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AP-42 Section 12,20  
Reference  
Report Sect. 4  
Reference 71

NOV 4 1992

Air Quality Section  
November 2, 1992

M E M O R A N D U M

TO: Mike Aldridge  
THROUGH: Laura S. Butler *LSB*  
FROM: Jerry P. Clayton *JPC*  
SUBJECT: JMC Hi-Tech Metals, Inc.  
Research Triangle Park, North Carolina  
Durham County

The Air Permits Branch (APB) received, as part of an air permit application, the attached stack test for chromium emissions from the U.S. Chrome Corporation facility located in Batavia, NY. As this test was the basis for determining the potential emissions from the above subject source, the APB would like to have the testing reviewed for correctness and emissions verification purposes.

The Department of Commerce has been working with this facility and as a proposed new facility, it also has a high permitting priority with the Air Quality Section. Your review of the testing procedures and data within thirty (30) days (or sooner if possible) will be appreciated.

DIVISION OF ENVIRONMENTAL MANAGEMENT

Air Quality Section

December 10, 1992

MEMORANDUM

To: Jerry Clayton  
From: Shannon Vogel *SMV*  
Subject: JMC Hi-Tech Metals, Inc.  
Research Triangle Park, Durham County, North Carolina  
Total and Hexavalent Chromium Emissions Testing of the  
U.S Chrome Corporation Facility in Batavia, New York  
Performed November 11, 1991 by IEA

The report of the subject testing has been reviewed. The test results are acceptable. The testing was performed to determine the hexavalent chromium emissions from two electrolytic baths.

The average hexavalent chromium emission rate demonstrated by the test results was 0.0063 pound per hour. The average total chromium emission rate demonstrated by the test results was 0.0142 pound per hour.

The de minimus limit for modeling stated in 15A NCAC 2H .0600 is 0.0056 pound of hexavalent chromium per year.

The emission rate for hexavalent and total chromium was calculated per process rate in ampere-hours. The average emission for hexavalent and total chromium was 0.080 and 0.181 milligram per ampere-hour, respectively.

cc: Central File via Lee Daniel  
Mike Aldridge

Revised 12/17/92

without blank subtracted

JMC Hi-Tech Metals, Inc.  
Research Triangle Park  
U.S. Chrome Corporation Facility  
November 11, 1991  
Hexavalent Chromium Results

		1	2	3
	RUN NUMBER			
	RUN DATE			
	RUN START TIME			
	RUN FINISH TIME			
N	NUMBER OF SAMPLE POINTS	16	16	16
Theta	TEST RUN TIME, MINUTES	64	64	64
Dn	NOZZLE DIAMETER, INCHES	0.195	0.195	0.195
An	NOZZLE AREA, SQ. IN.	0.0299	0.0299	0.0299
Cp	PITOT TUBE COEFFICIENT	0.840	0.840	0.840
Y	GAS METER CAL. FACTOR	1.008	1.008	1.008
Pbar	BAROMETRIC PRESSURE, IN. HG	30.00	30.00	30.00
Delta H	AVG. PRESS. DIFFERENTIAL OF ORIFICE METER, IN. H2O	1.395	1.424	1.483
Vm	ACTUAL METERED GAS VOL. CF	39.924	41.373	42.356
TmF	GAS METER TEMP, . DEG. F	85	92	94
Vm(std)	METERED GAS VOLUME AT DRY STANDARD CONDITIONS, DSCF	39.196	40.106	40.910
Vwc	VOLUME OF WATER COLLECTED IN IMPINGERS & DESICCANT, ML	8.5	11.5	9.0
Vwc(std)	VOLUME OF WATER VAPOR, SCF	0.400	0.541	0.424
%M	MOISTURE, % BY VOLUME	1.0	1.3	1.0
fm	MOLE FRACTION OF DRY GAS	0.990	0.987	0.990
%CO2	CO2, % BY VOLUME, DRY			
%O2	O2, % BY VOLUME, DRY	20.9	20.9	20.9
%CO	CO, % BY VOLUME, DRY			
%N2	N2, % BY VOLUME, DRY	79.1	79.1	79.1
Md	DRY MOLECULAR WT., #/#-MOLE	28.84	28.84	28.84

JMC Hi-Tech Metals, Inc.  
 Research Triangle Park  
 U.S. Chrome Corporation Facility

November 11, 1991  
 Hexavalent Chromium Results

	RUN NUMBER	1	2	3
Ms	WET MOLECULAR WT., #/#-MOLE	28.73	28.69	28.72
Pstatic	GAS STATIC PRESS, . IN. H2O	-5.60	-5.60	-5.60
Ps	ABSOLUTE GAS PRESS, IN. HG.	29.59	29.59	29.59
TsF	STACK GAS TEMP., DEG. F	80	84	94
SSR DP	SUM OF SQUARE ROOTS OF VELOCITY PRESS. VALUES	14.9587	14.7011	14.9715
vs	FLUE GAS VELOCITY, FT/SEC	53.5	52.8	54.3
Ds	DUCT DIAMETER, INCHES	31.3	31.3	31.3
As	DUCT AREA, SQUARE INCHES	767.0	767.0	767.0
AsF	DUCT AREA, SQUARE FEET	5.326	5.326	5.326
Qs	GAS FLOW RATE, WET ACFM	17106	16875	17340
Qstd	GAS FLOW RATE, DRY SCFM	16355	15980	16160
mn	SAMPLE WEIGHT, GRAMS	1.69E-04	6.66E-05	1.19E-04
cs	PART. CONCENTRATION GR/DSCF	6.65E-05	2.56E-05	4.49E-05
	PART. CONCENTRATION ug/DSCF	4.31	1.66	2.91
pmrc	POLLUTANT MASS RATE CALC. FROM CONCENTRATION, #/HR	9.33E-03	3.51E-03	6.22E-03
pmra	POLLUTANT MASS RATE CALC. FROM AREA RATIO, #/HR	8.97E-03	3.54E-03	6.32E-03
%I	% ISOKINETIC	96.2	100.7	101.6
pmr avg	AVERAGE OF pmra & pmrc	0.01	0.00	0.01
%EA	% EXCESS AIR	-118750.0	-118750.0	-118750.0
F	"F" FACTOR, CU FT/MIL BTU			



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**REPORT NO.:** 192-91-75

**PROJECT:** 1331-001

**DATE:** November 11, 1991

**TYPE:** **EMISSION REPORT**

**EMISSION TEST RESULTS  
FOR CHROMIUM EMISSION RATE  
OF THE SCRUBBER INLET  
AT THE U.S. CHROME CORPORATION FACILITY  
BATAVIA, NEW YORK**

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STATIONARY SOURCES (METHOD Cr<sup>+6</sup>)"

## 1. INTRODUCTION

Chromium emission testing was conducted on the inlet of the packed bed scrubber at the U.S. Chrome Corporation facility located in Batavia, New York. Testing was performed 02 October 1991, under the direction of Mr. Jim Pendergast of U.S. Chrome Corporation. Sampling was performed by Barry Rayfield and Chris Johnson of Industrial and Environmental Analysts, Inc. (IEA) of Research Triangle Park, North Carolina. Mr. Kenichi Sakamachi of JMC Hi-Tech Metals, Inc. was on-site to observe testing. Jeff Burdette was the IEA project manager. All chromium analyses were performed by Research Triangle Institute.

