARTICLE DISTRIBUTION NO. 1 CALCINER OFF-GASES AT FMC - POCATELLO, IDAHO (10/83)

Location	K	K	L	L
Sample Train	M5[a]	Andersen[b]	M5	Andersen[b]
1. Average emission rate of total particulate (lbs/hr)	69.7	37.5	30.3	51.2
2. Average concentration of particulate (gr/DSCF)	0.122	0.060	0.063	0.112
3. Average particulate sizing data as cumulative percent of total mass at or less than:				
Diameter (micron)				
.63		51.0		39.3
1.00		52.8		48.6
1.25		53.8		49.3
2.50		56.1		50.7
6.00		58.0		51.3
10.00		59.3		57.6
15.00		60.4		65.0
20.00		63.8		71.2

[a]Excludes the first test.

[b]Single Andersen run.

The results of the Andersen tests indicate that the percent of particulate matter less than 10 μm may vary in the range of 7.5 to 31 percent at location B depending on process variations. The average was 22.6 percent of cumulative particulate mass less than or equal to 10 μm.

2.2.3 Scrubber Outlet 2-2 (Location C)

The most representative determination of controlled emission rate for particulate matter at this location was determined to be 23 lbs/hr with 92.9 percent of the particulate matter less than 10 μm.

The average particulate matter mass emission rate was 26 lbs/hr for all three (3) Method 5 test runs performed at this sampling location. The range extended from 17 to 33 lbs/hr. The second and third test runs are thought to be the most accurate on the basis of isokinetic sampling rate and the accuracy of actual particulate matter emissions.

The average result of the Andersen Impactor tests indicate that 92.9 percent of the particulate matter mass was less than or equal to 10 μm. These series of particle sizing tests were nonisokinetic (127.9 to 141.6 percent) as discussed in Section 3. This is expected to result in a positive bias in the reported percentage of small sized particles.

Histograms of the particle size distribution determined by the cascade impactor methodology for the No. 2 calciner are illustrated in Figure 2-3. The total mass rate indicated on each histogram was the average particulate matter mass rate for the EPA Method 5 sampling methodology.

2.2.4 Scrubber Outlet 1-1 (Location K)

The controlled emission rate of particulate matter at this location averaged 70 lbs/hr based upon two (2) EPA Method 5 test runs. The first test run was not included in this average, due to sampling difficulties as noted in Section 3.2.1. This average is expected to include a slight bias owing to the elevated isokinetic rate. These two (2) runs ranged from 52 to 87 lbs/hr. The controlled emission rate of particulate matter using all three test runs averaged 79 lbs/hr and ranged from 52 to 98 lbs/hr.

The single Andersen impactor test performed at this sampling location indicated 59.3 percent of the total particulate mass at 10 μm or smaller.

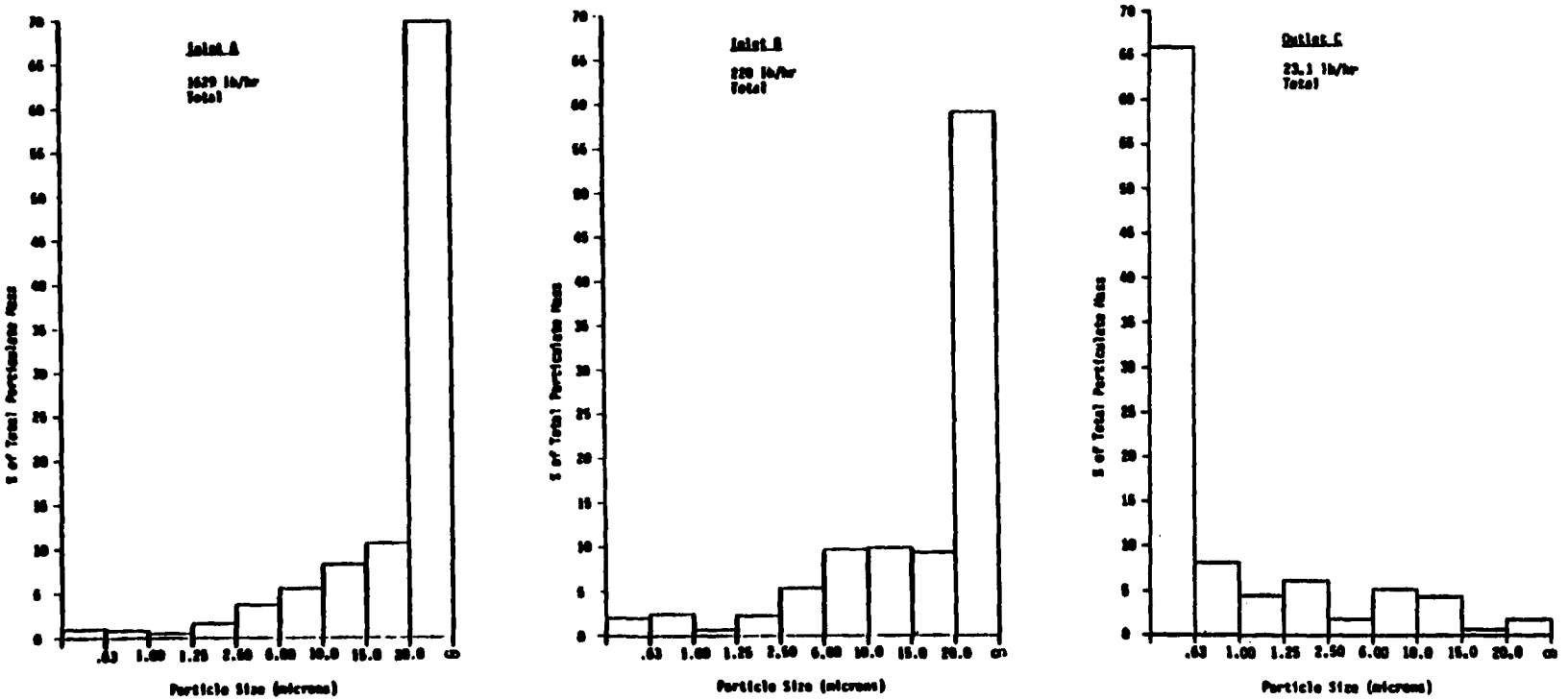

Figure 2-3. Andersen particle size histograms: No. 2 calciner unit, FMC - Pocatello, Idaho (October 1983).

The near ideal isokinetic sampling rate and absence of operational problems indicate that this test is both accurate and representative within the limits of a single measurement.

2.2.5 Scrubber Outlet 1-2 (Location L)

The controlled emission rate of particulate matter at this location averaged 30 lbs/hr for three (3) test runs. The range extended from 25 to 40 lbs/hr. The second and third test runs were corrected to the calculated percentage of moisture at saturation for the average stack temperature of each test run (see Section 3.2.1).

The single Andersen Impactor test run performed at this location was nonisokinetic (168.1 percent) as a result of a decrease in the estimated flue gas flow rate. The percentage of particulate mass at the 10 μm or smaller level was 57.6 percent. However, this test indicated a very similar 10 μm statistic to that of location K, the other outlet stack from the No. 1 calciner unit. A histogram of the particle size distribution is illustrated in Figure 2-4 for the No. 1 calciner scrubber stacks.

The emission data obtained indicate differences between the outlet stack C of the No. 2 calciner unit and outlets K and L of the No. 1 calciner unit. These differences indicate a higher particulate matter emission rate and a lower proportion of particles in the smaller size ranges. Locations K and L were saturated with moisture and contained entrained droplets of moisture. It is uncertain whether the differences in emissions are a result of the differences in construction between the control systems associated with the calciner units, differences in the slinger scrubber operating parameters (e.g., scrubber liquid flow rate), differences in moisture entrainment of the exhaust flue gas, or particulate matter stratification based upon inlet duct construction design.

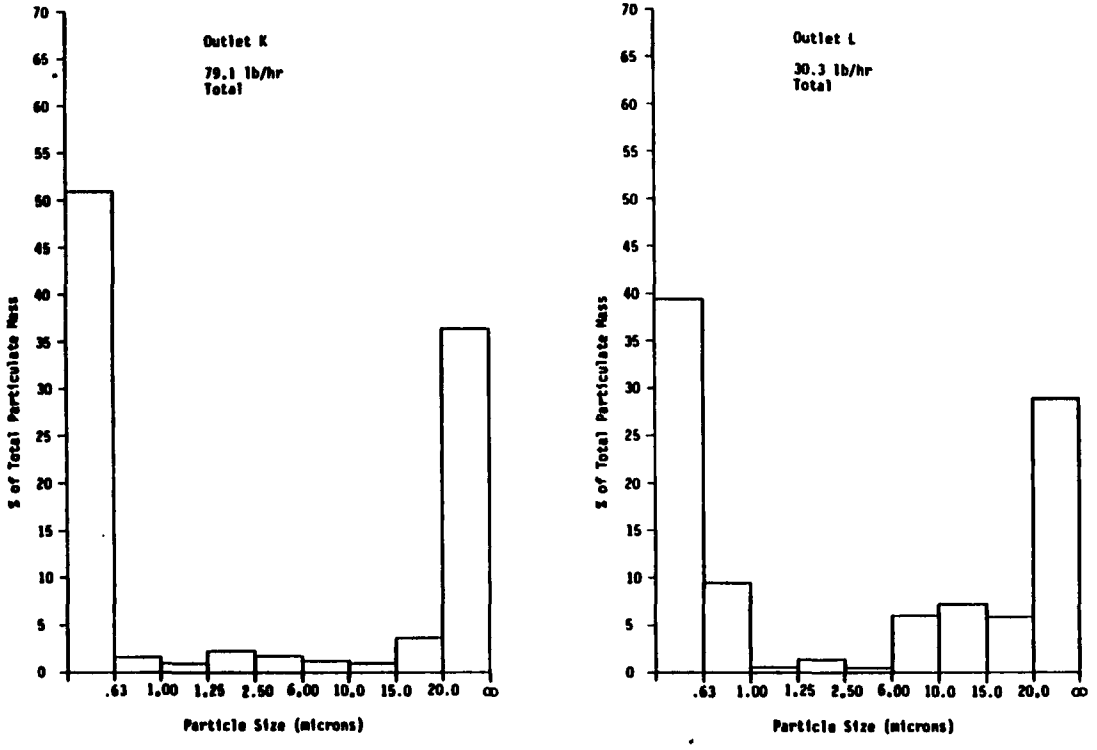

Figure 2-4. Andersen particle size histograms: No. 1 calciner outlet locations, FMC - Pocatello, Idaho (October 1983).

3. DISCUSSION OF RESULTS

This section is presented in two parts. The first is a tabular summary of the various sampling train test parameters which include average particulate matter emission rates and particle size distribution data for each test run at the respective flue gas sampling locations. The second part discusses factors contributing to the collection and validation of the data and subsequent results. Potential factors discussed include sampling train parameters which might affect data validity and plant process factors which might affect the resultant data. An interpretation of particulate matter emissions results is also included.

3.1 TABULAR RESULTS

During the period of October 26 to November 1, 1983, tests were performed for particulate matter emissions at one (1) outlet stack and two (2) slinger scrubber inlets which serve the No. 2 calciner located at the FMC facility in Pocatello, Idaho. Additionally, the two (2) outlet stacks of the No. 1 calciner were tested. This emission testing included both the determination of particulate matter concentration and particle size distribution. Tables 3-1, 3-2, 3-3, and 3-4 present the particulate matter results and test conditions for these emissions tests for the three (3) types of sampling trains employed.

The EPA Method 5 train was employed at all five (5) flue gas sampling locations (A, B, C, K, and L) to determine the particulate matter emission rate. These sampling locations are shown in Figure 2-2. The EPA Method 5 train is the reference method for particulate matter emission rate determination. The Andersen cascade impactor sampling train was used at all flue gas sampling locations to provide particle size distribution data. Additionally, one (1) Source Assessment Sampling System (SASS) test was performed at inlet location A to provide supplemental particulate matter and particle sizing data. The SASS (particle sizing cyclone)

Table 3-1. SUMMARY OF TEST PARAMETERS FOR FMC - POCATELLO, IDAHO WET SCRUBBER INLET DUCT 2-2 (LOCATION A) - NO. 2 CALCINER

Sample Train	M5	Andersen	Andersen	SASS	M5	Andersen	Andersen	M5	Andersen	Andersen	M5
Run No./Location[a]	A-1	A-1-1	A-1-2	A	A-2	A-2-1	A-2-2	A-3	A-3-1	A-3-2	A-4
Run Date	10-27	10-27	10-27	10-27	10-28	10-28	10-28	10-28	10-28	10-28	10-31
Start Time (MST)	1505	1500	1655	0735	0958	1007	1155	1453	1520	1727	0942
Sample Points[b]	Trav.	A-4,C-4	A-8,C-8	C-3	Trav.	A-4,C-4	A-8,C-8	Trav.	A-4,C-4	A-8,C-8	Trav.
Sampling Time (min)	144	30	31	241	144	30	30	144	30	30	144
Meter Volume (DSCF)	56.459	6.240	10.254	797.391	105.234	7.931	8.959	104.379	7.418	8.319	86.924
Nozzle Flow (ACFM)	-	.500	0.808	-	-	0.616	0.676	-	0.639	0.749	-
SASS Cyclone (ACFM)	-	-	-	6.92	-	-	-	-	-	-	-
Stack Flow (ACFM)	179,699	226,311	171,297	235,478	184,607	243,090	160,737	195,712	266,224	184,530	171,310
Stack Flow (DSCFM)	73,038	93,499	69,751	101,988	79,522	103,797	70,648	74,030	101,496	67,328	67,826
Stack Velocity (FPM)	5203	6552	4960	6818	5345	7038	4654	5667	7708	5343	4960
Stack Temp. (F)	549	532	547	483	495	504	476	573	565	611	530
% Isokinetics	55.8[c]	94.7	89.7	83.9	95.4	108.4	87.5	101.7	103.7	85.2	92.4
% Moisture	7.6	7.6	7.6	8.0	7.1	7.0	7.0	11.7	11.7	11.7	10.9
% CO_2	7.6	7.6	7.6	7.6	5.7	5.7	5.7	9.7	9.7	9.7	7.4
% O_2	16.4	16.4	16.4	16.4	17.6	17.6	17.6	15.0	15.0	15.0	16.6
% N_2	76.0	76.0	76.0	76.0	76.7	76.7	76.7	75.4	75.4	75.4	76.0
Particulate Total											
-lb/hr	932	483	430	999	525	569	329	3000	733	318	1994
-gr/dscf	1.488	0.603	0.719	1.141	0.770	0.639	0.543	4.726	0.842	0.550	3.429

[a]Location A - Inlet 2-2 to Slinger scrubber.

[b]See Method 5 traverse diagram.

[c]Low isokinetics.

