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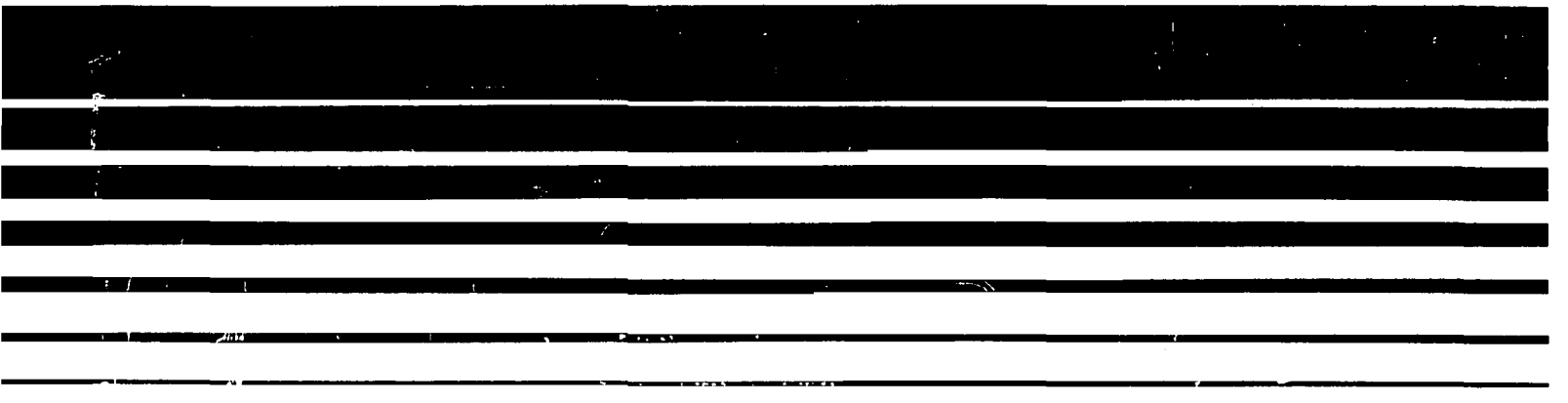
Chromium Electroplaters Test Report

Able Machine Co. Taylors, South Carolina



EMISSION TEST REPORT
METHOD DEVELOPMENT AND TESTING FOR CHROMIUM

CHROMIUM ELECTROPLATING INDUSTRY
ABLE MACHINE COMPANY
TAYLORS, SOUTH CAROLINA

ESED Project No. 85/2a
(86-CEP-3)

by

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Mr. Frank Clay, Emission Measurements Branch Task Manager, provided overall project coordination and guidance. Mr. Michael Hamlin of EMB observed the test program. Mr. Randy Strait and Ms. Robin Barker, representing Midwest Research Institute (MRI), monitored process and control equipment operation throughout the test period. Mr. Charles Bruffey was the PEI Project Manager. Principal authors were Messrs. Charles Bruffey and Thomas Wagner.

SECTION 1

INTRODUCTION

The U.S. Environmental Protection Agency (EPA) is currently evaluating chromium and several other potentially toxic metals and their compounds. Chromium emissions are not included in New Source Performance Standards (NSPS) for stationary sources or National Emissions Standards for Hazardous Air Pollutants (NESHAP).

As part of this study, EPA is evaluating atmospheric emissions of chromium from hard chromium plating operations. The purpose of these tests is to characterize uncontrolled and controlled emissions and size distribution of hexavalent chromium (Cr^{+6}) and total chromium (Cr) from a representative industrial operation.

The Emission Measurement Branch (EMB) of EPA's Environmental Standards and Engineering Division (ESED) requires contractor assistance in obtaining chromium emissions data from a representative source so that an accurate assessment of the potential problems can be made and appropriate regulatory action developed.

PEI Associates, Inc., under contract to EMB, conducted a testing program at the Able Machine Co. in Taylors, South Carolina, on June 30 and July 1 and 2, 1986. Triplicate tests to determine Cr^{+6} and total Cr emissions were performed at the inlet and outlet of a Duall mist eliminator controlling chromic acid emissions from one hard chromium plating tank.

In addition, particle size distribution measurements were taken at sampling points before and after the mist eliminator in an effort to characterize Cr^{+6} and total Cr emissions by size fraction. Samples of the plating tank solution and mist eliminator wash water were also collected during testing and analyzed for Cr^{+6} and total Cr.

The objectives of this project were met, and no major problems were encountered during the test project. Section 2 of this report presents a summary and discussion of test results; Section 3 addresses quality assurance; Section 4 describes the sampling locations and test procedures; and Section 5 describes source operation. Appendix A presents sample calculations and computer printouts; Appendices B and C contain the field data sheets and laboratory analytical results, respectively; Appendix D details sampling and analytical procedures; Appendix E summarizes equipment calibration procedures and results; Appendix F contains a list of project participants and a sampling log; and Appendix G describes the draft test method for analyzing hexavalent chromium emissions from stationary sources.

SECTION 2

SUMMARY OF TEST RESULTS

This section details the results of the sampling program. Subsections are used to identify results from each test type (i.e., Cr^{+6}, total Cr particle size distribution, etc.); results are expressed in both metric and English units where applicable.

2.1 TEST PROTOCOL

Table 2-1 presents the sampling and analytical protocol followed throughout this project, the test identification, and the sampling times for each specific test type.

In summary, triplicate tests were conducted simultaneously at the mist eliminator inlet and outlet to characterize uncontrolled and controlled Cr^{+6} and total Cr emissions from this type of source. Procedures detailed in EPA Test Methods 1 through 4* were used to measure flue gas flow rate, temperature, moisture content, and gas composition.

A Method 13B sampling train modified by eliminating the filter and placing 0.1 N NaOH in the impinger section was used to extract samples.** This methodology was developed by EPA during previous studies on similar plating operations. Cross-sectional, isokinetic sampling techniques were used in each case.

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* 40 CFR 60, Appendix A, EPA Reference Methods 1 through 4, July 1985.

** 40 CFR, Appendix A, Reference Method 13B, July 1985.

TABLE 2-1. SAMPLE/ANALYTICAL MATRIX FOR THE ABLE MACHINE COMPANY

Run No.	Date (1986) and time (24 h)	Location	Sample parameters: Modified Method 13b for Cr^{+6} and total Cr[a]	Sample parameters: Particle size distribution	Analytical parameters: Cr^{+6} diphenylcarbazide colorimetric method[b]	Analytical parameters: Total Cr by ICAP	Analytical parameters: Particle size distribution (gravimetric) Cr^{+6} and total Cr by size fraction
MEI-1	6/30 (1207-1609)	Inlet	X	-	X	X	-
MEO-1	6/30 (1208-1606)	Outlet	X	-	X	X	-
MEI-2	7/1 (0816-1143)	Inlet	X	-	X	X	-
MEO-2	7/1 (0815-1127)	Outlet	X	-	X	X	-
MEI-3	7/1 (1200-1500)	Inlet	X	-	X	X	-
MEO-3	7/1 (1209-1507)	Outlet	X	-	X	X	-
PSI-1	6/30 (1440-1540)	Inlet	-	X	-	-	X
PSO-1	6/30 (1209-1610)	Outlet	-	X	-	-	X
PSI-2	7/1 (0817-0932)	Inlet	-	X	-	-	X
PSO-2	7/1 (0817-1226)	Outlet	-	X	-	-	X
PSI-3	7/1 (1400-1515)	Inlet	-	X	-	-	X
PSO-3	7/2 (0836-1202)	Outlet	-	X	-	-	X
All	6/30-7/1 and 2	Process samples ° Tank solution	-	-	X	X	-
		° Mist eliminator wash water	-	-	X	X	-

[a]Method 13B sampling train modified by eliminating the sample filter and charging the impingers with 0.1 N NaOH. Cross-sectional, isokinetic sampling techniques were used.

[b]Test Methods for Evaluating Solid Waste. U.S. EPA SW-846, 2nd ed., July 1982.

[c]Inductively coupled argon spectroscopy (ICAP).

Hexavalent chromium content was determined by procedures recently developed by EPA for determining Cr^{+6} content in source emission samples. These latter procedures entail extraction of the sample fractions with an alkaline solution, followed by the diphenylcarbazide colorimetric method.*

Each emission sample was also analyzed for total chromium by use of Inductively Coupled Argon Spectroscopy (ICP) analytical techniques. A Perkin-Elmer Plasma II instrument was used for this analysis, which followed the general procedures outlined in EPA Method 3050 of EPA SW846.*

Samples were collected for particle size distribution measurements at the mist eliminator inlet and outlet by the use of in-stack cascade impactors. The Andersen Mark III multistage impactor was used at both locations.