The purpose of testing was to determine the hexavalent and total chromium emissions being emitted from two electrolytic baths. A total of three 64-minute sampling runs were completed.

The remainder of this report is comprised of three sections. A summary of the emission parameters is presented in Section 2. A discussion of the sampling and analytical procedures is presented in Section 3. Quality Assurance and Control procedures are itemized in Section 4. All supporting raw data is included in the attached Appendices.

## 2. SUMMARY OF RESULTS

### 2.1 General Summary

Table 2.1 presents a summary of the average emissions measured during the test program conducted on the inlet of the packed bed scrubber. All mass emissions are based on volumetric flow rates that were corrected to standard temperature and pressure (528°R and 29.92 inches Hg). Each individual run is presented along with the measured process rate in ampere-hours. Emissions are presented in pounds per hour (lb/hr), microgram per dry standard cubic foot ( $\mu\text{g}/\text{dscf}$ ), and milligrams per ampere hour (mg/ampere-hour). The average emission rate for hexavalent chromium was determined to be 0.080 mg/ampere-hour. The average emission rate for total chromium was 0.181 mg/ampere-hour.

Example calculations are presented in Appendix A. Field and recovery data is included in Appendix B. Laboratory data and chain-of-custody documentation are presented in Appendix C. Process rate data is presented in Appendix D. Equipment calibrations and certifications are presented in Appendix E. Project participants and project summary logs are presented in Appendix F. A copy of the test methodology used is included in Appendix G.

### 2.2 Chromium Summary

Table 2.2 presents a summary of the individual chromium runs. The average hexavalent chromium concentration was determined to be 2.92  $\mu\text{g}/\text{dscf}$  resulting in an average mass emission rate of 0.0063 pounds per hour (lb/hr).

The analysis of the potassium hydroxide solution for total chromium yielded values smaller than those obtained for hexavalent chromium. During the sample digestion, an uncharacteristic, gelatinous residue was observed. This residue was most likely caused by a matrix interference from the process. It is possible that the remaining total chromium component was bound in this residue. IEA used the hexavalent chromium concentration observed in this fraction as the total chromium portion.

Total chromium emission rates were based on the sum of the hexavalent fraction analyzed by ion chromatography (IC) and the total chromium fraction analyzed by atomic absorption (GFAAS). The average total chromium concentration was determined to be 6.58  $\mu\text{g}/\text{dscf}$  resulting in an average emission rate of 0.0142 lb/hr.

**TABLE 2.1  
PACKED-BED SCRUBBER INLET  
SUMMARY OF CHROMIUM EMISSIONS**

<b>RUN</b>	<b>EMISSION RATE</b>	<b>PROCESS RATE (ampere-hour/run)</b>	<b>HEXAVALENT CHROMIUM</b>	<b>TOTAL CHROMIUM</b>
Run 1	Chromium	38,074		
	Concentration, ug/dscf		4.27	5.10
	Emission Rate, lb/hr		0.0092	0.0110
	Emission Rate, mg/ampere-hour		0.117	0.140
Run 2	Chromium	37,554		
	Concentration, ug/dscf		1.62	1.97
	Emission Rate, lb/hr		0.0034	0.0042
	Emission Rate, mg/ampere-hour		0.044	0.054
Run 3	Chromium	37,749		
	Concentration, ug/dscf		2.87	12.67
	Emission Rate, lb/hr		0.0062	0.0273
	Emission Rate, mg/ampere-hour		0.079	0.350
Average	Chromium	37,792		
	Concentration, ug/dscf		2.92	6.58
	Emission Rate, lb/hr		0.0063	0.0142
	Emission Rate, mg/ampere-hour		0.080	0.181

**TABLE 2.2  
SCRUBBER INLET  
CHROMIUM SUMMARY**

SYMBOL	PARAMETER	RUN 1	RUN 2	RUN 3	AVERAGE
		10/2/91 0925-1030	10/2/91 1138-1242	10/2/91 1257-1681	
<b>MEASURED DATA</b>					
(Y)	Meter Box Y	1.008	1.008	1.008	1.008
	Avg Delta H, inches H2O	1.39	1.42	1.48	1.43
(Bp)	Barometric Pressure, inches Hg	30.00	30.00	30.00	30.00
(Vm)	Meter Volume, ft <sup>3</sup>	39.924	41.373	42.356	41.218
(Tm)	Avg Meter Temp, deg F	85.2	92.2	94.3	90.6
(Pg)	Static Pressure, inches H2O	-5.6	-5.6	-5.6	-5.6
(Ts)	Avg Stack Temp, deg F	80.4	83.8	84.6	82.9
(Vlc)	Water Collected, ml	8.5	11.5	9.0	9.7
(CO2)	CO2, %	0.0	0.0	0.0	0.0
(O2)	O2, %	20.9	20.9	20.9	20.9
	Avg Sqrt Delta P, (inches H2O) <sup>1/2</sup>	0.935	0.919	0.936	0.93
(t)	Sample Time, min	64	64	64	64
(Dn)	Nozzle Diameter, inches	0.195	0.195	0.195	0.195
(Ds)	Stack Diameter, inches	31.25	31.25	31.25	31.25
(Mcr6)	Hexavalent Chromium, ug	167.2	64.8	117.2	116.4
(Mcr)	Total Chromium (filter), ug	0.320	0.000	0.045	0.122
(Mcr)	Total Chromium (HNO3), ug	32.3	14.0	400.9	149.1
<b>CALCULATED DATA</b>					
(Vmstd)	Standard Meter Volume, ft <sup>3</sup>	39.195	40.106	40.910	40.070
(Ps)	Stack Pressure, inches Hg	29.59	29.59	29.59	29.59
(%Bws)	Moisture, %	1.0	1.3	1.0	1.1
(Ms)	Molecular Weight-wet, lb/lb-mole	28.7	28.7	28.7	28.7
(Vs)	Velocity, ft/s	53.5	52.8	53.8	53.4
(As)	Stack Area, ft <sup>2</sup>	5.33	5.33	5.33	5.33
(Qa)	Volumetric flow, acfm	1.71E+04	1.69E+04	1.72E+04	1.71E+04
(Qs)	Volumetric flow, dscfm	1.64E+04	1.60E+04	1.63E+04	1.62E+04
(I)	Isokinetic Rate, %	96.2	100.7	100.7	99.2
(Ccr)	Hex Chromium Concentration, ug/dscf	4.27	1.62	2.87	2.92
(Ccr)	Hex Chromium Concentration, mg/dscm	0.15	0.06	0.10	0.10
(ER)	Hex Chromium Emission Rate, lb/hr	0.0092	0.0034	0.0062	0.0063
(Tcr)	Total Chromium Conc, ug/dscf	5.10	1.97	12.67	6.58
(Tcr)	Total Chromium Conc, mg/dscm	0.18	0.07	0.45	0.23
(ER)	Total Chromium Emission Rate, lb/hr	0.0110	0.0042	0.0273	0.0142

