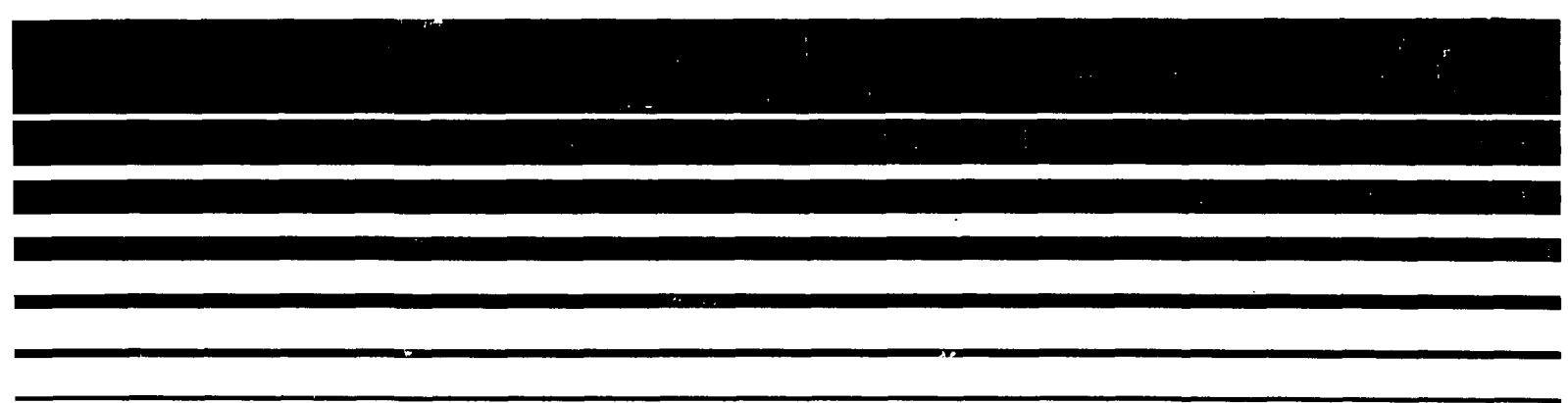

Air



Cadmium Screening Study Test Report

**Cadmium Refining
Industry
Amax Zinc
Company, Inc
East St. Louis,
Illinois**



NESHAP Development
Cadmium Emission Testing At The
AMAX Zinc Company, Inc
Primary Zinc Smelter
East St Louis, Illinois

June 1986

EPA Contract No. 86-02-4337
ESED Project No. 80/42
Work Assignment No. 1
CAD-1 E. St. Louis, IL
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PREFACE

The work described herein was conducted by personnel from TRC Environmental Consultants, Inc., Midwest Research Institute (MRI), the United States Environmental Protection Agency Emission Measurement Branch (EPA/EMB) and AMAX Zinc Co. in East St. Louis, Illinois.

The scope of work was issued under EPA Contract 68-02-4337, Work Assignment 1. The work was performed under the supervision of John H. Powell, TRC Work Assignment Manager, and Richard A. Pirolli, TRC Field Team Leader.

Mark Turner of MRI monitored process operations. MRI was responsible for preparing Section 3 of this report, which deals with process descriptions and operations. Raymond Ehrhard, Environmental Engineer for AMAX, provided invaluable assistance and guidance to TRC, EPA and MRI in the performance of the test program. Michael Toney, Office of Air Quality Planning and Standards (OAQPS), Emission Measurement Branch, EPA, served as Task Manager and was responsible for coordinating the test program.

1.0 INTRODUCTION

1.1 Background

Section 112 of the Clean Air Act of 1977 charges the administrator of the United States Environmental Protection Agency with the responsibility of establishing National Emission Standards for Hazardous Air Pollutants (NESHAP) that may significantly contribute to air pollution. When promulgated, these standards of performance are to reflect the degree of emission limitation achievable through application of the best demonstrated emission control technology. Emission data collected from controlled sources in the cadmium industry may provide a portion of the data base used by EPA to develop a NESHAP.

EPA Industrial Studies Branch (ISB) selected AMAX Zinc Co. in East St. Louis, Illinois, as a site for an emission test program because it is considered to employ process and emission control technology representative of modern cadmium oxide manufacturing plants. The test program was designed to develop controlled emission factors for Cadmium Oxide production.

TRC Environmental Consultants, Inc. was retained by the EPA Emission Measurement Branch (EMB) to perform emission measurements at the AMAX Zinc Co. in East St. Louis, Illinois. Testing was performed on the cadmium oxide baghouse outlet. This report has been prepared in accordance with EPA Contract No. 68-02-4337 under the provisions of Work Assignment No. 1.

Midwest Research Institute (MRI), the NESHAP contractor, was responsible for coordinating the overall test program with AMAX personnel and for assuring that process and control equipment operating conditions were suitable for testing. Related process data were monitored and recorded by MRI.

1.2 Summary of Process

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1.3 Applicability of EPA Reference Test Methods

EPA is required to publish a national reference test method for each regulated source category and pollutant for which a National Emission Standards for Hazardous Air Pollutants (NESHAP) is established. Reference test methods are usually specified by a state regulatory agency during the State Implementation Planning process and may be different from national reference test methods.

The purpose of establishing a national reference test method is to ensure that emission data collected from a specific source is representative of that source and comparable to data collected at other designated sources. The primary purpose of this test program was to collect emission data using standardized test methods which will allow the data to be evaluated to develop a NESHAP. Two modified configurations of EPA Method 5 were identified to measure emissions from cadmium oxide production. These methods are described in detail in Section 5.

1.3a EPA Method 5 Configuration For Cadmium

Cadmium emissions were measured by two different configurations of EPA Method 5. In the first configuration a flexible line was placed between the filter and impingers. In the second, the filter was eliminated from the train and flexible line was used to connect the probe to the first impinger. In the first configuration, five percent Nitric Acid was placed in the first two impingers, and the second five percent in the first three.

It should be noted that the flexible line was used because of a particularly difficult sampling location and would not normally be a component of a cadmium sampling train.

Five percent Nitric acid is used as the impinger solution because metals are readily soluble in Nitric acid and also Nitric acid increases capture efficiency.

1.4 Measurement Program Summary

The measurement program was conducted at the AMAX Zinc Co. in East St. Louis, Illinois during the week of June 16, 1986. Tests were performed at the cadmium oxide furnace baghouse outlet.

All emission testing was performed by TRC. MRI personnel monitored process operating conditions. Michael Toney of EMB observed the test program.

1.4a Baghouse Outlet

Preliminary Measurements

Preliminary testing was performed on June 16, 1986 to determine volumetric flowrate and stack gas moisture content. An integrated gas sample was also taken to determine concentrations of CO₂, O₂, and CO, which were found to be ambient. Stack diameter and the sampling port configuration were confirmed at this time.

Method 5 (configuration) modified for cadmium

Eight Method 5 tests modified for cadmium were performed, four on June 17 and four on June 18, 1986. One of the test on June 17, 1986 was invalid due to a leak in the sampling train.

Particle size distribution test

One particle size distribution test was performed on June 18, 1986 using an Anderson Mark IV Cascade Impactor.

Method 9 - Visible emissions

Visible emissions from the baghouse outlet were observed concurrently with each Method 5 modified for cadmium test.

Cadmium Dust

Samples of cadmium dust from the baghouse were drawn from the baghouse every half-hour during each emission test.

1.5 Report Sections

The remaining sections of this report present the Summary and Discussion of Results (Section 2), Process Description and Operations (Section 3), Description of Sampling Locations (Section 4), Sampling and Analytical Procedures (Section 5), and Quality Assurance (Section 6). Methods and procedures, field and laboratory data, and calculations are presented in various appendices as noted in the Table of Contents.

2.0 SUMMARY AND DISCUSSION OF RESULTS

A summary of all emission measurements and collected data is presented in this section. Section 2.1 presents cadmium results collected with the Method 5 train (with filter) modified for cadmium and a complete breakdown and discussion of parameters. Cadmium results collected with the Method 5 train (without a filter) modified for cadmium are presented in Section 2.2. Section 2.3 presents the particle size distribution and Section 2.4 summarises the visible emission observations. Section 2.5 presents results of the trace metals and baghouse dust analyses.

The cadmium results indicate that there was no significant difference between the sampling train with filter than the train without filter.

2.1 Method 5 (configuration) Modified For Cadmium

A total of four tests were conducted using the Method 5 train (with filter) modified for cadmium. The second test was discarded due to an unacceptable isokinesis of 134%. The first test had an isokinesis of 114% but it was decided to keep the test. All other tests were acceptable with 100 ± 10 percent isokinesis. All tests had an acceptable leak rate of < 0.02 cfm.

2.1a Cadmium Emissions

Tables 2-1a (English Units) and 2-1b (Metric Units) present a summary of measured cadmium emissions from the baghouse outlet using the Method 5 train with the filter.

The average total cadmium concentration was 1.55×10^{-2} gr/DSCF (35470 $\mu\text{g}/\text{NM}^3$) and ranged from 1.15×10^{-2} gr/DSCF (26350 $\mu\text{g}/\text{NM}^3$) to 1.76×10^{-2} gr/DSCF (40440 $\mu\text{g}/\text{NM}^3$). All significant amounts of cadmium concentrations and emission rates were found in the front half of sampling train.