Three particle size samples were collected at each location. Initially, the acetone rinse and filter fraction were subjected to gravimetric analysis using EPA Method 5 analytical procedures. At the completion of the gravimetric analysis, individual rinse and filter fractions were combined by stage cutpoint and location so that one composite sample was available for analysis of Cr^{+6} and total Cr. The filters were digested and analyzed for Cr^{+6} by use of procedures detailed in Appendix D of this report. Total Cr was determined from the digestion procedure filtrate using ICP analytical techniques.

During each emission test, plating tank solutions were collected. Grab samples were obtained approximately every 30 to 40 minutes during the Modified Method 13B tests. These grab samples were placed in a 1-gallon polyethylene container so that one composite sample of each type was available for analysis. Mist eliminator wash water was collected by MRI personnel at the end of each test day.

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* Test Methods for Evaluating Solid Waste. U.S. EPA SW-846, 2nd ed., July 1982.

All collected samples were analyzed for Cr^{+6} and total Cr by use of procedures similar to those used in the analysis of the Modified Method 13B samples. The following subsections detail the results of the sampling program.

2.2 HEXAVALENT AND TOTAL CHROMIUM EMISSION RESULTS

Table 2-2 summarizes pertinent sample and flue gas data, and Table 2-3 presents the results of the Modified Method 13B testing.

Sample volumes corrected to standard conditions [20°C and 760 mm Hg (68°F and 29.92 in.Hg) and zero percent moisture] are expressed in dry normal cubic meters (dNm^3) and dry standard cubic feet (dscf). Volumetric flow rates corrected to standard conditions are expressed as dry normal cubic meters per minute (dNm^3/min) and dry standard cubic feet per minute (dscf/min). Hexavalent and total chromium emission concentrations are expressed as milligrams per normal cubic meter (mg/dNm^3). Mass emission rates are expressed as kilograms per hour (kg/h) and pounds per hour (lb/h).

As reported in Table 2-2, sample volumes ranged between 3.16 and 4.66 dNm^3 for the inlet tests and between 2.21 and 3.48 dNm^3 for the outlet tests. Note that the first set of tests (MEI and MEO-1) were conducted for 180 minutes, while the remaining two tests were conducted for 120 minutes. Isokinetic sample rates ranged between 93.4 and 100.1 percent for all tests, which is within the applicable range of 90 to 110 percent.

At the mist eliminator inlet, volumetric gas flow rates ranged between 156 and 167 dNm^3/min and averaged 161 dNm^3/min (5680 dscf/min) for the three tests. Gas temperature and moisture content averaged 33°C (92°F) and 2.8

TABLE 2-2. SUMMARY OF SAMPLE AND FLUE GAS CONDITIONS
(Able Machine Co.)

Run No.	Date (1986)	Sample location	Sample parameter: Sample volume dNm³	Sample volume dscf	Percent isokinetic	Volumetric flow rate dNm³/min	Volumetric flow rate dscf/min	Flue gas condition: Temperature °C	Temperature °F	Moisture content, %	Static pressure, in. H_2O
MEI-1	6/30	Inlet	4.66	164.603	98.3	156	5,524	34	94	2.9	-1.9
MEO-1	6/30	Outlet	3.48	122.753	98.8	163	5,743	37	99	3.8	+1.5
MEI-2	7/1	Inlet	3.30	116.365	97.8	167	5,890	30	86	2.7	-1.7
MEO-2	7/1	Outlet	2.38	84.093	100.1	163	5,742	35	95	3.8	+1.5
MEI-3	7/1	Inlet	3.16	111.707	98.3	159	5,628	36	97	2.7	-1.65
MEO-3	7/1	Outlet	2.21	78.135	93.4	162	5,715	39	102	2.4	+1.5

TABLE 2-3. SUMMARY OF Cr^{+6} AND TOTAL Cr EMISSION DATA
(Able Machine Co.)

Run No.	Date (1986)	Sample location	Concentration				Mass emission rate				Total Cr collection efficiency[a], %
			Cr^{+6}		Total Cr		Cr^{+6}		Total Cr		
			mg/dNm³	gr/dscf	mg/dNm³	gr/dscf	kg/h	lb/h	kg/h	lb/h	
MEI-1	6/30	Mist eliminator inlet	10.2	0.004	10.0	0.004	0.095	0.21	0.095	0.21	98.6
MEO-1	6/30	Outlet	0.13	0.00006	0.14	0.00006	0.0014	0.003	0.0014	0.003	
MEI-2	7/1	Inlet	6.85	0.003	6.76	0.003	0.068	0.15	0.068	0.15	98.0
MEO-2	7/1	Outlet	0.14	0.00006	0.15	0.00006	0.0014	0.003	0.0014	0.003	
MEI-3	7/1	Inlet	6.84	0.003	6.90	0.003	0.064	0.14	0.064	0.14	98.6
MEO-3	7/1	Outlet	0.10	0.000045	0.11	0.00005	0.0009	0.002	0.0009	0.002	

[a] Total Cr collection efficiency calculated on mass rate basis.

$$\frac{\text{lb/h (in)} - \text{lb/h (out)}}{\text{lb/h (in)}} \times 100$$

percent, respectively. The static pressure of the inlet flue gas was continuously monitored using a 0- to 36-in. water manometer. Static pressures ranged between -1.65 and -1.90 in.H_2O.

At the mist eliminator outlet, volumetric gas flow rates averaged 163 dNm^3/min (5733 dscf/min) for the three tests, which compares to within 5 percent of the average inlet flow rate. Average temperature and moisture contents were 37°C (99°F) and 3.3 percent, respectively. The average static pressure measured during each outlet test was +1.5 in.H_2O.

The concentration of Cr^{+6} measured at the inlet to the mist eliminator ranged between 6.84 and 10.2 mg/dNm^3 (0.003 and 0.004 gr/dscf) and averaged 7.96 mg/dNm^3 (0.0033 gr/dscf) for the three tests. Mass rates for Cr^{+6} ranged between 0.064 and 0.095 kg/h (0.14 and 0.21 lb/h). Total Cr concentrations ranged between 6.76 and 10.0 mg/dNm^3 (0.003 and 0.004 gr/dscf) and averaged 7.89 mg/dNm^3 (0.0033 gr/dscf) for the three tests. Total Cr mass rates were essentially the same as the Cr^{+6} mass rates.

The content of Cr^{+6} in the inlet sample ranged between 21.6 and 47.4 mg, compared with values of 21.8 and 46.5 mg of total Cr. The overall comparability of the data suggests that the majority of Cr in the samples is in the form of Cr^{+6}.

Concentrations of Cr^{+6} measured at the mist eliminator outlet ranged between 0.10 and 0.14 mg/dNm^3 (0.000045 and 0.00006 gr/dscf). Mass rates for Cr^{+6} averaged 0.0012 kg/h (0.0027 lb/h). Total Cr concentrations ranged between 0.11 and 0.15 mg/dNm^3 (0.00005 and 0.00006 gr/dscf) with an average mass rate similar to that of Cr^{+6}. The content of Cr^{+6} in the outlet samples ranged between 0.226 and 0.451 mg and the content of total Cr ranged between 0.248 and 0.484 mg. On a mass rate basis, the overall Cr collection efficiency of the mist eliminator was 98 percent or greater for the three tests conducted.

2.3 PARTICLE SIZE DISTRIBUTION TEST RESULTS

Andersen Mark III in-stack impactors were used to measure particle size at each location. Each impactor consists of eight impaction stages followed by a backup filter. In these tests, glass-fiber filter media were used. A total of three samples were collected at each location at points in the duct(s) representing the average velocity and temperature.

Each test was conducted according to the procedures described in the Mark III operations manual supplied by the manufacturer. Isokinetic sampling rates were set initially, and constant cut-point characteristics were maintained throughout the sampling period. Test times were 180 minutes at the outlet location and between 60 and 75 minutes for the inlet samples.

At the completion of each test, the impactor samples were recovered according to procedures descriged in the Mark III operations manual.

Each individual impactor stage and acetone rinse of the sample nozzle and impactor casing was subjected to a gravimetric analysis using procedures similar to those in EPA Method 5. Cumulative size distribution data points representing the total weight of particulate matter smaller than the indicated aerodynamic particle diameter [in micrometers (μm)] were established for each individual test. The cut points for each test were calculated by computer programs contained in "A Computer-Based Cascade Impactor Data Reduction System"* (CIDRS) developed for U.S. EPA by Southern Research Institute (SRI). All particle-size results are based on a particle density of 1 g/cm.^{3} Data reduction for the particle-size tests was performed by computer programming; data on flue gas moisture and molecular weight were obtained from the Method 13B tests.