### 3. SAMPLING AND ANALYTICAL PROCEDURES

All sampling and analytical procedures used in this test are contained in 40 CFR Part 60, Appendix A (7-1-89 Edition). The hexavalent chromium sampling and analytical procedure used in this test is contained in FR Vol. 56, No. 137, pp. 32719-32728, July 19, 1991 and is entitled "Determination of Hexavalent Chromium from Stationary Sources (Method Cr<sup>+6</sup>)." Table 3.1 presents a summary of the procedures used for the test program.

#### 3.1 Sample Collection

Due to the nature of the obstructions surrounding the inlet duct, no accessible port location met all EPA Method 1 (Sample and Velocity Traverses) criteria. A sample location was chosen that minimized the flow disturbances. EPA Method 1 procedures were used as a guideline to determine the number of sample points and sampling point locations.

A preliminary velocity traverse was used to set up parameters for the first isokinetic test run. EPA Method 2 (Stack Gas Velocity and Volumetric Flow Rate) procedures were followed during each chromium test run to measure stack gas velocity head or  $\Delta p$ . A Type-S pitot tube was used in conjunction with an inclined oil gauge manometer to measure the individual  $\Delta p$ 's at each sample point.

The dry gas/molecular weight and O<sub>2</sub> concentrations for the were determined according to EPA Method 3 procedures (Gas Analysis for CO<sub>2</sub>, O<sub>2</sub>, Excess Air, and Dry Molecular Weight). Fyrite analysis was used to insure stack gas concentrations were at ambient levels.

Stack gas moisture content was measured using EPA Method 4 (Moisture Content of Stack Gases) procedures as part of the chromium test runs.

All portions of the train in contact with the sample gas were either Teflon or glass. All sample train components and sample containers were precleaned to minimize the potential for contamination. The cleaning sequence features hot soap and water, followed by 10% nitric acid soaking and a final rinse with deionized water. All exposed openings were sealed for transport.

The sample train featured a glass nozzle with a sharp, tapered leading edge. The nozzle was connected to a 3-way Teflon union. This union is the point where the absorbing solution was introduced to the gas sample. The vacuum operation of the sample train pulls sample gas and absorbing solution into the first of a series of four Teflon impingers. The first impinger contained 150 mL of 0.5 N potassium hydroxide (KOH). The bottom of the first impinger has an outlet that feeds a peristaltic pump. The peristaltic pump forces solution back to the Teflon union at the exit to the probe tip. The second and third impingers each contained 75 mL of KOH. The fourth impinger is empty. The final impinger was glass and contained a pre-weighed amount of silica gel to remove any residual moisture. The sample gas exits the silica gel impinger through connecting tubing to a sample metering system that monitored gas stream flow rate and temperature, sample gas volume, and other critical operational parameters.

TABLE 3.1  
SUMMARY OF TEST PROCEDURES

PARAMETER	SAMPLING METHOD	ANALYTICAL INSTRUMENT
Volumetric Flow	EPA 40 CFR Part 60 App A Method 2	Pitot Tube/Inclined Manometer
Oxygen	EPA 40 CFR Part 60 App A Method 3	Fyrite Analyzer
Carbon Dioxide	EPA 40 CFR Part 60 App A Method 3	Fyrite Analyzer
Moisture	EPA 40 CFR Part 60 App A Method 4	Analytical Balance (+/- 0.1 g)
Hexavalent Chromium	EPA Method 0013	Ion Chromatography / Post Column Reaction
Chromium	EPA Method 0013	Atomic Absorption / Graphite Furnace (GFAAS)

A diagram of the sample train is included in Figure 3.1. Initial and final leak rates, recovery data, and all train operating parameters for each test run can be found in the field data sheets in Appendix B.

### 3.2 Recovery Procedures

Upon completion of the sample run for  $\text{Cr}^{+6}$ , the train was carefully removed from the stack, leak-checked, and moved to the clean-up area.

The pH of the first impinger was tested to assure it was greater than 8.5. The sample train was then purged with nitrogen to safeguard against the conversion of hexavalent chromium to trivalent oxidation state. The sample was purged using the apparatus in Figure 3.2. The silica gel impinger was separated and weighed to document the weight gain.

The sample train absorbing solution was drained into a precleaned graduated cylinder and measured to document the volume within 1 mL. The contents were then transferred to a precleaned sample container. The entire sample train was rinsed four times with deionized water. The rinses were transferred to the same sample container. The entire sample train assembly was then rinsed three times with 0.1 N  $\text{HNO}_3$ . This rinse was transferred to a separate sample container. Prior to sampling again, the entire sample train was rinsed a final time with DI water. This rinse was discarded.