Table 3-2. SUMMARY OF TEST PARAMETERS FOR FMC - POCATELLO, IDAHO WET SCRUBBER INLET DUCT 2-1 (LOCATION B) - NO. 2 CALCINER

Sample Train	M5	Andersen	Andersen	M5	Andersen	Andersen	M5	Andersen	Andersen	M5
Run No./Location[a]	B-1	B-1-1	B-1-2	B-2	B-2-1	B-2-2	B-3	B-3-1	B-3-2	B-4
Run Date	10-27	10-27	10-27	10-28	10-28	10-28	10-28	10-28	10-28	10-31
Start Time (MST)	1401	1350	1900	1027	1056	1245	1453	1525	1719	0947
Sample Points[b]	Trav.	A-4,C-4	A-8,C-8	Trav.	A-8,C-8	A-4,C-4	Trav.	A-8,C-8	A-4,C-4	Trav.
Sampling Time (min)	144	39	30	144	31	30	144	30	32	144
Meter Volume (DSCF)	89.638	9.151	9.064	92.638	8.904	4.056	84.233	8.262	16.604	86.961
Nozzle Flow (ACFM)	-	0.583	0.738	-	.692	0.310	-	0.737	1.237[d]	-
Stack Flow (ACFM)	141,441	186,991	124,753	141,956	125,224	185,760	141,993	118,991	179,505	142,023
Stack Flow (DSCFM)	58,598	74,724	50,772	60,324	53,380	80,602	53,476	43,947	67,118	55,342
Stack Velocity (FPM)	4095	5414	3612	4110	3625	5378	4111	3445	5197	4112
Stack Temp. (F)	567	568	555	504	507	490	584	605	592	558
% Isokinetics	107.7	133.7	116.4	108.1	101.8	71.4	110.9	118.6	329.1	110.6
% Moisture	4.2[c]	7.5[e]	7.5[e]	7.5	7.5	7.5	11.2	11.2	11.2	9.8
% CO_2	8.6	8.6	8.6	7.2	7.2	7.2	10.3	10.3	10.3	8.2
% O_2	15.8	15.8	15.8	16.7	16.7	16.7	14.6	14.6	14.6	16.2
% N_2	75.6	75.6	75.6	76.2	76.2	76.2	75.1	75.1	75.1	75.6
Particulate Total										
-lb/hr	--[c]	75.6	48.8	134.8	76.0	214	203	83.3	--[d]	313
-gr/dscf	--[c]	0.118	0.112	0.260	0.166	0.310	0.443	0.221	--[d]	0.660

[a]Location B - Inlet 2-1 to Slinger scrubber.

[b]See Method 5 traverse diagram.

[c]Data invalidated because sampling train glassware was broken during test.

[d]Data invalidated because of high flowrate.

[e]Moisture from Run B-2 used in calculations.

Table 3-3. SUMMARY OF TEST PARAMETERS FOR FMC - POCATELLO, IDAHO
OUTLET FROM SLINGER SCRUBBER #2-2 (LOCATION C) - NO. 2 CALCINER

Sample Train	M5	Andersen	M5	Andersen	M5	Andersen
Run No./Location[a]	C-1	C-A-1	C-2	C-A-2	C-3	C-A-3
Run Date	10-26	10-26	10-26	10-26	10-26	10-26
Start Time (MST)	0839	0850	1244	1306	1650	1642
Sample Points[b]	Trav.	Trav.[c]	Trav.	Trav.[c]	Trav.	Trav.[c]
Sampling Time (min)	96	60	96	64	96	60
Meter Volume (DSCF)	49.082	17.227	73.444	19.298	76.736	16.851
Nozzle Flow (ACFM)	-	.435	-	.462	-	.433
Stack Flow (ACFM)	119,993	128,292	121,965	126,249	126,972	133,839
Stack Flow (DSCFM)	77,500	83,263	77,922	80,569	80,018	84,413
Stack Velocity (FPM)	3607	3856	3666	3795	3817	4023
Stack Temp. (F)	142	139	145	145	142	142
% Isokinetics	118.9	134.7	96.6	141.6	98.3	127.9
% Moisture	14.6	14.6	15.1	15.1	16.5	16.5
% CO_2	5.4	5.4	6.3	6.3	6.6	6.6
% O_2	17.7	17.7	16.9	16.9	16.9	16.9
% N_2	76.9	76.9	76.8	76.8	76.5	76.5
Particulate Total						
-lb/hr	32.9	9.44	26.4	24.1	19.8	16.7
-gr/dscf	0.049	0.013	0.039	0.035	0.029	0.023

[a]Location C - Scrubber Stack Outlet 2-2.

[b]See Method 5 and Andersen traverse diagrams.

[c]Four point traverses at 15% and 85% of ID of both ports.

Table 3-4. SUMMARY OF TEST PARAMETERS FOR FMC - POCATELLO, IDAHO OUTLET STACKS FROM SLINGER SCRUBBER #1-1 AND #1-2 (LOCATIONS K AND L) - NO. 1 CALCINER

Sample Train	Andersen	M5	M5	M5	Andersen	M5	M5	M5
Run No./Location[a]	K-A-1	K-1	K-2	K-3	L-A-1	L-1	L-2	L-3
Run Date	10-31	11-1	11-1	11-1	10-31	10-31	11-1	11-1
Start Time (MST)	1703	0953	1605	2206	1526	1828	0942	1524
Sample Points[b]	Trav.[c]	Trav.	Trav.	Trav.	Trav.[c]	Trav.	Trav.	Trav.
Sampling Time (min)	61	99	96	96	60	96	96	96
Meter Volume (DSCF)	13.179	49.245	43.907	46.689	14.798	34.610	36.822	39.632
Nozzle Flow (ACFM)	0.342	-	-	-	0.393	-	-	-
Stack Flow (ACFM)	120,701	117,224	110,214	113,689	88,690	86,073	92,481	99.081
Stack Flow (DSCFM)	73,338	71,236	65,337	67,991	53,288	51,772	55,808	59,857
Stack Velocity (FPM)	4097	3978	3741	3858	3010	2921	3139	3363
Stack Temp. (F)	142	142	138	137	138	136	136	136
% Isokinetics	107.0	112.7	113.0	115.4	168.1	103.2	101.8	102.2
% Moisture	19.4	19.3	21.8	21.3[d]	20.6	20.6	20.7[d]	20.7[d]
% CO_2	8.3	8.3	7.9	6.9	7.2	7.2	7.5	7.7
% O_2	16.1	16.1	16.2	17.0	16.8	16.8	16.8	16.7
% N_2	75.6	75.6	75.9	76.2	76.0	76.0	75.8	75.6
Particulate Total								
-lb/hr	37.5	97.9[e]	87.0	52.4	51.2	25.9	39.6	25.4
-gr/dscf	0.060	0.160[e]	0.155	0.090	0.112	0.058	0.082	0.049

[a]Location K - Scrubber Stack Outlet 1-1; Location L - Scrubber Stack Outlet 1-2.

[b]See Method 5 and Andersen traverse diagrams.

[c]Four point traverses at 15% and 85% of ID of both ports.

[d]Calculated percent moisture due to saturation (i.e. entrained moisture).

[e]Reported value potentially biased high.

test provided a large volume sample in four (4) discrete size fractions for subsequent analysis for an associated lung clearance rate study.

The No. 2 calciner particle sizing results to the slinger scrubber obtained using the Andersen impactor are summarized in Table 3-5. Table 3-6 presents the controlled particle sizing results from the slinger scrubber outlet stacks. The reported data for both tables were calculated using the Particulate Data Reduction (PADRE) program. The data are presented in terms of the cumulative percent mass of the total particulate matter sample which is smaller than, or equal to the indicated particle size. The standard diameters indicated in the table are derived from Mercer's aerodynamic impaction model which correlates with the inertial impaction mode of operation used by the Andersen cascade impactor.

The SASS train test results at location A for the particulate matter are presented in terms of pounds-per-hour (lbs/hr) (see Table 2-1). The particle sizing data are shown as cumulative percentages of the particulate matter sample which are less than or equal to the particle diameters of 1 μm, 3 μm, and 10 μm (see Table 2-1). The particle size fractions are determined by the design of the SASS cyclone system and are equal to the lower cut-off points of the three cyclones.

3.2 SAMPLING CONSIDERATIONS

3.2.1 Sampling Difficulties

Upon performing preliminary flue gas velocity traverses at the two (2) scrubber inlets from the No. 2 calciner (locations A and B), it was determined that the velocity gradient across the ducts was too great to yield an acceptable isokinetic sampling rate using a single Andersen impactor for all four (4) traverse points (see Section 5). Therefore, one Andersen impactor set-up was used for the two (2) sampling points closest to the port openings and a separate Andersen impactor set-up was used for sampling the selected points nearer the far wall of the duct. This approach was employed at both inlet locations. The Andersen sampling points denoted in various tables refer to the selected corresponding EPA Method 5 sampling points.

A moisture problem was encountered at the No. 1 calciner scrubber outlet locations (K and L). These flue gas streams were saturated and contained entrained droplets of moisture. This required that a moisture correction procedure be applied to the EPA Method 5 and Andersen impactor

Table 3-5. SUMMARY OF CALCINER OFF-GAS PARTICLE SIZING RESULTS: LOCATIONS A AND B - NO. 2 CALCINER AT FMC - POCATELLO, IDAHO

Run No./Location[b]	A-1-2[c]	A-2-2	A-3-2		A-1-1[c]	A-2-1	A-3-1		B-1-1	B-2-2		B-1-2	B-2-1	B-3-1	
Run Date	10-27	10-28	10-28		10-27	10-28	10-28		10-27	10-28		10-27	10-28	10-28	
Start Time (MST)	1655	1155	1727		1500	1007	1520		1350	1245		1900	1056	1525	
Sample Points[d]	A4,C4	A4,C4	A4,C4		A8,C8	A8,C8	A8,C8		A4,C4	A4,C4		A8,C8	A8,C8	A8,C8	
Total lb/hr Emissions	430.28	329.08	317.78		483.24	568.77	732.52		75.63	214.35		48.80	76.01	83.34	
Standard Diameters[a]	Cumulative percent of total mass below standard diameters														
				Average				Average			Average				Average
0.63	.8	.8	2.0	1.2	.5	.5	.8	.6	-	-	-	4.4	2.5	3.2	3.4
1.00	1.2	1.2	2.7	1.7	.6	.7	1.1	.8	5.4	1.1	3.2	5.9	4.0	4.4	4.8
1.25	1.6	1.5	3.1	2.0	.9	.9	1.5	1.1	6.4	1.3	3.9	6.8	4.9	5.1	5.6
2.50	2.7	2.3	4.6	3.2	1.4	1.6	3.1	2.0	9.5	2.5	6.0	9.3	7.7	7.8	8.3
6.00	6.7	6.4	8.8	7.3	3.4	4.3	7.9	5.2	17.9	4.5	11.2	16.9	14.4	13.8	15.1
10.00	13.2	16.0	13.2	14.1	7.1	9.2	11.4	9.3	29.4	7.5	18.4	26.8	31.1	22.8	26.9
15.00	22.2	27.2	20.5	23.3	14.4	16.8	17.5	16.3	41.0	11.6	26.3	36.9	46.4	32.6	38.6
20.00	33.8	38.9	31.4	34.7	23.5	27.4	27.3	26.0	51.2	15.0	33.1	47.4	58.0	43.3	49.5

[a] Diameter as defined by Mercers Aerodynamic Impaction Method.

[b] Location A - Inlet 2-2 to Slinger scrubber; Location B - Inlet 2-1 to Slinger scrubber.

[c] SASS train operated at traverse point C-3 of Duct A yielded 6.70% ≤10 microns; 1.38% ≤3 microns; and 0.57% ≤1 micron.

[d] See Method 5 traverse diagram.

Table 3-6. SUMMARY OF CONTROLLED PARTICLE SIZING RESULTS AT FMC - POCATELLO, IDAHO SLINGER SCRUBBER OUTLET SAMPLING LOCATIONS

Run No./Location[b]	C-1	C-2	C-3		K	L
Run Date	10-26	10-26	10-26		10-31	10-31
Start Time (MST)	0850	1306	1642		1703	1526
Sampling Points[c]	Trav.[c]	Trav.[c]	Trav.[c]		Trav.[c]	Trav.[c]
Total lb/hr Emissions	9.44	24.11	16.69		37.52	51.25
Standard Diameters[a]	Cumulative percent of total mass below standard diameter					
				Average		
0.63	7.2	72.5	60.4	46.7	51.0	39.3
1.00	31.6	79.0	70.0	60.2	52.8	48.6
1.25	52.7	81.9	75.6	70.1	53.8	49.3
2.50	80.5	88.5	82.3	83.8	56.1	50.7
6.00	87.1	91.7	83.3	87.4	58.0	51.3
10.00	92.7	93.3	92.6	92.9	59.3	57.6
15.00	96.7	94.6	99.8	97.0	60.4	65.0
20.00	98.6	95.6	100.0	98.1	63.8	71.2

[a]Diameter as defined by Mercers Aerodynamic Impaction Method.

[b]Location C - Scrubber Stack Outlet 2-2
Location K - Scrubber Stack Outlet 1-1
Location L - Scrubber Stack Outlet 1-2.

[c]See Andersen four-point traverse diagram.

data obtained at these locations. Without the application of this correction, the emission rate data obtained would be systematically low because of an incorrect assumption that all of the moisture condensed or absorbed in the sampling train impingers entered the sampling system in the gas phase. The percent moisture corresponding to the saturation level was calculated using the average stack gas temperature and barometric pressure for each test run (i.e., assuming the flue gas dewpoint equal to the average stack temperature). The corrected moisture volume was entered into the computer for calculating particulate matter concentrations, emissions rates, sampling train isokinetics, and sampling train flow rates (ACFM).

Additional sampling problems were encountered at location K. Extensive lengths of electrical cords had to be used to supply power to operate the sampling trains. The resulting inadequate power service resulted in inadequate heat control in the sampling probes and heated boxes which house the Method 5 filter assembly. The sample box operating temperatures dropped below optimum during particulate matter sampling run #K-1; as a result moisture was accumulated in the filter assembly.

3.2.2 Sampling Train Isokinetics and Flow Rates

The proposed limits for isokinetic sampling in this task were 100±10 percent for EPA Method 5 sampling and 100±20 percent for the Andersen impactor and SASS trains. These limits were maintained with partial success as a result of changing process flow rates (see Section 4).

3.2.2.1 Location A. Isokinetic sampling rates ranging from 92.4 to 101.7 percent were obtained for the EPA Method 5 test runs performed at location A (No. 2-2 scrubber inlet). This excludes the first Method 5 test which was run at a low isokinetic sampling rate as a result of an incorrect nozzle size selection. The remaining test runs reported complied with the proposed Method 5 limits. A fourth EPA Method 5 run was performed due to the low isokinetic rate of the first particulate matter sampling run.