TABLE 2-1a (English Units)
Summary of Controlled Emissions
Method 5 Train with Filter
AMAX Zinc Co.
Baghouse Outlet

TEST NO	1A	3A	4A	AVG
DATE	6/17/86	6/18/86	6/18/86	---
TIME	0900-1106	1115-1322	1420-1635	---
<u>Sample Conditions</u>				
Volume (DSCF) ¹	67.61	52.86	54.73	--
Front Half Catch (µg)	77200	39500	61400	--
Back Half Catch (µg)	42.1	25.2	9.6	--
Total Catch (µg)	77242	39525	61410	--
Isokinesis (%)	114.2	104.6	102.5	--
<u>Stack Conditions</u>				
Temperature (°C)	233	227	230	229
<u>Cadmium Emissions</u>				
<u>Front Half</u>				
Concentration (gr/DSCF)	1.76×10^{-2}	1.15×10^{-2}	1.73×10^{-2}	1.55×10^{-2}
<u>Back Half</u>				
Concentration (gr/DSCF)	9.61×10^{-6}	7.36×10^{-6}	2.71×10^{-6}	6.56×10^{-6}
<u>Total Cadmium Emissions</u>				
Concentration (gr/DSCF)	1.76×10^{-2}	1.15×10^{-2}	1.73×10^{-2}	1.55×10^{-2}

¹ Standard Conditions: 29.92 in Hg @ 68°F

TABLE 2-1b (Metric Units)
Summary of Controlled Emissions
Method 5 Train with Filter
AMAX Zinc Co.
Baghouse Outlet

TEST NO	1A	3A	4A	AVG
DATE	6/17/86	6/18/86	6/18/86	---
TIME	0900-1106	1115-1322	1420-1635	---
<u>Sample Conditions</u>				
Volume (NM ³) ¹	1.91	1.50	1.55	--
Front Half Catch (µg)	77200	39500	61400	--
Back Half Catch (µg)	42.1	25.2	9.6	--
Total Catch (µg)	77242	39525	61410	--
Isokinesis (%)	114.2	104.6	102.5	--
<u>Stack Conditions</u>				
Temperature (°C)	112	108	110	110
<u>Cadmium Emissions</u>				
<u>Front Half</u>				
Concentration (µg/NM ³)	40420	26330	39610	35450
<u>Back Half</u>				
Concentration (µg/NM ³)	22.04	16.80	6.19	15.01
<u>Total</u>				
Concentration (µg/NM ³)	40440	26350	39620	35470

¹ Standard Conditions: 760 mm Hg @ 20°C

2.1b Stack Conditions

Stack temperature during this test series averaged 229°F and ranged from 233°F to 227°F.

2.2 Method Configuration 5 (w/out filter) Modified For Cadmium)

A total of four tests were conducted using the Method 5 train (without filter) modified for cadmium. The second test was deleted due to an unacceptable leak rate of >0.02 cfm. All other tests were acceptable with a leak rate of <0.02 cfm and 100±10 percent isokinesis.

2.2a Cadmium Emissions

Table 2-2a (English units) and Table 2-2b (Metric units) present a summary of measured cadmium emissions from the baghouse outlet.

The total average cadmium concentration was 1.62×10^{-2} gr/DSCF (37000 $\mu\text{g}/\text{NM}^3$) and ranged from 1.45×10^{-2} gr/DSCF (33220 $\mu\text{g}/\text{NM}^3$) to 1.61×10^{-2} gr/DSCF (36940 $\mu\text{g}/\text{NM}^3$).

2.2b Stack Conditions

Stack exhaust gas temperature during this test series averaged 236°F and ranged from 241°F to 230°F.

TABLE 2-2a (English Units)
Summary of Controlled Emissions
Method 5 Train w/o Filter
AMAX Zinc Co.
Baghouse Outlet

TEST NO	1B	3B	4B	AVG
DATE	6/17/86	6/18/86	6/18/86	---
TIME	0900-1106	0834-1215	1420-1636	---
<u>Sample Conditions</u>				
Volume (DSCF) ¹	64.63	66.03	62.40	--
Front Half Catch (µg)	49400	52600	40800	--
2nd Impinger Catch (µ)	7070	9920	31100	--
3rd&4th				
Impinger Catch (µg)	4330	6560	378	--
Total Catch (µg)	60800	69080	72278	--
Isokinesis (%)	109.2	109.4	107.6	--
<u>Stack Conditions</u>				
Temperature (°F)	230	241	238	236
<u>Cadmium Emissions</u>				
<u>Front Half (Probe Wash & 1st Impinger)</u>				
Concentration (gr/DSCF)	1.18×10^{-2}	1.23×10^{-2}	1.01×10^{-2}	1.14×10^{-2}
<u>2nd Impinger</u>				
Concentration (gr/DSCF)	1.69×10^{-3}	2.32×10^{-3}	7.69×10^{-3}	3.90×10^{-3}
<u>3rd&4th Impinger</u>				
Concentration (gr/DSCF)	1.03×10^{-3}	1.53×10^{-3}	9.35×10^{-5}	8.84×10^{-4}
<u>Total Cadmium Emissions</u>				
Concentration (gr/DSCF)	1.45×10^{-2}	1.61×10^{-2}	1.79×10^{-2}	1.62×10^{-2}

¹ Standard Conditions: 29.92 in Hg @ 68°F

TABLE 2-2b (Metric Units)
Summary of Controlled Emissions
Method 5 Train w/o Filter
AMAX Zinc Co.
Baghouse Outlet

TEST NO	1B	3B	4B	AVG
DATE	6/17/86	6/18/86	6/18/86	---
TIME	0900-1106	0834-1215	1420-1636	---
<u>Sample Conditions</u>				
Volume (NM ³) ¹	1.83	1.87	1.77	--
Front Half Catch (µg)	49400	52600	40800	--
2nd Impinger Catch (µg)	7070	9920	31100	--
3rd&4th				
Impinger Catch (µg)	4330	6560	378	--
Total Catch (µg)	60800	69080	72278	--
Isokinesis (%)	109.2	109.4	107.6	--
<u>Stack Conditions</u>				
Temperature (°C)	110	116	114	113
<u>Cadmium Emissions</u>				
<u>Front Half (Probe Wash & 1st Impinger)</u>				
Concentration (µg/NM ³)	26990	28130	23050	26060
<u>2nd Impinger</u>				
Concentration (µg/NM ³)	3860	5300	17570	8910
<u>3rd&4th Impinger</u>				
Concentration (µg/NM ³)	2370	3510	220	2030
<u>Total Cadmium Emissions</u>				
Concentration (µg/NM ³)	33220	36940	40840	37000

¹ Standard Conditions: 760mm Hg @ 20°C

2.3 Particle Size Distribution

Table 2-3 presents particle size data summary from the baghouse outlet. Figure 2-1 presents a graph of particle size to cumulative percent less than cut diameter. Table 2-3 includes sampling time, sample volume, effective cut diameter, size range and percent in size range.

Particle size data show that 26.0% of the particles are in the 0.68 to 1.11 μm size range, 21.3% in the 2.25-3.53 μm range and 18.5% of the particles are in the 1.11-2.25 μm size range. Figure 2-1 indicates that the particle size distribution is unimodal.

2.4 Visible Emissions

A summary of visible emission observations from the baghouse outlet is presented in Table 2-4. Average opacities are presented for 6 minute time periods during each test. The average opacity was 3% for tests 1, 2 and 4 and 5% for test 3. These 6-minute average opacities are presented graphically in figures 2-2 through 2-5.

2.5 Trace Metals

One sample and blank per set of tests, baghouse dust and lab blanks were analyzed for trace metals by A.A. Barium, chromium, copper, iron, lead, magnesium, manganese, mercury, nickel and zinc were analyzed. Mercury was analyzed for the train without the filter but not for the train with the filter. Mercury was not analyzed for the train with the filter because of the difficult digestion procedure. The baghouse dust and road dust was also analyzed for cadmium. Table 2-5 presents a summary of the trace metals results. All sample train results were corrected for field and/or laboratory blanks. Trace metals for the sample train blanks were high, possibly due to the nitric acid rinse of the nozzles.

TABLE 2-3
Particle Size Data Summary
Baghouse Outlet
AMAX Zinc Co.

June 18, 1986

SAMPLING TIME: 90 MINUTES

SAMPLE VOLUME: 46.50 DSCF

STAGE	EFFECTIVE CUT DIAMETER	SIZE RANGE (μ)	STAGE SAMPLE (Mg)	% IN SIZE RANGE	CUMULATIVE % LESS THAN CUT DIAMETER
PS&O	>11.16	>11.16	0.07	0.15	99.8
1	7.06	7.06-11.16	0.69	1.5	98.3
2	5.19	5.19-7.06	4.69	10.4	87.9
3	3.53	3.53-5.18	6.72	14.9	73.0
4	2.25	2.25-3.53	9.63	21.3	51.7
5	1.12	1.12-2.25	8.34	18.5	33.2
6	0.680	0.680-1.12	11.71	26.0	7.2
7	0.457	0.457-0.680	1.50	3.3	3.9
Backup	<0.457	<0.457	<u>1.77</u> 45.12	3.9	--

Table 2-4
Summary of Visible Emissions
Baghouse Outlet
AMAX Zinc Co.
East St. Louis, Illinois

TEST NO 1 6/17/86 0900-1116		TEST NO 2 6/17/86 1308-1520		TEST NO 3 6/18/86 0828-1328		TEST NO 4 6/18/86 1420-1644	
SIX MINUTE TIME PERIOD	AVERAGE OPACITY (%)	SIX MINUTE TIME PERIOD	AVERAGE OPACITY (%)	SIX MINUTE TIME PERIOD	AVERAGE OPACITY (%)	SIX MINUTE TIME PERIOD	AVERAGE OPACITY (%)
0909-0915	4	1308-1314	4	0828-0834	4	1420-1426	0
0915-0921	5	1314-1320	5	0834-0840	4	1426-1432	0
0921-0927	5	1320-1326	4	0840-0846	5	1432-1438	3
0927-0933	4	1326-1332	4	0846-0852	5	1438-1444	1
0933-0939	6	1332-1338	3	0852-0858	5	1444-1450	0
0939-0945	3	1338-1344	1	0858-0904	5	1450-1456	3
0945-0951	4	1344-1350	2	0904-0910	4	1456-1502	1
0951-0957	4	1350-1356	1	0910-0916	4	1502-1508	0
0957-1003	3	1356-1402	3	0916-0922	5	1508-1514	2
1003-1009	3	1402-1408	2	0922-0928	5	1514-1520	2
1009-1015	3	1408-1414	0	0928-0934	5	1520-1526	3
1015-1021	3	1414-1420	3	0934-0940	5	1526-1532	2
1021-1027	2	1420-1426	4	0940-0946	4	1532-1538	4
1027-1033	2	1426-1432	3	STOP TEST		1538-1544	5
1033-1039	1	1432-1438	1	1110-1116	5	1544-1550	4
1039-1045	2	1438-1444	4	1116-1122	5	1550-1556	4
1045-1051	1	1444-1450	5	1122-1128	5	1556-1602	1
1051-1057	2	1450-1456	4	1128-1134	5	1602-1608	2
1057-1103	1	1456-1502	3	1134-1140	8	1608-1614	5
1103-1109	0	1502-1508	3	1134-1140	8	1614-1620	4
AVG	3	1508-1514	2	1140-1146	7	1620-1626	4
		1514-1520	1	1146-1152	7	1626-1632	3
		AVG	3	1152-1158	5	1632-1638	5
				1158-1204	6	AVG	3
				1204-1210	4		
				1210-1216	1		
				1216-1222	0		
				1222-1228	3		
				1228-1234	5		
				1234-1240	5		
				1240-1246	5		
				1246-1252	5		
				1252-1258	5		
				1258-1304	2		
				1304-1310	2		
				1310-1322	5		
				1322-1328	5		
				AVG	5		