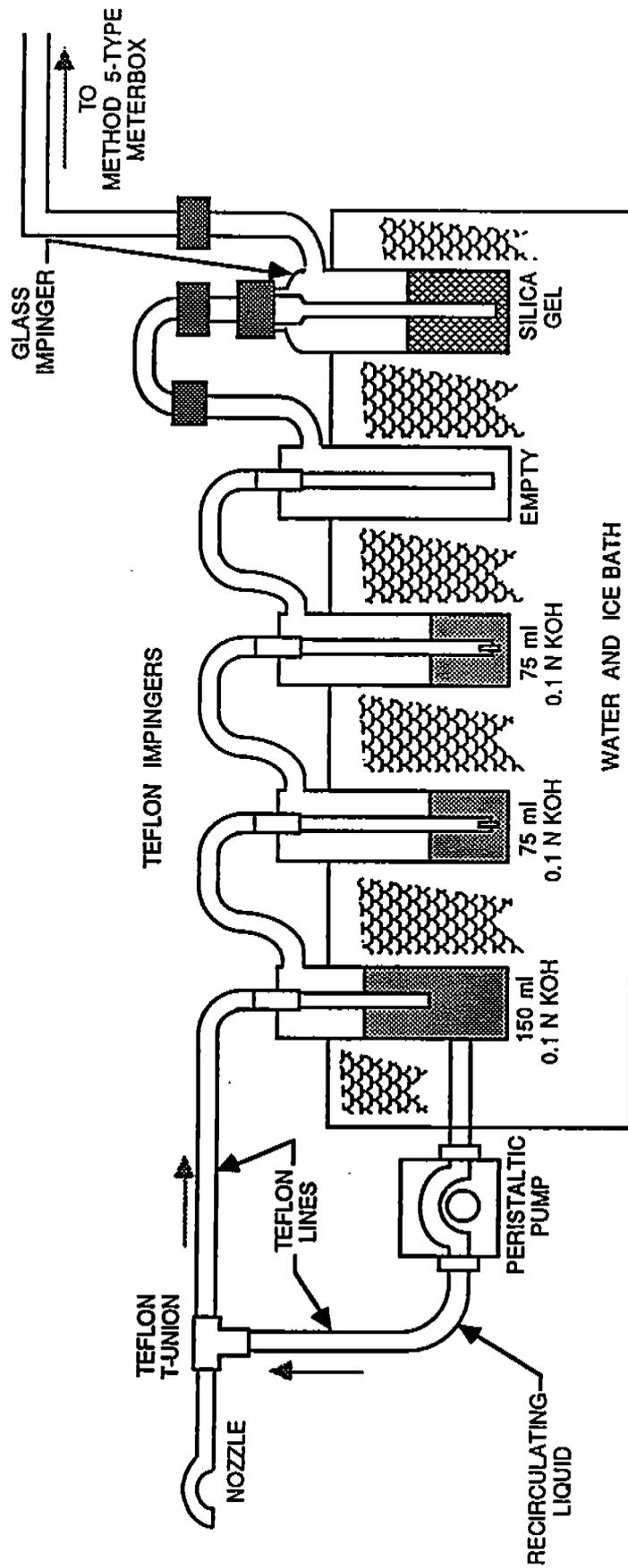
The KOH sample was filtered with a 0.45 micron acetate filter immediately following recovery to remove any insoluble particulate matter. Nitrogen was used to pressure assist the filtration process. The apparatus in Figure 3.3 was used to filter the sample. The filtrate was collected in a 1000 mL graduated cylinder. The sample container and teflon reservoir were rinsed three times with DI water, combined with the filtrate, and measured to document the volume for shipment. The sample was then transferred back to the sample container. The filter and residue were recovered and placed in a vial for analysis of total chromium. All samples were labeled appropriately and packed for transport to the laboratory. A schematic of the recovery process is provided in Figure 3.4.

### 3.3 Analytical Methodology

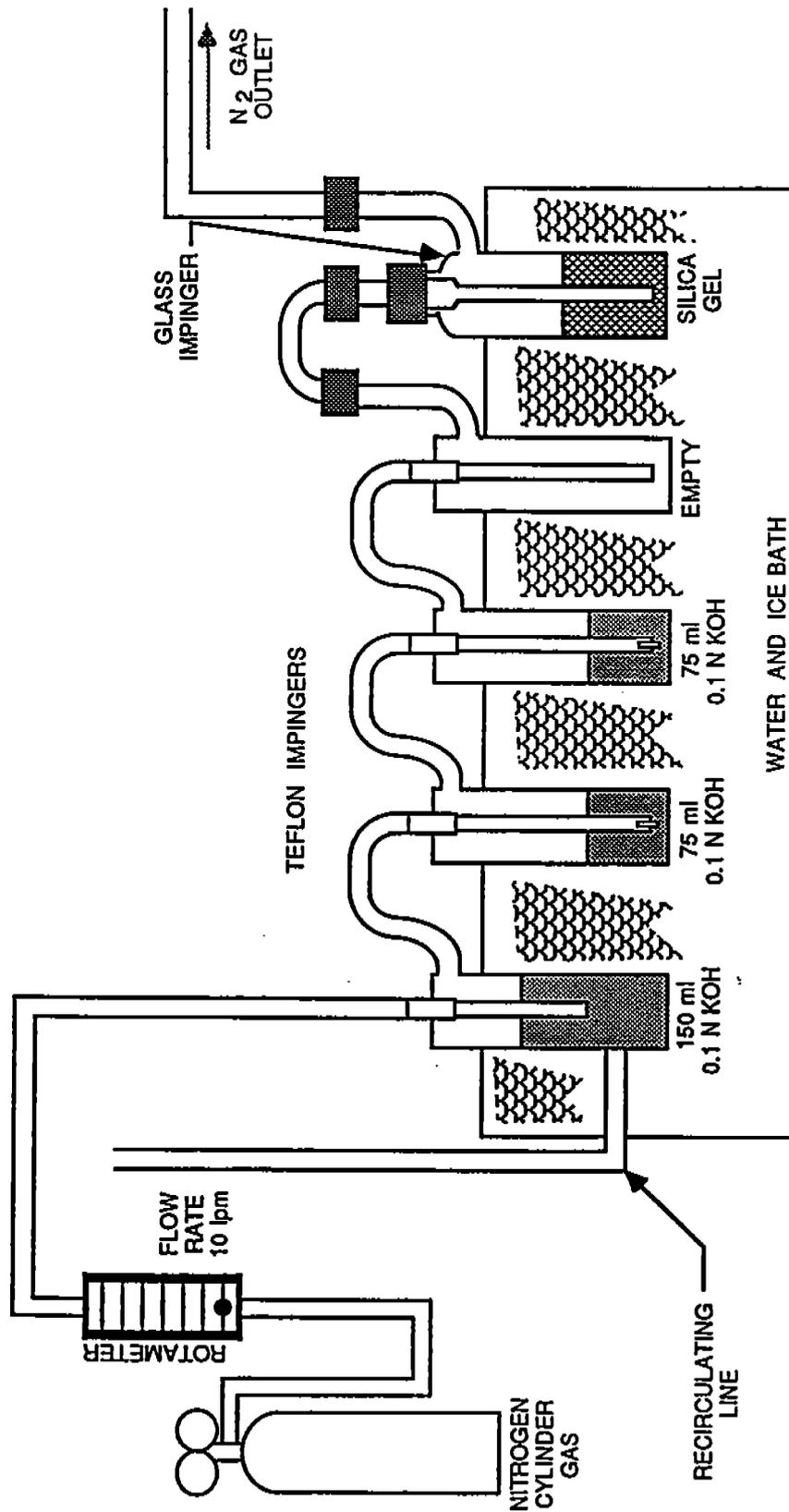
Recovered stack gas samples were submitted to Research Triangle Institute located in Research Triangle Park, NC, for analysis of hexavalent chromium ( $\text{Cr}^{+6}$ ) and total chromium.