Isokinetic sampling rates ranging from 85.2 to 108.4 percent were obtained for the Andersen impactor test runs performed at location A. All of the test runs performed were declared valid as all test runs met the proposed limits. The Andersen impactor volumetric flow rates ranged from 0.500 to 0.808 ACFM (actual cubic feet-per-minute) for these same

test runs. The published range of calibration for the Andersen impactor is 0.25 to 0.75 CFM.[6] It has been reported that a volumetric sample gas flow rate in the range of 0.25 to 0.50 CFM is optimum for "hard" aerosols using the Andersen Mark III Impactor.[7] All particle size measurement runs met this criteria with the exception of a single test run which exceeded the flow rate criteria slightly. The flow rate (0.808 ACFM) was extrapolated slightly beyond the upper range limit to preserve the data.

Table 3-5 presented the Andersen particle sizing results separately for inlet locations A and B. In this table, the data are grouped by sampling points and averaged. Table 2-1 shows the average of these two sample points as an overall particle size distribution for flue gas sampling locations A and B. By comparing the values presented in these two tables, it is evident that there was an appreciable degree of particulate matter stratification in the process duct work. Inlet ducts 2-1 (location B) and 2-2 (location A) diverge from a common feed duct with duct 2-1(B) exiting at a higher physical elevation than duct 2-2(A). The corresponding particulate mass rates display an order of magnitude difference.

Furthermore, the more elevated duct, 2-1(B), shows a higher proportion of the smaller particle sizes than the lower duct. A comparison of the percentage of the particulate mass which is smaller or equal to 10 μm shows only 10.2 percent in this size range at inlet location A, whereas for inlet location B, this size range accounted for 22.6 percent of the particulate emissions. Both of these observations are consistent with the normal trends of stratification in horizontal ducts. The force of gravity causes heavier particulate matter to concentrate in the lower region of the duct, while smaller sized particles remain entrained in the upper region of the duct.

The Andersen impactor results indicate that the percent of particulate matter less than 10 μm may vary in the range of 7.1 to 16 percent at location A depending on process variations. The reported average was 11.7 percent less than or equal to 10 μm. Test runs A-1 and A-2 yielded closely matching particle size distributions for both sampling points. However, an increase in all size fractions below 6.0 μm is shown for both sampling point for the third particle sizing

test series (A-3). This appears to be a result of the increase in process feed rate, which occurred immediately prior to the third test series at locations A and B. This may possibly indicate that the higher process rate produces larger amounts of small particles. No similar effect is apparent from the data for the data collected at location B to verify this hypothesis.

An isokinetic sampling rate of 83.9 percent was obtained for the single SASS test run performed at location A. This complied with the proposed limits. The SASS cyclone flow rate for this test run was 6.92 ACFM compared to the ideal value of 6.50 ACFM (0.184 ACMM) in the cyclones which are maintained at 400°F.[8] This flow rate determines the particle size cutpoints of the three cyclones. Considering the limited adjustability of the SASS sampling flow system, this SASS test run was valid and the resulting data accurate within the limits of the sampling methodology.

The SASS test run at this location sampled all four (4) sampling points represented by the pair of Andersen test runs. The SASS particle size results show acceptable agreement with the Andersen data for this sampling location. A comparison with the average of the first and second test series is appropriate since the process feed rate was increased thereafter. The SASS train indicated a percentage of particulate mass of 6.7 percent for the particulate matter less than or equal to 10 μm while the Andersen yielded 10.2 percent. For sizes less than or equal to 3 μm, the SASS data indicated 1.4 percent while the Andersen results indicate 2.5 percent. For sizes less than or equal to 1.0 μm, the SASS data indicated 0.6 percent while the Andersen indicated 0.9 percent.

3.2.2.2 Location B. Isokinetic sampling rates ranging from 107.7 to 110.9 percent were obtained for the four EPA Method 5 test runs performed at location B (No. 2-1 scrubber inlet). A total of four particulate matter test runs were performed at this location. The fourth sampling run was added due to a glassware break discovered during the initial sampling run. The particulate matter and moisture data gathered during this run were therefore not reported. Two of the reported EPA Method 5 runs at this location exceeded the proposed upper isokinetic sampling rate limit fractionally. The resultant minor systematic error would

bias the reported grain loading low for these two sampling runs. The reported EPA Method 5 results are considered both valid and representative of the actual particulate matter concentrations.

Isokinetic sampling rates ranging from 71.4 to 133.7 percent were obtained for the Andersen impactor test runs performed at location B. This excludes one invalid test run (B-3-2) which was nonisokinetic as a result of an error in setting the nomograph. Ideal isokinetics were difficult to obtain as a result of the large velocity gradient across the duct and the limited selection of nozzle sizes. This range of isokinetics is expected to introduce minor systematic errors in the resultant data. Inertial particle sizing devices (such as the Andersen cascade impactor) operate to yield the best data when an isokinetic sample enters the sample nozzle. The sample in the impactor will then be representative of the particle distribution in the gas stream. Particles of different size and mass are then separated by their inertia. Generally, higher than desired isokinetic rates lead to the collection of more smaller sized particles with the total weight of the sample being biased low. Low isokinetic rates lead to collection of less smaller particles with the total weight of the sample being biased high. The impactor volumetric flow rates at location B ranged from 0.310 to 0.738 ACFM for the valid test runs, all within the range of calibration.

NOTE: It was a matter of coincidence that the Andersen particle size test runs at location A yielded better isokinetic sampling rates than location B under the same constraints of duct design and velocity stratification.

3.2.2.3 Location C. Isokinetic sampling rates ranging from 96.6 to 118.9 percent were obtained for the Method 5 test runs performed at location C (No. 2-2 scrubber outlet stack). The first EPA Method 5 test run was 118.9 percent isokinetic. The three (3) Andersen impactor test runs were also nonisokinetic. The nonisokinetic sampling rates were a result of a significant decrease in the stack gas velocity between the preliminary velocity traverse and the start of testing. The sampling rate was corrected after the first EPA Method 5 test run, but the decrease in velocity was not apparent to the Andersen operator as the Andersen sampling probe does not have pitot tubes for measuring stack gas velocity. The reported EPA Method 5 particle matter results would be biased low

for the first sampling run (32.9 lbs/hr). The reported average measurement of 26.4 lbs/hr particulate matter at location C is considered valid and accurate.

The isokinetic sampling rates ranged from 127.9 to 141.6 percent for the Andersen particle sizing runs. This condition is expected to introduce a positive bias in the Andersen-derived particulate matter emission rate and in the reported percentage of smaller sized particles for particle sizing runs at this location. The Andersen impactor volumetric flow rates ranged from 0.433 to 0.462 ACFM, which is in the ideal range for the Andersen.

3.2.2.4 Location K. Isokinetic sampling rates ranging from 112.7 to 115.4 were obtained for the EPA Method 5 sampling performed at location K. This elevated sampling rate resulted from the unexpected extent of moisture saturation of the flue gas.

An isokinetic sampling rate of 107.0 percent was obtained for the single Andersen impactor run performed at location K. The volumetric sample flow rate was 0.342 ACFM for this test run. Both are ideal for the Andersen.

3.2.2.5 Location L. Isokinetic sampling rates were obtained for all three (3) EPA Method 5 test runs performed at location L (the No. 1-2 scrubber outlet stack). The range of isokinetic sampling rates were 101.8 to 103.2 percent.

The single Andersen impactor test run at location L was nonisokinetic (168.1 percent) as a result of the stack gas velocity decreasing considerably from the preliminary traverse value. This will introduce a positive bias into the data for both the Andersen derived particulate emission rate and the determination of smaller size fraction particles.

3.2.3 Process Considerations

There were process equipment failures, process shut-downs, and deviations from steady state operation encountered throughout the series of tests performed. These will be described in detail in Section 4. As a consequence of these conditions, there were unanticipated fluctuations in process parameters such as flue gas velocity and temperature which may have potentially affected the magnitude of the emissions data obtained. As an example, during the testing of the No. 2 calciner unit, there was a 40 percent increase in the briquet feed rate processed between the

second and third inlet test series. There was an apparent corresponding 6 fold increase in the mass rate to slinger scrubber #2-2 as measured at location A. The mass rate to slinger scrubber #2-1 increased <2 fold. These types of process variations experienced during testing may be typical for this plant and therefore the range of resultant particulate concentrations may be representative of normal process operation at this plant.

4. PROCESS DESCRIPTION AND CONTROL EQUIPMENT

The process description and operation section of the test report will be divided into three subsections. The first section describes the overall calcining process and identifies key sample points. The second section defines key process parameters and determines "normal" process rates. The third section presents process data taken during the test and summarizes process interruptions and downtimes.

4.1 PROCESS DESCRIPTION

FMC produces elemental phosphorus from phosphate ore ("shale"). In general terms, the process flow includes briqueting the shale, calcining the briquets to remove organic material and to form heat-hardened nodules, reducing the nodules in an electric furnace, and collecting the elemental phosphorus from the furnace off-gases. The calcining processes tested in this study are discussed in more detail below.

The FMC facility has two moving grate calciners designated as No.1 and No. 2. A schematic overview of the process, which is similar for both systems, is shown in Figure 4-1. Prior to entering the calciner, the ore is formed into briquets using a mechanical process. These briquets are fed by a vibratory feeder onto the pallets of the calciner grate. The calciner is divided into three sections. The first section, the calcining section, has six overflow burners that heat the bed to about 1,200°C (2,200°F). Carbon monoxide (CO) from the electric furnace exhaust gas streams is the primary fuel with natural gas as an auxiliary fuel source. The second section is a cooling section that is open to the atmosphere. The down-draft air from this section is drawn through a fan and used as combustion air in the CO burners. The final section is a cooling section. The cooling air is drawn down through the bed and exhausted out a stack with no air pollution control equipment. The cooled nodules are discharged through a hopper to a conveyor belt and

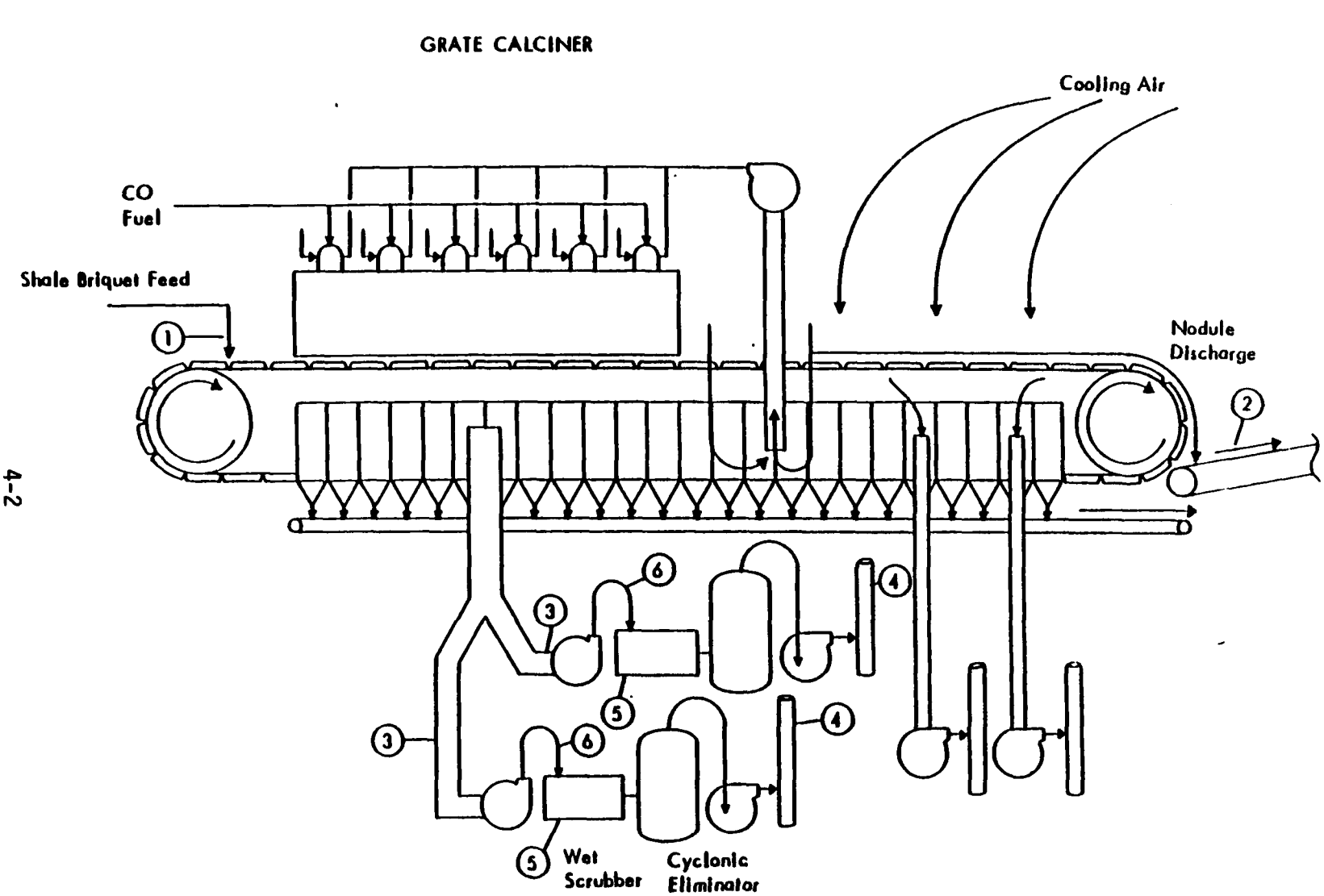

Figure 4-1. Schematic of the FMC calciner.

transported to the furnace area. Feed samples were taken at the discharge of the vibratory feeder (Figure 4-1, Point 1) and product samples were collected from the transport conveyor (Figure 4-1, Point 2).

The exhaust air from the calciner section enters a manifold and splits into two parallel streams. Each gas stream flows along a horizontal duct and into a control system depicted in Figure 4-2. The gases pass sequentially through a fan, a spray quench chamber, a horizontal scrubber, a mist eliminator/spray chamber, a second fan, and out the stack.

The low pressure drop scrubber was designed to control both fluoride and particulate emissions. The horizontal scrubber has a water bed and surface "slingers" that project droplets into the gas stream. The system uses single pass water that enters in the quench chamber and the cyclonic separator; the overflow from the bed in the horizontal scrubber is transported to a settling pond.

The scrubber systems for Unit Nos. 1 and 2 differ in terms of demisting. Unit No. 1 is equipped with Chevron demisting pads. A cyclonic demisting system was installed on Unit No. 2. Technical design information on the scrubber systems (built and installed by FMC) is not available.

During the test series, emissions were sampled at the inlet and outlet of Unit No. 2. The inlets to both scrubber units of Unit No. 2 (No. 2-1 and No. 2-2) were sampled. Due to deterioration of the duct work from scrubber No. 2-1 (scheduled for repair after the testing was performed), the outlet of scrubber No. 2-2 and the two outlets of Unit No. 1 (No. 1-1 and No. 1-2) were sampled. Sampling locations at the inlets and outlets are identified in Figure 4-1.