TABLE 2-5
TRACE METALS
AMAX ZINC CO
BAGHOUSE

JUNE 1986

SAMPLE	ROAD DUST	HNO ₃ BLANK	LAB BLANK
<u>TRACE METAL</u>			
Barium (µg)	----	nd>25	nd<25
Cadmium ¹	7850µg/g	--	--
Chromium (µg)	----	nd<5.0	nd<5.0
Copper (µg)	----	nd<4.0	nd<4.0
Iron (µg)	----	0.131µg/ml	nd<7.0
Lead (µg)	----	nd<23	nd<23
Magnesium (µg)	----	0.023µg/ml	2.7
Manganese (µg)	----	nd<4.0	nd<4
Mercury (µg) ³	----	--	--
Nickel (µg)	----	nd<7.0	nd<7.0
Zinc (µg)	----	0.276µg/ml	nd<0.9

¹ Cadmium considered major metal refer to Tables 2-1 and 2-2

² nd = Non Detected

³ Mercury was not analyzed due to the difficult digestion procedure for the train with filter

CUMULATIVE PERCENT GREATER THAN

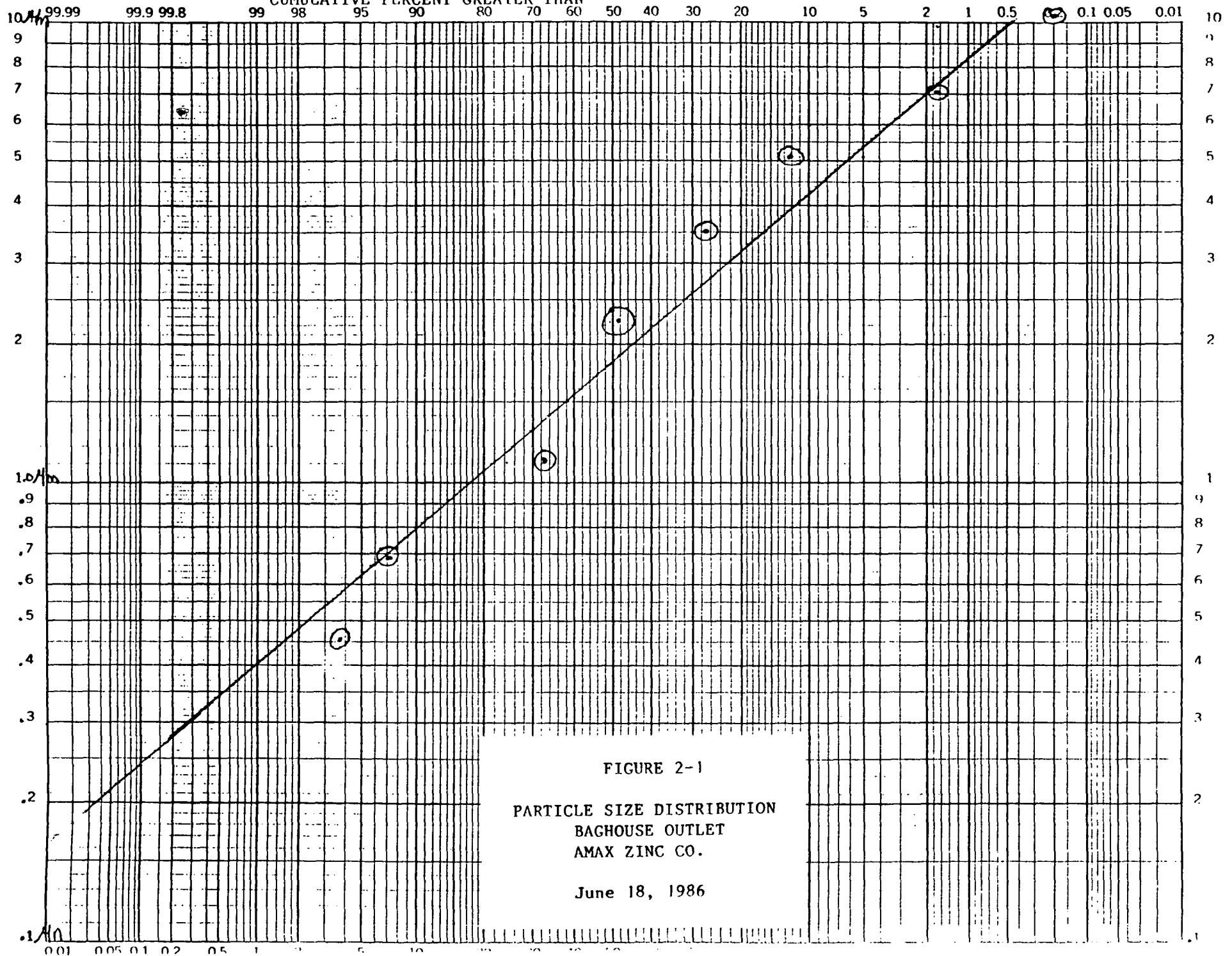


FIGURE 2-1

PARTICLE SIZE DISTRIBUTION
BAGHOUSE OUTLET
AMAX ZINC CO.

June 18, 1986

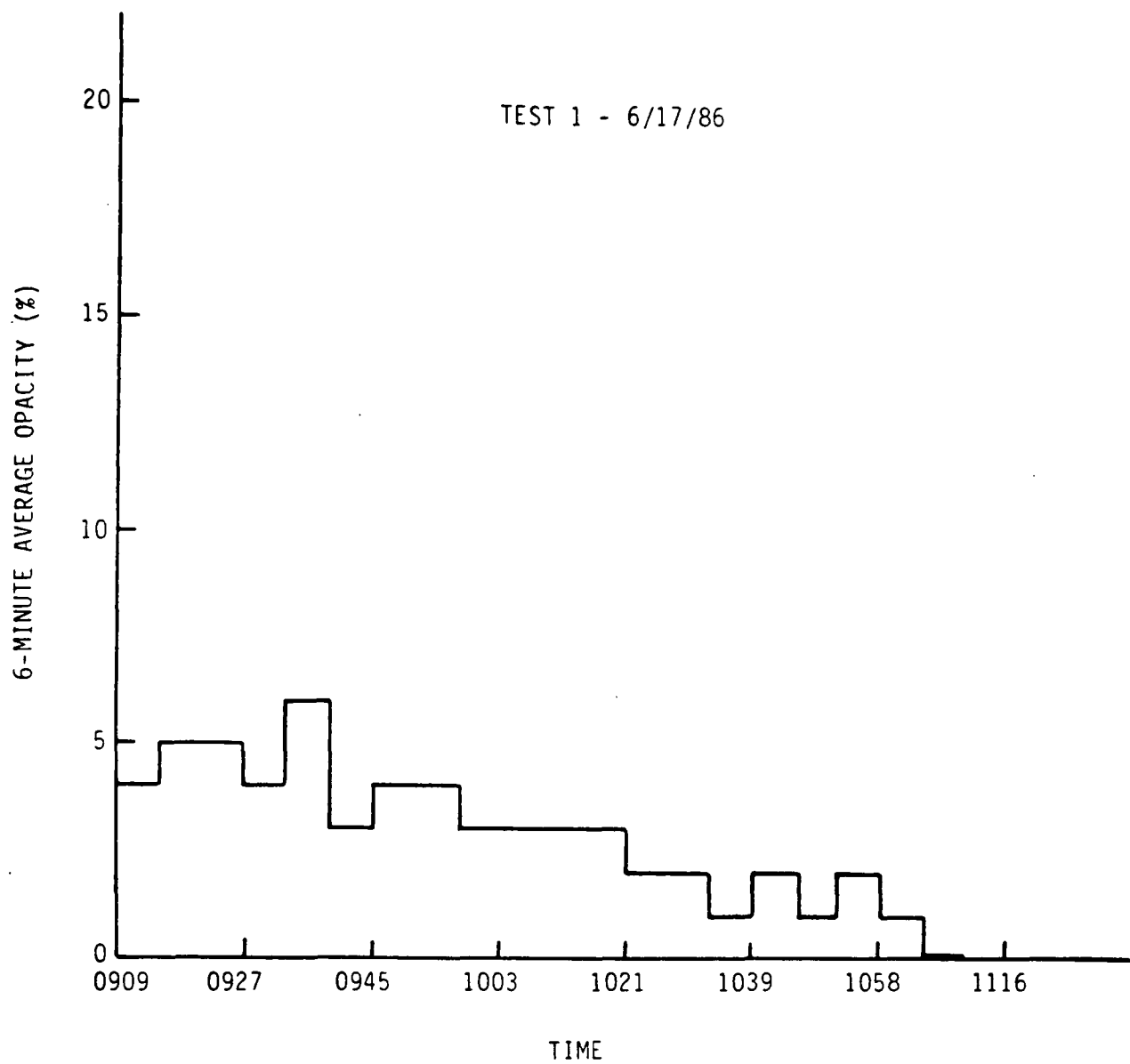


Figure 2-2. Summary of Visible Emissions form Baghouse Outlet
Amax Zinc Co., E. St. Louis, Illinois