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* Southern Research Institute. A Computer-Based Impactor Data Reduction System. Prepared for U.S. EPA under Contract No. 68-022-131, Revised March 1980.

Figures 2-1 through 2-6 depict individual size distributon curves by test location. These curves were plotted using size cutpoint and cumulative percent weight data from CIDRS computer programs. Actual impactor stage data points are depicted by the solid dots, and the open dots represent an extrapolated best-fit curve. (See Appendix A.)

For the inlet impactor runs (Figures 2-1 through 2-3), individual impactor stages did not contain enough particulate matter to yield reliable data (no more than 0.4 mg was collected on any one stage, compared with a desired amount of between 1 and 10 mg). Although the total catch for these runs ranged between 15.9 and 42.4 mg, the majority of material was collected in the sample nozzle and impactor casing prior to the filter media. Since the collected material was observed to be a liquid mist, particles that normally would be collected on the various stages may have been collected in the nozzle and casing, which would tend to bias the cumulative percent less than 10 to 15 μm on the low side. The cumulative size distribution curves for these runs show that the percent less than 10 μm ranged from about 2 to 5 percent with about 2 to 3 percent less than 2.5 μm. The validity of this data is questionable.

For the outlet impactor runs (Figures 2-4 through 2-6), individual impactor stage loadings ranged between zero and 0.4 mg, which is less than the desired loadings of between 1 and 10 mg per stage.

Once again, the majority of the total catch for these runs was found in the sample nozzle and impactor casing prior to the filter media. The total catch ranged between 0.9 and 6.0 mg. It should be noted that 0.1 N NaOH was inadvertently used to rinse the nozzle and impactor casing for Test PSO-1; therefore, only the filter weights are reported, which (for all practical purposes) are considered void. For Tests PSO-2 and 3, the cumulative percent

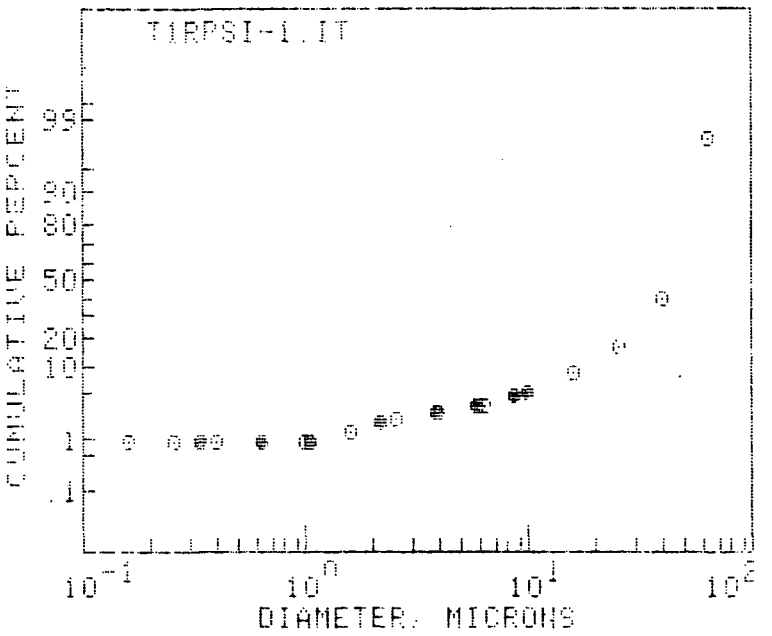

Figure 2-1. Particle size distribution for mist eliminator inlet Run PSI-1.

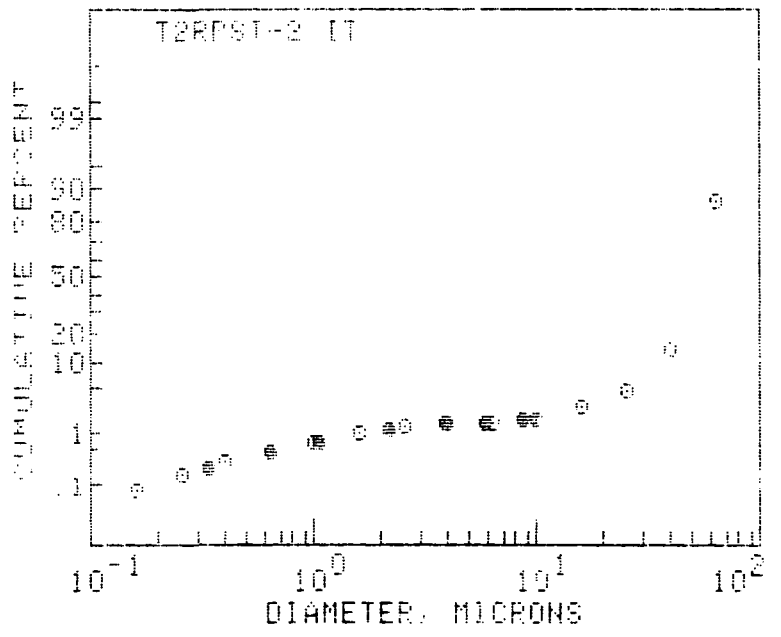

Figure 2-2. Particle size distribution for mist eliminator inlet Run PSI-2.

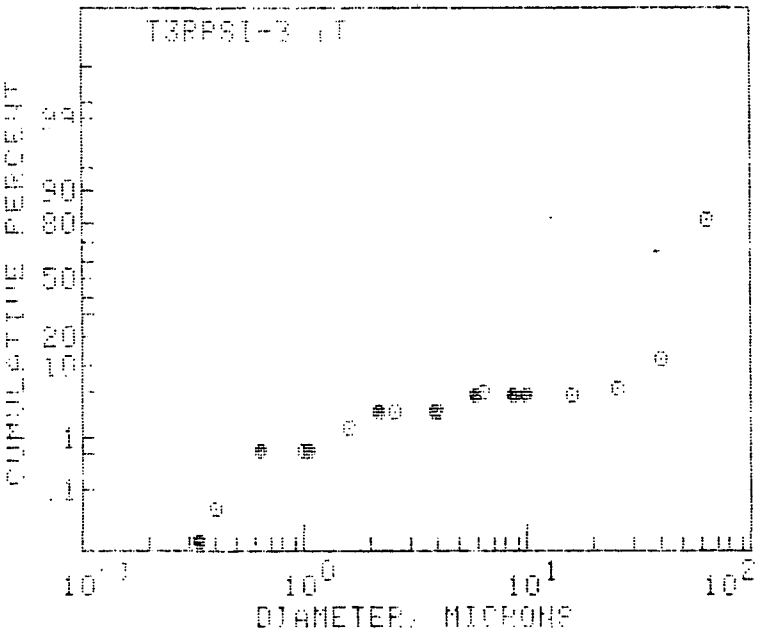

Figure 2-3. Particle size distribution for mist eliminator inlet Run PSI-3.

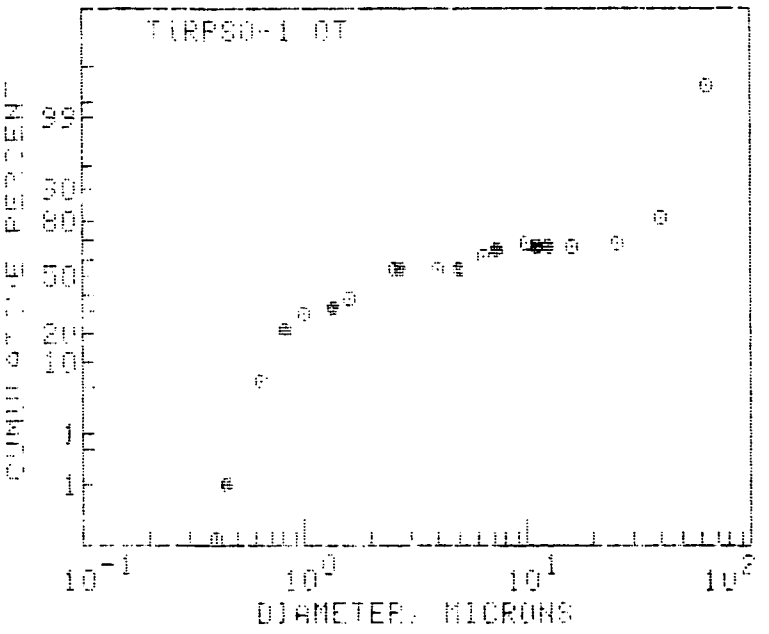

Figure 2-4. Particle size distribution for mist eliminator outlet Run PSO-1.

