

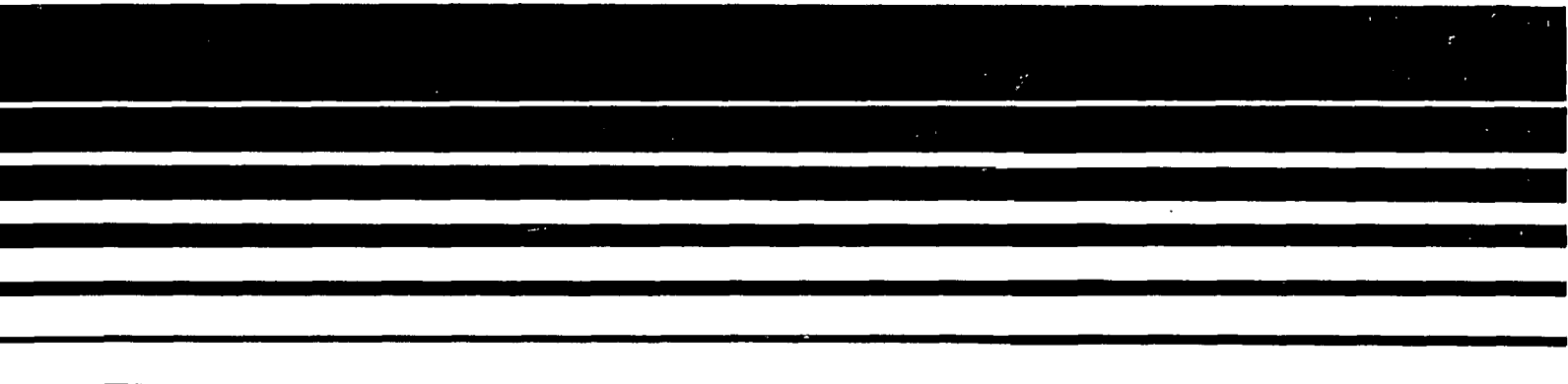
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# **Cadmium Screening Study Test Report**

**Cadmium Sulfide  
Pigments  
Harshaw/Filtrol  
Partnership  
Louisville,  
Kentucky**



NESHAP DEVELOPMENT  
CADMIUM EMISSION TESTING AT THE  
HARSHAW/FILTROL PARTNERSHIP  
LOUISVILLE, KENTUCKY

JULY 1986

EPA Contract No. 86-02-4337  
ESED Project No. 80/42  
Work Assignment No. 1  
CAD-2 Louisville, KY  
TRC Project No. 3497-E81-90

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November 1986

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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 Harshaw/Filtrol Partnership in Louisville, Kentucky.

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. Sam Narvarte, Technical Superintendent of Harshaw/Filtrol, 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) from sources 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 Harshaw/Filtrol Partnership in Louisville, Kentucky, as a site for an emission test program because there is no data currently available to quantify cadmium emissions accurately from belt dryers, rotary calciners or vacuum pan dryers used in pigment manufacturing plants. The test program was designed to develop controlled emission factors for cadmium sulfide pigment industries.

TRC Environmental Consultants, Inc. was retained by the EPA Emission Measurement Branch (EMB) to perform emission measurements at the Harshaw/Filtrol Partnership in Louisville, Kentucky. Testing was performed on the red and yellow belt dryer venturi scrubber stacks (SD-301 & 201) and the rotary calciners/vacuum pan dryer spray tower stack (S-103). 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 Harshaw/Filtrol 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 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 that will allow the data to be evaluated to develop a NESHAP. One test method was selected by EPA to measure emissions from cadmium pigment manufacturers. This method is briefly described in the following subsection and is described in detail in Section 5.

#### 1.3.1 EPA Method 5 Configuration Modified for Cadmium

Cadmium sampling was performed by using a modification of the standard EPA Method 5 train.<sup>1</sup> This modification consisted of placing flexible Teflon tubing between the filter and the impingers and the use of 5% Nitric acid

solution in the first two impingers, teflon tubing was used due to the particularly difficult sampling locations.

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 Harshaw/Filtrol Partnership in Louisville, Kentucky during the week of July 14, 1986. Tests were performed at the red and yellow belt dryer venturi scrubber stacks and the rotary calciners/vacuum pan dryer spray tower stack.

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

##### 1.4.1 Spray Tower and Venturi

###### Preliminary Measurements

Preliminary testing was performed on July 14, 1986 to determine volumetric flowrate and stack gas moisture content. An integrated gas sample was also taken to determine concentrations of CO<sub>2</sub>, O<sub>2</sub>, and CO, which were found to be ambient. Stack diameter and the sampling port configuration were confirmed at this time.

###### Method 5 Modified For Cadmium

Six Method 5 tests modified for cadmium were performed, two tests on the yellow belt dryer venturi scrubber (SD-201), one test on the red belt dryer venturi scrubber stack (SD-301) and three tests on the rotary calciner/vacuum

<sup>1</sup> Code of Federal Regulations 40, Part 60 Appendix A, July, 1981.

pan dryer spray tower (S-103). Testing at these locations was performed on July 15, 16 and 17, 1986.

#### Method 9 - Visible Emissions

Visible emissions from the spray tower stack and the venturi scrubber stacks were observed concurrently with each Method 5 modified for cadmium 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 and visible emission results collected at spray tower 103 and a complete breakdown and discussion of parameters. Cadmium, and visible emission results collected at venturi scrubber 201 and 301 are presented in Section 2.2. Section 2.3 presents results of the trace metals.

### 2.1 Spray Tower

A total of three tests were conducted at spray tower 103 using a Method 5 sampling train modified for cadmium. All tests were acceptable with a leak rate of <0.02cfm and isokinetics of  $100 \pm 10\%$ .

#### 2.1.1 Cadmium Emissions

Tables 2-1a (English Units) and 2-1b (Metric Units) present a summary of measured cadmium emissions from spray tower 103.

The average total cadmium emission rate was  $4.04 \times 10^{-3}$  lb/hr ( $5.09 \times 10^{-4}$  g/sec) and ranged from  $2.75 \times 10^{-3}$  lb/hr ( $3.46 \times 10^{-4}$  g/sec) to  $4.97 \times 10^{-3}$  lb/hr ( $6.26 \times 10^{-4}$  g/sec). The average total cadmium concentration was  $1.98 \times 10^{-4}$  gr/DSCF and ranged from  $1.07 \times 10^{-4}$  gr/DSCF to  $2.44 \times 10^{-4}$  gr/DSCF. All significant cadmium concentrations and emission rates were collected in the front half of the sampling train.

#### 2.1.2 Visible Emissions

A summary of visible emission observations from the spray tower is presented in Table 2-2. Average opacities are presented for 6 minute time periods during each two hour test. The average opacity was 1% for tests 1 and

TABLE 2-1a

(ENGLISH UNITS)

SUMMARY OF CONTROLLED CADMIUM EMISSIONS  
 SPRAY TOWER 103  
 HARSHAW/FILTROL PARTNERSHIP  
 JULY 1986

TEST NO	1	2	3	AVG
DATE	7/15/86	7/16/86	7/17/86	--
TIME	1056-1500	0832-1245	0846-1251	--
<u>Sample Conditions</u>				
Volume (DSCF) <sup>1</sup>	107.65	118.29	150.66	--
Front Half Catch (µg)	1680	1860	1030	--
Back Half Catch (µg)	13.6	7.2	10.2	--
Total Catch (µg)	1694	1867	1040	--
Isokinesis (%)	101.7	96.2	97.0	--
<u>Stack Conditions</u>				
Flowrate (DSCFM)	2120	2380	3010	2500
Temperature (°F)	101	99	103	101
Moisture (%)	11.4	3.2	5.5	6.7
Oxygen (%)	20.9	20.9	20.9	20.9
Opacity (%)	1	1	0	1
<u>Process Conditions</u>				
Production Rate (ton/hr)	0.15	0.15	0.15	0.15
<u>Cadmium Emissions</u>				
<u>Front Half</u>				
Concentration (gr/DSCF)	$2.41 \times 10^{-4}$	$2.43 \times 10^{-4}$	$1.06 \times 10^{-4}$	$1.97 \times 10^{-4}$
Mass Emission Rate (lb/hr)	$4.38 \times 10^{-3}$	$4.95 \times 10^{-3}$	$2.72 \times 10^{-3}$	$4.02 \times 10^{-3}$
(lb/ton)	$2.92 \times 10^{-2}$	$3.30 \times 10^{-2}$	$1.81 \times 10^{-2}$	$2.68 \times 10^{-2}$
<u>Back Half</u>				
Concentration (gr/DSCF)	$1.95 \times 10^{-6}$	$9.39 \times 10^{-7}$	$1.05 \times 10^{-6}$	$1.31 \times 10^{-6}$
Mass Emission Rate (lb/hr)	$3.54 \times 10^{-5}$	$1.92 \times 10^{-5}$	$2.70 \times 10^{-5}$	$2.72 \times 10^{-5}$
(lb/ton)	$2.36 \times 10^{-4}$	$1.28 \times 10^{-4}$	$1.80 \times 10^{-4}$	$1.81 \times 10^{-2}$
<u>Total</u>				
Concentration (gr/DSCF)	$2.43 \times 10^{-4}$	$2.44 \times 10^{-4}$	$1.07 \times 10^{-4}$	$1.98 \times 10^{-4}$
Mass Emission Rate (lb/hr)	$4.41 \times 10^{-3}$	$4.97 \times 10^{-3}$	$2.75 \times 10^{-3}$	$4.04 \times 10^{-3}$
(lb/ton)	$2.94 \times 10^{-2}$	$3.31 \times 10^{-2}$	$1.83 \times 10^{-2}$	$2.69 \times 10^{-2}$

<sup>1</sup> Standard Conditions: 29.92 in. Hg @ 68°F

TABLE 2-1b

(METRIC UNITS)