4.2 KEY OPERATING PARAMETERS

The preliminary test plan and conversations with various FMC personnel suggested key parameters needed to evaluate the calciner/control system performance during the test period. Those operating parameters have been claimed as confidential. The discussion of these key operating parameters appears in Volume III - Confidential Material of the Emission Test Report and includes Sections 4.2 and 4.3 of the draft emission test report.

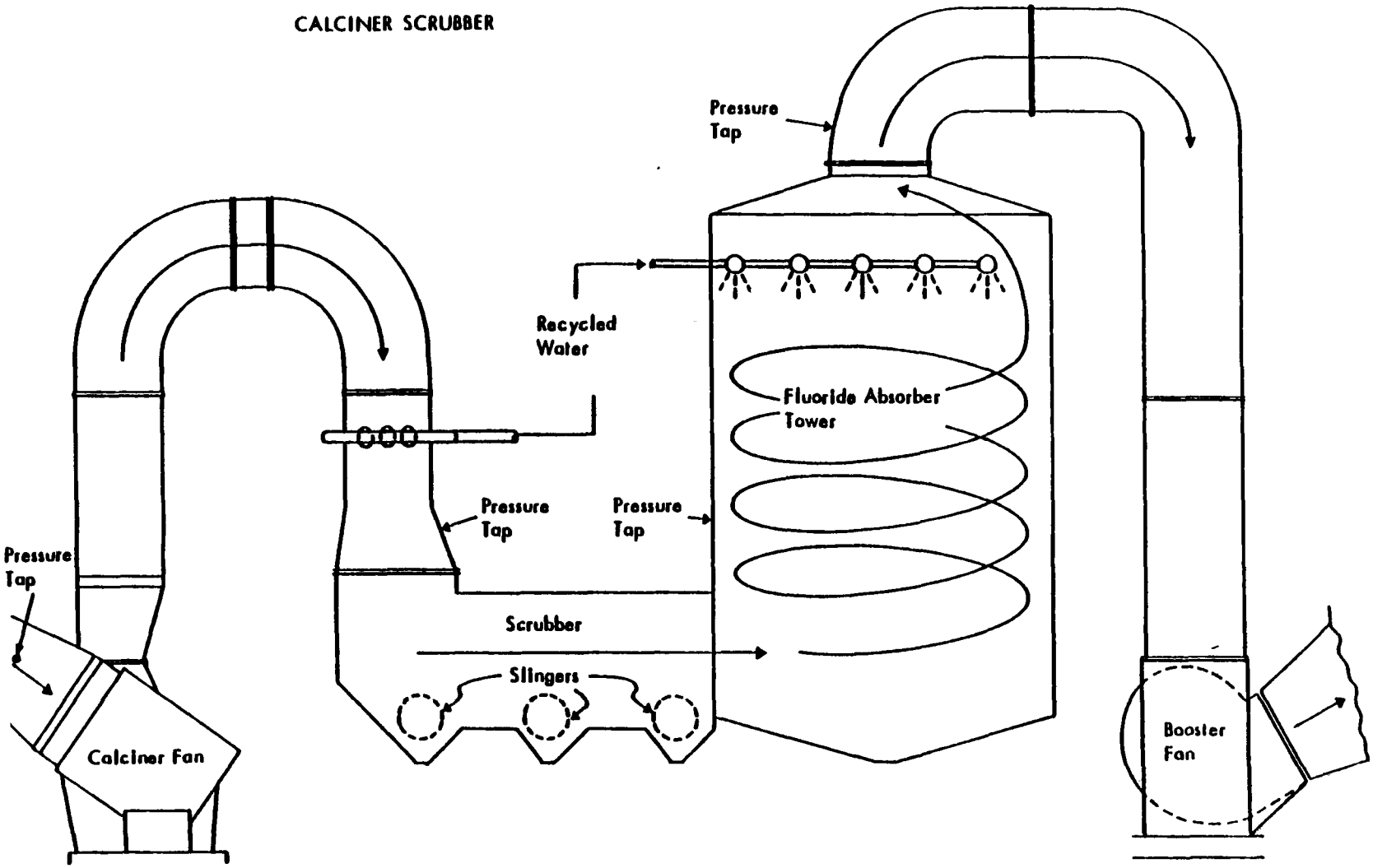

Figure 4-2. Schematic of calciner emissions control system.

5. SAMPLING LOCATIONS

The process diagram (Figure 2-2) indicates the sample point locations around the calciner and associated control equipment. The sample locations were designated as follows:

Sampling Locations	Location Designation
Inlet - Slinger Scrubber #2-2	A
Inlet - Slinger Scrubber #2-1	B
Scrubber Stack Outlet #2-2	C
Scrubber Stack Outlet #2-1	D*
Shale Briquet Feed (conveyor assembly)	E
Calcined Nodule Product (conveyor assembly)	F
Scrubber #2-2 Influent (recycle)	G
Scrubber #2-1 Influent (recycle)	H*
Scrubber #2-2 Effluent	I
Scrubber #2-1 Effluent	J*
Scrubber #1-1 Stack Outlet	K
Scrubber #1-2 Stack Outlet	L

Figure 5-1 provides a process layout of the sample locations maintained around the FMC dual kiln systems. The relative positions of the flue gas and process sampling locations are designated.

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*Was not sampled.

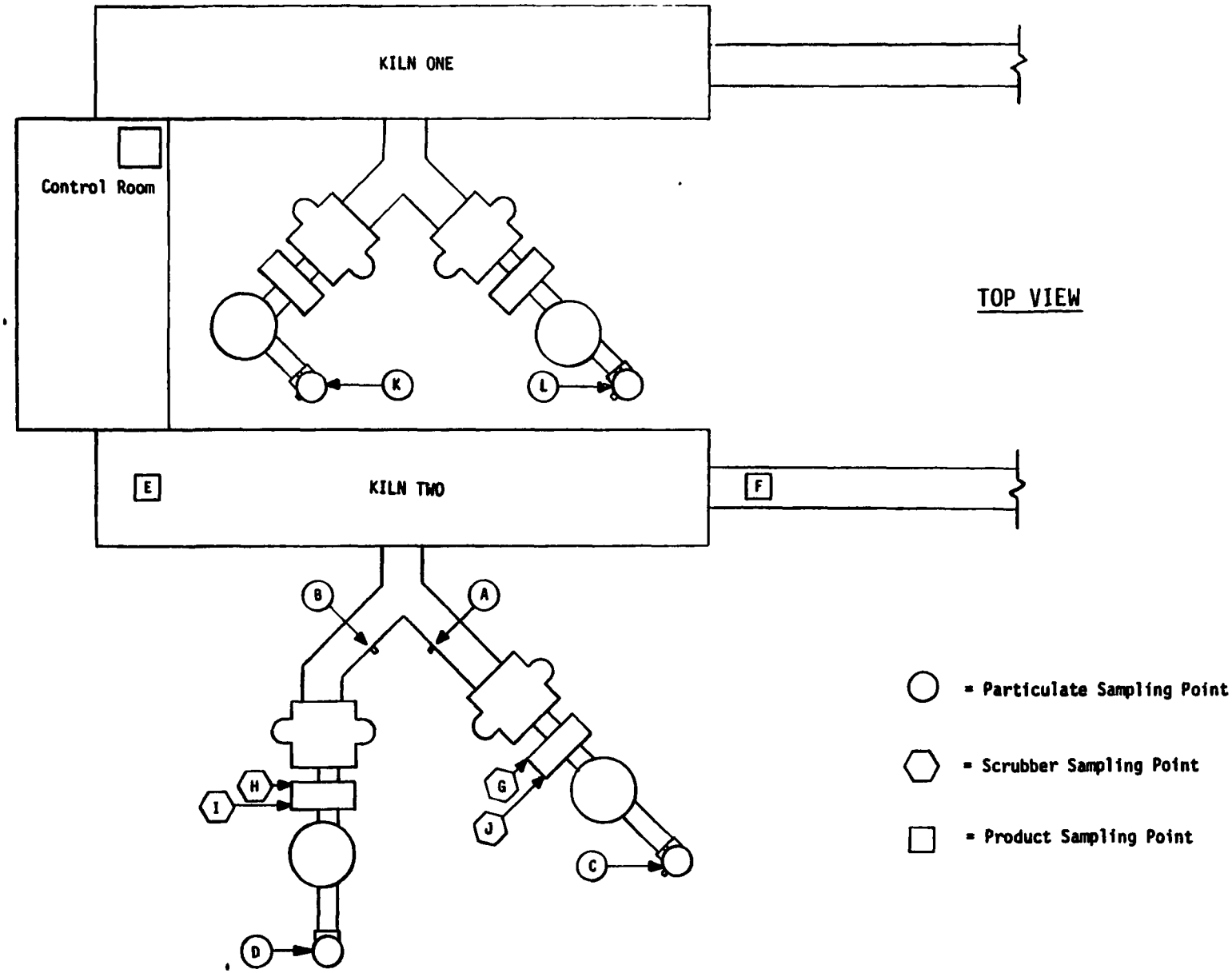


Figure 5-1. Sample locations overall perspective layout, FMC - Pocatello, Idaho.

5.1 SCRUBBER INLET #2-2 AND #2-1 - LOCATION A AND B

Figure 5-2 is a schematic diagram of the inlet sampling locations for slinger scrubber (#2-1 and #2-2). The associated ductwork is shown in plan and side views. The sampling ports were located 16½ feet downstream and 4 feet upstream from the nearest flow disturbances. A four (4) by ten (10) sampling matrix was used for particulate matter measurement. A four point matrix for particle sizing was used. The four point particle size sampling matrix was slightly offset (above) from the recommended number of points specified in the IP protocol. Given that an accumulation of particulate matter was possible along the bottom of the horizontal duct, the selected points were preferred to prevent biasing the particle sizing measurements.

The specific point locations for the inlet test positions described above are enlarged in Figure 5-3. The dimensions of the duct work are 98 inches x 50 3/4 inches. The duct (#2-2) was equipped with four (4) 3½-inch ID sampling ports. For the test project, plant personnel installed similar ports in the scrubber #2-1 inlet ductwork.

Access to the inlet sampling locations was by means of temporary scaffolding. The scaffolding was approximately 20 feet x 4 feet x 8 feet in size and capable of fully supporting 2 men and 300 pounds of test equipment. All scaffolding was equipped with appropriate safety guardrails around the work area.

5.2 SCRUBBER #2-2 STACK OUTLET - LOCATION C

The scrubber #2-2 stack outlet location was designated as location C. An approximate sketch of the flow disturbance is illustrated in Figure 5-4. Access was by approximately 50 feet of caged ladder. The nearest downstream flow disturbance is the ductwork entering the stack from the induced draft fan at the base of the stack. The ductwork enters the stack approximatley 15 feet above grade. The stack diameter is 6.5 feet. The upstream flow disturbance is greater than 2 equivalent duct diameters ($>2_{ED}$). The cross-sectional traverse point position at the sampling location is indicated in the inset of Figure 5-4. Two (2) sampling ports were available with monorail assemblies for particulate testing. Plant maintenance personnel exchanged the three 3-inch ports for four 4-inch ports for facilitating the cascade impactor for particle sizing purposes.

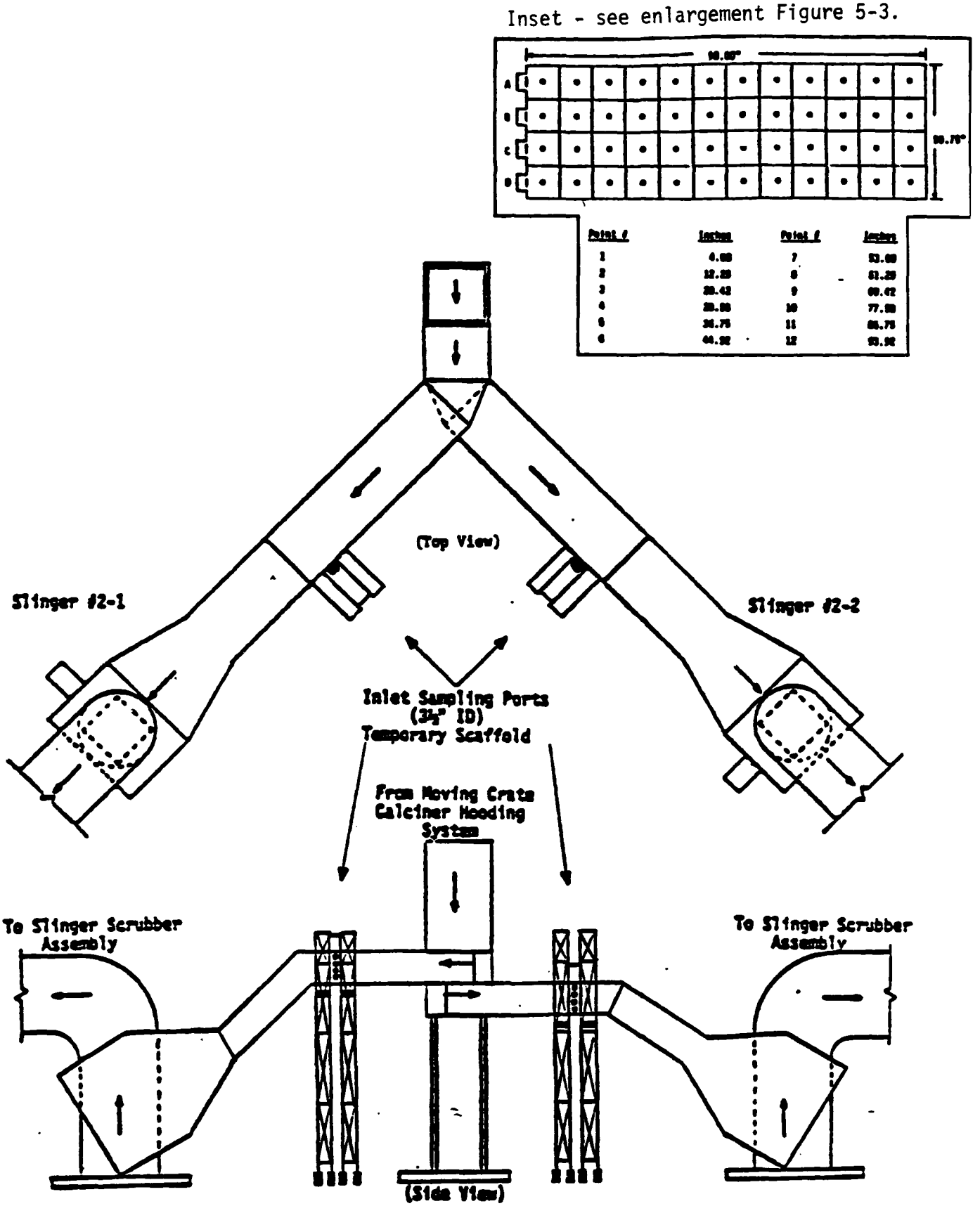
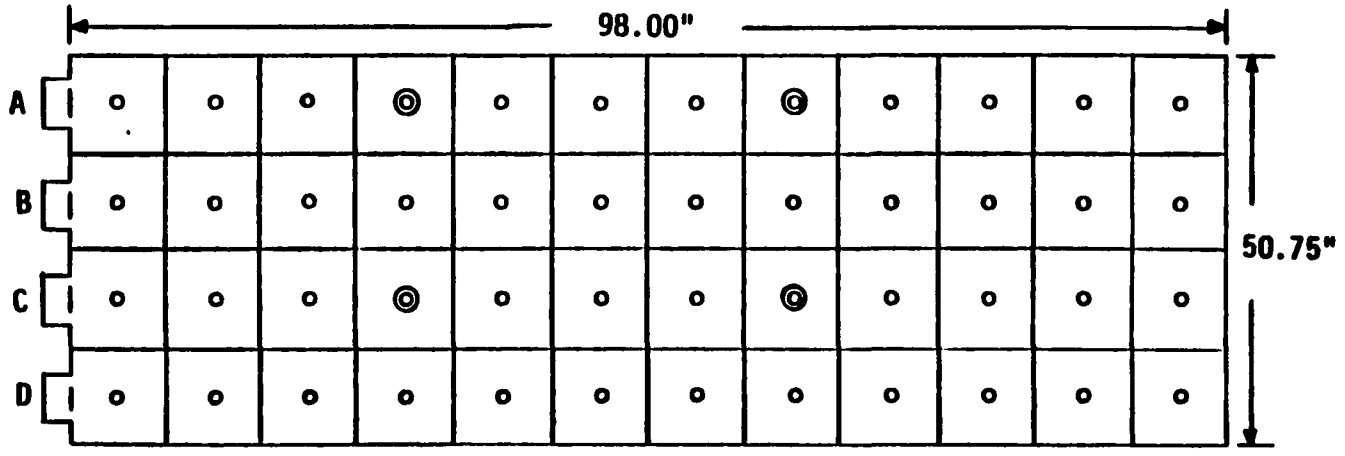


Figure 5-2. Calciner emission control system - inlet duct configuration to slinger scrubbers, FMC - Pocatello, Idaho (locations A and B).