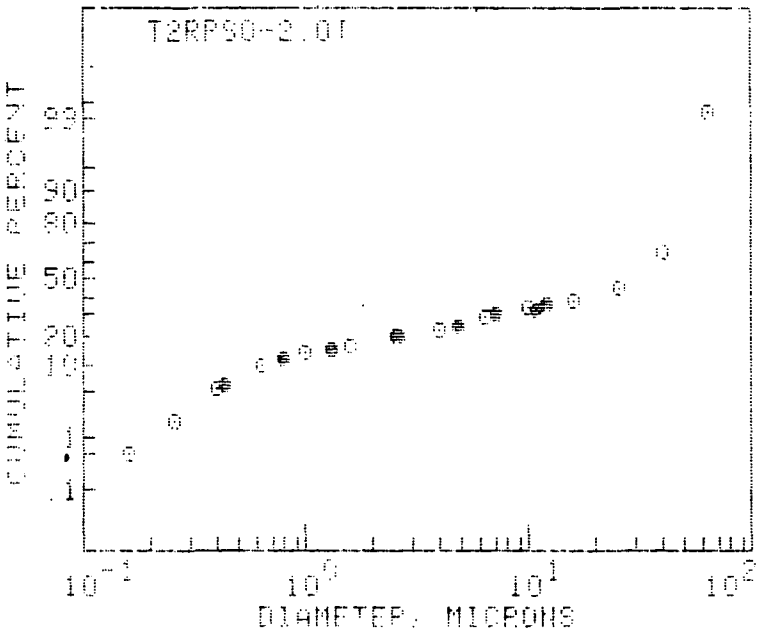

Figure 2-5. Particle size distribution for mist eliminator outlet Run PSO-2.

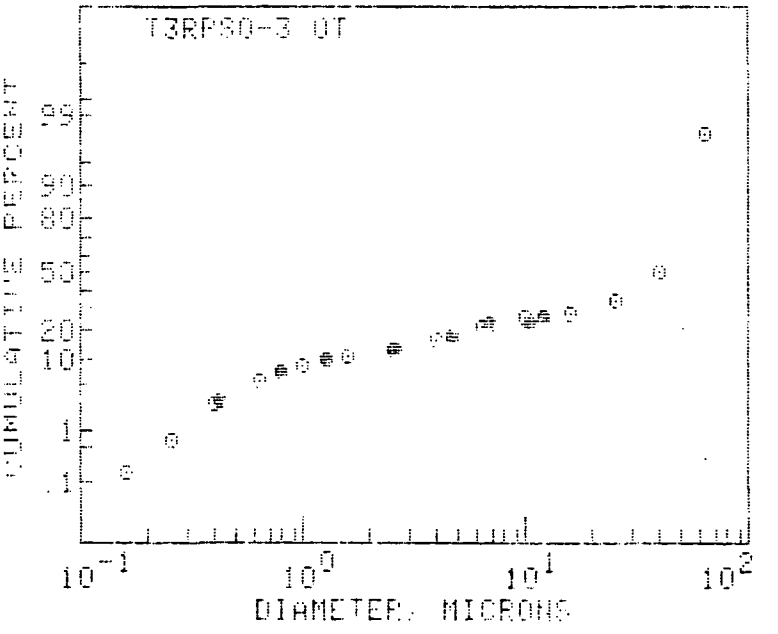

Figure 2-6. Particle size distribution for mist eliminator outlet Run PSO-3.

less than 10 μm ranged between 26 and 35 percent, while the percent less than 2.5 μm ranged between 13 and 20 percent.

The average isokinetic sample rates* were all within the acceptable range of the IP Protocol (80 to 120 percent), and the impactor sampling rates were all within the manufacturer's suggested operating limits (0.3 to 0.75 acfm).

In an attempt to characterize Cr^{+6} and total Cr by size fraction, inlet and outlet samples were combined by stage cutpoint into a single composite sample from each location and analyzed for Cr^{+6} and total Cr. Combined filters were digested following procedures described in Method 3060 of EPA SW-846 (alkaline digestion method) and analyzed for Cr^{+6} using the diphenol-carbazide colorimetric method. The alkaline extract residue was then digested using Method 3050 of EPA SW-846 and analyzed for total Cr using ICP analytical techniques. Table 2-4 summarizes the analytical results. The inlet data show the majority of Cr^{+6} and total Cr in the acetone rinse as greater than 10 μm in diameter. These data correspond to the gravimetric data presented in this section. The remainder of Cr^{+6} and total Cr is concentrated on stages 2 through 6 with cutpoints ranging from 3.8 to less than 1.0 μm.

For the outlet sample, the majority of Cr^{+6} and total Cr (exclusive of the acetone rinse) is concentrated on stages 4 through 7 with cutpoints ranging from 2.6 to less than 0.5 μm. Note that the total amount of Cr^{+6} and total Cr on each stage do not compare favorably as did the results of the

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* Southern Research Institute. Procedures Manual for Inhalable Particulate Sampler Operation. Prepared for U.S. EPA under Contract No. 68-02-3118, November 1979.

TABLE 2-4. SUMMARY OF Cr^{+6} AND TOTAL Cr SIZE DISTRIBUTION DATA

Run No.	Stage No.	Range of size cutpoints,[a] µm	Cr^{+6}, µg (blank corrected)	Total Cr, µg (blank corrected)
PSI 1-3	0	9.7 - 9.8	4.6	18.25
	1	8.6 - 8.7	6.5	18.0
	2	5.8	27.8	51.45
	3	3.8 - 3.9	64.8	101.1
	4	2.1	78.4	125.25
	5	1.03 - 1.04	35.9	61.4
	6	0.64	22.4	42.75
	7	0.34	13.8	30.9
	Backup	<0.34	1.3	0
	Acetone	>10 µm	13,800	24,800
PSO 1-3	0	11.8 - 12.1	4.3	15.85
	1	10.4 - 10.6	2.2	10.8
	2	6.9 - 7.1	3.1	14.1
	3	4.6 - 4.7	3.8	15.8
	4	2.6	20.1	40.1
	5	1.3	68.9	102.1
	6	0.8	30.6	50.5
	7	0.43	25.9	45.8
	Backup	<0.43	5.2	15.6
	Acetone	>12 µm	121	164

[a] Range of size cutpoints as determined from the CIDRS computer program. (See Appendix A.)

modified Method 13B samples. This probably results from a reduction of Cr^{+6} to a lower valence state on the glass-fiber filter media.

2.4 PROCESS SAMPLE ANALYTICAL RESULTS

Samples of plating tank solution were collected during each modified Method 13B emission test and analyzed for Cr^{+6} and total Cr using procedures similar to those used for the emission samples. Mist eliminator washwater was collected at the end of each day (6/30 and 7/1) on which the Method 13B tests were conducted. Table 2-5 summarizes the analytical results.

TABLE 2-5. PROCESSS SAMPLE ANALYTICAL RESULTS

Laboratory No.	Run No./ description	Fraction	Chromium(VI), mg/liter	Total chromium, mg/liter
FT499	ME wash water, 6/30	Liquid	2,790	3,490
FT450	ME wash water, 7/1	Liquid	3,470[a]	4,220
FT451	MEI (MEO) -1 tank	Liquid	79,000	84,500
FT452	MEI (MEO) -2 tank	Liquid	81,000	85,800
FT453	MEI (MEO) -3 tank	Liquid	82,700	85,100

[a] Spike recovery was 105.8 percent for Cr(VI) and 70.5 percent for total Cr. In the total chromium spike, 2 µg was added to the 40 µg present in the sample. This spike level was too low for the amount already in the sample and probably explains the lower recovery determined for this sample.

SECTION 3

PROJECT QUALITY ASSURANCE

The application of quality assurance procedures to source emission measurements ensures accurate emission-testing results. Quality assurance guidelines provide the detailed procedures and actions necessary for defining and producing acceptable data. In this project, four documents were used in the preparation of a source-specific test plan that would ensure the collection of acceptable data: 1) EPA Quality Assurance Handbook, Volume II, EPA-600/4-77-0271; 2) PEI Emission Test Quality Assurance Plan; 3) PEI Laboratory Quality Assurance Plan; and 4) Determination of Hexavalent Chromium Emissions From Stationary Sources, December 13, 1984. Two of these are PEI's general guideline manuals that define the standard operating procedures followed by the company's emission testing and laboratory groups.