SUMMARY OF CONTROLLED CADMIUM EMISSIONS  
 SPRAY TOWER 103  
 HARSHAW/FILTROL PARTNERSHIP  
 JULY 1986

TEST NO	1	2	3	AVG
DATE	7/15/86	7/16/86	7/17/86	--
TIME	1056-1500	0832-1245	0846-1251	--
<u>Sample Conditions</u>				
Volume (NM <sup>3</sup> ) <sup>1</sup>	3.05	3.35	4.27	--
Front Half Catch (µg)	1680	1860	1030	--
Back Half Catch (µg)	13.6	7.2	10.2	--
Total Catch (µg)	1694	1867	1040	--
Isokinesis (%)	101.7	96.2	97.0	--
<u>Stack Conditions</u>				
Flowrate (NM <sup>3</sup> /MIN)	60.04	67.40	85.24	70.89
Temperature (°C)	38	37	39	38
Moisture (%)	11.4	3.2	5.5	6.7
Oxygen (%)	20.9	20.9	20.9	20.9
Opacity (%)	1	1	0	1
<u>Process Conditions</u>				
Production Rate (ton/hr)	0.15	0.15	0.15	0.15
<u>Cadmium Emissions</u>				
<u>Front Half</u>				
Concentration (Mg/NM <sup>3</sup> )	551	555	241	449
Mass Emission Rate (g/sec)	5.51x10 <sup>-4</sup>	6.24x10 <sup>-4</sup>	3.43x10 <sup>-4</sup>	5.06x10 <sup>-4</sup>
(g/ton)	13.22	14.98	8.23	12.14
<u>Back Half</u>				
Concentration (Mg/NM <sup>3</sup> )	4.46	2.15	2.39	3.00
Mass Emission Rate (g/sec)	4.46x10 <sup>-6</sup>	2.41x10 <sup>-6</sup>	3.39x10 <sup>-6</sup>	3.42x10 <sup>-6</sup>
(g/ton)	0.107	0.058	0.081	0.082
<u>Total</u>				
Concentration (Mg/NM <sup>3</sup> )	555	557	244	452
Mass Emission Rate (g/sec)	5.55x10 <sup>-4</sup>	6.26x10 <sup>-4</sup>	3.46x10 <sup>-4</sup>	5.09x10 <sup>-4</sup>
(g/ton)	13.32	15.02	8.30	12.21

<sup>1</sup> Standard Conditions: 760 mm in. Hg @ 20°C

TABLE 2-2

SUMMARY OF VISIBLE EMISSIONS  
 SPRAY TOWER 103  
 HARSHAW/FILTROL PARTNERSHIP  
 JULY 1986

TEST NO 1 7/15/86 1040-1413		TEST NO 2 7/16/86 0824-1135		TEST NO 3 7/17/86 0745-1114	
SIX MINUTE TIME PERIOD	AVERAGE OPACITY (%)	SIX MINUTE TIME PERIOD	AVERAGE OPACITY (%)	SIX MINUTE TIME PERIOD	AVERAGE OPACITY
1040-1046	1	0824-0830	1	0745-0751	0
1046-1052	2	0830-0836	1	0751-0757	0
1052-1058	3	0836-0842	0	0757-0803	0
1058-1104	2	0842-0848	0	0803-0809	0
1104-1110	3	0848-0854	0	0809-0815	0
1110-1116	2	0854-0900	0	0815-0821	0
1116-1122	1	0900-0906	0	0821-0827	1
1122-1128	0	0906-0912	0	0827-0833	0
1128-1134	0	0912-0918	1	0833-0839	0
1134-1140	0	0918-0924	1	0839-0845	0
STOP TEST		STOP TEST		STOP TEST	
1313-1319	0	1035-1041	1	1014-1020	0
1319-1325	0	1041-1047	0	1020-1026	0
1325-1331	0	1047-1053	2	1026-1032	0
1331-1337	0	1053-1059	0	1032-1038	0
1337-1343	0	1059-1105	0	1038-1044	0
1343-1349	1	1105-1111	1	1044-1050	0
1349-1355	0	1111-1117	0	1050-1056	0
1355-1401	0	1117-1123	1	1056-1102	0
1401-1407	0	1123-1129	1	1102-1108	0
1407-1413	<u>1</u>	1129-1135	0	1180-1114	0
AVG	<u>1</u>	AVG	<u>1</u>	AVG	<u>0</u>

2 and 0% for test 3. These 6-minute average opacities are presented graphically in Figures 2-1 through 2-3.

### 2.1.3 Stack Conditions

An average volumetric flowrate of 2500 DSCFM was measured exiting the spray tower stack at 101°F, 6.7% moisture and 20.9% oxygen. For the first test, the volumetric flowrate was 2120 DSCFM at 101°F, 11.4 % moisture and 20.9% oxygen. This volumetric flowrate was 30% lower than test 3. This may be accounted for by the high moisture seen in the first test. Test 3 had a volumetric flowrate of 3010 DSCFM at 103°F, 5.5% moisture and 20.9% oxygen.

## 2.2 Venturi Scrubber

Two tests were performed at venturi scrubber stack 201 and one test on venturi scrubber 301 using a Method 5 sampling train modified for cadmium. All tests were acceptable with a leak rate of <0.02cfm and 100± 10 percent isokinetics.

### 2.2.1 Cadmium Emissions

Tables 2-3a (English Units) and 2-3b (Metric Units) present a summary of measured cadmium emissions from the venturi scrubber. The average total cadmium emission rate was  $4.85 \times 10^{-3}$  lb/hr ( $6.10 \times 10^{-4}$  g/sec) and ranged from  $4.45 \times 10^{-3}$  lb/hr ( $5.61 \times 10^{-4}$  g/sec) to  $5.16 \times 10^{-3}$  lb/hr ( $6.50 \times 10^{-4}$  g/sec).

The average total cadmium concentration was  $5.17 \times 10^{-4}$  gr/DSCF and ranged from  $4.49 \times 10^{-4}$  gr/DSCF to  $6.18 \times 10^{-4}$  gr/DSCF. All significant cadmium concentrations and emission rates were collected in the front half of the sampling train.



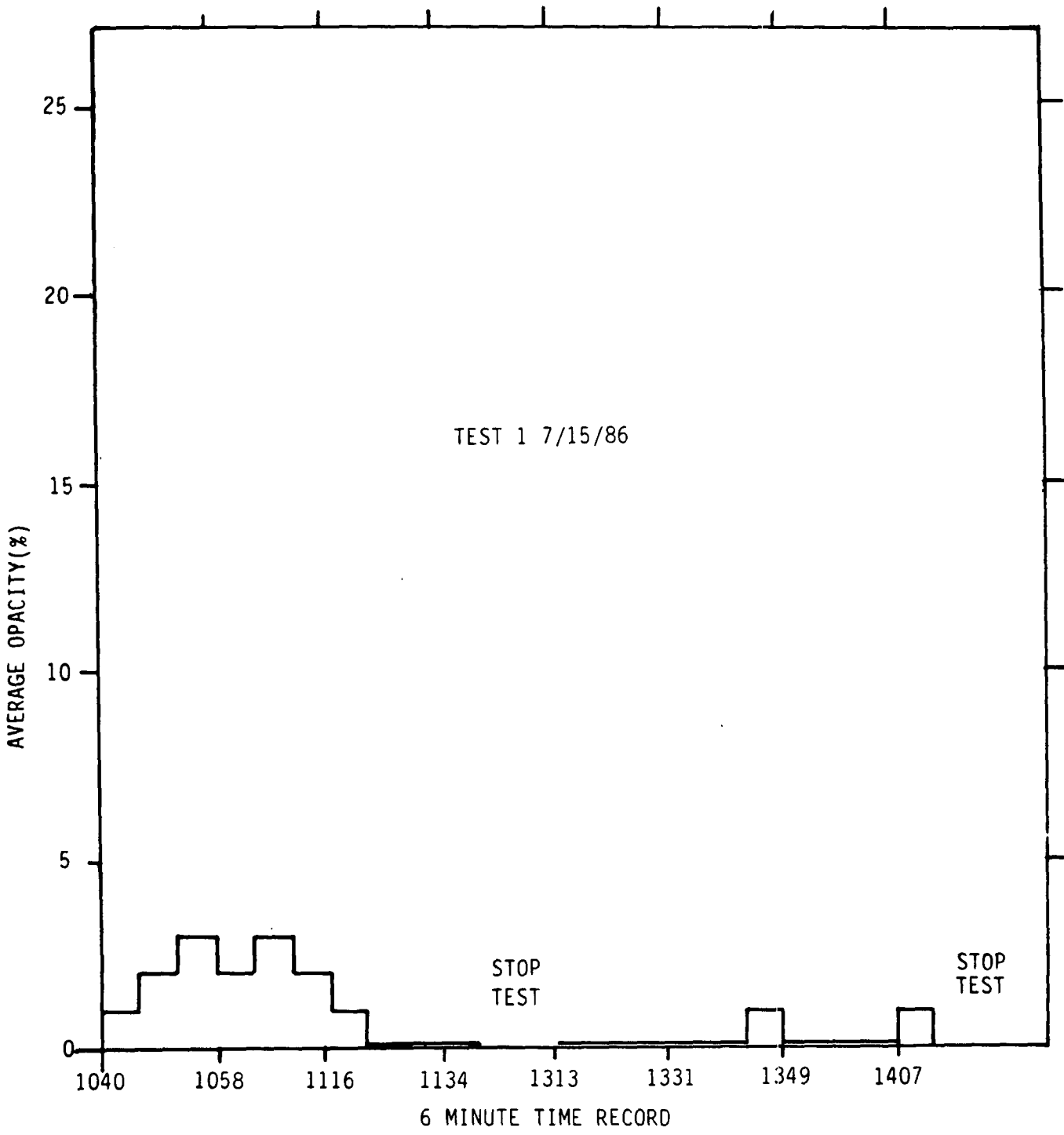


Figure 2-1. Summary of Visible Emissions  
 Spray Tower 103  
 Harshaw/Filtrol Partnership