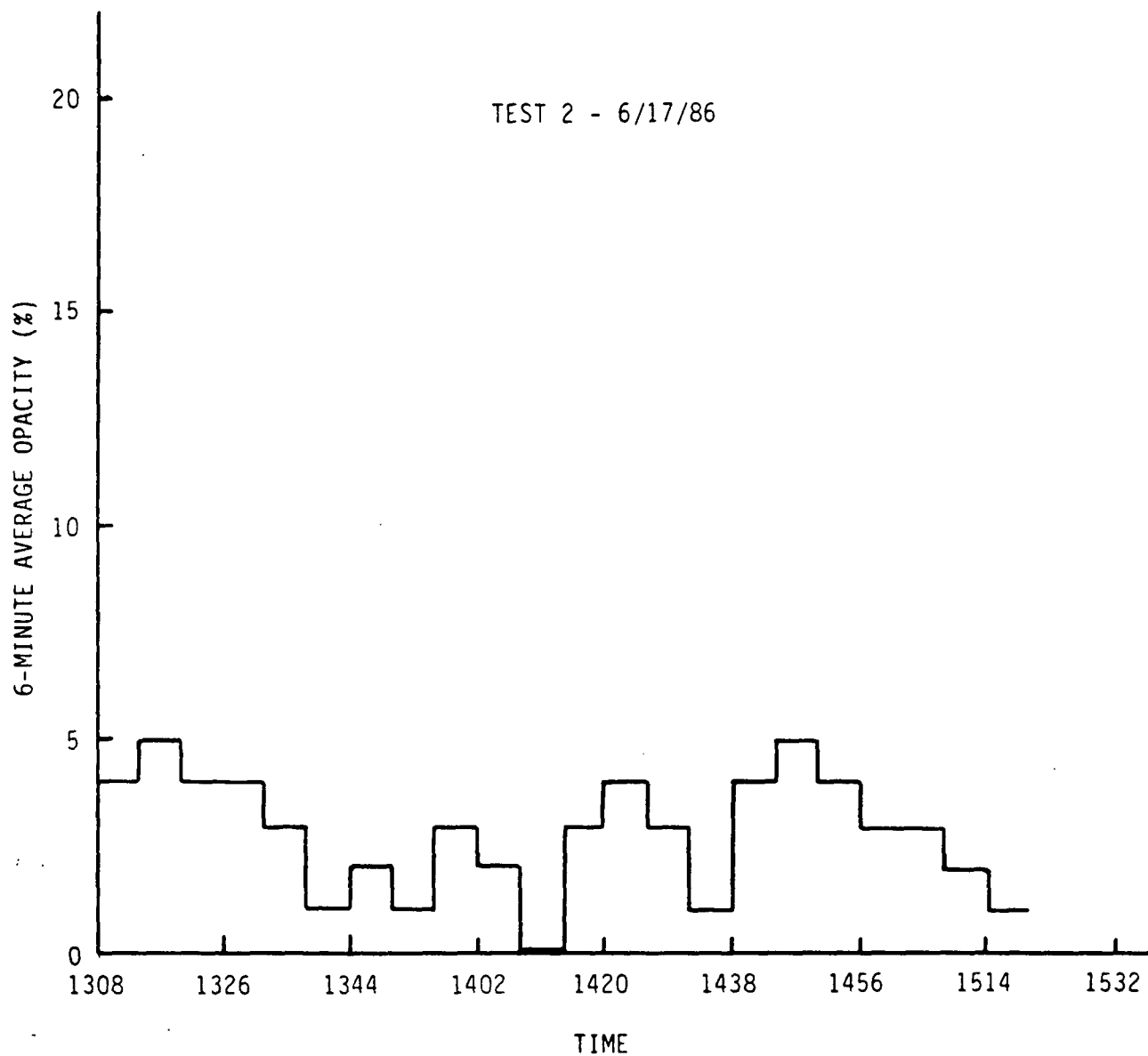


Figure 2-3. Summary of Visible Emissions from Baghouse Outlet
Amax Zinc Co., E. St. Louis, Illinois

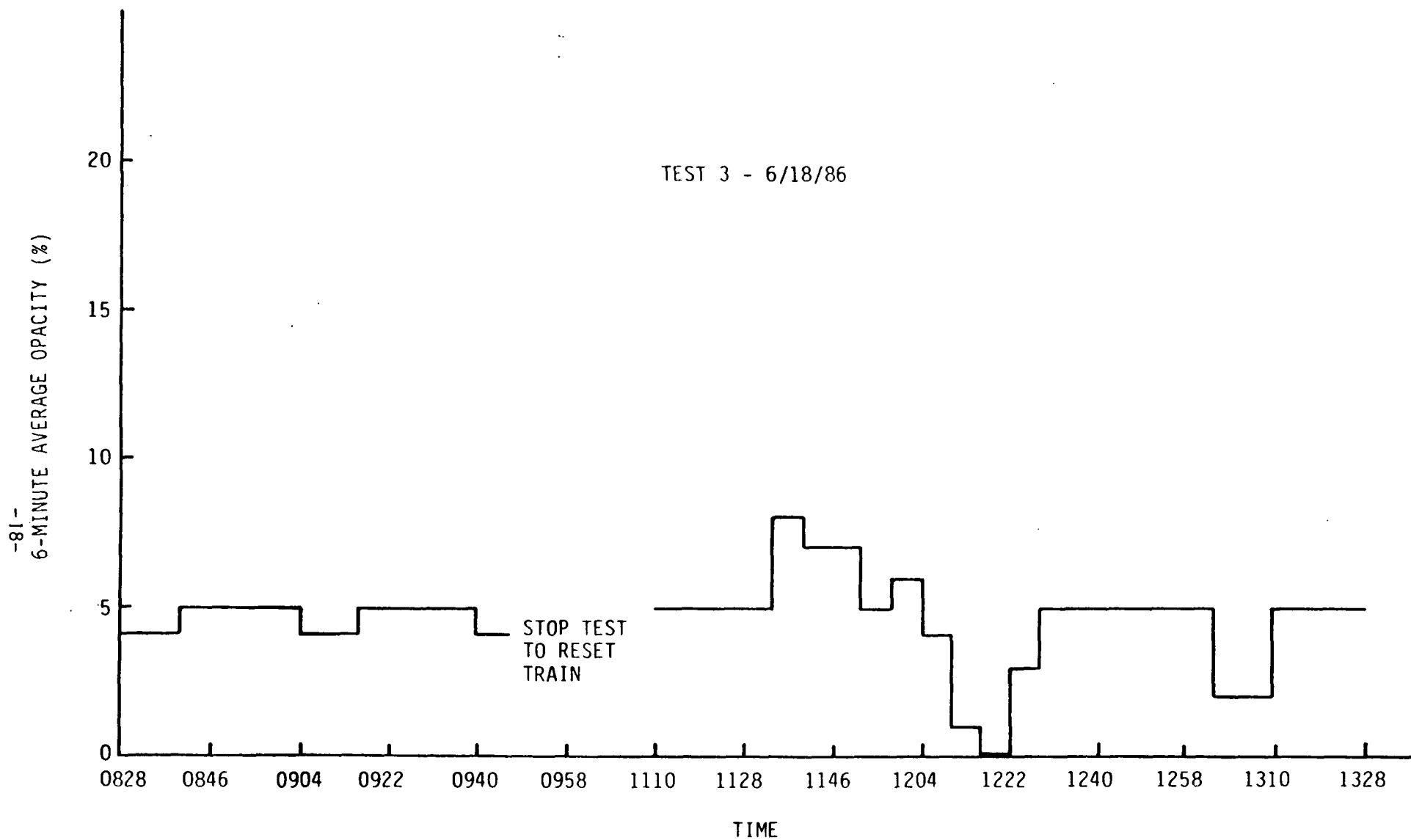


Figure 2-4. Summary of Visible Emissions from Baghouse Outlet
Amax Zinc Co., E. St. Louis, Illinois

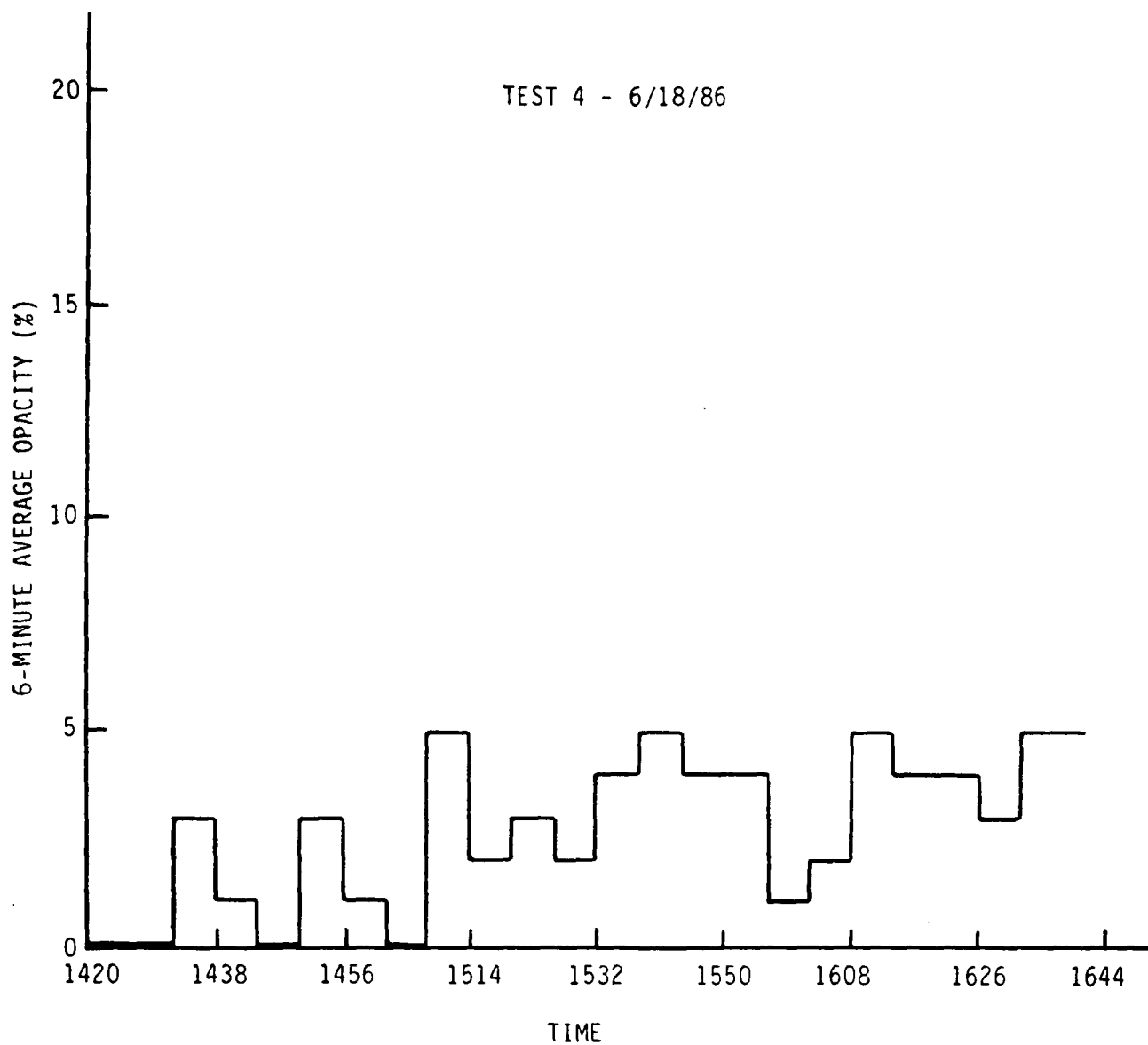


Figure 2-5. Summary of Visible Emissions from Baghouse Outlet
Amax Zinc Co., E. St. Louis, Illinois