In this specific test program, which was reviewed by EPA's Emission Measurement Branch, the following steps were taken to ensure that the testing and analytical procedures produced quality data:

- Onsite quality assurance checks, such as leak checks of the sampling train, pitot tube, and Orsat line. Onsite quality assurance checks of all test equipment prior to its use.
- Use of designated analytical equipment and sampling reagents.
- Internal and external audits to ensure accuracy in sampling and analysis.
- Calibration of all field sampling equipment.
- Checks of train configuration and calculations.

Table 3-1 lists the specific sampling equipment used to perform the Cr^{+6}, total Cr, and particle size distribution tests as well as the calibration guidelines and limits. In addition to the pre- and post-test calibrations, a field audit was performed on the metering systems and temperature-measurement devices used during sampling. These data are summarized in Table 3-2, and copies of the field audit data sheets are presented in Appendix B of this report.

The PEI project manager and EPA Task Manager performed the onsite sample calculations, and computer programming was used to validate the data upon return to PEI's Cincinnati laboratory. Minor discrepancies between the hand calculations and computer printouts are due primarily to rounding off of values. Computerized example calculations are presented in Appendix A.

The following subsections summarize the quality assurance activities performed during the analytical phase of this project. As a check of the gravimetric analytical procedure for particle sizing, a blank set of filters and a reagent (acetone) were analyzed in a fashion similar to that used for the actual field samples. Table 3-3 summarizes the blank analysis data, which indicate good gravimetric analytical technique.

Emission and process samples were analyzed in two separate batches. Table 3-4 summarizes the linear regression data of the spectrophotometer calibration for these samples. Standards containing 0, 5, 10, 15, 20, and 25 µg of chromium(VI) per 50 ml were analyzed with each batch of samples. The detection limits listed in Table 3-4 are based on an absorbance value of 0.005.

TABLE 3-1. FIELD EQUIPMENT CALIBRATION

Equipment	ID No.	Calibrated against	Allowable error	Actual error	Within allowable limits	Comments
Meter box	FB-3	Wet test meter	ΔH@ ± 0.15	0.05; 2.25%	✓	
	FB-9		(Y ± 0.05 Y	0.11; 0.91%	✓	
	FB-11		post-test)	0.05; -0.204%	✓	
	FT-1			0.05; -1.2%	✓	
Pitot tube	242	Standard pitot tube	Cp ± 0.01		✓	Visually inspected onsite. Cp = 0.84 per Method 2.
	504				✓	
	015				✓	
	016				✓	
Digital indicator	FT-1	Millivolt signals	0.5%	+0.22%	✓	Maximum deviation.
	219			+0.20%	✓	
Thermocouple and stack thermometers	411			0.40%	✓	
	101	ASTM-2F or 3	1.5%	0.22%	✓	Maximum deviation.
	412		(±2% saturated)	0.3%	✓	
	409			0.15%	✓	
Orsat analyzer	422	Standard gas	± 0.5%	CO: 0.2% O_2: 0.0 CO_2: 0.2%	✓	
Impinger thermocouple	I-15	ASTM-2F or 3F	± 2°F	+1°F	✓	
	I-1			+2°F	✓	
Trip balance	Mettler 1	Type S weight	± 0.5 g	0.0	✓	
Barometer	406	NBS traceable barometer	± 0.10 in.Hg (0.20 post-test)	+0.01	✓	

(continued)

TABLE 3-1 (continued)

Equipment	ID No.	Calibrated against	Allowable error	Actual error	Within allowable limits	Comments
Dry gas thermometer	FB-3	ASTM-2F or 3F	± 0.5°F	In: +2°F; Out: +1°F	✓	Maximum deviation
	FB-9			In: +3°F; Out: +2°F	✓	
	FB-11			In: +2°F; Out: +2°F	✓	
	FT-1			In: +2°F; Out: +2°F	✓	
Probe nozzle	MEO Caliper		Dn ± 0.004 in.	0.001	✓	
	MEI Caliper			0.003	✓	
	3-110			0.004	✓	
	3-104			0.000	✓	

TABLE 3-2. ON-SITE FIELD EQUIPMENT CALIBRATION VERIFICATION

Equipment	ID No.	Calibrated against	Allowable deviation	Actual deviation	Within allowable limits	Comments
Meter box	FB-3 FB-11 FT-1 FB-9	Critical orifice	Y ± 0.05 Y ΔH@ ± 0.15	-3.10; + 0.06 -0.81; -0.05 -0.01; +0.03 -2.7; - 0.01	✓ ✓ ✓ ✓	PEI constructed critical orifices used for this audit.
Pitot tube		(Geometrical specs) (Cylinder pitot tube)	Cp ± 0.01		✓	Visually inspected on site.
Digital indicator	FT-1 219	Millivolt signals	1.0%	NA -0.45	✓ ✓	
Thermocouple and stack thermometers	411 101 412	ASTM-3F	± 7°F (±2°F saturated)	NA NA NA	 ✓ ✓	See Table 3-1.
Orsat analyzer	422	Ambient air O_2	± 0.7%	NA	✓	See Table 3-1.
Impinger thermocouple	I-1 I-15	ASTM-3F	± 2°F	-2°F -1°F	✓ ✓	
Trip balance		Type S weight	± 0.5 g	NA	✓	
Dry gas thermometer		ASTM-3F	± 5°F	NA	✓	
Probe nozzle		Caliper	Dn ± 0.004 in.		✓	See Table 3-1.

TABLE 3-3. FILTER AND REAGENT BLANK ANALYSIS DATA

Sample type	PEI lab No.	Tare weight, mg	Average gross weight, mg	Net difference, mg
Acetone[a]	FT337	98,791.3	98,799.2	7.9 mg (0.0410 mg/g)
Andersen filter set				
Stage 0, No. AS-37	FT328	159.6	159.7	0.1
Stage 1, No. AP-37	FT329	164.2	164.2	0.1
Stage 2, No. AS-19	FT330	162.4	161.9	0.5
Stage 3, No. AS-49	FT331	163.0	162.6	0.4
Stage 4, No. AM-32	FT332	144.1	143.7	0.4
Stage 5, No. AP-90	FT333	142.3	142.5	-0.2
Stage 6, No. AP-88	FT334	142.5	142.5	0
Stage 7, No. AO-06	FT335	149.3	149.3	0
Backup, No. A-294	FT336	220.2	219.6	0.6

[a] If a blank residue value greater than 0.01 mg/g or 0.001 percent of the blank weight was obtained, a maximum value of 0.01 mg/g was subtracted from the sample weight.

TABLE 3-4. LINEAR REGRESSION DATA FOR SPECTROPHOTOMETER CALIBRATION

Date (1986)	Y-Intercept	Slope	Correlation coefficient	Duplicate curves	Detection limit, µg/ml
7/20	-0.0045	0.0293	0.9998	No	<0.4
8/12	-0.0000	0.0265	0.9999	Yes	<0.2

The ICP was also calibrated for each of the two batches. The initial calibration consists of a blank and a 5-ppm standard, both containing 50 ppm of scandium as an internal standard. The internal standard is also added to all samples at the same concentration. Table 3-5 summarizes the results of the ICP QC check sample (1.00 ppm) analyzed after approximately every tenth sample.

TABLE 3-5. QC CHECK SAMPLE DATA FOR ICP

Date (1986)	Value determined, ppm
7/23	1.00
	1.02
	1.06
	1.05
8/15	0.93
	0.92
	0.97
	0.93
	0.92
	0.94

Table 3-6 summarizes all blank data for chromium(VI) and total chromium analyses.

TABLE 3-6. SUMMARY OF BLANK DATA

Description	Chromium(VI), μg	Total chromium, μg
Reagent blank for particulate	<0.4	<2
Acetone blank	0.6	<2
Filter stages 0, 2, 4, 6	0.9	11.0
Filter stages 1, 3, 5, 7	0.8	11.6
Backup filter	1.5	19.8
Method 5 sample blank	<6[a]	<20[a]

[a] Based on largest volume of sample received.

Two fractions were analyzed for the modified Method 5 samples and the process: the liquid and the digested solids. The amount of solids were small, and the amount of chromium(VI) and total chromium in the solids were insignificant compared with that in the liquid.

Table 3-7 summarizes the results of the spike sample and duplicate sample analysis for chromium(VI) and total chromium.

TABLE 3-7. RESULTS OF SPIKE AND DUPLICATE ANALYSES[a]

Run No.	Spike recovery, %	Duplicate results, mg/liter
PSI 1-3 Stage 5	87.4 total Cr	-
PSI 1-3 acetone	88.4 Cr(VI)	-
MEI-3	101.0 Cr(VI)	-
MEO-1	92.5 total Cr	-
MEI wash water 7/1	105.8 Cr(VI) 70.5 total Cr[b]	3,470, 4070 mg/liter Cr(VI) 4,220, 3,950 mg/liter total Cr
MEI wash water 6/30	- -	2,790, 3,270 mg/liter Cr(VI) 3,490, 3,320 mg/liter total Cr

[a] Spike recoveries on solid samples were within the same range.