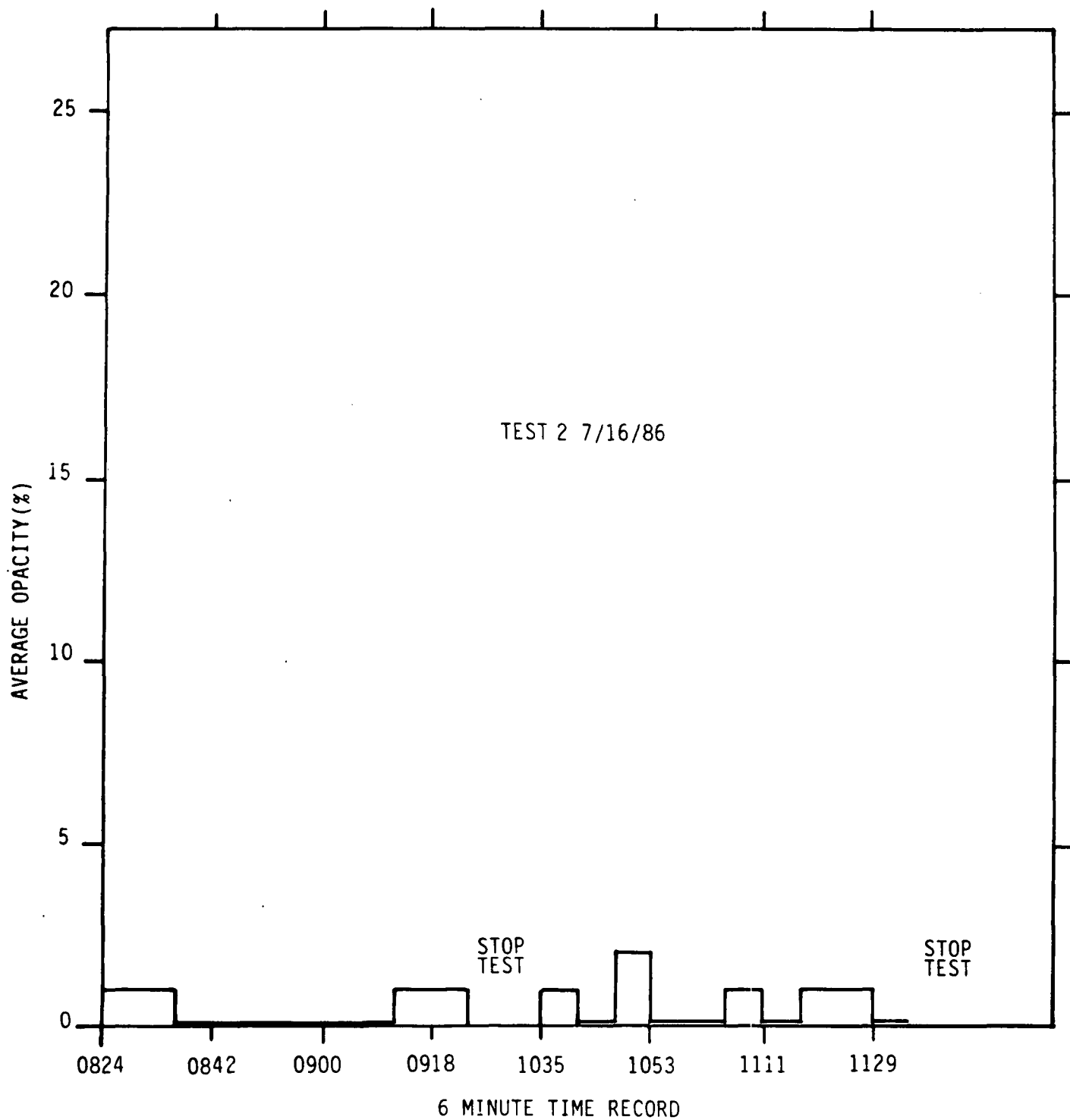


Figure 2-2. Summary of Visible Emissions  
 Spray Tower 103  
 Harshaw/Filtrol Partnership

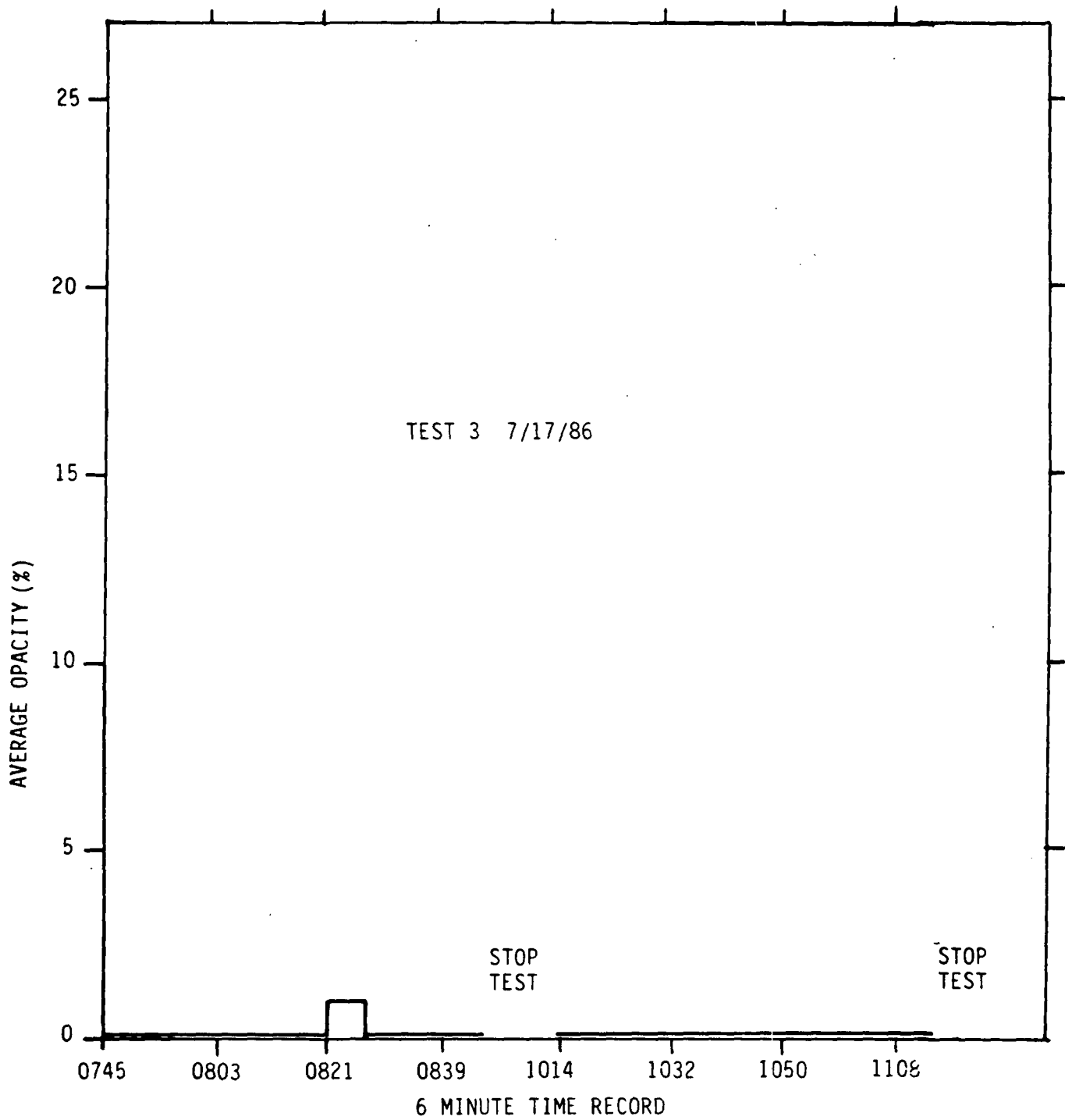


Figure 2-3. Summary of Visible Emissions  
 Spray Tower 103  
 Harshaw/Filtrol Partnership

TABLE 2-3a

(ENGLISH UNITS)

SUMMARY OF CONTROLLED CADMIUM EMISSIONS  
 VENTURI SCRUBBER 201  
 HARSHAW/FILTROL PARTNERSHIP

JULY 1986

TEST NO	1	2	3 <sup>1</sup>	AVG
DATE	7/15/86	7/16/86	7/17/86	--
TIME	1058-1515	0820-1227	0754-1200	--
<u>Sample Conditions</u>				
Volume (DSCF) <sup>2</sup>	96.05	85.72	120.03	--
Front Half Catch (µg)	3010	3420	3460	--
Back Half Catch (µg)	11.0	11.8	33.5	--
Total Catch (µg)	3021	3432	3494	--
Isokinesis (%)	97.0	99.8	96.8	--
<u>Stack Conditions</u>				
Flowrate (DSCFM)	1070	930	1340	1110
Temperature (°F)	130	138	140	136
Moisture (%)	6.6	9.4	7.3	7.8
Oxygen (%)	20.9	20.9	20.9	20.9
Opacity (%)	0	0	4	1
<u>Process Conditions</u>				
Production Rate (ton/hr)	0.063	0.063	0.063	0.063
<u>Cadmium Emissions</u>				
<u>Front Half</u>				
Concentration (gr/DSCF)	4.84x10 <sup>-4</sup>	6.16x10 <sup>-4</sup>	4.45x10 <sup>-4</sup>	5.15x10 <sup>-4</sup>
Mass Emission Rate (lb/hr)	4.44x10 <sup>-3</sup>	4.91x10 <sup>-3</sup>	5.11x10 <sup>-3</sup>	4.82x10 <sup>-3</sup>
(lb/ton)	7.05x10 <sup>-2</sup>	7.79x10 <sup>-2</sup>	8.11x10 <sup>-2</sup>	7.65x10 <sup>-2</sup>
<u>Back Half</u>				
Concentration (gr/DSCF)	1.77x10 <sup>-6</sup>	2.13x10 <sup>-6</sup>	4.31x10 <sup>-6</sup>	2.74x10 <sup>-6</sup>
Mass Emission Rate (lb/hr)	1.62x10 <sup>-5</sup>	1.69x10 <sup>-5</sup>	4.95x10 <sup>-5</sup>	2.75x10 <sup>-5</sup>
(lb/ton)	2.57x10 <sup>-4</sup>	2.68x10 <sup>-4</sup>	7.86x10 <sup>-4</sup>	4.37x10 <sup>-4</sup>
<u>Total</u>				
Concentration (gr/DSCF)	4.85x10 <sup>-4</sup>	6.18x10 <sup>-4</sup>	4.49x10 <sup>-4</sup>	5.17x10 <sup>-4</sup>
Mass Emission Rate (lb/hr)	4.45x10 <sup>-3</sup>	4.93x10 <sup>-3</sup>	5.16x10 <sup>-3</sup>	4.85x10 <sup>-3</sup>
(lb/ton)	7.06x10 <sup>-2</sup>	7.83x10 <sup>-2</sup>	8.19x10 <sup>-2</sup>	7.69x10 <sup>-2</sup>

<sup>1</sup> Test No. 3 on Venturi Scrubber 301<sup>2</sup> Standard Conditions: 29.92 in Hg @ 68°F

TABLE 2-3b

(METRIC UNITS)

SUMMARY OF CONTROLLED CADMIUM EMISSIONS  
VENTURI SCRUBBER 201  
HARSHAW/FILTROL PARTNERSHIP

JULY 1986

TEST NO	1	2	3 <sup>1</sup>	AVG
DATE	7/15/86	7/16/86	7/17/86	--
TIME	1058-1515	0820-1227	0754-1200	--
<u>Sample Conditions</u>				
Volume (NM <sup>3</sup> ) <sup>2</sup>	2.72	2.43	3.40	--
Front Half Catch (µg)	3010	3420	3460	--
Back Half Catch (µg)	11.0	11.8	33.5	--
Total Catch (µg)	3021	3432	3494	--
Isokinesis (%)	97.0	99.8	96.8	--
<u>Stack Conditions</u>				
Flowrate (NM <sup>3</sup> /MIN)	30.30	26.34	37.95	31.53
Temperature (°C)	54	59	60	58
Moisture (%)	6.6	9.4	7.3	7.8
Oxygen (%)	20.9	20.9	20.9	20.9
Opacity (%)	0	0	4	1
<u>Process Conditions</u>				
Production Rate (ton/hr)	0.063	0.063	0.063	0.063
<u>Cadmium Emissions</u>				
<u>Front Half</u>				
Concentration (µg/NM <sup>3</sup> )	1107	1407	1018	1177
Mass Emission Rate (g/sec)	5.59x10 <sup>-4</sup>	6.18x10 <sup>-4</sup>	6.44x10 <sup>-4</sup>	6.07x10 <sup>-4</sup>
(g/ton)	31.94	35.31	36.80	34.68
<u>Back Half</u>				
Concentration (µg/NM <sup>3</sup> )	4.04	4.86	9.85	6.25
Mass Emission Rate (g/sec)	2.04x10 <sup>-6</sup>	2.13x10 <sup>-6</sup>	6.23x10 <sup>-6</sup>	3.47x10 <sup>-6</sup>
(g/ton)	0.117	0.122	0.356	0.198
<u>Total</u>				
Concentration (µg/NM <sup>3</sup> )	1111	1412	1028	1184
Mass Emission Rate (g/sec)	5.61x10 <sup>-4</sup>	6.20x10 <sup>-4</sup>	6.50x10 <sup>-4</sup>	6.10x10 <sup>-4</sup>
(g/ton)	32.06	35.43	37.14	34.88

<sup>1</sup> Test No. 3 on Venturi Scrubber 301<sup>2</sup> Standard Conditions: 760 mm Hg @ 20°C

### 2.2.2 Visible Emissions

A summary of visible emission observations from the venturi scrubber stacks is presented in Table 2-4. Average opacities are presented for 6-minute time periods during each two hour test. The average opacity was 0% for tests 1 and 2 and 4% for test 3. Opacity observations were conducted on venturi scrubber 301 for test 3. The opacity was high (12% to 0%) for the first hour of observation. During the second hour of observation, the average opacity was 0%. These 6-minute average opacities are presented graphically in Figures 2-4 through 2-6.