3.0 PROCESS DESCRIPTION AND OPERATIONS (Provided by MRI)

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3.1 Process Equipment

CONFIDENTIAL

3.2 Control Equipment

CONFIDENTIAL

4.0 DESCRIPTION OF SAMPLING LOCATION

This section presents a description of each sampling location. Figure 4-1 presents a schematic layout of the cadmium oxide process and identifies all sampling locations.

4.1 Cadmium and Particle Size

The cadmium oxide baghouse outlet was sampled in the 19-inch diameter duct which exits the top of the baghouse, enters the I.D. fan and exits through a stack. Sample ports are located 90° apart on the duct section preceding the fan 12 diameters downstream of a bend and 1.8 diameters upstream of the ID fan. Point A in figure 4-1 presents this location.

In accordance with EPA Method 1, sampling was performed at 12 traverse points through the two sample ports simultaneously. Figure 4-2 presents the sample port configuration and a cross section of the duct showing the exact distance of each sampling point from the duct wall.

Each Method 5 test lasted 120 minutes (10 minutes traverse point). The particle size test was run for 90 minutes. Particle size sampling was performed at an average point of flow in the stack.

4.2 Visible Emissions Observation Locations

A certified visible emissions observer read at the exhaust opacity at the cadmium oxide baghouse stack for a period of 2 hours during each test. Sample Point C in Figure 4-1 depicts the exhaust point.

The observers location was determined in accordance with EPA Method 9 and considering obstructions and traffic problems in the area.

The Method 9 data reduction was calculated by taking 6 minute averages for the entire length of the test. Location of the observer can be found in Figure 4-3.

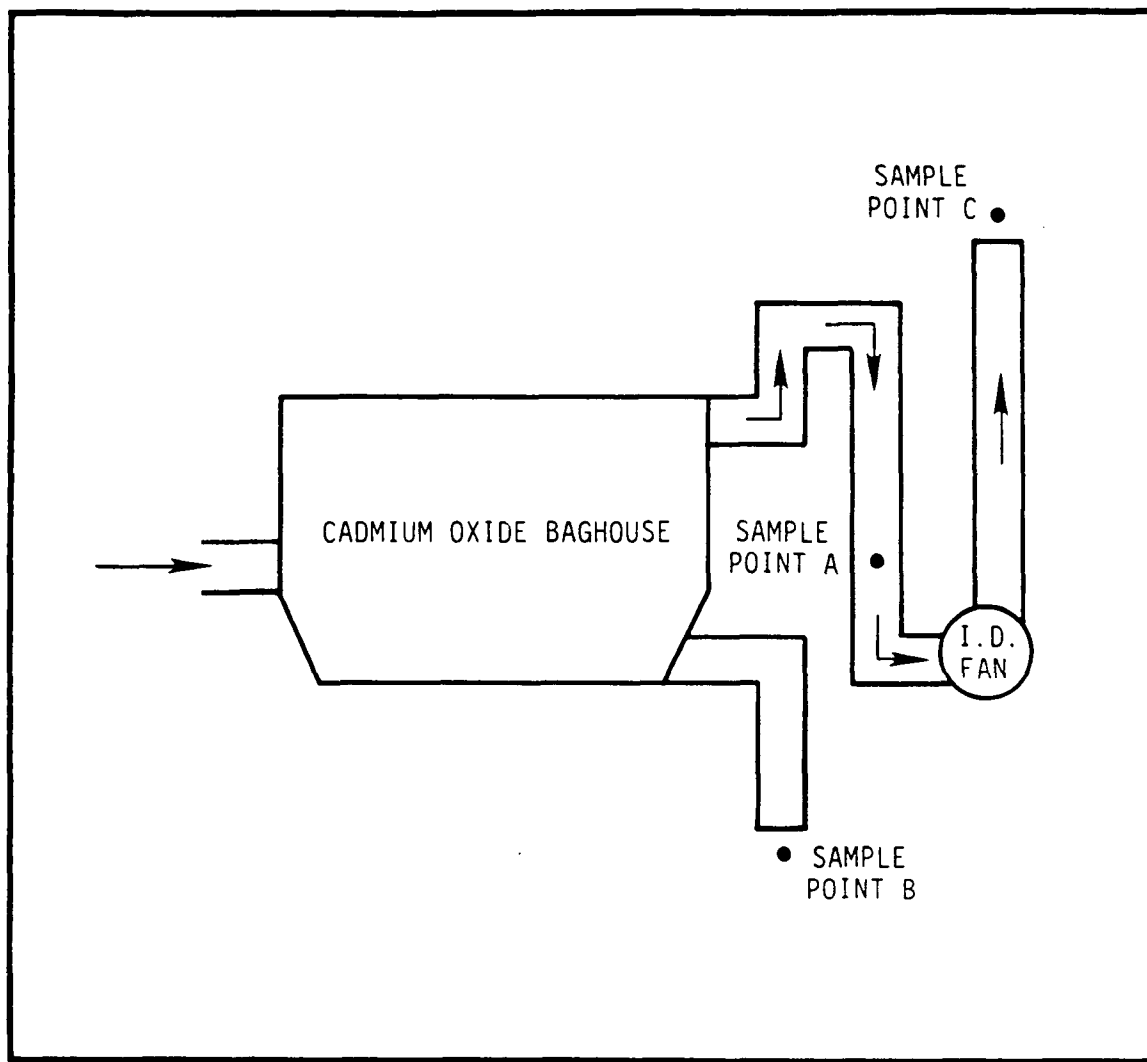


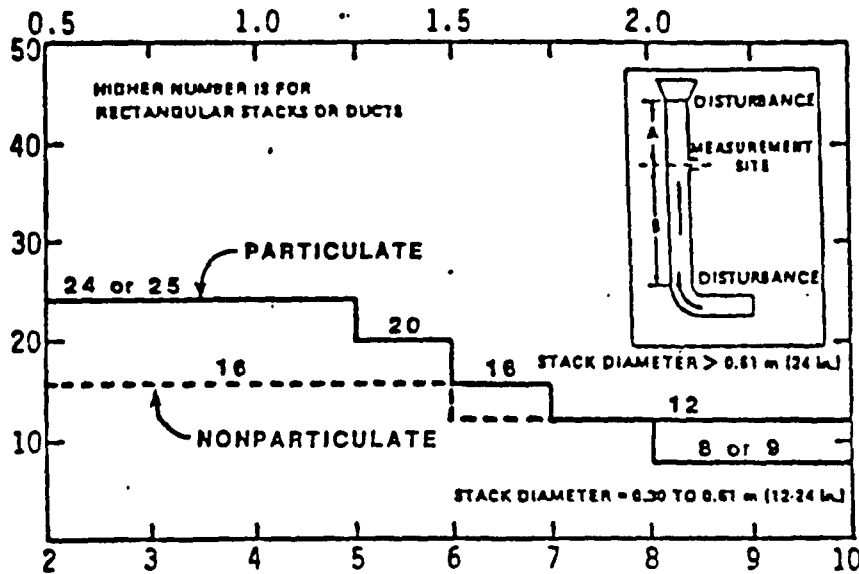
Figure 4-1. Process and Sampling Points
AMAX Zinc Co., Sauget, Ill.

FIGURE 4-2

Firm EPA/AMAX ZINC CO. Total Traverse Points Required 12
 Location CADMIUM OXIDE BAGHOUSE Number of Ports 2
 Diameters Upstream 12 Points Per Port 6
 Diameters Downstream 1.8 Traverse (Horizontal or Vertical) HORIZONTAL

**MINIMUM NUMBER OF TRAVERSE POINTS FOR PARTICULATE
AND NONPARTICULATE TRAVERSES**

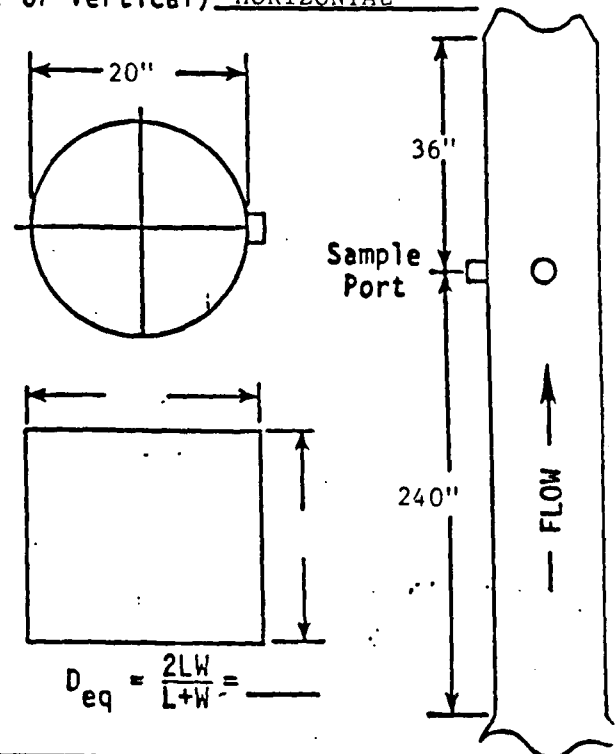
Duct Diameters Upstream from Flow Disturbance
(Distance A)