[b] Spike level was inappropriate for accurate recovery determination (2 µg were spiked in 40 µg).

SECTION 4

SAMPLE LOCATIONS AND TEST METHODS USED

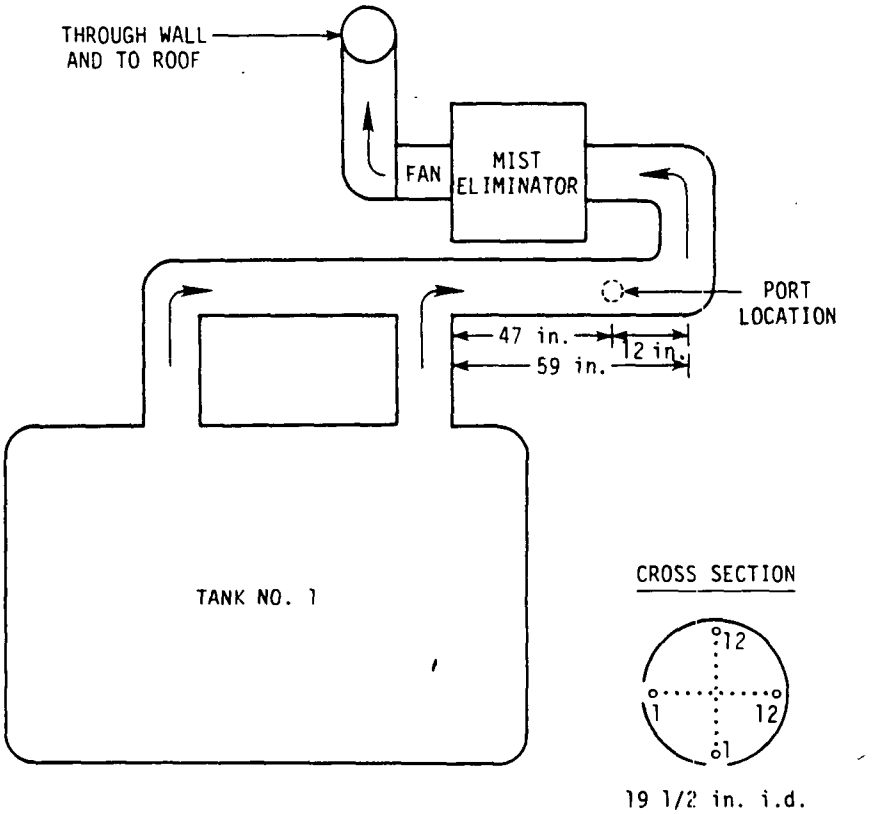
4.1 SAMPLE LOCATIONS

Samples were simultaneously extracted from the inlet and outlet ducts of the mist eliminator. Figures 4-1 and 4-2 show the inlet and outlet sample locations.

At the inlet, two sampling ports were located 90 degrees off-center, approximately 2.4 duct diameters (dd) downstream and 0.62 dd upstream from the nearest flow disturbance in the 19½-inch i.d. round duct. A total of 24 traverse points (12 per port) were used to traverse the cross-sectional area of the duct. Sample times were 180 minutes (7.5 minutes per point) for the first test (MEI-1) and 120 minutes (5 minutes per point) for the remaining two tests (MEI-2 and -3).

At the outlet, two sampling ports were located 90 degrees off-center, approximately 1.33 dd downstream and 0.58 dd upstream from the nearest flow disturbances in the 24-inch i.d. round duct. A total of 24 sample points were used to traverse the cross-sectional area of the stack. Sample times were identical to those used at the inlet. The minimum port location criteria specified in EPA Method 1* could not be met at the mist eliminator outlet location; however, this was the only feasible location to extract samples. As detailed in Section 2 of this report, the quality of inlet and

* 40 CFR 60, Appendix A, Reference Method 1, July 1985.



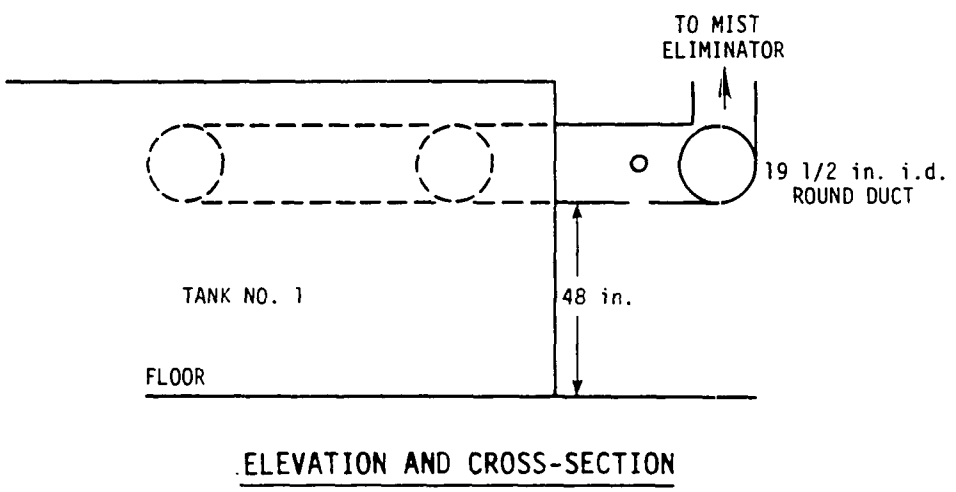


Figure 4-1. Tank No. 1 Inlet Sample Location, Able Machine Co.

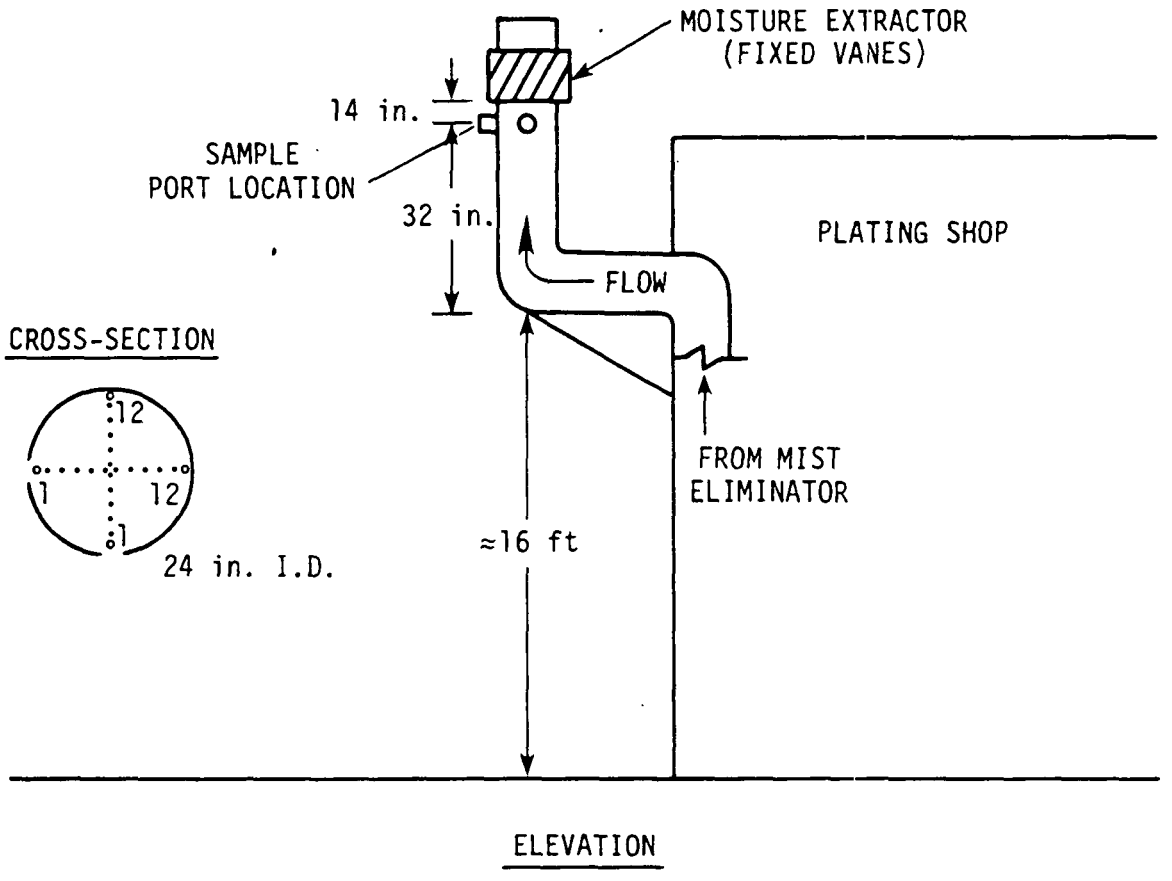


Figure 4-2. Tank No. 1 Outlet Sample Location, Able Machine Co.

outlet flow rate data indicates that this problem did not adversely affect test results. Note also that samples were extracted after the mist eliminator but before a fixed vane moisture extractor designed to remove mist which may pass through the mist eliminator.