#### 3.3.1 Hexavalent Chromium

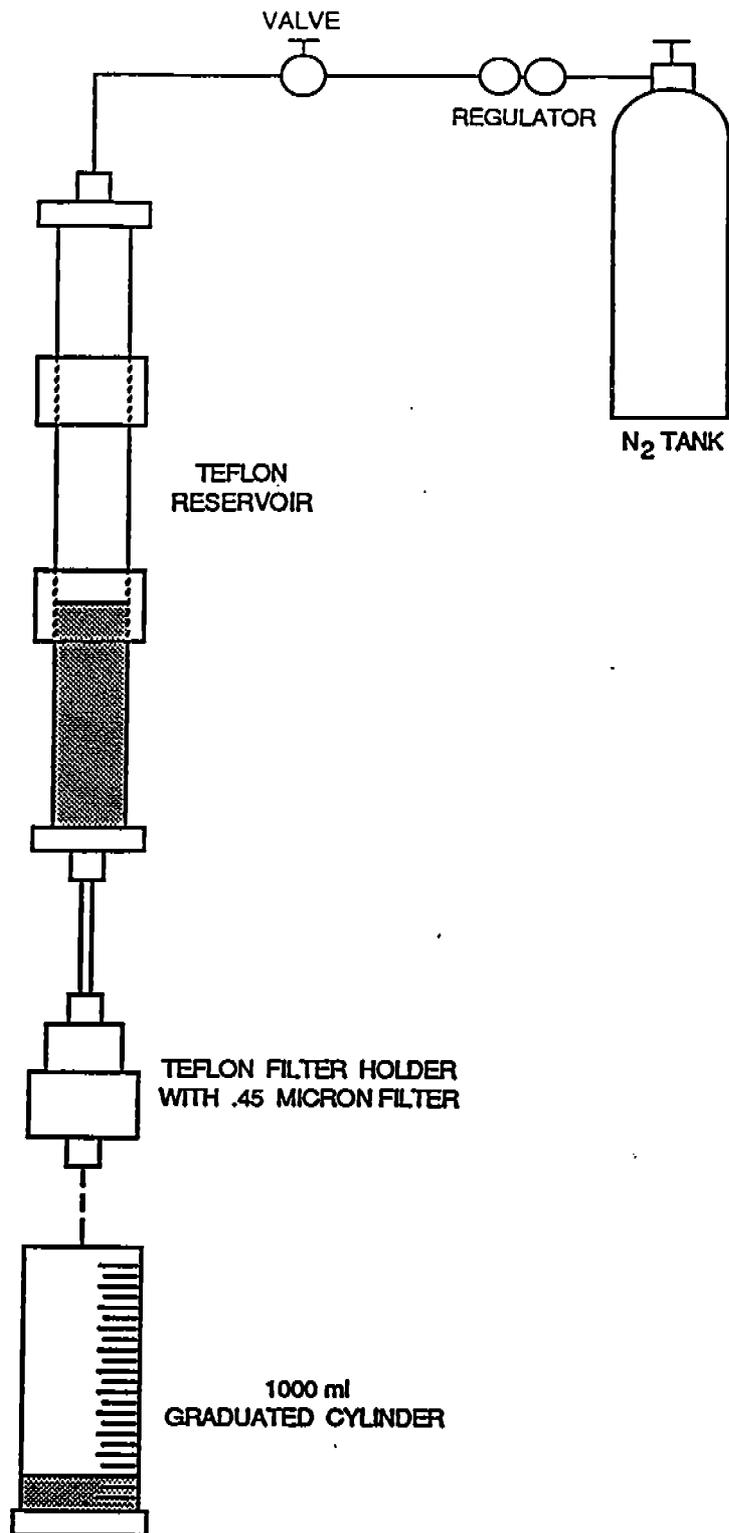
The  $\text{Cr}^{+6}$  content of the liquid sample was determined by ion chromatography coupled with a post column reactor (IC/PCR). To increase sensitivity for trace levels of chromium, a preconcentration system was also used in conjunction with the IC/PCR. Prior to sample injection for analysis, all samples were filtered through a 0.45-um filter. The preconcentration was accomplished by selectively retaining the analyte on a HPIC non-metallic column, followed