Duct Diameters Downstream from Flow Disturbance
(Distance B)

LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS

Point Number On A Diameter	(Percent of stack diameter from inside wall to traverse point)				
	Number Of Traverse Points On A Diameter				
	4	6	8	10	12
1	6.7	4.4	3.2	2.6	2.1
2	25.0	14.6	10.5	8.2	6.7
3	75.0	29.6	19.4	14.6	11.8
4	93.3	70.4	32.3	22.6	17.7
5		85.4	67.7	34.2	25.0
6		95.6	80.6	65.8	35.6
7			89.5	77.4	64.4
8			96.8	85.4	75.0
9				91.8	82.3
10				97.4	88.2
11					93.3
12					97.9



CROSS-SECTIONAL LAYOUT FOR RECTANGULAR STACKS	
Total	Matrix
9	3x3
12	4x3
16	4x4
20	5x4
25	5x5

TRAVERSE POINT LOCATIONS

No.	Distance From Wall	Nipple Size	Total Distance
1	0.9		
2	2.9		
3	5.9		
4	14.1		
5	17.1		
6	19.1		
7			
8			
9			
10			
11			
12			

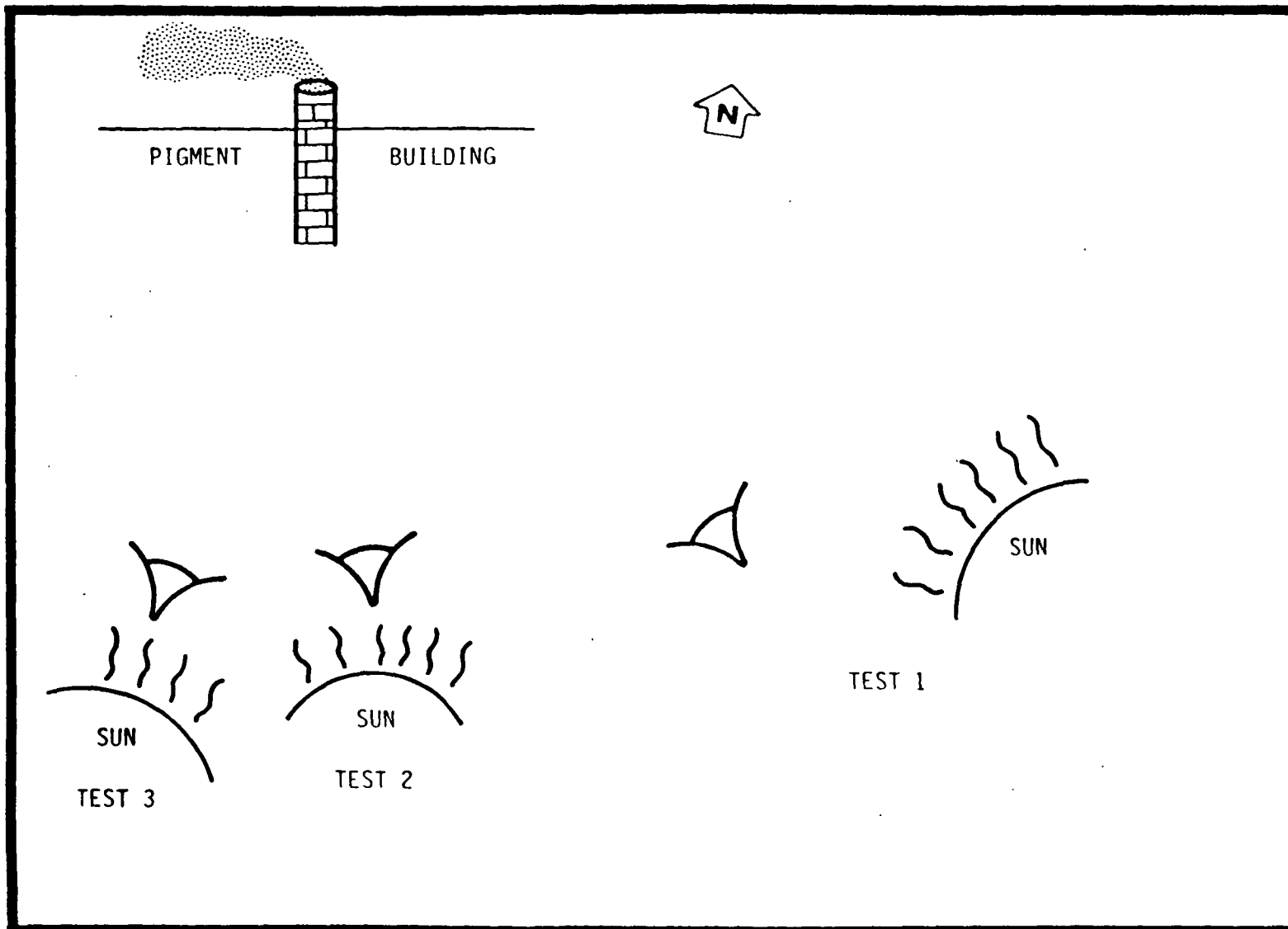


Figure 4-3 Observers Location
Visible Emission
Amax Zinc Co
Baghouse Outlet
June 1986

4.3 Cadmium Product Composite Sample

A sample of the baghouse dust was collected at Sample Point B as shown in Figure 4-1. Samples were collected at half hour intervals during each test run. All half hour samples were then mixed into one representative sample. The resulting composite was returned to TRC and analyzed for trace metals by GFAA using a Parr Bomb digestion with nitric acid.

5.0 SAMPLING AND ANALYTICAL PROCEDURES

This section presents descriptions of sampling and analysis procedures which were employed during the emission testing conducted at the AMAX Zinc Company facility in East St. Louis, IL.

A combination of EPA Methods 1, 2, 3, 4, 5 (modified for Cadmium), and 9 were used to measure cadmium emissions from the Cadmium Oxide Baghouse exhaust. In addition, one Andersen cascade impactor sample was taken to determine particle size distribution.

A composite sample of cadmium product was analyzed for trace metals using a Parr Bomb digestion and graphite furnace atomic absorption (GFAA).

5.1 EPA Method 5 with filter (Modified for Cadmium)

Cadmium sampling was performed by using two configurations of the Method 5 sampling train¹. The first is shown schematically in Figure 5-1 and consists of a nozzle, probe, filter with glass frit, a flexible Teflon umbilical line, four impingers, vacuum pump, dry gas meter, and an orifice flow meter. One modification of the standard EPA Method 5 train consists of placing flexible Teflon tubing between the filter and the impingers. This modification makes the sampling equipment much easier to handle. A second modification is the use of 5% nitric acid in first two impingers. The sampling train was calibrated before and after this test program. This method is based on the proposed methodology presented in Appendix F.

¹ Code of Federal Regulations 40, Part 60 Appendix A, July 81

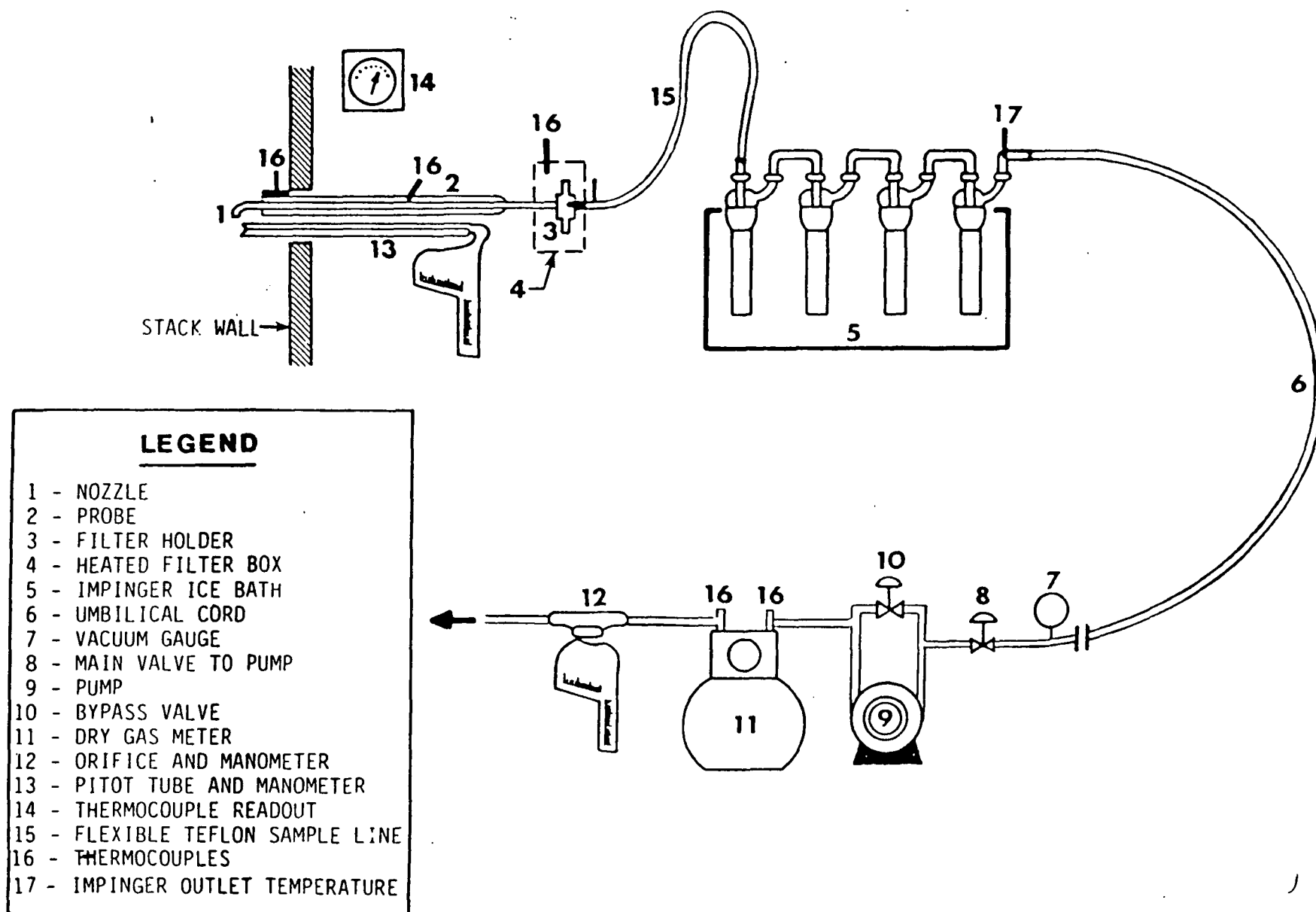


Figure 5-1. Modified EPA Method 5 cadmium sampling train
(August 18, 1977 Federal Register)

A nozzle was attached to a stainless steel glass-lined probe which was heated to prevent condensation. Whatman EPM-1000 fiberglass filter paper supported in a 4 1/2 inch glass filter holder with a glass frit was used as a particulate collection medium. Filters were desiccated and pre weighed. A visual inspection for irregularities in the filter material was conducted. The filter assembly was enclosed in a heated box to keep the filter temperature at approximately 150° F (± 10), which is approximately the same as the stack temperature. A thermocouple, located inside the back half of the filter holder, monitors the gas stream temperature to ensure proper filter temperature.