### 2.2.3 Stack Conditions

The volumetric flowrate exiting venturi scrubber stack 201 was 10700 DSCFM at 130°F, 6.6% moisture and 20.9% oxygen for Test 1 and 930 DSCFM at 138°F, 9.4% moisture and 20.9% oxygen for Test 2. Test 3 was conducted at venturi scrubber stack 301 and the volumetric flowrate was 1340 DSCFM at 140°F, 7.3% moisture and 20.9% oxygen.

## 2.3 Trace Metals

One sample and blank per set of tests were analyzed for trace metals. The trace metals analyzed for were barium, chromium, copper, iron, lead, magnesium, manganese, nickel and zinc. Mercury was not analyzed due to the different digestion procedure. Table 2-5 presents a summary of trace metals. Sample train results were corrected for field and/or laboratory blanks. Trace metals in the field blanks were high. This may be caused from the nitric acid rinse of the nozzles. The sample train results are representative due to the significant difference between blanks and runs.

TABLE 2-4

SUMMARY OF VISIBLE EMISSIONS  
 VENTURI SCRUBBER 201  
 HARSHAW/FILTROL PARTNERSHIP  
 JULY 1986

TEST NO 1 7/15/86 1157-1513		TEST NO 2 7/16/86 0930-1244		TEST NO 3* 7/17/86 0851-1217	
SIX MINUTE TIME PERIOD	AVERAGE OPACITY (%)	SIX MINUTE TIME PERIOD	AVERAGE OPACITY (%)	SIX MINUTE TIME PERIOD	AVERAGE OPACITY
1157-1203	0	0930-0936	0	0851-0857	11
1203-1209	1	0936-0942	0	0857-0903	12
1209-1215	0	0942-0948	0	0903-0909	7
1215-1221	0	0948-0954	0	0909-0915	11
1221-1227	0	0954-1000	0	0915-0921	10
1227-1233	1	1000-1006	1	0921-0927	9
1233-1239	1	1006-1012	1	0927-0933	9
1239-1245	0	1012-1018	0	0933-0939	7
1245-1251	0	1018-1024	0	0939-0945	6
1251-1257	0	1024-1030	0	0945-0951	3
STOP TEST		STOP TEST		STOP TEST	
1413-1419	0	1144-1150	0	1117-1123	0
1419-1425	0	1150-1156	0	1123-1129	0
1425-1431	0	1156-1202	0	1129-1135	0
1431-1437	0	1202-1208	0	1135-1141	0
1437-1443	0	1208-1214	0	1141-1147	0
1443-1449	0	1214-1220	0	1147-1153	0
1449-1455	0	1220-1226	0	1153-1159	0
1455-1501	0	1226-1232	0	1159-1205	0
1501-1507	1	1232-1238	0	1205-1211	0
1507-1513	0	1238-1244	0	1211-1217	0
AVG	0	AVG	0	AVG	4

\* Opacity Observations at scrubber 301

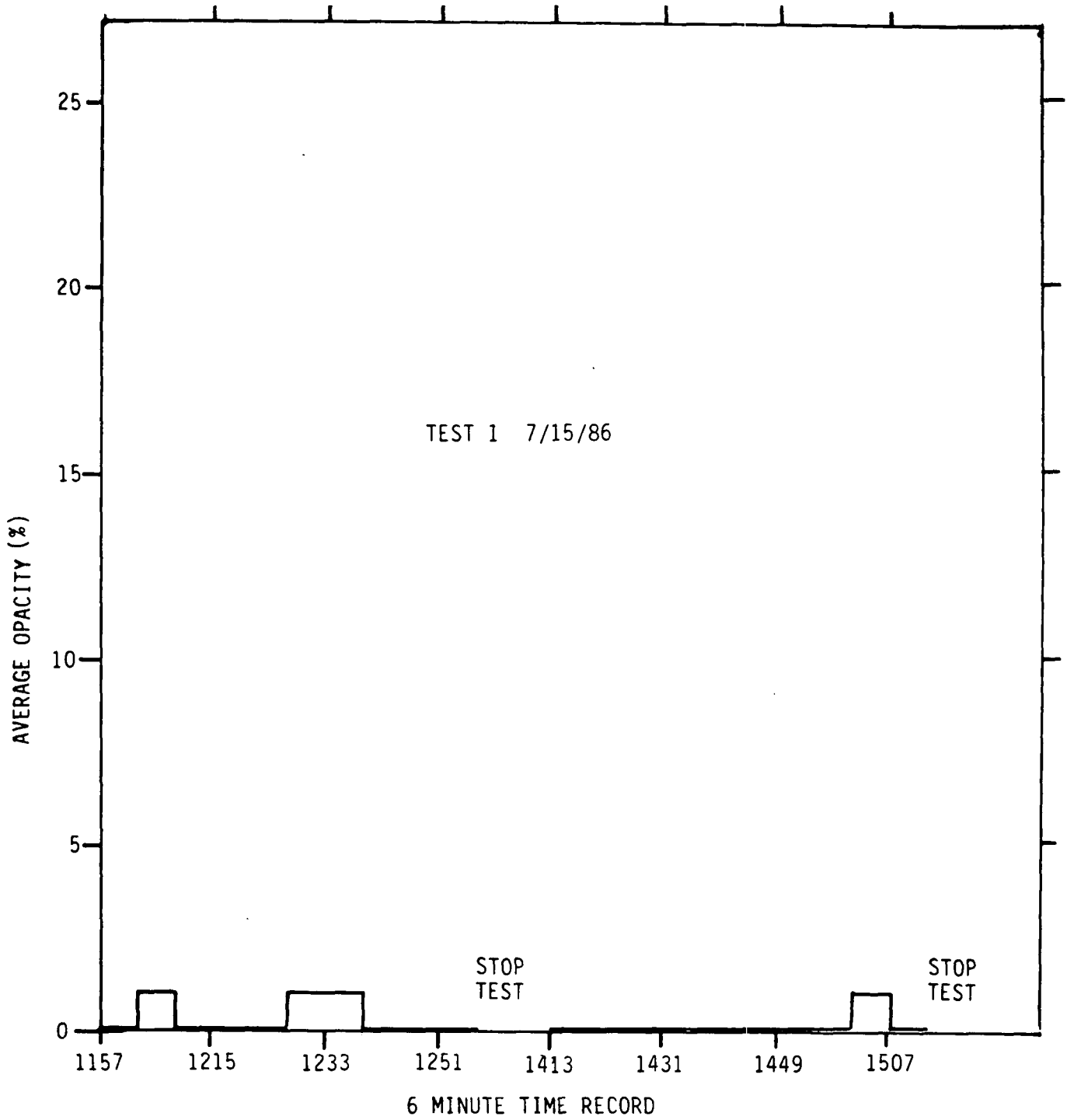


Figure 2-4. Summary of Visible Emissions  
 Venturi Scrubber 201  
 Harshaw/Filtrol Partnership



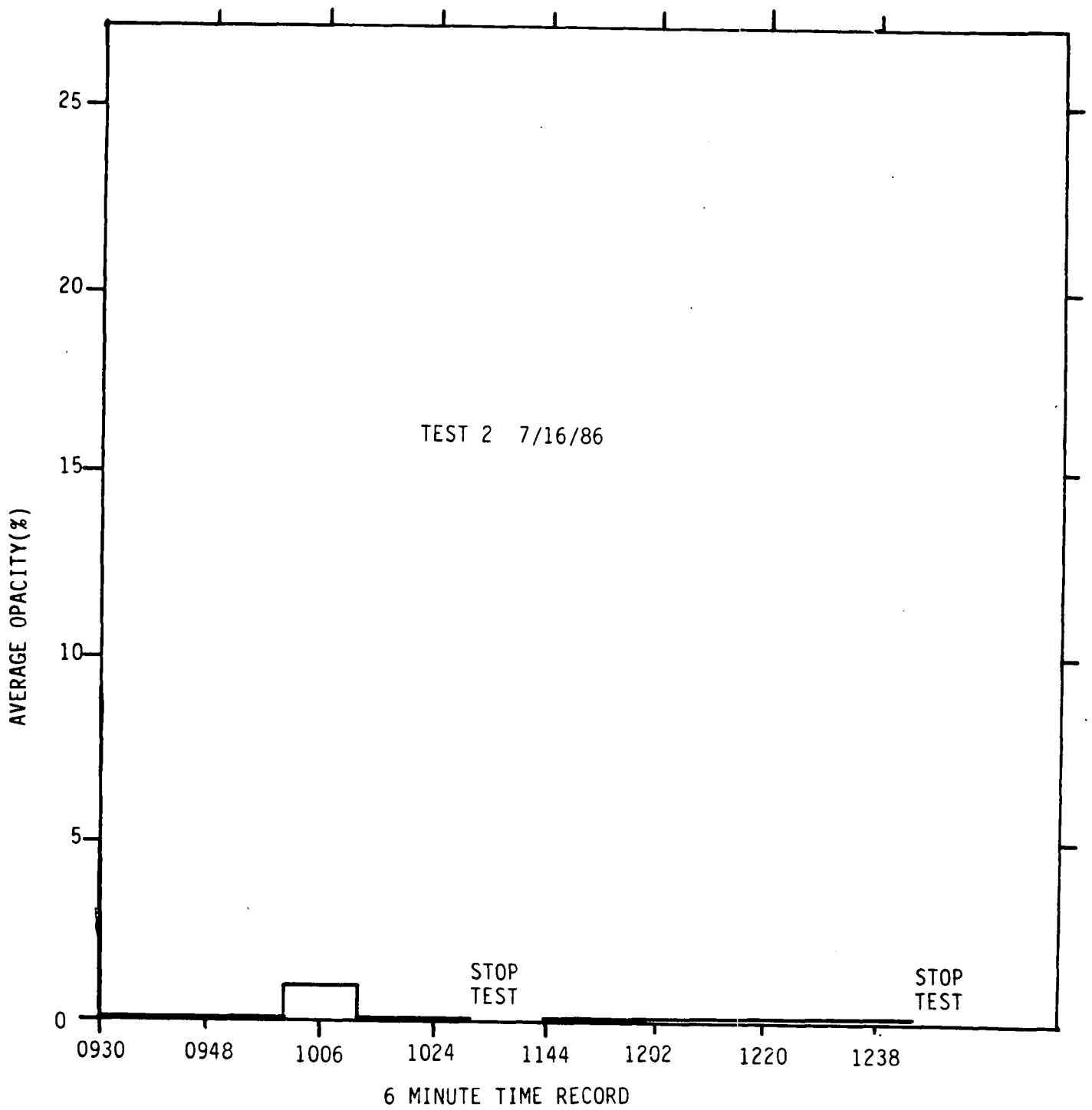


Figure 2-5. Summary of Visible Emissions  
 Venturi Scrubber 201  
 Harshaw/Filtrol Partnership