**FIGURE 3.1**  
**SCHEMATIC OF RECIRCULATORY IMPINGER TRAIN WITH PUMP SPRAYER ASSEMBLY**



**FIGURE 3.2**  
**SCHEMATIC OF POST TEST NITROGEN PURGE SYSTEM**



**FIGURE 3.3**  
**SCHEMATIC OF SAMPLE FILTER SYSTEM**

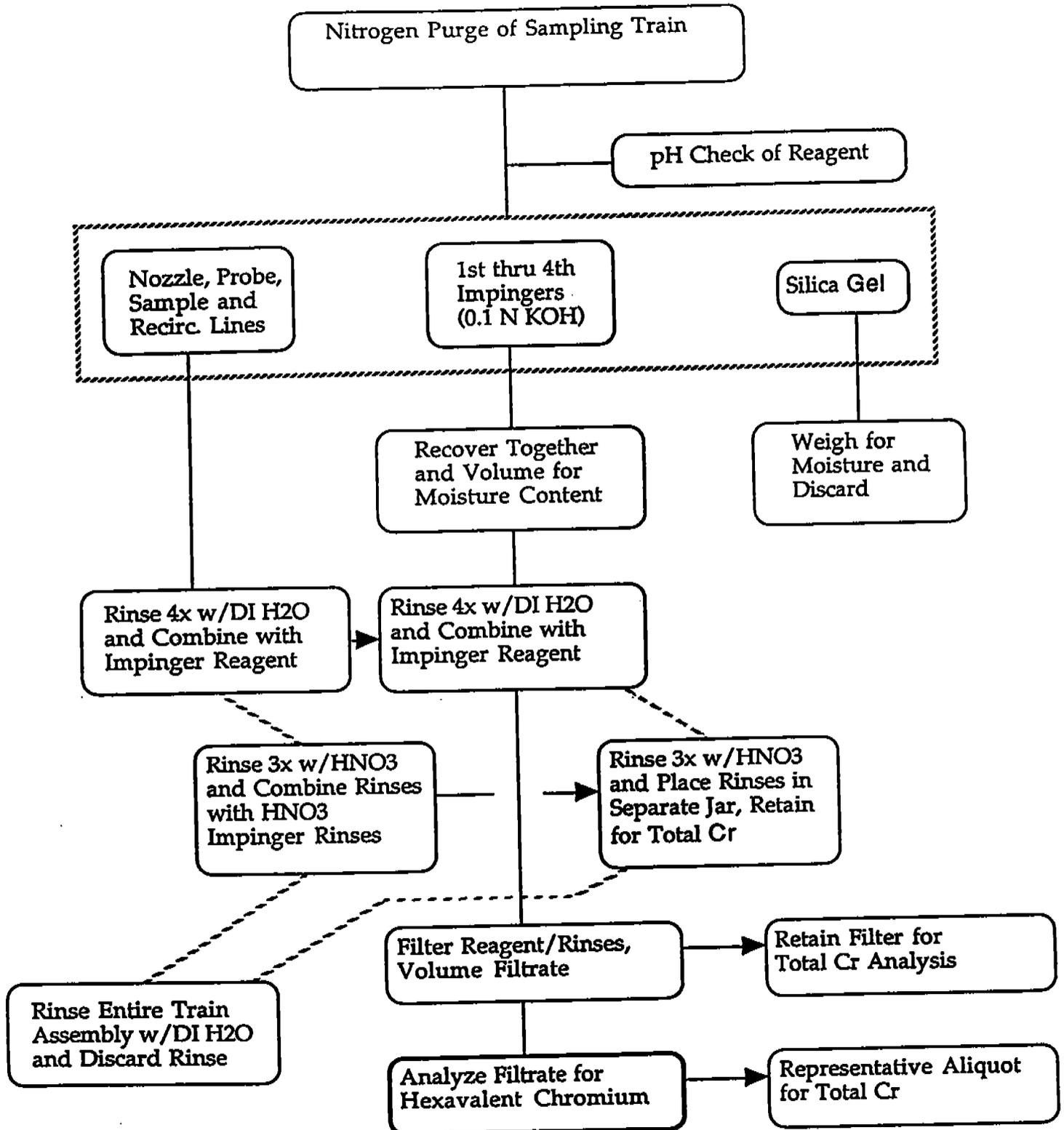


FIGURE 3.4  
Cr<sup>+6</sup> METHOD RECOVERY PROCEDURE

by removal of the analyte from the absorbent. The sample was injected into a sample loop and the  $\text{Cr}^{+6}$  was collected on the resin bed of the column. The injection valve was switched, and the eluent displaces the concentrated  $\text{Cr}^{+6}$  sample moving it off the preconcentration column and onto the IC anion separation column and onto the IC anion separation column. After separation from other sample components,  $\text{Cr}^{+6}$  forms a specific complex in the PCR with a diphenylcarbazide reaction solution, and the complex was then detected by visible absorbance at a wavelength of 520 nm. The amount of absorbance measured is proportional to the concentration of the  $\text{Cr}^{+6}$  complex formed. The IC retention time and absorbance of the  $\text{Cr}^{+6}$  complex was compared with known  $\text{Cr}^{+6}$  external calibration standards analyzed under identical conditions to provide both qualitative and quantitative analyses. The analytical results are presented in Appendix C.

### 3.3.2 Total Chromium

The nitric acid rinse and filter were analyzed for total chromium. An aliquot of acid rinse and the entire filter sample were digested and analyzed for total chromium by atomic absorption using graphite furnace. A fraction of the liquid sample originally subjected to hexavalent chromium analysis (potassium hydroxide solution) was also retained to determine total chromium.

#### 4. QA/QC PROCEDURES AND RESULTS

The objective of a quality assurance/quality control (QA/QC) program is to assure that the precision and accuracy of all environmental data generated by IEA, Inc. are commensurate with data quality objectives (DQOs). DQOs are based on a common understanding of the intended end use(s) of the data, the measurement process, and the availability of resources. Once DQOs are established, formally or informally, QC protocol can be defined for the measurements.

The data quality objectives in this project are to provide defensible data that can be used for determination of emission rates suitable demonstrate compliance. The final data user will be JMC Hi-Tech Metals, Inc.

The goal of a QA/QC program is that data generated and used for decision-making are scientifically sound, of known quality, and documented to be "in control." To accomplish this goal, standardized methods or procedures are used whenever possible. They must be validated for their intended use, rigorously followed, and data reported with quality indicators (precision, accuracy, completeness, etc.).

Two basic concepts used in a QC program are to:

1. Control errors; and
2. Verify that the entire Sampling and Analytical (S&A) method is operating within acceptable performance limits.

Use of qualified personnel, reliable and well-maintained equipment, appropriate calibrations and standards, and close supervision of all operations are important components of the QC system. QC in this test program included the use and documentation of calibrated sampling and analytical instruments, use of EPA validated methods (EPA 40 CFR Part 60), adherence to established protocol, method blanks as a check against possible contamination, sample chain-of-custody documentation, and redundant data calculation.

All of the equipment used was calibrated according to procedures outlined in the Quality Assurance Handbook for Air Pollution Measurement System, Volume III, EPA-600/4-77-027b. Actual calibration data sheets are provided in Appendix E.

##### 4.1 Barometer

Barometric pressure values for the testing period were recorded from a calibrated barometer on-site and verified by phone from a local airport and were corrected for elevation to stack sample port level (0.01 inches Hg per 10 ft. elevation).

## 4.2 Probe Nozzle

The probe nozzles used in this test were calibrated initially by the manufacturer and thereafter by the field sampling crew by checking for dimension roundness. This was done by making three separate measurements using alternative inside diameters and calculating the average. A micrometer with a minimum tolerance of 0.001 inch was used for measuring. If a deviation of more than 0.004 inch is found between any measurements, the nozzle is either discarded or repaired and remeasured.

## 4.3 Pitot Tubes

Each pitot tube used in sampling meets the design specifications for type-S pitot tubes in EPA Method 2. Therefore in accordance with Method 2 procedures, a baseline coefficient ( $C_p$ ) of 0.84 is assigned to each pitot tube. Calibration at the manufacturer for pitot face-opening alignment included measuring the external tubing diameter (dimension  $D$ ), the base-to-opening plane distance (dimensions  $P_a$  and  $P_b$ ), and the face opening misalignment angles, with all terms as described in Figures 2-2 and 2-3 of EPA Method 2. Pitot tubes were visually inspected at the completion of the test to insure structural integrity. Pitot tube inspection sheets are presented in Appendix E.

## 4.4 Calibration Meter and Metering System

The secondary reference meter equipment arrangement for calibration is shown in Figure 5.7 of EPA Method 5. The prescribed procedures were followed. A wet test meter with a 1 ft<sup>3</sup>/rev capacity and  $\pm 1$  percent accuracy is used as the primary calibrant. The dry gas meter's pump is operated for a minimum of 5 minutes at a flow rate of 0.35 cfm to condition the interior surface of the wet test meter. Leak checks are performed and if satisfactory, triplicate runs at each of no less than three different flow rates are performed. A calibration curve is prepared and the meter is recalibrated after 200 hours of operation or annually, whichever comes first.