4.2 HEXAVALENT AND TOTAL CHROMIUM SAMPLE EXTRACTION AND ANALYSIS

As shown previously in Table 2-1, three tests were conducted at points located before and after the mist eliminator in order to determine the Cr^{+6} and total Cr content.

Prior to sampling, velocity, static pressure, molecular weight, moisture content, and temperature were measured to define sampling rates and nozzle sizes as described in EPA Reference Methods 1 through 4.* In addition, the degree of turbulent flow at each location was assessed based on procedures described in EPA Reference Method 2. In this method, the face openings of the Type-S pitot tube are aligned perpendicularly to the duct cross-sectional plane, designated "0-degree reference." Null (zero) pitot readings obtained at 0-degree reference indicate an acceptable flow condition at a given point.

If the pitot reading is not zero at 0-degree reference, the pitot is rotated (up to 90 degrees ± yaw angle) until a null reading is obtained. The value of the rotation angle (yaw) is recorded for each point and averaged across the duct. Method 2 criteria stipulate that average angular rotations greater than ±10 degrees indicate turbulent (nonaxial) flow conditions in the duct(s). Angular rotations of less than 10 degrees were observed at each location, which indicated acceptable flow patterns and enabled the extraction of representative samples from this source.

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* 40 CFR 60, Appendix A, Reference Methods 1 through 5, July 1985.

An EPA Method 13B sampling train was used to extract samples. The train was modified by eliminating the sample filter and placing 300 ml of 0.1 N NaOH in the impinger section. Each train consisted of a heated, glass-lined probe followed by a series of four Greenburg-Smith impingers, a calibrated orifice, a dry gas meter, and associated equipment to measure gas flow and temperature and maintain isokinetic sampling conditions.

The impingers were weighed before and after each test to determine the moisture content of the flue gas stream. The contents of the impingers were placed in a polyethylene container, and all glassware including the sampling nozzle and probe were rinsed with 0.1 N NaOH; this rinse was added to the same container. Appropriate blank solutions (0.1 N NaOH) were also taken for analysis. Upon return to the laboratory, each sample (including blanks) was analyzed for Cr^{+6} using analytical methodology recently developed by EPA. A copy of the draft method entitled "Determination of Hexavalent Chromium Emissions From Stationary Sources" is contained in Appendix G of this report. In summary, this method entails the extraction of the sample with an alkaline solution, followed by the diphenylcarbazide colorimetric method.*

At the completion of the Cr^{+6} analysis, a separate portion of each sample was digested and analyzed for total Cr by use of ICP analytical techniques.** Appendix D of this report contains the detailed analytical methodology used for these analyses.

4.3 PARTICLE SIZE DISTRIBUTION

Three samples were collected at each test location to determine particle size distribution. These tests were designed to characterize Cr^{+6} and total

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* Test Methods for Evaluating Solid Waste. U.S. EPA SW-846, 2nd ed., July 1982. Method 3060.

** Test Methods for Evaluating Solid Waste. U.S. EPA SW-846, 2nd ed., July 1982. Method 3050.

Cr emissions by size fraction. All size distribution tests were performed in accordance with procedures detailed in the equipment manufacturer's operations manual. Guidelines established in the IP Protocol* were used to evaluate collected data.

Samples for particle-size distribution measurements were collected using an Andersen Mark III impactor with glass-fiber filters as the substrated media. This in-stack impactor consists of eight cut-point stages and a backup filter. The sampled gas stream enters the system through the precutter. Particles with sufficient inertia are impacted against the sides of the precutter. Smaller particles flow with the gas stream, exit the precutter, and enter the main impactor. Then, particles with sufficient inertia are impacted on the front of the zero stage plate (no filter), smaller particles pass through the holes in the zero stage plate, and the portion of these particles with sufficient inertia impacts on the zero stage filter. The remainder of the particles pass through the holes in the first stage plate and similarly on to each succeeding stage. Finally, a glass-fiber backup filter removes all particles remaining in the gas stream downstream of the final, seventh stage plate.

A single impactor was used to collect samples at each location. Two points of average velocity were selected at the outlet location and a single point of average velocity was selected at the inlet. Sampling times were 180 minutes at the outlet and between 60 and 75 minutes for the inlet samples.

Isokinetic sampling rates were set initially based on the expected average gas velocity at the selected sample points, and constant cutpoint characteristics were maintained throughout the sampling period. The average

* Procedures Manual for Inhalable Particulate Sampler Operation. Prepared by Southern Research Institute for EPA, Contract No. 68-02-3118, November 1979.

isokinetic sampling rate for each run was based on the actual flue gas velocity pressures and temperatures measured at each of the test points. At the completion of each test, the impactor samples were recovered according to procedures described in the mark III operations manual.

Each recovered fraction was subjected to a gravimetric analysis using procedures similar to those in EPA Method 5, except that the "constant weight" criteria for the filters was ±0.2 mg instead of ±0.5 mg. At the completion of the gravimetric analysis, samples were combined by location and stage cutpoint for analyses of Cr^{+6} and total Cr. Analytical procedures followed those previously described.

Cut-points for the eight Mark III impactor stages were calculated by computer programs contained in "A Computer-Based Cascade Impactor Data Reduction System" (CIDRS) developed by Southern Research Institute (SRI).* All particle size results are based on a particle density of 1 g/cm^3. Data reduction and intermediate results calculations were performed by the CIDRS program, and moisture contents and gas molecular weights were obtained from the Cr^{+6}/total Cr tests. Size distribution curves were established to represent the total weight percent of particulate matter smaller than the indicated aerodynamic particle diameter in micrometers.

4.4 PROCESS SAMPLES

Process samples (plating tank solution) were collected by PEI personnel during each test period. Each sample was collected at least four times

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* Southern Research Institute. A Computer-Based Cascade Impactor Data Reduction System. Prepared for U.S. Environmental Protection Agency under Contract No. 68-022-131, Revised March 1980.

during the test period and placed in polyethylene containers. A sample of mist eliminator wash water was collected at the end of each test day and placed in a polyethlyene container. These samples were analyzed for Cr^{+6} and total Cr according to procedures similar to those used for the actual emission samples.

SECTION 5

PROCESS DESCRIPTION AND OPERATION

5.1 PROCESS DESCRIPTION

Able Machine Company is a small-sized job shop that performs hard chromium electroplating of industrial rolls. Hard chromium plating of industrial rolls provides a wear-resistant surface and protection from corrosion. The plating facility consists of two tanks, a new tank and an old tank. The old tank, however, is used only when the new tank is down for repairs or otherwise unavailable. The emission measurements documented in this report were performed on the new tank (see Figure 5-1) and its associated control device.

The new tank was installed in July 1985. Based on size; operating parameters such as current, voltage, plating time; and chromic acid concentration, the tank is typical of other hard chromium plating tanks used in the electroplating industry. The tank is 4.3 meters (m) (14.0 feet [ft]) long, 1.2 m (4.0 ft) wide, and 3.0 m (10.0 ft) deep, and holds about 15,820 liters (ℓ) (4,180 gallons [gal]) of plating solution. The plating bath used is a conventional hard chromium plating solution containing about 210 grams per liter (g/ℓ) (28 ounces per gallon [oz/gal]) of chromic acid and 1.3 g/ℓ (0.18 oz/gal) of sulfuric acid. The normal operating temperature of the plating bath ranges from 43 to 60°C (110° to 140°F). The tank is cooled with circulating water. The tank is equipped with a transformer rectifier rated at 12 volts and 12,000 amperes.

The plating tank is operated 8-hours (h) per day, 5 days per week. However, the tank is sometimes operated overnight to plate rolls that require a thick metal deposit. Typically, the tank is operated at full capacity (12 rolls). An overhead hoist is used to transport rolls to and from the plating tank. After plating, the rolls are rinsed with water from a hose over the top of the plating tank. This rinsing allows excess plating solution on the rolls to drain into the plating tank, thus reducing drag-out. It takes a total of about 40 minutes to unload and load the plating tank.