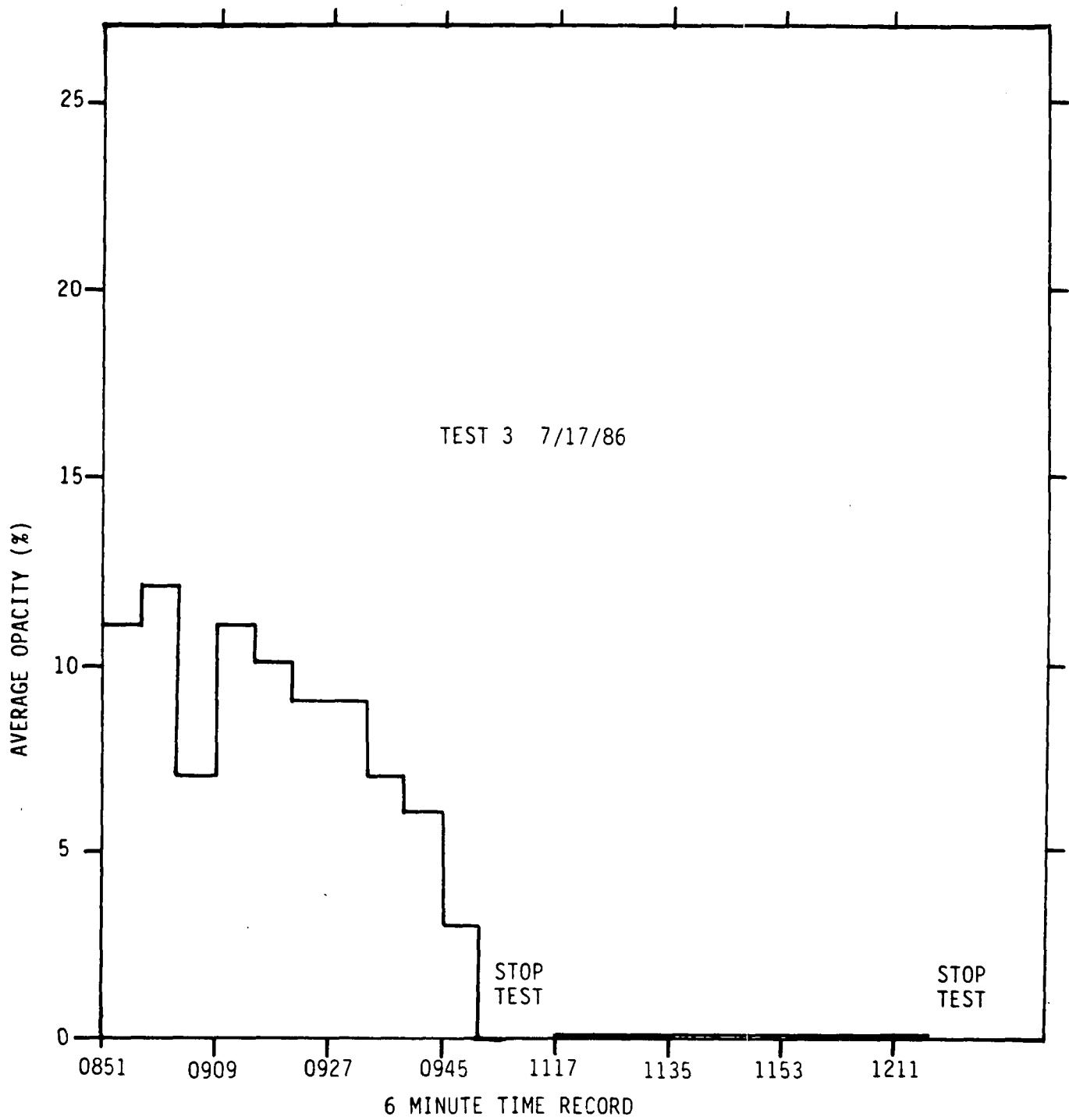


Figure 2-6. Summary of Visible Emissions  
 Venturi Scrubber 301  
 Härshaw/Filtrol Partnership

TABLE 2-5  
SUMMARY OF TRACE METALS  
HARSHAW/FILTROL PARTNERSHIP  
JULY 1986

Sample <sup>1</sup> Location	Blank 1 Venturi 201	Run 1 Venturi 201	Blank 1 Spray Tower 103	Run 1 Spray Tower 103	HNO <sub>3</sub> Blank	Lab Blank
Barium (µg)	nd <sup>2</sup> <50	nd<50	nd<50	nd<50	nd<25	nd<25
Chromium (µg)	253	1280	53	100	nd<5	nd<5
Copper (µg)	9	39	15	37	nd<3	nd<4
Iron (µg)	1010	6800	282	892	8	nd<7
Lead (µg)	nd<46	nd<46	nd<46	nd<46	nd<23	nd<23
Magnesium (µg)	115	153	104	174	1.4	2.7
Manganese (µg)	22	183	7	13	nd<4	nd<4
Nickel (µg)	94	676	44	126	nd<7	nd<7
Zinc (µg)	90	6320	110	4050	nd<1	7

<sup>1</sup> Sample train results corrected for field and/or lab blank

<sup>2</sup> Non Detected

3.0 PROCESS AND OPERATIONS (Provided by MRI)

**CONFIDENTIAL**

3.1 Process Equipment

**CONFIDENTIAL**

3.2 Control Equipment

**CONFIDENTIAL**

#### 4.0 SCOPE OF SAMPLING PROGRAM

Sampling train operators maintained a detailed log of their test times for each run. Data sheets present all necessary information in concise legible form.

Method 5 test data such as: percent isokinetics, moisture, flowrate (DSCFM) and stack temperatures was calculated daily. Results were recorded on a daily summary sheet.

#### 4.1 Belt Dryer

Prior to emission testing, the flowrate of the stack was measured in accordance with EPA Methods 1-2. Duct measurements were verified at this time. Stack gas moisture content was determined using wet bulb/dry bulb thermometers.

Cadmium samples were collected from a 10 inch circular duct. Two 3-inch sample ports were placed 90° apart in the duct. Figure 4-1 presents the sample port configuration. Sample point A indicates the Method 5 sampling location. According to EPA Method 1, 24 traverse points are required. Each traverse point was sampled for ten minutes for a total test time of 4 hours. An "EPA Method 1, Sampling and Velocity Traverse" sheet is presented in Figure 4-2.

#### 4.2 Rotary Calciner and Vacuum Pan Dryer

Prior to emission testing, routine preliminary measurements were conducted as described in Section 4.1.

Cadmium samples were collected in two ports positioned 90° apart and located 26 inches (2 diameters) downstream from the fan and 14 inches (1 diameter) upstream from the top of the stack. The location of the sampling ports are presented in Figure 4-3.