The calibration set-up for the dry gas metering system using the secondary reference meter in lieu of the wet test meter is given in Figure 5.5 of EPA Method 5. A leak check of the metering system before calibration was performed as shown in Figure 5.4 of EPA Method 5. The metering system's pump is operated for 5 minutes at an orifice manometer setting of 0.5 inches H<sub>2</sub>O to heat up the pump and system to stabilize the meter inlet and outlet temperatures. Values for the orifice setting ( $\Delta H$ ), wet test meter volume ( $V_w$ ), corresponding dry test meter volume ( $V_d$ ), dry test meter inlet and outlet gas temperatures ( $t_{di}$  and  $t_{do}$ ), and time are recorded for the initial calibration. The ratio of the wet test meter to the dry test meter ( $\gamma$ ) and the orifice pressure differential that equates to 0.75 cfm at standard conditions ( $\Delta H@$ ) are then calculated. A copy of the calibration is in Appendix E.

#### 4.5 Post-Test Meter Calibration Check

Post-test meter calibrations were made on the dry gas meters used during the test to check their accuracy against the original pre-test calibration. This post-test calibration was made using the average orifice setting obtained during each test run and setting the vacuum at the maximum value obtained during each test run. These test runs were made against IEA's secondary reference dry gas meter which was calibrated against a wet test meter. A copy of the calibration is in Appendix E.

#### 4.6 Thermocouples and Digital Indicators

Thermocouples are calibrated by comparing them against an ASTM-3F mercury-in-glass thermometer at approximately 32° F (ice water), ambient temperature and approximately 100° F (hot oil). Each thermocouple is calibrated against temperature ranges to which it is typically exposed during test conditions, and they must agree within 1.5 percent (expressed in °R) of the reference thermometer throughout the entire calibration range.

Digital indicators are checked by introducing a series of millivolt signal strengths to the input and comparing the indicator reading with the actual signal strength. Acceptable calibration error must not exceed 0.5 percent when temperatures are expressed in °R. Copies of the calibrations are presented in Appendix E.

#### 4.7 Blanks

Solution and filter blanks were provided for analysis as a check against potential sample contamination. All of these were within the tolerance allowed by the method. The results were blank corrected according to the procedures outlined in the method.

#### 4.8 Spikes, Duplicates, and QC Samples

The spike recovery for hexavalent chromium was 97%. Duplicates of the audit samples for total chromium demonstrated 99.4% and 105% recovery. The QC samples that were analyzed as a calibration check demonstrated 93% of the expected value for hexavalent chromium and 105% of the expected value for total chromium. Results of these audit procedures are included in Appendix C.

#### 4.9 Sensitivity and Precision

A minimum detection limit of 8 ng/dscm with a 3 dscm gas sample can be achieved by preconcentration (0.05 ppb in solution).

The precision of the IC/PCR with sample preconcentration is 5 to 10 percent. The overall precision for sewage sludge incinerators emitting 120 ng/dscm of Cr<sup>+6</sup> and 3.5 µg/dscm of total chromium is 25% and 9% for Cr<sup>+6</sup> and total chromium, respectively; for hazardous waste incinerators emitting 300 ng/dscm of Cr<sup>+6</sup> it is 20 percent.

#### 4.10 Interferences

Components in the sample matrix may cause  $\text{Cr}^{+6}$  to convert to trivalent chromium ( $\text{Cr}^{+3}$ ) or cause  $\text{Cr}^{+3}$  to convert to  $\text{Cr}^{+6}$ . A post-sampling nitrogen purge and sample filtration are included to eliminate many of these interferences. The chromatographic separation of  $\text{Cr}^{+6}$  using ion chromatography reduces the potential for other metals to interfere with the post-column reaction. For the IC/PCR analysis, only compounds that coelute with  $\text{Cr}^{+6}$  and affect the diphenylcarbazide reaction will cause interference. Periodic analysis of deionized (DI) water blanks is used to demonstrate that the analytical system is essentially free from contamination. Sample cross-contamination that can occur when high-level and low-level samples or standards are analyzed alternately is eliminated by thorough purging of the sample loop. Purging can easily be achieved by increasing the injection volume of the samples to ten times the size of the sample loop.

Total chromium analysis of the impinger solution should have produced concentrations equal or greater than the concentrations observed for hexavalent chromium of the same sample. However, during analysis of the potassium hydroxide sample for total chromium, an uncharacteristic, gelatinous residue was observed. Since the total chromium values were determined to be less than the hexavalent chromium values, it is presumed that some portion of the total chromium could be bound in this residue. Due to the fact that this residue was only seen in the samples and not in the blank, it is indicative of a process related matrix interference. Results from the hexavalent chromium analysis were used to determine the total chromium fraction. It is impossible to determine if any additional total chromium (in excess of the hexavalent chromium concentration) was present in the samples.



APPENDIX A  
EQUATIONS AND EXAMPLE CALCULATIONS



EPA METHOD 5 CALCULATIONS

Plant US CHEMIE Location Sampled SCRUBBER INLET

Date 10/31/91 Run No. 1 Calc By S. BURETTE

Dry gas meter calibration factor = Y = 1.008

Volume of gas at meter conditions (ft<sup>3</sup>) = V<sub>m</sub> = 39.924

Average meter temperature (°F + 460)°R = T<sub>m</sub> = 85.2 + 460 = 545.2

Barometric pressure absolute (in. Hg) = P<sub>bar</sub> = 30.00

Average orifice pressure drop (in. H<sub>2</sub>O) = ΔH = 1.39

V<sub>m(std)</sub> = Volume of gas sampled at the dry gas meter corrected to standard conditions (dscf)

$$V_{m(std)} = \frac{Y (17.64)(V_m)(P_{bar} + (\Delta H/13.6))}{(T_m)}$$

$$V_{m(std)} = \frac{(1.008)(17.64)(39.924)[(30.00) + (1.39)/13.6]}{(545.2)}$$

$$V_{m(std)} = \underline{39.195} \text{ dscf}$$

Total volume of water collected (mL) = V<sub>lc</sub> = 8.5

V<sub>wc(std)</sub> = volume of water vapor at standard conditions (scf)

$$V_{wc(std)} = (0.04707)(V_{lc}) = (0.04707)(8.5) = \underline{0.40}$$

Mole fraction of water vapor = B<sub>ws</sub>

$$B_{ws} = \frac{V_{wc(std)}}{V_{m(std)} + V_{wc(std)}} = \frac{(0.40)}{(39.195) + (0.40)}$$

$$B_{ws} = \underline{0.010}$$

Percent moisture = (100)(B<sub>ws</sub>) = 100 (0.010)