Four impingers immersed in an ice bath were attached to the back end of the filter holder with a flexible Teflon tube. The first two impingers each contained 100 ml of 5% nitric acid, the third was empty and the fourth contained 200 grams of silica gel to remove any remaining moisture. Impinger outlet temperatures were kept between 50°F to 80°F.

Flexible tubing, vacuum gauge, needle valve, leakless vacuum pump, bypass valve, dry gas meter, calibrated orifice and inclined manometer complete the sampling train. A check valve was not used in the TRC sampling train.

A nomograph was used to quickly determine the orifice pressure drop required for any pitot velocity pressure and stack temperature in order to maintain isokinetic sampling conditions. Sampling flow was adjusted by means of the bypass valve. Before and after each particulate test run the sampling train was leak checked. Sample time was 120 minutes per run.

Test data recorded at ten minute intervals for each sampling point included: test time, sampling duration at each traverse point, pitot pressure, stack temperature, dry gas meter volume and inlet-outlet temperatures, probe temperature, and orifice pressure drop.

Due to the large negative static pressure in the duct, sampling trains were started and stopped prior to stack entry and stopped after stack exit. This procedure mitigated any effects of suction on the sampling media.

Sample Recovery

Sample recovery was performed in a clean, wind-free area to avoid sample contamination. A 22 foot by 8 foot high-cube truck provided shelter. The following fractions were recovered:

- Container No. 1 The glass fiber filter was removed from its holder and deposited in an inert petri dish and then sealed.
- Container No. 2 The probe, nozzle and front-half of the filter holder was rinsed and brushed three times with 5% HNO₃ into a 500 ml glass sample jar with a Teflon-lined lid.
- Container No. 3 The Teflon line was drained into the first impinger. The first three impinger's volumes were determined gravimetrically to the nearest 0.5g. The contents were then deposited into a 1000 ml glass sample jar with a Teflon lined lid. The back-half of the filter holder, the Teflon line, and the first three impingers were each rinsed three times with 5% HNO₃, measured then added into the 1000 ml sample jar.
- Container No. 4 The silica gel was be returned to its original container and weighed to the nearest 0.5g.

Sample Analyses

Sample analyses was performed in TRC Environmental Laboratories in East Hartford, CT. The sample fractions were analyzed as follows:

- Container No.1 The filter was desiccated and weighed for particulate. The filter was digested, extracted and then analyzed for cadmium by graphite furnace atomic absorbtion. One set of samples was be analyzed for trace metals.
- Container No.2 Nozzle and probe rinse was dried and weighed, then brought back to solution with HNO₃. The solution was extracted and analyzed as Container No. 1.
- Container No. 3 The solution was analyzed as Container No. 2.
- Container No. 4 The silica gel was weighed to the nearest 0.5g.

Trace metals standards purchased from J. T. Baker Standards, which are NBS traceable, were used for all trace metals analyses. Samples were concentrated or diluted in order to bring the analysis range into the center of the Atomic Absorption linearity curve. All samples were analyzed in triplicate as a measure of precision. A complete description of the analytical procedure can be found in the Appendix of this test report.

The trace metals analyzed for are:

Barium
Chromium
Copper
Iron
Lead
Magnesium
Manganese
Nickel
Zinc

5.2 EPA Method 5 (without filter) Modified for Cadmium

A second Method 5 sampling train for simultaneous cadmium testing was used as a method development tool to determine the best sample train component arrangement for cadmium. This sampling train is shown in Figure 5-2. This train consisted of a heated probe ($150^{\circ} \pm 10^{\circ}\text{F}$) and five impingers. The first three each contained 100 ml of 5% HNO_3 . The fourth was empty and the last contained 200 grams of silica gel. No filter was present in this train so any particulate captured was collected in the impinger solutions.

Impinger outlet temperatures did not exceed 80°F . Data was recorded as previously described in Section 5.1.

Sample Recovery

Sample recovery was performed in the location described in Section 5.1.

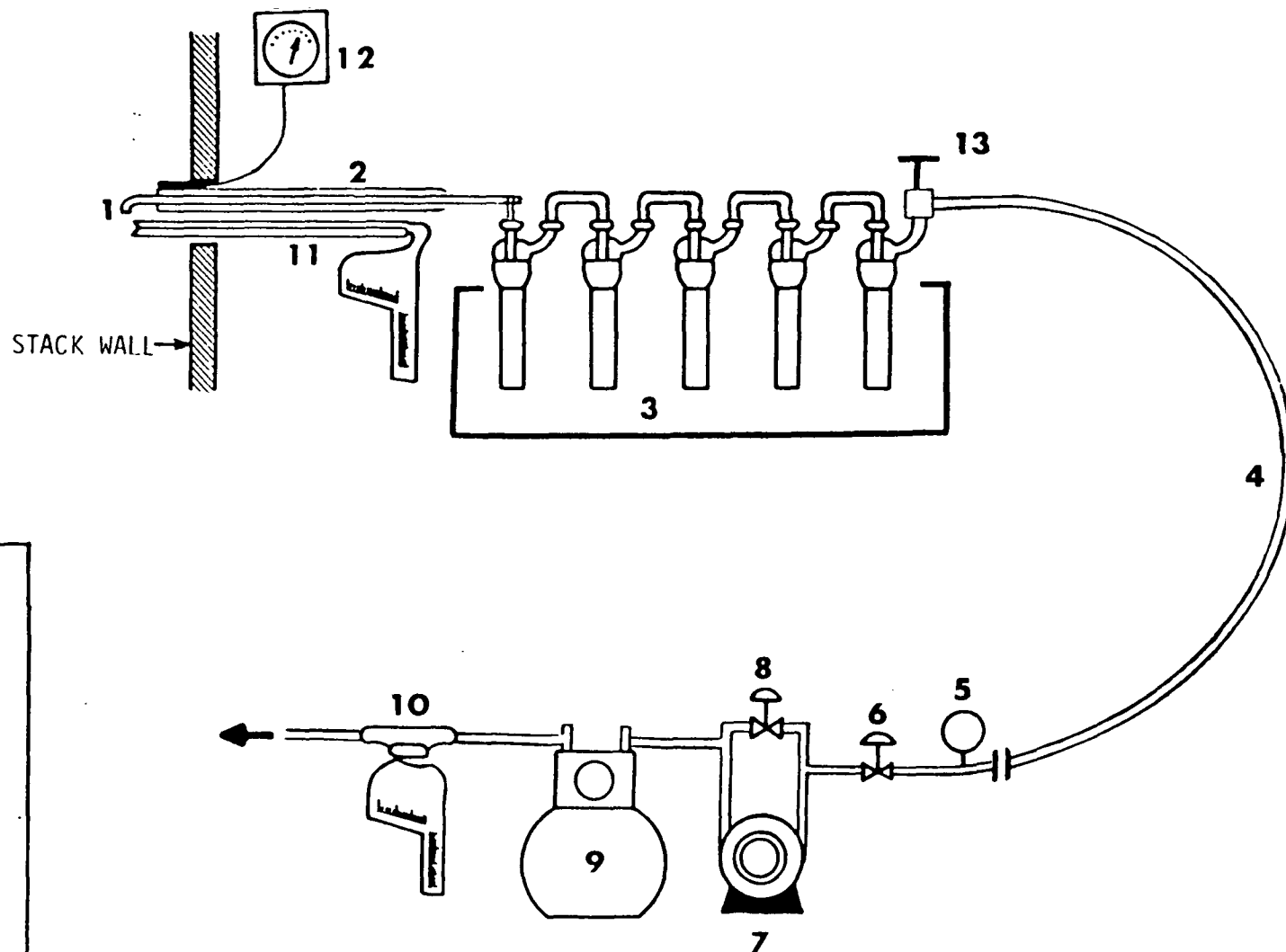
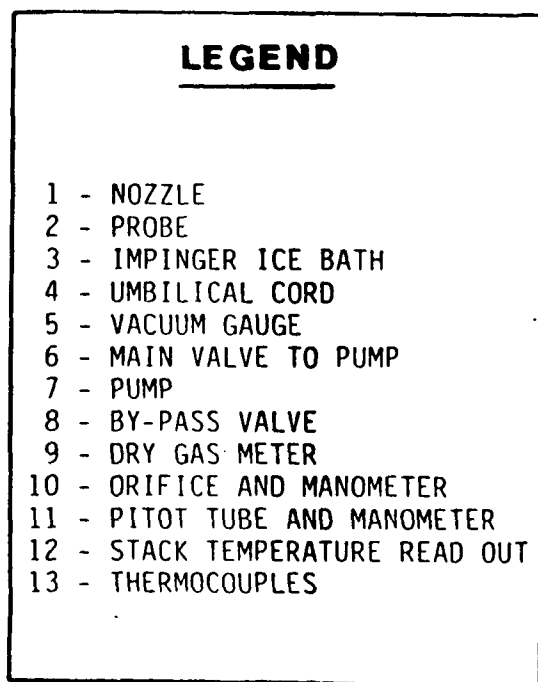


Figure 5-2. Modified EPA Method 5 Cadmium Sampling Train
(modified for cadmium sampling method comparison)

- Container No. 1 The probe and nozzle were rinsed and brushed three times with 5% HNO₃ into a 1000 ml glass sample jar with a Teflon-lined lid. The first impinger was weighed to determine moisture gain and the contents deposited in the jar. The impinger was then rinsed three times with 5% HNO₃, measured and added into the sample jar.
- Container No. 2 The second impinger was weighed to determine moisture gain and the contents deposited into a 500 ml glass sample jar with a Teflon-lined lid. The impinger was then rinsed three times with 5% HNO₃, measured and added into the jar.
- Container No. 3 Impingers 3 and 4 were combined and treated as Impinger 2.
- Container No. 4 The silica gel was weighed to the nearest 0.5 g to determine moisture gain.