SAMPLE POINT B → ●

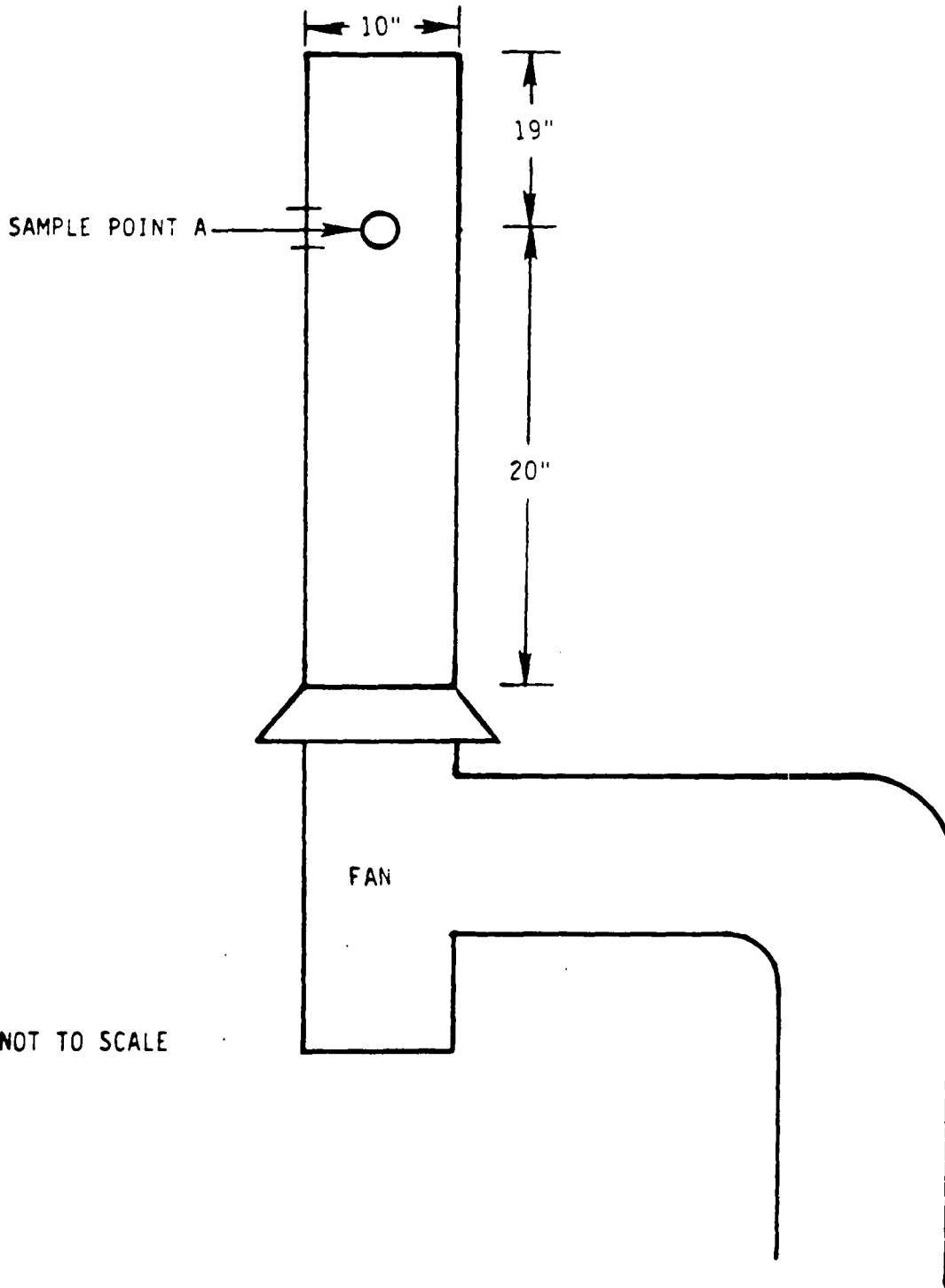


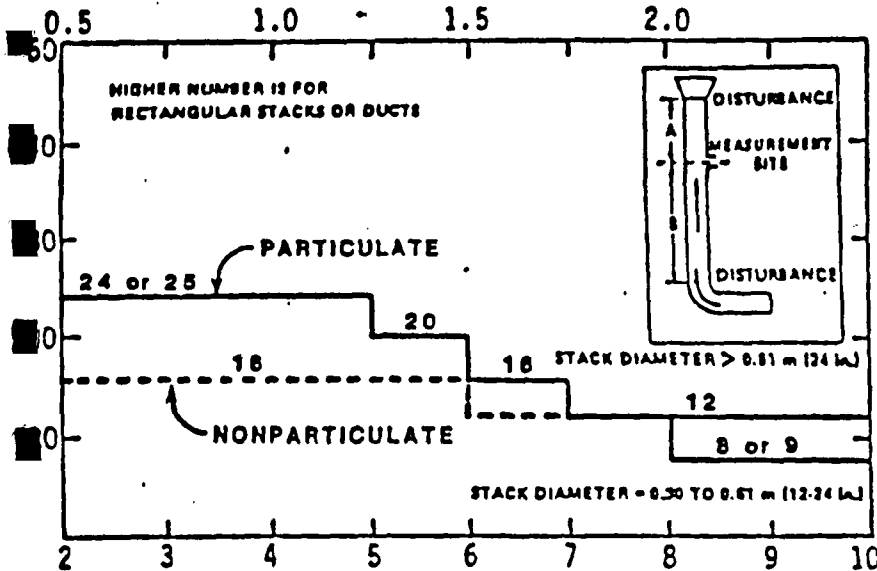
Figure 4-1. Belt Dryer Venturi Scrubber Outlet

Figure 4-2

Firm HARSHAW/FILTROL Total Traverse Points Required 24  
 Location SPRAY TOWER OUTLET Number of Ports 2  
 Diameters Upstream 2 Points Per Port 12  
 Diameters Downstream 1 Traverse (Horizontal or Vertical) Horizontal

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

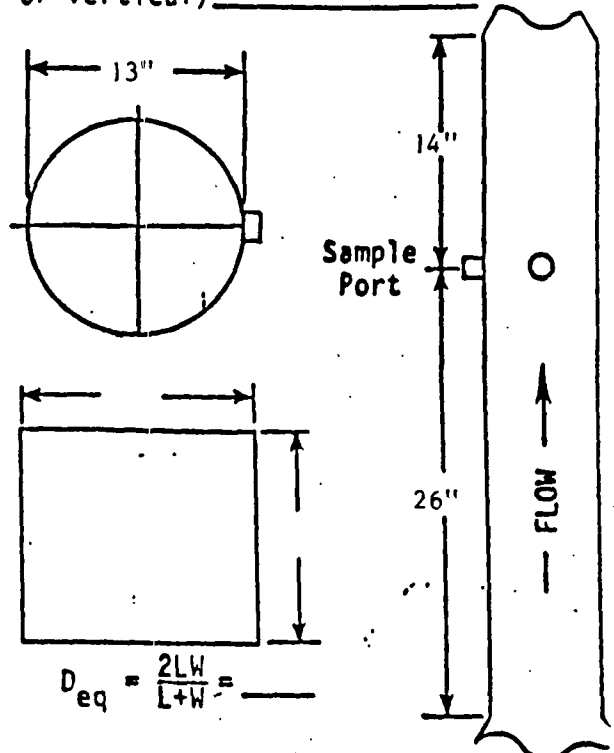
Duct Diameters Upstream from Flow Disturbance (Distance A)



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)				
	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 Traverse Points	Matrix
9	3x3
12	4x3
16	4x4
20	5x4
25	5x5

**TRAVERSE POINT LOCATIONS**

No.	Distance From Wall	Nipple Size	Total Distance
1	0.3"		
2	0.9"		
3	1.5"		
4	2.3"		
5	3.3"		
6	4.6"		
7	8.4"		
8	9.6"		
9	10.7"		
10	11.5"		
11	12.1"		
12	12.7"		

SAMPLE POINT B → ●

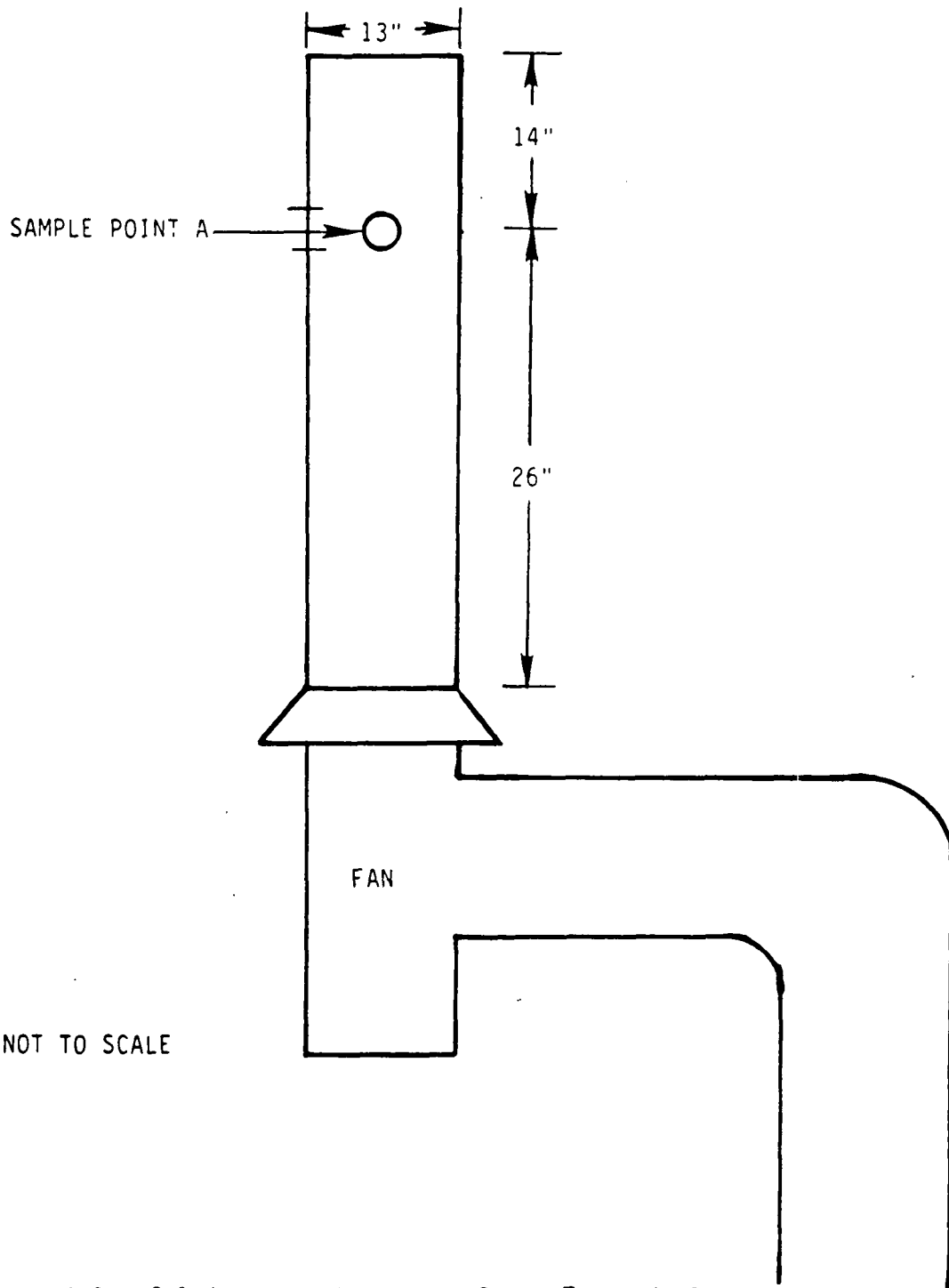


Figure 4-3. Calciner and Pan Dryer Spray Tower Outlet



According to EPA Method 1, 24 traverse points were sampled at this location for ten minutes per point for a total test time of 4 hours. Sample port and traverse point locations are presented in Figure 4-4.

#### 4.3 Visible Emission Observations (Method 9)

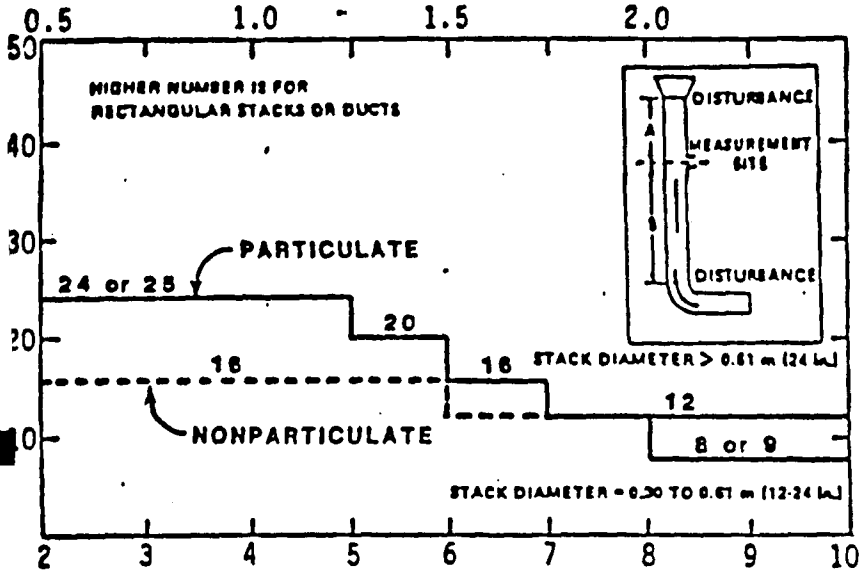
Visible emission observations were conducted in accordance with EPA Method 9. Observations were performed at the venturi scrubber stack and the spray tower stack separately. Each location was observed for two hours during each cadmium emission test. A diagram showing the observers location can be found in Figure 4-5.