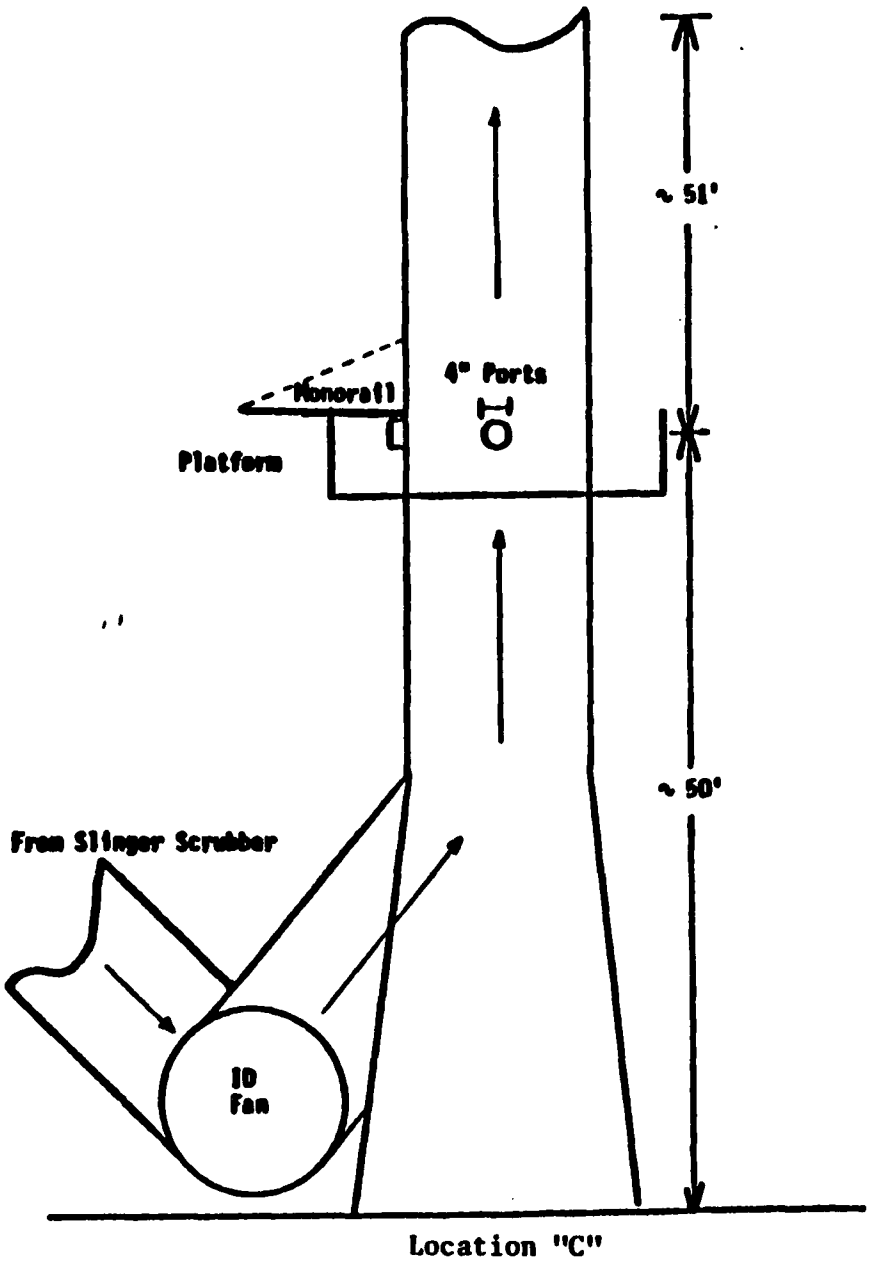
TRAVERSE POINT DISTANCES FROM INSIDE OF STACK

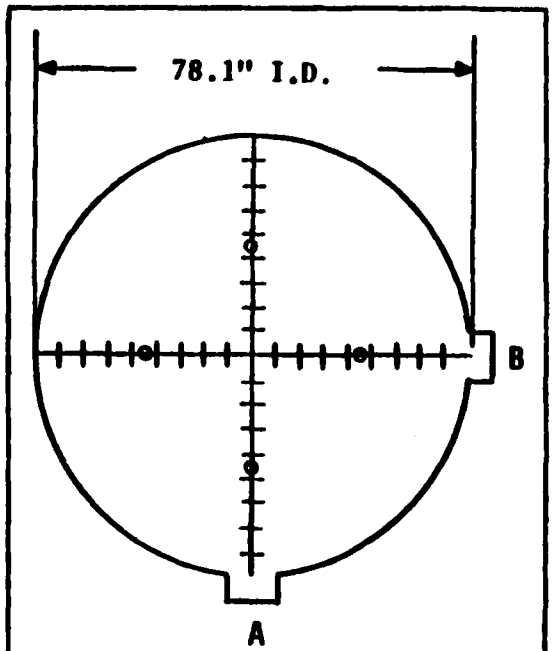
1.	4.08	7.	53.08
2.	12.25	8.	61.25
3.	20.42	9.	69.42
4.	28.58	10.	77.58
5.	36.75	11.	85.75
6.	44.92	12.	93.92

LOCATION OF ANDERSON POINTS FROM INSIDE OF STACK

A-3	C-3
A-8	C-8

Figure 5-3. Cross-sectional drawing of inlet ductwork with EPA Method 5 and particle size sampling points, FMC - Pocatello, Idaho (inset from Figure 5-2) (locations A and B).





TRAVERSE POINT DISTANCES FROM INSIDE OF STACK, (inches)

1.	1.3	9.	49.7
2.	3.9	10.	57.0
3.	6.8	11.	62.0
4.	9.9	12.	66.0
5.	13.4	13.	69.6
6.	17.5	14.	72.2
7.	22.5	15.	75.6
8.	29.8	16.	78.2

Location of Anderson Points From Inside of Stack (inches)

1.	11.9
2.	67.6

Figure 5-4. Scrubber stack outlet sampling locations, FMC - Pocatello, Idaho (approximate dimensions not to scale).

5.3 SCRUBBER #2-1 STACK OUTLET - LOCATION D

The #2-1 scrubber stack outlet was not sampled during the test period due to impending maintenance work scheduled for late November 1983.

5.4 SHALE FEEDSTOCK - LOCATION E

The shale feedstock (briquets) sampling location was designated as location E. The sampling location was located at the discharge of the vibrator feed conveyor on to the moving crate. Access to sampling location was gained by stairwell. Sampling personnel were limited to a selected individual in order to minimize the number of personnel in the actual calciner operating vicinity. The grab sample was collected with a long handled metal scoop from the moving belt and composited hourly during the course of the test into a covered precleaned stainless steel bucket. The sampling times are listed on the Solids Field Data Sheets in Appendix B-7.

5.5 CALCINED NODULES - LOCATION F

The calcined nodules (briquets) were sampled with a small metal scoop from the moving conveyor assembly as the product nodules were transported to storage. The collector bin and conveyor assembly provided adequate mixing of the nodules which aided in a homogeneous mixture of briquets. Access to the conveyor assembly was from the inclined walkway adjacent to the assembly, located a few feet above grade. The grab sample was collected with a metal scoop from the moving belt and composited hourly into a covered precleaned stainless steel bucket during the course of the test. The sampling times are listed on the Solids Field Data Sheets in Appendix B-7.

5.6 SCRUBBER #2-2 INFLUENT (RECYCLE WATER) - LOCATION G

The water to slinger scrubber was collected from sampling location G. Access to the top of the scrubber unit was by stairwell and a small ladder. No access problems were encountered. The grab sample was collected from a valve tap in a recycle line prior to the scrubber. The influent sample was taken hourly during each daily test series from the tap valve and composited directly to a precleaned Nalgene dewar. The Liquids Data Sheets indicating sample times are in Appendix B-7.

5.7 SCRUBBER #2-2 EFFLUENT - LOCATION I

The effluent from the slinger scrubber was sampled at the discharge from the unit. The sample was collected prior to the sump located at the base of the unit. The effluent contained both suspended solids and liquids. A grab sample of the effluent was obtained by a long handled Nalgene scoop as the effluent overflowed into the collection sump. The sample was collected hourly as listed on the Liquids Data Sheets in Appendix B-7 and composited immediately into a precleaned Nalgene dewar during each test series.

5.8 SCRUBBER #1-1 STACK OUTLET - LOCATION K

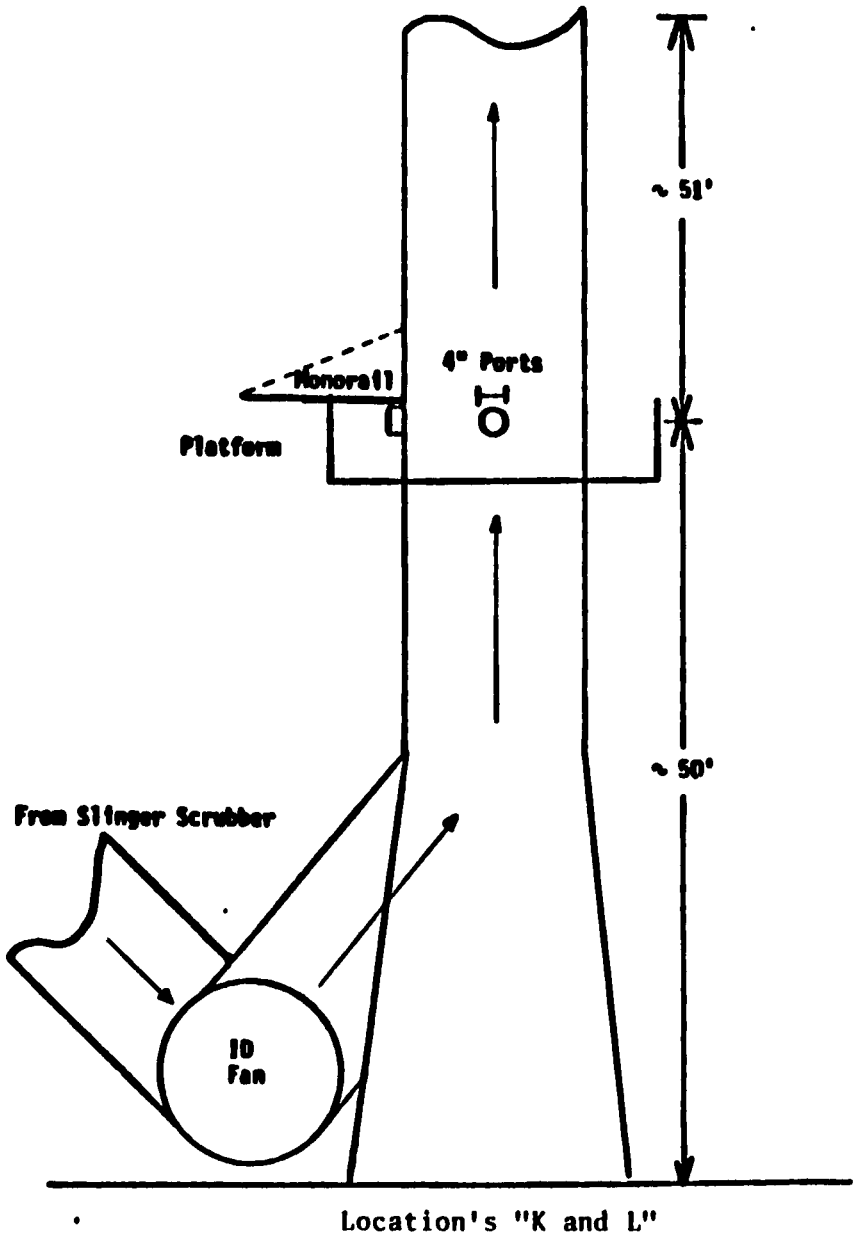
The scrubber #1-1 stack outlet was designated as location K for this test program. Access to the location was by caged ladder. The design duct diameter is 6.5 feet. The design of the sampling location and port arrangements are identical to the #2 calciner scrubber stack outlets. (See Figure 5-5.) The same modifications to the sample ports and monorail assemblies were required. All modifications were completed by plant personnel prior to the test period. The inset in Figure 5-5 indicates the traverse points utilized.