Sample Analyses

Containers 1 through 3 was analyzed for cadmium and trace metals by GFAA as described in Section 3.1.

5.3 Particle Size Distribution Sample Collection

The particle size distribution sampling train is shown schematically in Figure 5-3. The train consisted of a nozzle, Andersen right-angle inlet pre-separator, Andersen Mark III cascade impactor, probe, flexible umbilical line, impinger, vacuum pump, dry gas meter, and orifice flowmeter. Reeve Angel 934AH fiberglass substrates are used as the sample collection media in the impactor. One impinger in an ice bath was connected to the probe by a flexible umbilical tube. The impinger contained silica gel to remove sample stream moisture prior to the gas meter. A vacuum gauge, needle valve, leakless vacuum pump, bypass valve, dry gas meter, calibrated orifice, and inclined manometer complete the sampling train.

Before each particle size sample collection run, the assembled sampling train was leak checked. The acceptable leak rate was less than 0.02 cfm at 5 inches of mercury vacuum. A nomograph was used to determine the orifice

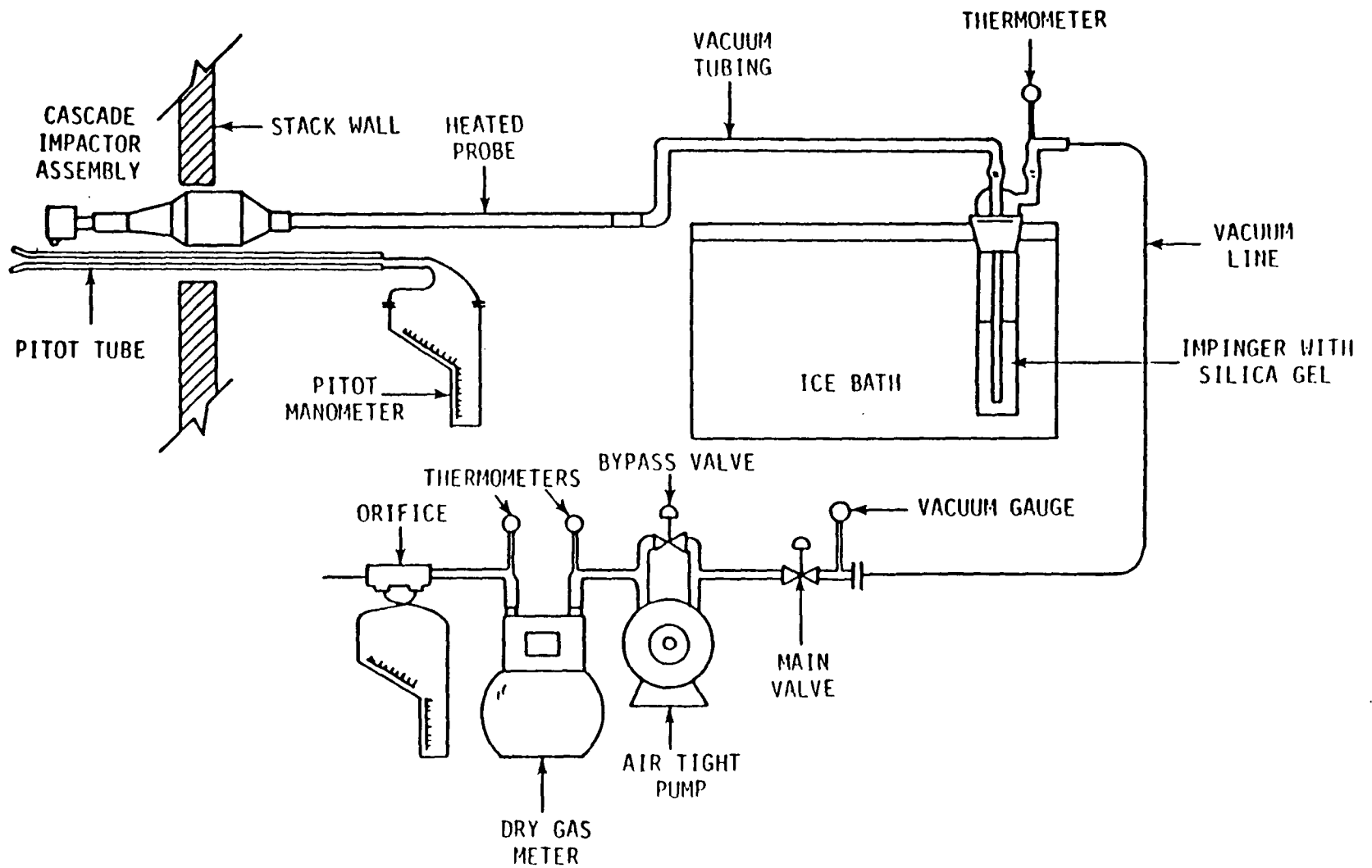


Figure 5-3. Particle size distribution sampling train.

pressure drop required to maintain isokinetic sampling conditions at the sample point velocity and temperature. Sampling time of 90 minutes was observed.

At the conclusion of the sampling time period, the sample flow was shut off and the pre-separator/impactor assembly carefully removed from the duct and maintained in a horizontal position. The nozzle and outlet ends were then sealed with Parafilm® and the assembly kept vertical and carefully transported to the clean-up area.

Sample recovery performed in the same area described in Section 5.1. The recovered sample fractions are identified as follows:

- Container No. 1: Acetone wash from the nozzle, pre-separator, interconnecting coupling, and impactor inlet throat.
- Container No. 2 through 8: Individual substrates and back-up filters in their respective containers.

The following analyses was performed on these samples:

- Container No. 1: Transfer the acetone washing to a tared beaker and evaporate to dryness at ambient temperature and pressure. Desiccate and dry to a constant weight. Report results to the nearest 0.1 mg.
- Containers No. 2 through 8: Transfer the filter, and any loose particulate matter from the sample container to a tared glass weighing dish, desiccate the dry to a constant weight. Report results to the nearest 0.1 mg.

A calculator program developed by TRC is used to calculate volume sampled at standard conditions, particulate concentration (grains/DSCF), flowrate (ACFM), viscosity, mean free path, and cutoff diameter per stage.

5.4 Cadmium Dust Analyses

Samples of cadmium dust from the baghouse were drawn during each test for trace metals and particle size distribution analyses. One 500 ml glass sample jar with a Teflon-lined lid was filled at half-hour intervals during each emission test. These samples were composited for one set of trace metals analyses. A complete outline of analytical procedure can be found in Appendix F.

Trace Metals Analyses

One dust sample was digested in nitric acid in a Parr Bomb and then analyzed for trace metals by GFAA. The sample was broken into nine aliquots individually analyzed as a measure of precision. The following summarizes principal Trace Metals for analyses for this test program:

- Barium
- Chromium
- Copper
- Iron
- Lead
- Magnesium
- Manganese
- Nickel
- Zinc

6.0 QUALITY ASSURANCE

TRC's quality assurance program for source emission measurement is designed so that the work is done by competent, experienced individuals using properly calibrated equipment and approved procedures for sample collection, recovery and analysis with proper documentation in various appendices as noted in the Table of Contents.

Specific details of TRC's quality assurance program for stationary air pollution sources may be found in the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III (EPA-600/4-7-027b).

At the beginning of each day, an organizational meeting was held to orient personnel to the day's activities, to discuss results from the previous day, and to determine if any special considerations are appropriate for the day's work.

Method 5

TRC's measurement devices, pitot tubes, dry gas meters, thermocouples, probes and nozzles are uniquely identified and calibrated with documented procedures and acceptance criteria before and after each field effort. Records of all calibration data are maintained in TRC files. Samples of these calibration forms are presented in Appendix.

All Method 5 sampling was 100 ± 10 percent isokinetic. Filter outlet temperatures were maintained at $150 \pm 10^\circ\text{F}$.

Clean-up evaluations were performed on each initial set of glassware prior to collecting field samples.

The Method 5 sampling train with the glass fiber filter had a clean-up sequence as follows:

- Blank 1
- Run 1
- Blank 2
- Run 2
- Blank 3
- Run 3
- Blank 4

The Method 5 sampling train without the glass fiber filter had a clean-up sequence as follows:

Blank 1
Run 1
Run 2
Run 3
Blank 2

All blank sample recovery was conducted identically to sample recovery procedures outlined in Section 5 of this test plan.

Blanks of all solution used during the test program (HNO_3) Filter media was collected and analyzed.

In summary, the evaluation tests shall be designed to precondition the sample collectors, to establish blank background values, and to educate the clean-up personnel in specific sample recovery procedures.

All sample recovery was performed by a one person clean-up crew. Appropriate sample recovery data was recorded on the sample identification log, sample handling log, chain of custody form, and analytical data forms as presented in Appendix C.

Recovered samples were secured in padlocked, shock-proof, steel containers for storage and shipment for analysis.

All preparation and analysis of Method 5 samples will be performed by TRC. TRC will adhere to the standards of quality assurance as set forth in Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III (EPA-600/4-7-027b) and the Handbook for Analytical Quality Control in Water and Wastewater Laboratories. (EPA-600/4-79-019, March 1979).

Method 9