Figure 4-4

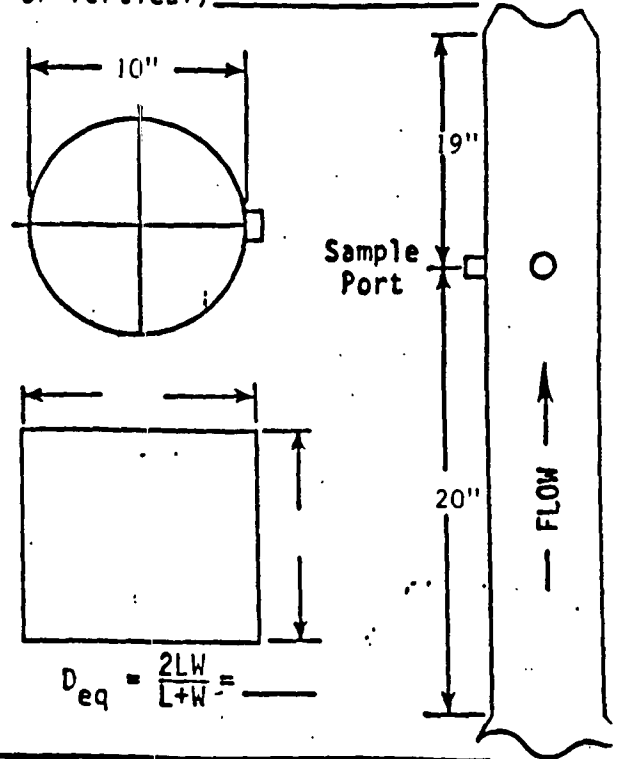
Firm HARSHAW/FILTROL Total Traverse Points Required 24  
 Location Ventruil Scrubber Outlet Number of Ports 12  
 Diameters Upstream 1 Points Per Port 2  
 Diameters Downstream 2 Traverse ( Horizontal or Vertical) \_\_\_\_\_

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)



$$D_{eq} = \frac{2LW}{L+W}$$

LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS

Point Number On A Diameter	(Percent of stack diameter from inside wall to traverse point)				
	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	0.2"		
2	0.7"		
3	1.2"		
4	1.8"		
5	2.5"		
6	3.6"		
7	6.4"		
8	7.5"		
9	8.2"		
10	8.8"		
11	9.3"		
12	9.8"		

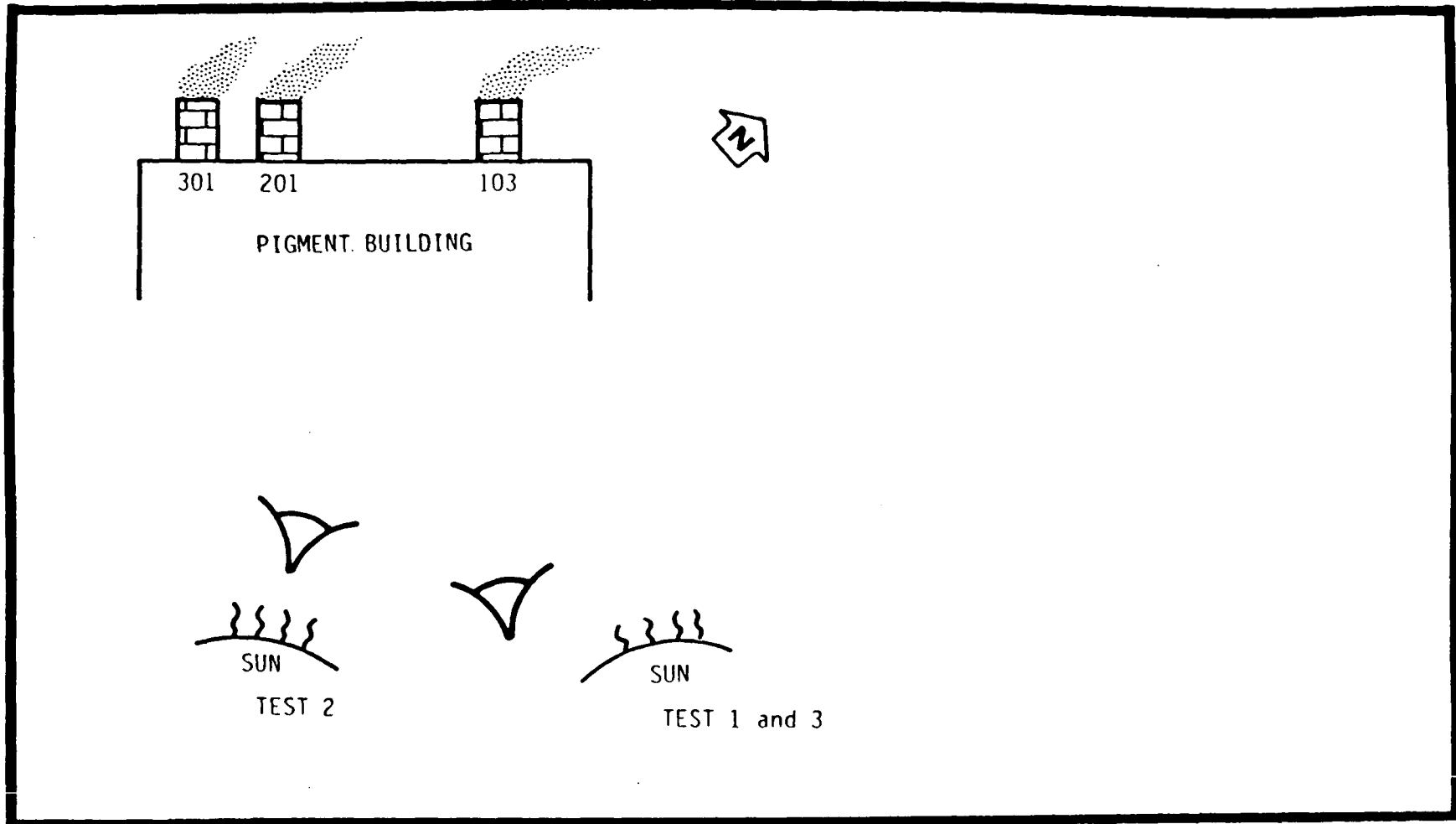


Figure 4-5. Observers Location  
Visible Emissions  
Spray Tower and Venturi Scrubber  
Harshaw/Filtrol Partnership  
July 1986

## 5.0 SAMPLING AND ANALYTICAL PROCEDURES

This section presents descriptions of sampling and analyses procedures which were employed during the emission testing conducted at Harshaw/Filtrol Partnership facility in Louisville, Kentucky.

A combination of EPA Methods 1, 2, 3, 4, 5 (modified for cadmium) and 9 were used to measure cadmium, visible and trace metals emissions from the venturi scrubber stack and the spray tower stack.

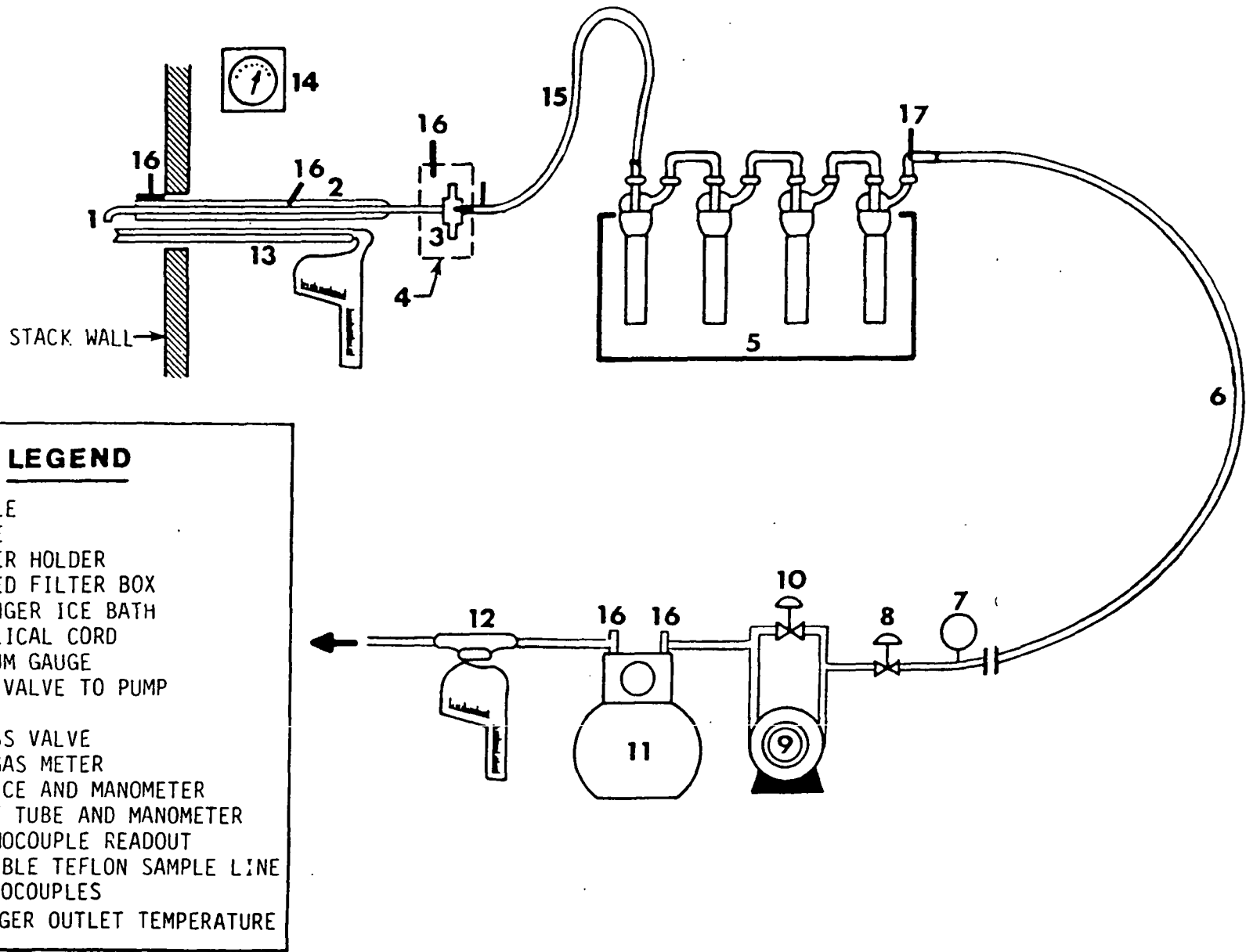
### 5.1 EPA Method 5 Configuration Modified for Cadmium

Sampling was performed using a modification to the standard EPA Method 5 sampling train<sup>1</sup>. It 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. The modification of the standard EPA Method 5 train consisted of placing flexible Teflon tubing between the filter and the impingers. This modification made the sampling equipment much easier to handle. A second modification was the use of 5% nitric acid in first two impingers. The sampling train was calibrated before and after this test program at TRC. This method is based on the proposed methodology presented in the Appendix.