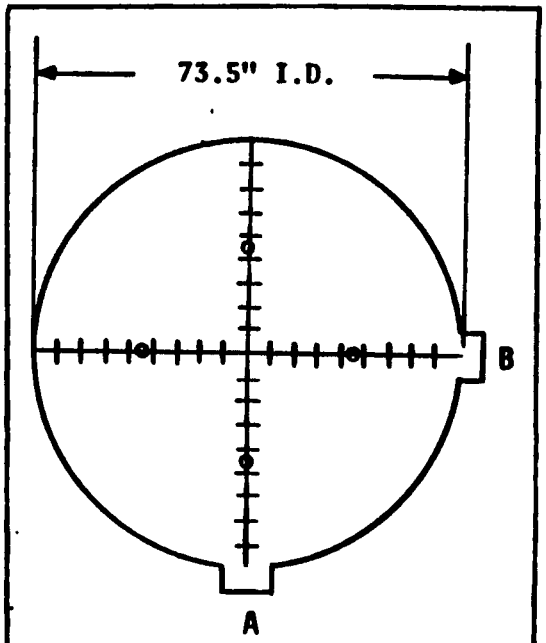
The traverse points utilized for particulate matter and particle size determinations at sampling locations K and L were different than those for locations C and D. There was a build up of particulate matter on the interior walls of the #1 scrubber stacks, which decreased the stack effective internal diameter. The sampling points were therefore calculated based on an effective internal diameter of 73½ inches according to Section 2.3 of EPA Reference Method 1.

The design of the emission control system serving the No. 1 calciner is slightly different than the emission control system serving the No. 2 calciner. The No. 1 calciner emission control system is equipped with Chevron demister pads rather than a cyclonic absorber. According to plant personnel, the absorber tower is more efficient at removing entrained water from the flue gas.

5.9 SCRUBBER STACK #1-2 STACK OUTLET - LOCATION L

This location was been designated as location L for this test program. The design duct diameter is 6.5 feet. The design of the sampling location and port arrangements was identical to sample location K, as previously discussed (Section 5.8). See inset of Figure 5-5 for the selected sampling point matrix.





TRAVERSE POINT DISTANCES FROM INSIDE OF STACK, (inches)

1.	1.25	9.	45.88
2.	3.63	10.	52.75
3.	6.25	11.	57.25
4.	9.25	12.	61.13
5.	12.38	13.	64.25
6.	16.25	14.	67.25
7.	20.75	15.	69.88
8.	27.63	16.	72.25

Location Anderson Points From Inside of Stack (inches)

1.	11.00
2.	62.50

Figure 5-5. Scrubber stack outlet sampling locations, FMC - Pocatello, Idaho (approximate dimensions not to scale).

6. SAMPLING AND ANALYTICAL PROCEDURES

This section presents general descriptions of sampling and analytical procedures employed during the source testing project conducted at the elemental phosphorus calciner units at the FMC facility in Pocatello, Idaho. The pollutant of concern in this test program is particulate matter because it is believed to contain significant quantities of Pb-210 and Po-210 radionuclides. The ultimate objectives of the test program were to obtain particulate matter emission data, analyze particulate matter samples for associated radionuclide activity,[10] and collect associated process data to determine representative emission rates. Flue gas samples were collected from the inlet and outlet of the control device(s) to obtain: (1) total particulate mass rate to the slinger scrubbers and the controlled particulate matter emission rate from the slinger scrubbers, and (2) the particle size distributions in the gas streams to and from the scrubbers. In addition, process grab samples were obtained of: (1) all feed materials entering the calciner (shale briquets), (2) calcined nodules, (3) water entering, and (4) water exiting the scrubber.

Section 6 is divided into the EPA Reference Sampling Methods (Section 6.1), the Non-reference Sampling Methods (Section 6.2), the Process Sample Methods (Section 6.3), and the Sample Analysis Methods (Section 6.4) utilized for this test project. Standard EPA sampling and analysis procedures are detailed in the Federal Register[11-15] and the non-reference procedures are presented in Appendix C. The non-reference methods utilized at FMC were the Source Assessment Sampling System (SASS) and the Andersen cascade impactor.

6.1 EPA REFERENCE METHODS DURING THE TEST PERIOD

The following EPA Reference Methods were used during this testing program. These methods are taken from "Standards of Performance for New Stationary Sources," Appendix A, Federal Register, Volume 42, No. 160, Thursday, August 18, 1977, pp 41755 ff

- Method 1 - Sample and Velocity Traverses for Stationary Sources - This method specifies the number and location of sampling points within a duct, taking into account duct size and shape and local flow disturbances. In addition, this method discusses the pitot-nulling technique used to establish the degree of cyclonic flow in a duct. (No cyclonic flow was encountered during the test program.)
- Method 2 - Determination of Stack Gas Velocity and Volumetric Flow Rate - This method specifies the measurement of gas velocity and flow rate using a pitot tube, manometer, and temperature sensor. The physical dimensions of the pitot tube and its spatial relationship to the temperature sensor and any sample probe are also specified.
- Method 3 - Gas Analysis for CO_2, O_2, Excess Air And Dry Molecular Weight - This method describes the extraction of a grab or integrated gas sample from a stack and the analysis of that sample to characterize the flue gas. As permitted under Section 1.2, paragraph 2 of the reference document, a modification to the sampling procedures and use of an alternative analytical procedure was implemented. A single point integrated sample was collected. In lieu of an Orsat analyzer, a gas chromatograph with a thermal conductivity detector (GC/TCD) was utilized to measure the concentrations of oxygen (O_2), carbon dioxide (CO_2), and nitrogen (N_2) in the integrated bag sample. The field chromatograms are presented in Appendix D. This alternative field analytical method offers greater accuracy than an Orsat and a permanent hard copy record of the analysis. Previous test programs have demonstrated the acceptability of this substitution and have been approved by regulatory authorities. The gas chromatograph utilized was a Shimadzu GC-3BT with a Shimadzu Chromatopac® to integrate and record the chromatogram peak area and peak heights. Helium was the carrier gas. Compound separation was achieved with a packed stainless steel Chromosorb® 102/Molecular Sieve column. Calibration gas standards were injected prior to and after sample by injection for a quantification by retention time and

peak area. A one point calibration method was employed utilizing a Scotty II-Mix 35 calibration (±2% certified) mixture. This mixture contained stationary gas components as follows:

- CO_2 - 3.0 percent;
- O_2 - 17.0 percent; and
- N_2 - 79.9 percent.

- Method 4 - Determination of Moisture Content in Stack Gases - This method describes the extraction of a gas sample from a stack and the removal and measurement of the moisture in that sample by condensation impingers. The assembly and operation of the required sampling train is specified.
- Method 5 - Determination of Particulate Emissions from Stationary Sources - This method describes the extraction of particulate matter from a source and collection on a glass fiber filter under isokinetic conditions. The assembly and operation of the required sampling train is specified (see Figure 6-1). The standard impinger solutions of the EPA Method 5 particulate matter sampling train were changed for this test. Rather than distilled water for moisture condensation, the impingers were filled with a 1.0 molar solution of nitric acid (HNO_3) for potential radiochemical metals analysis. The particulate mass, which includes any material that condenses at or above the filtration temperature, was determined gravimetrically after removal of uncombined water.

The parameters monitored during the test periods were the stationary gas contents (O_2, CO_2, and N_2) , the gas flow rate, the moisture content, the particulate matter levels, and the particle size.

Single point integrated bag samples were obtained over each test run at each separate test location. The samples were analyzed in the field by gas chromatography with thermal conductivity detection (GC/TCD). Analyzed sample runs were compiled, calculated, and recorded for providing stationary gas levels.

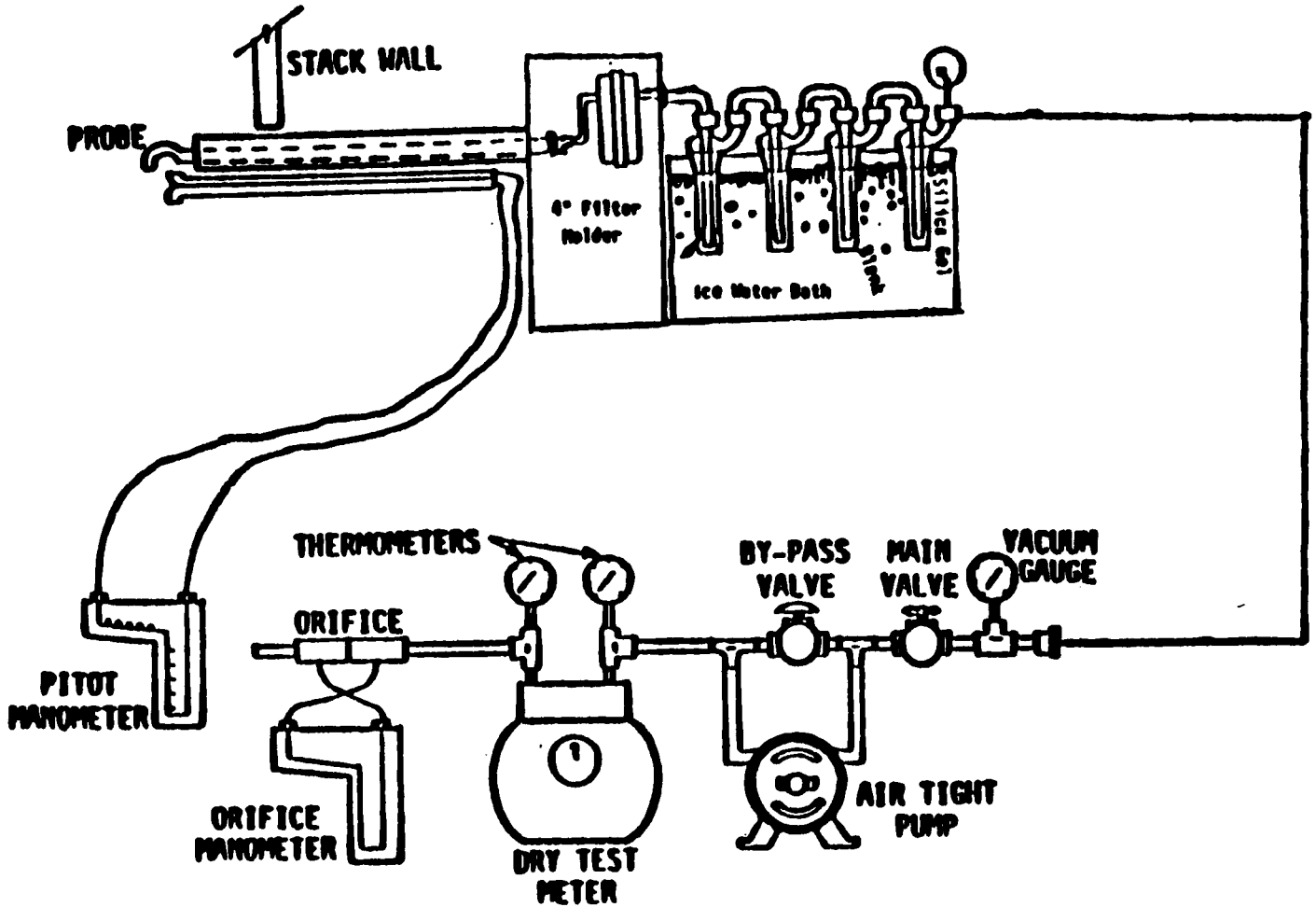


Figure 6-1. EPA Method 5 sampling train configuration.

The gas volumetric flow rates were determined from the EPA Method 5 sampling results. Based on standard EPA methodology, the volumetric flow and isokinetic rates during the test periods were calculated and determined. (See Appendix A for sample calculations.)

The moisture levels present in the sample gas streams were determined by differential volumes of each set of EPA Method 5 and SASS impingers. The impinger solution volume changes and silica gel weight changes were measured and recorded (before rinses) so that water vapor concentrations could be determined.

The particulate matter concentrations were determined according to EPA Method 5 at all five flue gas sample locations. The EPA Method 5 filters were weighed prior to and after the test run for determining the particulate catch over the test period. All acetone rinses were heated to 100-120°F on a hot plate for field determination of the total particulate catch.

The SASS train was utilized at one inlet sample location to supply a sufficient size sample for an associated lung clearance rate study. An inertial cascade impactor system was required for determining particle size at each flue gas sample location. The discussion of these methods follows in Section 6.2.

6.2 NON-REFERENCE SAMPLING METHODOLOGY

The particle sizing and the size sample (at inlet location A only) required the use of non-reference sampling methdologies. The particle sizing determinations were achieved with an inertial cascade impactor system (Andersen) and the adequate size sample was accomplished with a Source Assessment Sampling System (SASS). The Andersen cascade impactor utilizes staged substrates in providing ten (10) particle size fractions. The SASS system has a system of size differential cyclones and filters for four (4) particle sizing fractions.

6.2.1 Andersen Cascade Impactor System

Andersen cascade impactor test runs were completed at each sampling location. A typical run time for an inlet test location would be preferably 30 minutes in duration, while an outlet test location run time would be preferably 60 minutes in duration. The sampling systems were equipped with a 10 μm (nominal value) preseparator. Generalized operating instructions for cascade impactors are contained in Appendix C

and were used as a guideline in the operation of the Andersen impactors. Additional guidance for impactor operation is given in the EPA/Industrial Environmental Research Laboratory (IERL) report: Procedures for Cascade Impactor Calibration and Operation in Process Streams (Revised 1979).[16] Further recommendations for the particle size methodology were provided by the EPA/Emission Standards and Engineering Division (ESED) Test Support Section.[17]

During the FMC test project, the specified guidelines for conducting the particle size measurements and analyzing the data were determined from previous test experience and based on information in the IERL report. A further discussions of the quality assurance (QA) guidelines followed for the particle size procedures with results of QA checks are provided in Section 7.

The sample recovery and analysis were performed in the field. This required a precision balance operated in a field laboratory for tare and final weighings. The substrate filters were prepared and analyzed in the field by desiccating before and after each test.

Pretest procedures conducted were the selection of glass fiber mats for sample substrates, the inclusion of aluminum envelopes for each sample substrate, the verification of impactor hole sizes and nozzle sizes, and the incorporation of a reactivity run and blank run in the test scheme. Reeve Angel 943 glass fiber mats were chosen because of their nonreactive characteristics and general applicability to a wide range of emission sources. The aluminum envelopes for each substrate prevented sample loss during handling. The envelope weights were included in the initial and final weighings. The verification of impactor hole sizes was accomplished with precision wire gauges and the nozzles measured with a precision micrometer.

A reactivity run (IERL references as blank runs in Reference 16) was included during the field test to check for any reaction between the flue gas constituents and the sample substrates. This check was run by attaching a prefilter to an impactor to remove particulate and operating the impactor at the same conditions as a normal sampling run. The change in weight of the substrate in the reactivity run indicated the amount of change in weight of the substrate due to reactivity in the regular test runs. The reactivity weight change was the background

value subtracted (or added) from the weight gains observed from the regular test runs. The results of the reactivity runs are presented in Section 7, Table 7-2.

A blank run (IERL references as control runs in Reference 16) was included during the field test to check for sample handling errors with sample runs and analyses of the impactor system. The blank impactor was prepared like a regular test run with caps on the impactor inlet and outlet. The impactor was carried to the sampling location but not operated. The blank impactor was returned to the laboratory and unloaded for analysis in the same way the test impactors were treated. Lack of changes in the weight of the substrate indicate the stability of the glass fiber mat during handling. The results of the blank test run are provided in Table 7-3.