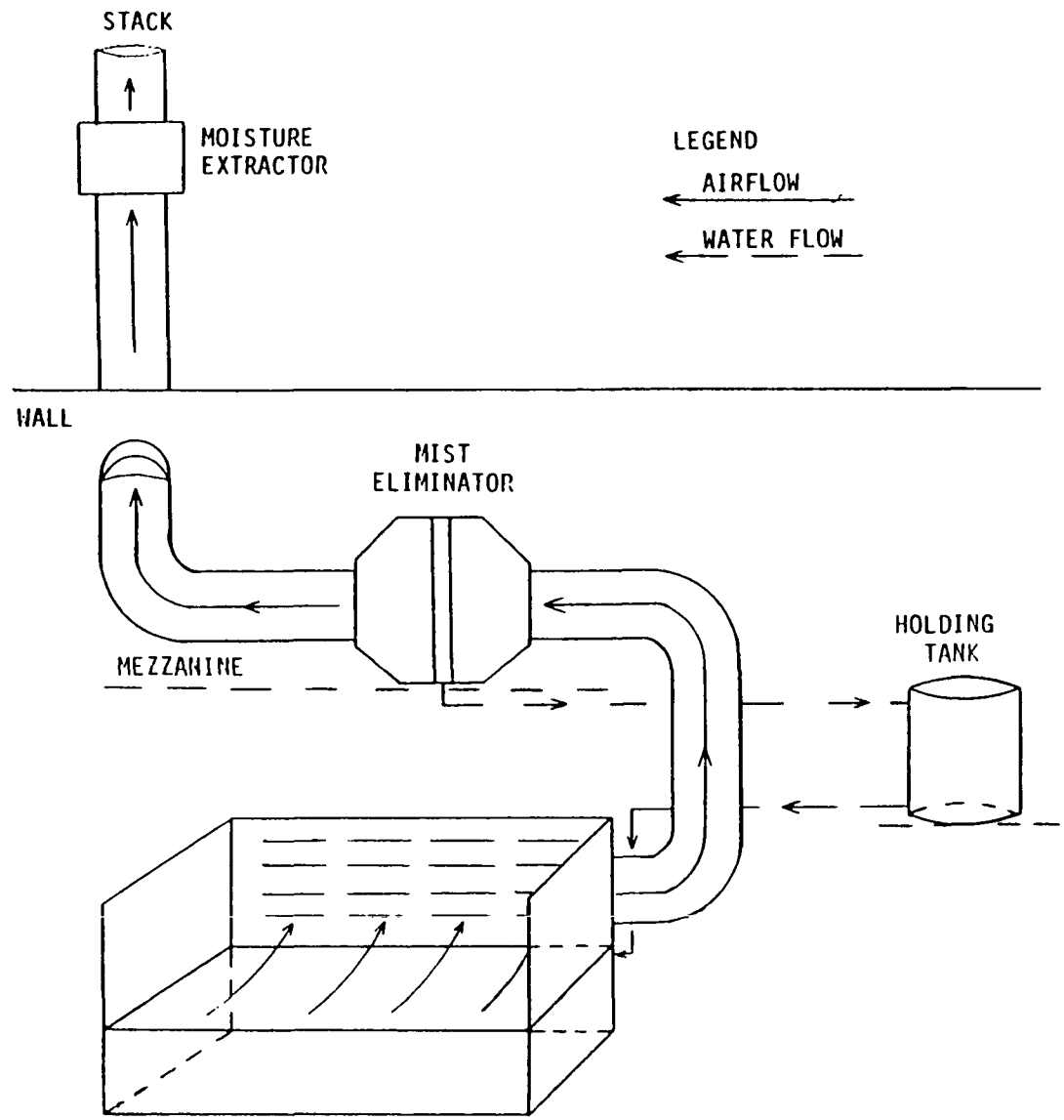

Figure 5-1. Schematic of new hard chromium plating tank at Able Machine Company.

5.2 AIR POLLUTION CONTROL

The plating tank is equipped with a push-pull capture system and a chevron-blade mist eliminator that were manufactured and installed in July 1985 by Duall Industries, Incorporated. The push side of the capture system consists of a 5.1-cm (2-in.) diameter pipe along the entire length of the tank. The pipe contains 72 holes that are each 0.32 cm (0.125 in.) in diameter. The holes are spaced 5.1 cm (2 in.) apart. The pull side of the capture system consists of an exhaust hood installed on the back of the tank. The hood measures 3.6 m (12 ft) in length and 1.8 m (6 ft) in height and contains 3 rows of slots with 15 slots per row. The slots are 25.4 centimeters (cm) (10 in.) in length and 2.54 cm (1 in.) in width. Both sides of the tank are equipped with baffles 1.2 m (4 ft) in length and 1.8 m (6 ft) in height. Removable panels are placed over the top of the tank during plating to enclose the surface of the plating solution to maximize capture efficiency.

Chromium emissions from the tank are vented to a chevron-blade mist eliminator located on a mezzanine structure behind the tank. The mist eliminator contains two sets of chevron blades. Each set changes the direction of gas flow four times at thirty degree angles. The gas flow rate of the system is 170 cubic meters per minute (6,000 actual cubic feet per minute). The pressure drop of the mist eliminator is rated at 0.5 kilopascals (2 in. of water column). A moisture extractor is installed in the stack downstream of the mist eliminator. The moisture extractor consists of a stationary set of blades that centrifugally forces acid mist or droplets entrained in exhaust gas to impinge against the sides of the extractor wall. The droplets drain down the sides of the extractor into collection areas. The moisture extractor was installed at the company's request to control chromium emissions that might be drawn through the mist eliminator. The mist eliminator and moisture extractor are washed down with about 284 liters (ℓ) (75 gallons [gal]) of water at the end of each work day, and at the beginning of the work day if the tank was operated overnight. Washdown water is drained into a 606-ℓ (160-gal) holding tank inside the plating shop. The plating tank is equipped with a float that regulates the flow of makeup water from the holding tank to the plating tank.

5.3 PROCESS CONDITIONS DURING TESTING

Mass emission and particle size distribution tests were conducted at the inlet and outlet of the mist eliminator on the new tank to characterize the uncontrolled emissions from the hard chromium plating tank and the performance of the mist eliminator. The first and second mass emission and particle size distribution runs were conducted concurrently. The third particle size distribution run was conducted after the third mass emission run was completed. The process was operating normally during the tests.

Process operating parameters such as the voltage, current, and plating solution temperature were monitored and recorded during each mass emission test run. Descriptions (dimensions and surface areas) and plating requirements (current and plating time) of each individual part plated also were recorded for each test run. Process data sheets documenting the process and control device operating parameters during mass emission testing (test run Nos. MEI-1 through 3 and MEO-1 through 3) are presented in Appendix H. Data on the average operating parameters recorded during the mass emission test runs are presented in Table 5-1. The pressure drop across the mist eliminator was not monitored; however, there were no indications of any malfunctions in the mist eliminator or capture system during testing.

Grab samples were taken from the tank to determine the chromic acid concentration of the plating solution during each mass emission run. Grab samples of the mist eliminator and moisture extractor washdown water also were taken at the end of the day. The mist eliminator and moisture extractor were washed down with about 318 ℓ (84 gal) of water after the first mass emission test run and with about 254 ℓ (67 gal) of water after the third mass emission test run. The chromic acid concentration of the grab samples is reported in Section 2.4 of this report.

Test run Nos. 1, 2, and 3 were each interrupted for approximately 45 minutes to unload and reload the tank.

The total amount of current supplied to the tank during each test run is calculated in terms of ampere-hours and included in Appendix H. A tabular summary of the total current values is presented in Table 5-2.

TABLE 5-1. AVERAGE OPERATING PARAMETERS FOR THREE MASS EMISSION SOURCE TEST RUNS

Test Run No. Inlet/Outlet	Operating voltage, volts	Operating current, amperes	Temperature of plating solution, °C (°F)
MEI-1/MEO-1	7.5	8,579	52 (125)
MEI-2/MEO-2	7.1	9,527	52 (125)
MEI-3/MEO-3	7.5	7,054	52 (126)

TABLE 5-2. TOTAL CURRENT SUPPLIED TO THE TANK DURING THREE MASS EMISSION SOURCE TEST RUNS

Test Run No. Inlet/Outlet	Total current, ampere-hours	
	Inlet	Outlet
MEI-1/MEO-1	25,790	24,367
MEI-2/MEO-2	18,717	18,773
MEI-3/MEO-3	16,868	13,771