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 visible 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). A thermocouple, located inside

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<sup>1</sup> Code of Federal Regulations 40, Part 60 Appendix A, July 81



**LEGEND**

- 1 - NOZZLE
- 2 - PROBE
- 3 - FILTER HOLDER
- 4 - HEATED FILTER BOX
- 5 - IMPINGER ICE BATH
- 6 - UMBILICAL CORD
- 7 - VACUUM GAUGE
- 8 - MAIN VALVE TO PUMP
- 9 - PUMP
- 10 - BYPASS VALVE
- 11 - DRY GAS METER
- 12 - ORIFICE AND MANOMETER
- 13 - PITOT TUBE AND MANOMETER
- 14 - THERMOCOUPLE READOUT
- 15 - FLEXIBLE TEFLON SAMPLE LINE
- 16 - THERMOCOUPLES
- 17 - IMPINGER OUTLET TEMPERATURE

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

the back half of the filter holder, monitored 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 and 80°F.

Flexible tubing, vacuum gauge, needle valve, leakless vacuum pump, bypass valve, dry gas meter, calibrated orifice and inclined manometer completed 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 as well as port changes, the sampling train was leak checked. Sample time was 4 hours 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.

## 5.2 Sample Recovery

Sample recovery was performed in a clean, wind-free area to avoid sample contamination. The TRC mobile environmental laboratory was adjacent to the test site and utilized for sample recovery. Working surfaces were covered with fresh Kimwipes prior to each sample recovery to mitigate contamination. Personnel wore disposable gloves when recovering samples. Brushes and tweezers were kept in plastic bags when not in use. Each sample probe had its

own brush to lessen cross contamination. All sampling equipment had no exposed openings before and after sampling. The following sample fractions were recovered:

<u>Container No. 1</u>	Glass fiber filter.
<u>Container No. 2</u>	Probe, nozzle, and front half of the filter housing HNO <sub>3</sub> rinses.
<u>Container No. 3</u>	Impinger solution, Teflon line, impingers and back-half of the filter housing HNO <sub>3</sub> rinses.
<u>Container No. 4</u>	Silica gel.

The probe, nozzle and front half of the filter were rinsed and brushed three times with 5% HNO<sub>3</sub>. A 200 ml flask with 28/12 socket joint was attached to the ball end of the probe. Approximately 50 ml of HNO<sub>3</sub> were rinsed through the probe and collected in the flask. A blunt end brush constructed of nylon bristles and a stainless steel shaft was then passed 3 times each with a fresh immersion of 5% HNO<sub>3</sub> solution held in the flask. The brush was then removed and rinsed into the sample jar. Recovery personnel sealed the opposite end of the probe with a gloved finger and inverted twice allowing the HNO<sub>3</sub> to pass along the inside of the probe. A final flush of fresh 5% HNO<sub>3</sub> through the probe was made into the sample container rotating the probe so that HNO<sub>3</sub> made contact with all parts of the glass liner. This sample was labeled and weighed for shipping.

The contents of the first three impingers were weighed, recorded, and deposited in Container No. 3. The back half of the filter housing, the flexible Teflon line, and the first three impingers and their connecting glassware were rinsed 3 times with 5% HNO<sub>3</sub> solution and combined with the impinger contents. A 200 ml flask with 28/12 socket joint was attached to one end of the flexible Teflon line. Approximately 50 ml of 5% HNO<sub>3</sub> was passed

through the line and collected three times with fresh 5% HNO<sub>3</sub>. The final volume was weighed and recorded. Silica gel moisture gain was weighed on a triple beam balance and recorded.

The glass fiber filter was removed, placed into a contamination free petri dish, sealed with parafilm and labeled.

The sample container was labeled with the following information:

1. Sample I.D. (determined by location, run number and content)
2. Project Name
3. Date of Sampling
4. Shipping weight
5. Comments

Sample recovery data sheets were used to record: volumes recovered, filter ID numbers, silica gel weights and visual descriptions of the samples. Completed forms can be found in the Appendix.

A sample log sheet was completed recording: date, run number, sample ID, sample description and remarks. Completed copies of this form is presented in the Appendix.

### 5.3 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 measurement. The filter and probe wash was digested, extracted and analyzed for cadmium by flame atomic absorption. One set of samples was analyzed for trace metals by flame atomic absorption.

Container No. 2 Nozzle and probe rinse were dried and weighed; then brought back to solution with HNO<sub>3</sub>. The solution was extracted and analyzed with Container No. 1.

Container No. 3 The solution was analyzed separately as Container No. 2.



A flow diagram of the sample analysis procedure is presented in Figure 5-2.

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

The trace metals to be analyzed for were:

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

#### 5.4 Visible Emissions Observation

A certified visible emissions observer read exhaust opacity at the outlets of the venturi scrubber and spray tower. Sample Point B in Figures 4-1 and 4-2 depicts the exhaust points.

The observer's location was determined on-site in accordance with EPA Method 9 and considering obstructions and traffic problems in the area.

The Method 9 data reduction was calculated by averaging 24 consecutive observations recorded at 15-second intervals to calculate an average for observation for a 6 minute time period.

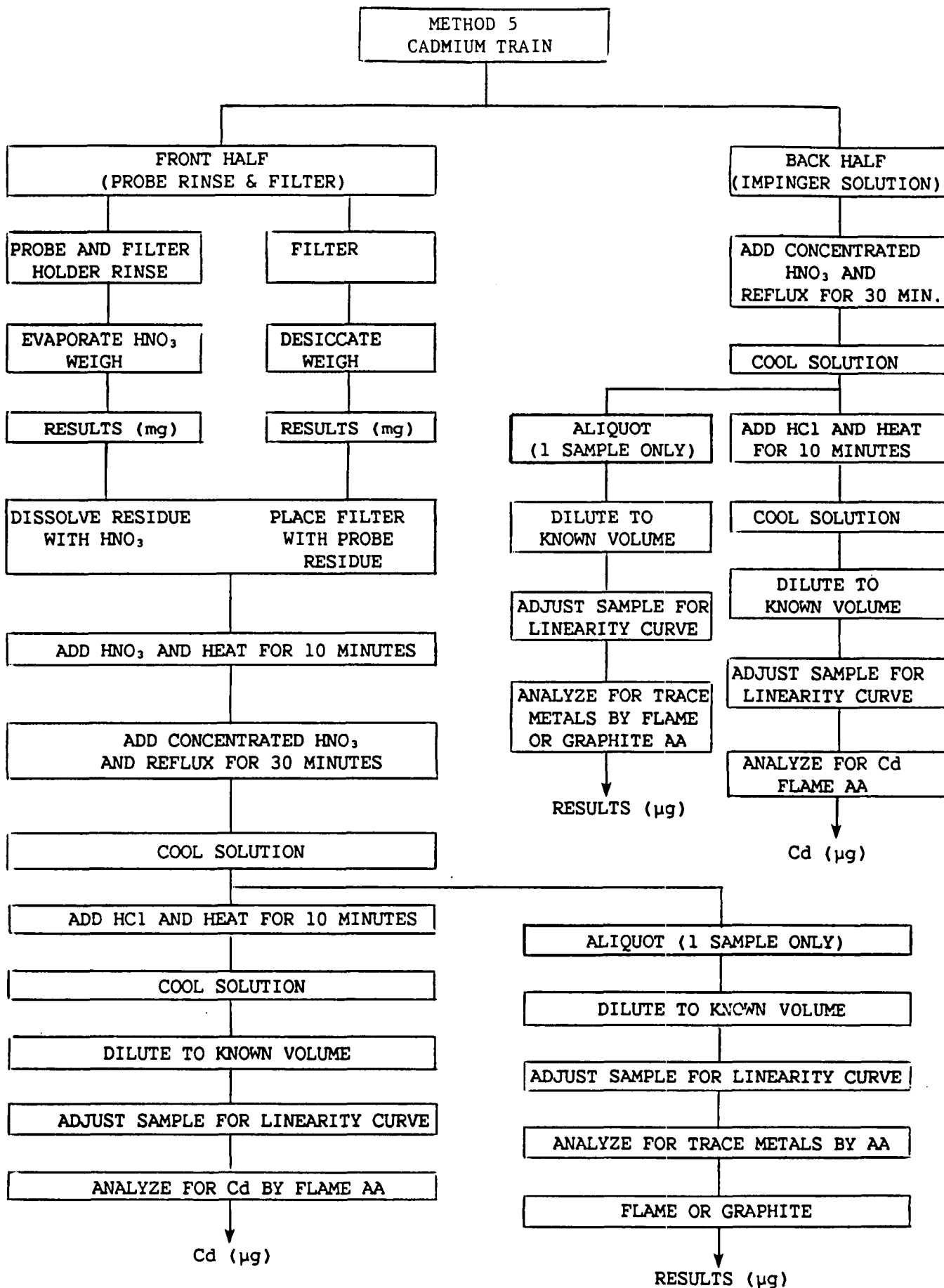


Figure 5-2. Schematic of Sample Analyses

## 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.

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 for equipment used during the test program are presented in the Appendix.

All Method 5 sampling was  $100 \pm 10$  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 evaluation tests were performed at Harshaw/Filtrol Partnership facility.

Each Method 5 sampling train had a clean-up sequence as follows:

Blank 1  
Method 5 Run 1  
Method 5 Run 2  
Method 5 Run 3  
Blank 2

All blank sample recovery was conducted identically to sample recovery procedures outlined in Section 5.

Blanks of all solution used during the test program (HNO<sub>3</sub>) were collected and analyzed.

In summary, the evaluation tests were 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 were performed by a one person clean-up crew. Appropriate sample recovery data were recorded on the sample identification log, sample recovery, chain of custody form, and analytical data forms as presented in the Appendix.

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

All preparation and analysis of Method 5 samples were performed by TRC. TRC adhered 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).

#### Analytical Quality Control

The analytical quality control procedures consisted of analyzing duplicate spikes, and blanks on 10% of the samples as described in TRC Technical Standard T/S-902 section E & F. Balances were calibrated with four class S rates daily. Since stack samples (filters, probe wash and impingers) cannot

be split before the metals digestion, duplicates were not done. Each type of sample was analyzed for cadmium by the method of standard addition to determine if there are any matrix interferences. If this result differed by more than 5% from the routine analysis, all the samples were analyzed by standard addition. The atomic absorption spectrophotometer was calibrated with four standards. Three measurements were taken on each sample for flame analysis and two for furnace analyses. Refer to Appendix A for sample calculations.

The laboratory participates in the following inter-laboratory quality control programs:

- State of Connecticut, Department of Health Services, Laboratory Division, Laboratory Improvement Program (annually).
- EPA, Environmental Monitoring and Support Laboratory, Quality Assurance Branch, Water Pollution Laboratory Performance Evaluation and Water Supply Performance Evaluation (voluntary).
- NIOSH, Proficiency Analytical Testing Program (quarterly).
- EPA Environmental Monitoring Systems Laboratory Quality Assurance Division. Stationary Source and Ambient Air, Inter-Laboratory Studies (semi-annually).

#### Method 9

The TRC emissions observer was certified within the past 6 months to perform visible emission evaluations. Documentation verifying the observers certification is provided in Appendix D.