Procedures for the operation of the cascade impactor included leak checks of the impactor system, pre-heating the impactor, specifying four-point or traverse sampling scheme, and determining the sampling time duration for controlling the stage loading. A complete leak check from the nozzle back through the impactor provided sample integrity. The acceptable leak rate for the impactor test run was 0.02 CFM. The leak rate results are noted on the individual field run sheets in Appendix B. The pre-heating of the impactors was completed before exposing the sample substrate to the flue gas. The outlet location (Points C, K, and L) required heating during the test run because of the low flue gas temperature (≅145°F) and high (14-20) percent moisture. EPA Method 1 was used to determine the traverse sampling points at each sample port. The length of the sample time was determined by preliminary impactor measurements and visual inspection of the substrates which would indicated an overload of an impactor stage. The general rule followed was not to allow a single stage to be loaded over 10 mg.

Procedural guidelines observed after each test period included a purging period following each sample collection, an attempt to maintain the impactor in the horizontal position, and a visual inspection during analysis. The impactor was purged with at least 1 cubic foot of dry ambient air to remove wet stack gas. Low flow rates (0.05 CFM) were pulled during purging with the impactor maintained in the horizontal position. The impactor was maintained in the horizontal position until

shutting off the airflow and turned to the upright position during the transportation and recovery steps. A visual inspection of the sampled substrate determined the balance between the sample flow rate through the impactor and the sample substrate collection efficiency. Evidence of bounce or re-entrainment of the particles was investigated by examining the stage catches. This type of problem will result in particles being collected on stages downstream of where they should. Notations of visual observations made during impactor recovery procedures were recorded in the Field Analytical Log (Appendix D-1).

Analytical procedures observed during the recovery of the impactor test runs included daily balance checks against a known weight, a blank substrate retained in the desiccator for a check weight each day, and blanks acquired of reagants and filters. The weighing of the small particle catches (<10 mg) requires precise weighing techniques. A Mettler H20T analytical balance was used at FMC. The balance had an accuracy in grams to five decimal places. The manufacturer's directions were followed when operating the balance and the manufacturer's calibration practice updated. Additional field calibration guidelines included verifying the repeatability of measurements by check weighing, throughout the recovery period, a control weight of approximately the same weight as a substrate. An additional verification of the substrate dry weight was accomplished by desiccating a substrate, weighing, and then desiccating it again and reweighing at a later time. The dry weight checks should be within the precision of the balance. The acquisition of reagent and filter blanks provides background levels for analytical results.

In the field, data analysis procedures included the use of programmable calculators, microcomputers, the PADRE system, and on-site data compilation. The use of programmable calculators and a microcomputer in the field provided the ability for computing the flow rates of the system and isokinetic sampling rates of the test trains. These results, along with pertinent test conditions, were loaded in a direct module connection with the PADRE system for providing impactor stage cut points and impactor results. The compilation provides a degree of data analysis for determining test results validity. Anomalies in the analyzed data can be noticed and decisions made regarding the accuracy of a specific impactor run.

6.2.2 SASS Train System

A Source Assessment Sampling System (SASS) test run was completed at inlet sampling location A. This particle size sample was collected with the SASS train for obtaining one (1) gram per size sample cut in four size fractions (10 μm, 3 μm, 1 μm, and <1 μm). The purpose of this sample was to obtain a sufficient size sample for an associated lung deposition study. Appendix C-4 indicates the methodology planned for the lung clearance rate study.

The SASS train was utilized to provide the adequate particle size distribution sample of the flue gas at sample location A. Size fractionation is accomplished in the cyclone portion of the SASS train, which incorporates the three cyclones in series to provide large collection capacities for particulate matter nominally size-classified into three ranges: (1) >10 μm, (2) 3 μm to 10 μm, and (3) 1 μm to 3 μm. By means of a standard 142-mm back-up filter, a fourth cut, <1 μm, is also obtained. The SASS train operated at a flow rate of four SCFM and at a temperature of 400°F for the probe and cyclone oven in order to maintain the particle size cut points of the cyclones. The individual size fractions were recovered separately using acetone for rinsing after mechanical brushing into tared aluminum foil recovery packets. The acetone rinse of each fraction was evaporated to dryness at 100°F with a hot plate, desiccated, and weighted to obtain a constant weight in the field. The SASS train allowed the required collection of larger size fractions in a reasonable amount of sampling time.

The SASS train consists of a stainless steel probe that connects to three size selective cyclones and a filter in an oven module, a gas treatment section, and an impinger series (see Figure 6-2). The gas treatment system for organic determination was not utilized for this program. A series of four (4) impingers were used to cool the gas stream and remove moisture prior to the pumps and dry gas meter. The pumping capacity is supplied by two 10-ft^3/min, high-volume vacuum pumps, while required pressure, temperature, power, and flow conditions are regulated through a main control box.

The flue gas velocity based upon previous test data and preliminary velocity profiles was used to select a nozzle size to approximate isokinetic sampling conditions. The flue gas was sampled at a constant

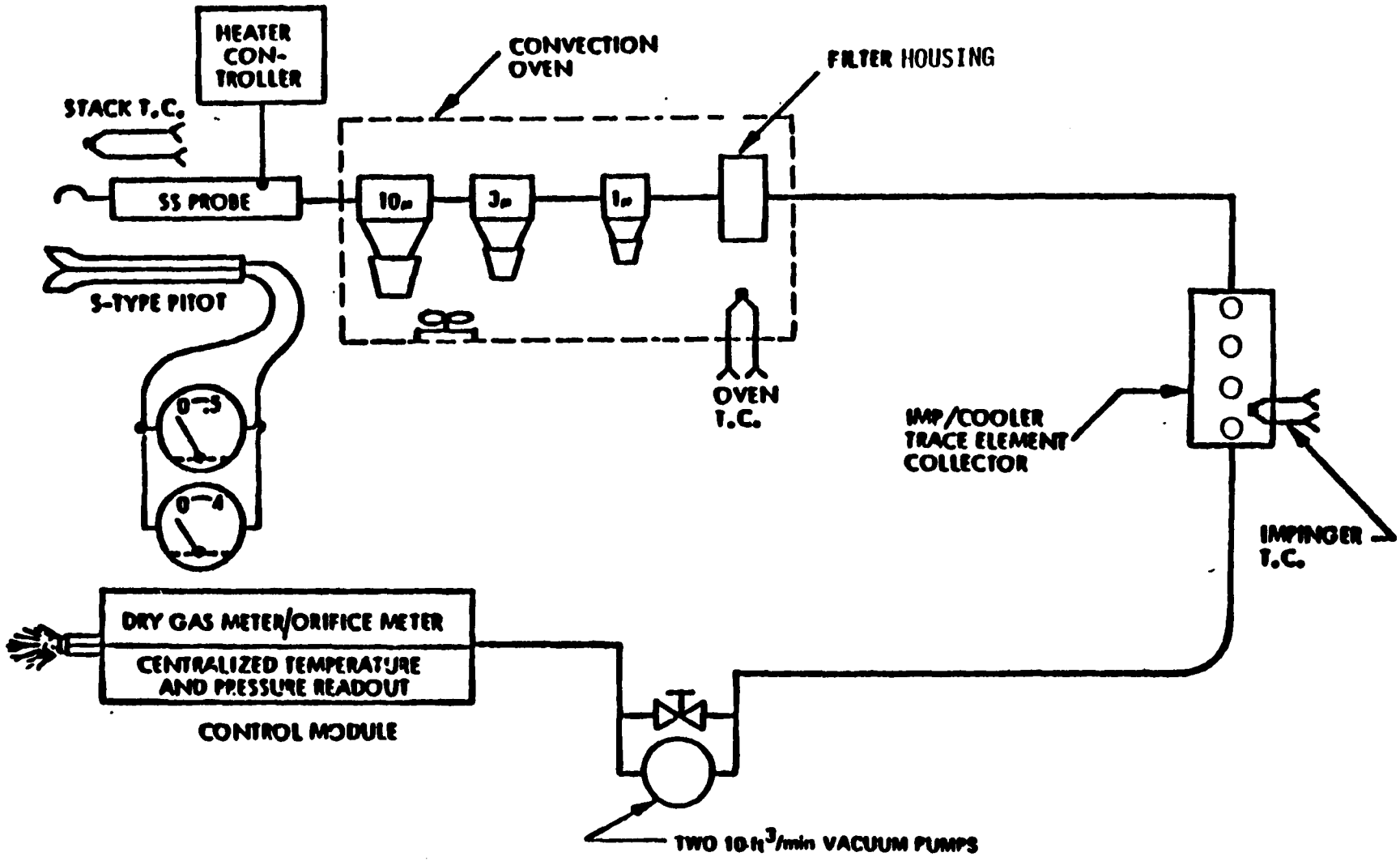


Figure 6-2. Source assessment sampling train (SASS) schematic.

flow rate in order to maintain the SASS cyclones cut points. The four (4) particulate fractions were summed to determine particulate matter grain loading. Acetone was used to rinse the various size fractions as appropriate. Detailed SASS operating instruction appears in Appendix C-1.

6.3 PROCESS SAMPLES

The following process related samples were obtained in order to determine radionuclide activity: shale rock feedstock briquets, calcined nodules, and scrubber water influent and effluent. Figure 2-2 provides a process diagram with the process sample locations designated. Process related samples were collected only during the testing of the No. 2 calciner. A composite of each process stream was collected and retained as listed below on each of five (5) test days. An aliquot from all five (5) test days was combined into a single composite sample at the contractor's base laboratory prior to shipment to EPA's Eastern Environmental Radiation Facility (EERF) for radionuclide analysis in Montgomery, Alabama. The daily process composite samples were retained by the contractor.

6.3.1 Shale Feedstock (Briquets)

A grab sample of the shale feedstock briquets was taken periodically from the conveyor vibrator assembly prior to discharging onto the moving grate. An hourly grab sample was obtained by a metal scoop across the briquet pallet. The sample was composited in a one (1) gallon precleaned stainless steel bucket during the test period. The composite of individual grab samples obtained during the course of each test day was then divided by the cone and quarter technique. The resultant sample was split and a duplicate retained by the plant for QA purposes.

6.3.2 Calcined Nodules

A grab sample of the calcined nodules was collected by the same method and frequency as noted above. The calcined product is conveyed to storage and was sampled after exiting the cooling compartment of the calciner and dump bin. The composite of individual grab samples obtained during the course of each test day was then divided by the cone and quarter technique. The resultant sample was split with a duplicate retained by the plant for QA purposes.

6.3.3 Scrubber Influent (Recycle Water)

A grab sample of scrubber influent (recycle water) was obtained from one of 4 spray nozzle taps on the scrubber unit itself. The sampling location was located atop each slinger scrubber on a platform ~10 feet above grade. A tap to measure pressure drop across the scrubber located at this location was not operable. An hourly one (1) liter grab sample was taken, with a final composite obtained at the end of each test day. A duplicate sample was split and retained by the plant for QA purposes.

6.3.4 Scrubber Effluent

A grab sample of the scrubber effluent was collected at the scrubber discharge (sump) located at the base of the scrubber unit. The sample contained suspended solids within the liquid sample. A grab sample was collected with a dipper every hour and composited into a precleaned Nalgene container. A final one (1) liter composite sample was retained at the end of the test day. A duplicate sample was split for retention by the plant for QA purposes.

6.4 ANALYTICAL METHODS

6.4.1 EPA Method 5

The field analytical procedures used in conjunction with EPA Method 5 were the standard analytical protocols specified under Section 5 for gravimetric and moisture analysis of the reference method document. The option of using higher than ambient temperatures to speed the evaporation of the acetone rinse (Container No. 2) was utilized as specified under the analytical note in the reference method. Analytical recovery data sheets are in Appendix B-4. Field gravimetric results are listed in Appendix D-1.

6.4.2 SASS

The field analytical procedures used in conjunction with the SASS train were essentially the same as the EPA Method 5 sampling train. The particulate matter catch of the EPA Method 5 is comprised of the two fractions (the filter and probe rinse) which are gravimetrically weighed and summed. The particulate matter loading of the SASS train is comprised of five fractions: the three cyclones, the filter, and the probe rinse. Each fraction is recovered a discrete sample except for the acetone (probe rinse) fraction which is used to rinse (recover) all components containing the particulate matter fractions. The particulate

matter determination from the SASS train is determined by summing the five (5) front half fractions. A detailed analytical flowchart for sample recovery and gravimetric analysis is contained in Appendix C. The SASS gravimetrical measurements are reported in Appendix D-1.

6.4.3 Andersen Cascade Impactor

The analytical procedures followed for the Andersen cascade impactor samples were according to the manufacturer's instructions[6] and previous cited references. All gravimetric measurements were made in the field onsite. All gravimetric measurements were recorded in a dedicated field laboratory notebook. Applicable journal entries are collected in Appendix D-2.

6.4.4 Radionuclide Analysis

The particulate matter and particle size fractions from all sampling trains were sent to the ORP Eastern Environmental Radiation Facility for radionuclide analysis. Selected process samples were also shipped for possible radionuclide analysis. The procedures "Radiochemical Determination of Lead-210 and Polonium-210 in Dry Inorganic and Biological Samples" will be followed by EPA/ORP.[10]

The principle of the method involves the addition of polonium-209 and bismuth-207 traces and lanthanum carrier to a weighed aliquot of sample which has been dried at 100°C for 24 hours. The sample is solubilized by wet ashing. The radioelements are coprecipitated as hydroxide with NH_4OH. The hydroxide is redissolved in acid and the bismuth and polonium are spontaneously deposited on a clean copper disc.

The disc is beta counted for ^{210}Bi, gama assayed by Ge (Li) for ^{207}Bi, and radioassayed by alpha spectroscopy for ^{209}Po and ^{210}Po. The ^{210}Pb can be determined by measuring in growth of its decay daughter ^{210}Bi which has a half life of five days.[10]

The specified procedure is contained in Appendix C-3.

7. QUALITY ASSURANCE PROCEDURES AND RESULTS

Section 7 discusses the quality assurance (QA) procedures practiced during the sampling project at the FMC facility. The results of the QA checks will be presented with the discussion. Standard sampling methodology and non-reference methods were used as discussed in Section 6. The standard methods have reference documentation detailing the requirements for precision and accuracy, sample representativeness and sample calculations. All provisions of Quality Assurance Procedures for EPA Method 5[18] were followed in the field. Quality assurance provisions for modified sampling methods used for SASS and particle size measurements are not documented as standard reference methods. Therefore, the operational and QA procedures were obtained from appropriate particle size manuals, an EPA/IERL report, and interaction with EPA/ESED Test Support Section.

Section 7.1 will discuss and provide the results of the QA procedures used with the particle sizing sampling system. Section 7.2 will provide the sample handling provisions and Section 7.3 will discuss the QA procedure used during the radionuclide analysis by the Office of Radiation Programs. Section 7.4 discusses the particulate matter data handling procedures.

7.1 PARTICLE SIZING QA PROCEDURES

The particle sizing sampling and analytical procedures, as specified in the EPA/IERL report, requires a sophisticated level of accuracy that is more applicable to laboratory situations. Therefore, detailed field QA procedures are necessary to minimize inaccuracies in sampling and analytical techniques. The periodic maintenance of EPA Method 5 (RAC) sampling console included the necessary calibration checks of the particle sizing measurements. The Andersen Impactor system required verification of the hole sizes in the staged filter plate system and determination of the effect of flue stack gas conditions on the sampling system with

blank and reactivity runs. The analytical procedures required balance calibrations, dry weight checks, and visual observations of collection efficiency.

7.1.1 Plate Calibration

Calibration of the cascade impactor was not performed, in spite of the fact that procedures for cascade impactor sampling recommend use of calibrated impactors. Calibration of individual impactors is costly and beyond budgetary limitations of most field testing programs. In the absence of calibration data for their specific impactors, users (and manufacturers) frequently choose a constant value of the impaction parameter, $\sqrt{\psi}50$, for data analysis.

In order to provide the most useful estimate of the calibration constants for several commercially available impactors, a set of "generic" average calibration constants have been incorporated in PADRE as plate set 0 for the Andersen Mark III, as well as several other impactors. These average constants were compiled under the assumption that the different sets of the same impactor stage will behave alike. This assumption appears to be valid based on experimental calibrations. In recent studies, consistency is typical for the averages used to form the generic plate sets. Variances typically are less than 5 percent for individual stages even when the average stage $\sqrt{\psi}50$ may differ by 20 to 25 percent from the typical value of approximately 0.36 (as in Andersen stage 8). Since there appears to be a tendency for systematic variation using measurements of hole diameter, the generic $\sqrt{\psi}50$ values have been corrected to assume use of the manufacturers nominal jet diameter (D_j) rather than the mean measured diameter. This value of D_j was used in data reduction as well.[7,19]

In lieu of calibrating the cascade impactors staged plates, a hole size verification was performed by a trial and error system utilizing a series of wire gauges. Table 7-1 presents the plate verification data completed prior to the testing project. The labeling system for the calibration data was a two (2) number system. The first number designates the impactor system (0-3) and the second number the plate number of the impactor (0-7).

Table 7-1. ANDERSEN CASCADE IMPACTOR STAGE VERIFICATION - HOLE DIMENSIONS
FMC - POCATELLO, IDAHO

Impactor ID no.	0	1	2	3	0	1	2	3	0	1	2	3
Plate no.	0	0	0	0	1	1	1	1	2	2	2	2
Plate holes checked (in.)												
1	.0631	>.0631	.0636	.0636	.0465	.0465	.0470	.0470	.0355	.0355	.0365	.0365
2	.0636	.0636	.0641	.0641	.0465	.0465	.0470	.0470	.0360	.0355	.0365	.0365
3	.0631	>.0631	.0636	.0636	.0465	.0465	.0470	.0470	.0355	.0355	.0365	.0365
4	.0636	.0631	.0641	.0641	.0465	.0465	.0470	.0470	.0360	.0355	.0365	.0365
5	.0631	>.0631	.0636	.0636	.0465	.0465	.0470	.0470	.0355	.0355	.0365	.0365
6	.0636	.0631	.0641	.0641	.0465	.0465	.0470	.0470	.0360	.0355	.0365	.0365
7	.0631	>.0631	.0636	.0636	.0465	.0465	.0470	.0470	.0355	.0355	.0365	.0365
8	.0636	.0631	.0641	.0641	.0465	.0465	.0470	.0470	.0360	.0355	.0365	.0365
9	.0636	>.0631	.0636	.0636	.0465	.0465	.0470	.0470	.0355	.0355	.0365	.0365
10	.0636	.0631	.0641	.0641	.0465	.0465	.0470	.0470	.0360	.0355	.0365	.0365

(continued)

Table 7-1. ANDERSEN CASCADE IMPACTOR STAGE VERIFICATION - HOLE DIMENSIONS
FMC - POCATELLO, IDAHO

Impactor ID no.	0	1	2	3	0	1	2	3	0	1	2	3
Plate no.	3	3	3	3	4	4	4	4	5	5	5	5
Plate holes checked (in.)												
1	.0275	.0280	.0285	.0285	.0205	.0210	.0215	.0215	.0131	.0136	.0141	.0136
2	.0280	.0280	.0285	.0285	.0210	.0210	.0215	.0215	.0136	.0136	.0141	.0136
3	.0275	.0280	.0285	.0285	.0205	.0210	.0215	.0215	.0131	.0136	.0141	.0136
4	.0280	.0280	.0285	.0285	.0205	.0210	.0215	.0215	.0136	.0136	.0141	.0136
5	.0275	.0280	.0285	.0285	.0210	.0210	.0215	.0215	.0136	.0136	.0141	.0136
6	.0280	.0280	.0285	.0285	.0205	.0210	.0215	.0215	.0136	.0136	.0141	.0136
7	.0275	.0280	.0285	.0285	.0205	.0210	.0215	.0215	.0136	.0136	.0141	.0136
8	.0280	.0280	.0285	.0285	.0205	.0210	.0215	.0215	.0131	.0136	.0141	.0136
9	.0280	.0280	.0285	.0285	.0205	.0210	.0215	.0215	.0131	.0136	.0141	.0136
10	.0280	.0280	.0285	.0285	.0210	.0210	.0215	.0215	.0136	.0136	.0141	.0136

(continued)

Table 7-1. ANDERSEN CASCADE IMPACTOR STAGE VERIFICATION - HOLE DIMENSIONS
FMC - POCATELLO, IDAHO

Impactor ID no.	0	1	2	3	0	1	2	3
Plate no.	6	6	6	6	7	7	7	7
Plate holes checked (in.)								
1	.0100	.0100	.0095	.0095	.0095	.0100	.0100	.0095
2	.0100	.0100	.0100	.0095	.0095	.0100	.0100	.0095
3	.0100	.0100	.0095	.0095	.0095	.0100	.0100	.0095
4	.0100	.0100	.0100	.0095	.0095	.0100	.0100	.0095
5	.0100	.0100	.0095	.0095	.0095	.0100	.0100	.0095
6	.0100	.0100	.0100	.0095	.0095	.0100	.0100	.0095
7	.0100	.0100	.0095	.0095	.0095	.0100	.0100	.0095
8	.0100	.0100	.0100	.0095	.0095	.0100	.0100	.0095
9	.0100	.0100	.0095	.0095	.0095	.0100	.0100	.0095
10	.0100	.0100	.0100	.0095	.0095	.0100	.0100	.0095

7.1.2 Reactivity and Blank Runs

The reactivity and blank runs determine the background data for the weight changes the collection substrate undergoes during the actual impactor tests. The background runs allows adjustment in the data for minimizing the effects of the substrate weight changes.

The reactivity run determines the background value of collection substrate weight change from exposure to the test gas stream. The reactivity run is made by attaching a prefilter to an impactor and operating the assembly in the same gas stream and under the same conditions of flow rate and sampling duration as the regular test runs. Table 7-2 presents the reactivity run results from FMC.

The blank run determines the background weight lost from mechanical or manual abrasion the impactor was exposed to during the testing sequence. The blank run was accomplished by loading an impactor as for a regular run. Then, plug the inlet and outlet and carrying the impactor to the sampling site. The impactor was not operated, but kept at the sampling site until the actual run was completed. The impactor was transported back to the field laboratory and recovered in the same sequence as a regular test run. Table 7-3 presents the blank run results from FMC.

7.1.3 Plant QA Particle Sizing

In order to verify the contractor generated particle sizing results (and associated radionuclide content), the host facility performed a replicate particle size measurement on October 31, 1983. The FMC particle size meaurement was conducted immediately after the fourth contractor EPA Method 5 test series. The FMC generated test run data sheets and particle sizing results are contained in Appendix G-2 for reference.

7.1.4 Field Analytical QA Procedures

The level of accuracy required of the field analytical laboratory was critical because of the small weight changes measured. A 5-place Mettler Balance (Model H20T) was used in the FMC field laboratory. The balance was operated according to manufacturer specifications. Spot checks were added to the manufacturer written direction to assure the calibration and operation. The calibration was checked by repeated measurements of a control weight of approximately the same weight as the sample substrate. The consistency of the substrate dry weight was checked by repeated measurement of a substrate before and after. Calibration and spot checks are contained in Table 7-4.

Table 7-2. ANDERSEN CASCADE IMPACTOR REACTIVITY RUN
FMC - POCATELLO, IDAHO (10/28/83)

Stage	Pre-weight (gm)	Post-weight (gm)	Differential weight (gm)
0	0.42139	0.42209	+0.00070
1	0.41386	0.41387	+0.00001
2	0.42407	0.42432	+0.00025
3	0.41092	0.41102	+0.00010
4	0.44257	0.44261	+0.00004
5	0.41409	0.41411	+0.00002
6	0.42775	0.42780	+0.00005
7	0.40464	0.40464	0.00000
8	0.50580	0.50612	+0.00032

Standard deviation = 0.00023

Mean = 0.00017

Table 7-3. ANDERSEN CASCADE IMPACTOR BLANK RUN
FMC - POCATELLO, IDAHO (11/01/83)

Stage	Pre-weight (gm)	Post-weight (gm)	Differential weight (gm)
Precutter	0.26703	0.26686	-0.00017
0	0.43577	0.43568	-0.00009
1	0.41026	0.41018	-0.00008
2	0.41573	0.41576	+0.00003
3	0.39766	0.39773	+0.00007
4	0.42922	0.42928	+0.00006
5	0.40145	0.40140	-0.00005
6	0.41429	0.41427	-0.00002
7	0.39778	0.39784	+0.00006
8	0.48307	0.48309	+0.00002

Standard deviation = 0.00017

Mean = 0.00006

Table 7-4. QUALITY ASSURANCE REFERENCE WEIGHT CHECKS
RADIAN FIELD ANALYTICAL BALANCE[a] AT FMC - POCATELLO, IDAHO (10-11/83)

Date	Standard weight (grams) t	Measured weight (grams) x	Accuracy[b] (%)
10/24/83	0.10000	0.09993	0.07
	1.0000	0.99979	0.021
10/25/83	0.10000	0.09999	0.01
	1.0000	0.99997	0.003
10/26/83	0.10000	0.10000	0.000
	1.0000	0.99988	0.012
10/27/83	0.10000	0.09897	1.03
	1.0000	0.99882	0.118
10/28/83	0.10000	0.10000	0.000
	1.0000	0.99988	0.012
10/29/83	0.10000	0.10000	0.000
	1.0000	1.00002	0.002
10/30/83	0.10000	0.10000	0.000
	1.0000	0.99994	0.006
10/31/83	0.10000	0.10000	0.000
	1.0000	0.99988	0.012
11/01/83	0.10000	0.10000	0.000
	1.0000	0.99986	0.014
11/02/83	0.10000	0.10000	0.000
	1.0000	0.99985	0.015

[a] Mettler H20T.

[b] $(\frac{x-t}{t}) \times 100$

Bounce and re-entrainment are problems caused by a flow imbalance through the impactor collection stages. These problems result in particles being collected on stages downstream of where they should theoretically be deposited. Possible operational abnormalities were recorded upon visual inspection of the collection substrate during the recovery process in the field notebook and data sheet.

7.2 SAMPLE HANDLING PROCEDURES

In order to ensure expeditious shipment of the samples for radionuclide activity to EPA/ORP, all gravimetric measurements for particlate matter and particle size were done onsite in the field. TRW was equipped with a Mettler 5-place analytical balance, drying ovens and desiccators for field measurement. Upon completion of gravimetric analysis in the field, the particulate matter and particle size samples were shipped in a secure manner by air freight to the ORP Eastern Environmental Radiation Facility (EERF) for radionuclide analysis. To assure that samples were analyzed at or near their maximum activity, all samples were shipped by TRW from the field to ORP/EERF for analysis as soon as possible after collection and field gravimetric analysis, but in all cases within 15 days of sample collection.

The fractions sent to ORP/EERF for radionuclide activity analysis included the filter media and solvent rinses used to recover the sample from each type of sampling train. The acetone rinse of the probe and cyclones were evaporated and shipped to ORP/EERF in 400 ml beakers covered with parafilm in a secure manner.

7.2.1 Sample Blanks

Blank filters for each type of filter and lot utilized during the test series were submitted along with the samples for field gravimetric and radionculde analysis. Field blanks of the acetone and water used to recover the sample train were submitted for the test series. The blank samples were treated the same as test samples (i.e., liquid blanks came from the wash bottles and were shipped in collection bottles identical to those used for the test samples.

7.2.2 Retained Samples

Samples of impingers contents from the test series from the back half of an EPA Method 5 sampling train were retained for potential radionuclide analysis. No analysis was performed on the fraction retained

by TRW. (All impinger contents were shipped to EERF for radionuclide analysis on 1/26/84.)

All process samples collected during each test series were split with the host facility. The purpose was to have available a safety mechanism in case of sample breakage during shipment and allow the host facility the opportunity to compare the activity measurements through the use of an independent contract laboratory.

7.3 RADIONUCLIDE ANALYSIS QA PROCEDURES

Two (2) quality assurance samples were submitted with the particulate matter, particle size and process samples to EERF. These QA samples were submitted as blind audits. Spiked and unspiked samples of shale rock had been prepared by an independent third party. An aliquot of each sample was taken and split between the host facility and the test contractor. The test contractor shipped the following QA samples to the EERF subsequently.

EERF	Field ID	Date
—	FMC-QA-1-Shale	November 2, 1983
—	FMC-QA-2-Shale	November 2, 1983

The EERF radionuclide analysis of the audit samples will be reported in the radionuclide summary report for the FMC facility. The host facility intended to submit the duplicate sample to their contract laboratory for radionuclide analysis along with the FMC replicate particle size determination (see Section 7.1.3).

7.4 DATA HANDLING

7.4.1 EPA Method 5

A microcomputer based data analysis program was used to reduce all EPA Method 5 sampling train data and SASS train data directly from the raw field test worksheets. This technique is used to provide consistent and cost-effective reporting of experimental results. This computer program was also used to calculate flue gas flow rate, velocity, and the isokinetic sampling rate for the Andersen Impactor tests. The computer generated reports for the individual test runs are included in Appendix A.

7.4.2 Andersen Impactor Particle Size Data Analysis

The computer program PADRE was used to store, review, edit and analyze, and through a variety of data checks to identify invalid or

suspect particulate size data. This software program facilitates entry, reduction, and analysis of cascade impactor data for particle size distributions. Impactor stage at points are calculated and cumulative and differential mass concentrations are determined and interpolated and extrapolated to standard diameters. PADRE was developed to ensure the quality of data included in the Fine Particle Emissions Information System (FPEIS), which is a component of the Environmental Assessment Data System (EADS). The PADRE User's Guide[20] which describes how PADRE can be accessed and summarizes PADRE's logic and capabilities.

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