Percent moisture = 1.0 %

Mole fraction of dry gas = MF = 1 - (B<sub>ws</sub>) = 1 - (0.010)

$$MF = \underline{0.99}$$

$$\begin{aligned} \text{Percent O}_2 \text{ by volume dry basis} &= \text{O}_2 = \frac{20.9}{\phantom{0}} \% \\ \text{Percent CO by volume dry basis} &= \text{CO} = \frac{\phantom{0}}{\phantom{0}} \% \\ \text{Percent CO}_2 \text{ by volume dry basis} &= \text{CO}_2 = \frac{0}{\phantom{0}} \% \end{aligned}$$

$$\text{Percent N}_2 \text{ by volume dry basis} = 100 - (\text{O}_2 + \text{CO} + \text{CO}_2)$$

$$\text{N}_2 = 100 - [(20.9) + (0) + (0)]$$

$$\text{N}_2 = \underline{79.1} \%$$

$$\text{Molecular weight of dry gas (lb/lb-mole)} = M_d$$

$$M_d = 0.44 (\text{CO}_2) + 0.32 (\text{O}_2) + 0.28 (\text{N}_2 + \text{CO})$$

$$M_d = 0.44 (0) + 0.32 (20.9) + 0.28 (79.1 + 0.0)$$

$$M_d = \underline{28.8A} \text{ lb/lb-mole}$$

$$\text{Molecular weight of wet stack gas (lb/lb-mole)} = M_s$$

$$M_s = (M_d) (1 - B_{ws}) + 18 (B_{ws})$$

$$M_s = (28.8A) [1 - (0.01)] + 18 (0.01)$$

$$M_s = \underline{28.7} \text{ lb/lb-mole}$$

$$\text{Percent excess air (\%)} = \% \text{ EA}$$

$$\% \text{ EA} = \frac{(\% \text{ O}_2) - [0.5 (\% \text{ CO})]}{[0.264 (\% \text{ N}_2)] - (\% \text{ O}_2) - [0.5 (\% \text{ CO})]} \times 100$$

$$\% \text{ EA} = \frac{(\phantom{0}) - [0.5 (\phantom{0})]}{[0.264 (\phantom{0})] - (\phantom{0}) - [0.5 (\phantom{0})]} \times 100$$

$$\% \text{ EA} = \underline{\phantom{0}}$$

$$\text{Average stack temperature (}^\circ\text{F}+460) = T_s = \underline{80.4+460 = 540.4}$$

$$\text{Avg of sq root of velocity head (in H}_2\text{O)}^{1/2} = \sqrt{\Delta p} = \underline{0.935}$$

$$\begin{aligned} \text{Absolute pressure of stack gas (in. Hg)} &= P_s = \underline{30.00 + \frac{-5.6}{13.6} = 29.59} \\ \text{note } P_s &= \text{barometric pressure (in. Hg)} \pm \\ &[\text{gauge pressure (in. H}_2\text{O)} / 13.6] \end{aligned}$$

$$\text{Pitot tube coefficient, type S} = c_p = \underline{0.8A}$$

$V_s$  = average stack gas velocity (ft/s)

$$V_s = 85.49 \times C_p \times \sqrt{\frac{T_s}{P_s \times M_s}} \times (\sqrt{\Delta p})_{avg}$$

$$V_s = 85.49 \times (0.89) \times \sqrt{\frac{(540.4)}{(29.59) \times (28.7)}} \times (0.935)$$

$$V_s = \underline{53.5} \text{ ft/s}$$

Area of stack (ft<sup>2</sup>) = A = 5.33

Testing time (min) =  $\theta$  = 64

Volumetric gas flow rate in stack (dscfm) =  $Q_{sd}$

$$Q_{sd} = 60 \times (1 - B_{ws}) \times V_s \times A \times (528/T_s) \times (P_s/29.92)$$

$$Q_{sd} = 60 \times (1 - 0.01) \times (53.5) \times (5.33) \times \left(\frac{528}{540.4}\right) \times \left(\frac{29.59}{29.92}\right)$$

$$Q_{sd} = \underline{16,400} \text{ dscfm}$$

Diameter of sampling nozzle (in.) =  $d_n$  = 0.195

Area of nozzle (ft<sup>2</sup>) =  $A_n = 54.54E-4 (d_n)^2$

$$A_n = (0.005454) (0.195)^2 = \underline{2.074 \times 10^{-4}} \text{ ft}^2$$

Isokinetic sampling rate (%) = I

$$I = \frac{100 T_s [0.002669 V_{1c} + (V_m Y/T_m)(P_{bar} + \Delta H/13.6)]}{60 \theta V_s P_s A_n}$$

$$I = \frac{100 (540.4) [0.00267(8.5) + ((399.24)(1.008)/(545.2))((30.0) + (1.39)/13.6)]}{60 (64) (53.5) (29.59) (2.074 \times 10^{-4})}$$

$$I = \underline{96.2} \%$$

$$\text{Hexavalent Cr - Blank, } \mu\text{g} = M_{\text{Cr}6} = 169 - 1.78 = 167.2$$

$$\text{Total Cr (filter) - Blank, } \mu\text{g} = M_{\text{Cr}} = 0.485 - 0.165 = 0.32$$

$$\text{Total Cr (HNO}_3\text{) - Blank, } \mu\text{g} = M_{\text{Cr}} = 33.4 - 1.11 = 32.3$$

$$\text{Total Cr} = \text{Cr}^{+6} + \text{Total Cr (filter)} + \text{Total Cr (HNO}_3\text{)} = M_{\text{TCr}} = 167.2 + 0.32 + 32.3 = 199.8 \mu\text{g}$$

Hexavalent Chromium Concentration,  $\mu\text{g/dscf}$ 

$$C_{\text{Cr}6} = M_{\text{Cr}6} / V_{\text{MSTD}}$$

$$C_{\text{Cr}6} = 167.2 / 39.195 = 4.27 \mu\text{g/dscf}$$

Total Chromium Concentration,  $\mu\text{g/dscf}$ 

$$T_{\text{Cr}} = M_{\text{TCr}} / V_{\text{MSTD}}$$

$$T_{\text{Cr}} = 199.8 / 39.195 = 5.10 \mu\text{g/dscf}$$

Hexavalent Chromium Emission Rate, lb/hr

$$\text{ER}_{\text{Cr}6} = C_{\text{Cr}6} (\mu\text{g/dscf}) \times (1.0\text{g} / 10^6 \mu\text{g}) \times (1 \text{ lb} / 453.6 \text{ g}) \times Q_{\text{sd}} (\text{dscf} / \text{min}) \times (60 \text{ min} / \text{hour})$$

$$\text{ER}_{\text{Cr}6} = C_{\text{Cr}6} (\mu\text{g/dscf}) \times Q_{\text{sd}} (\text{dscf/min}) \times 1.323 \times 10^{-7} [ (\text{lb} \times \text{min}) / (\mu\text{g} \times \text{hour}) ]$$

$$\text{ER}_{\text{Cr}6} = (4.27) \times (16,400) \times 1.323 \times 10^{-7} = 0.0092 \text{ lb/hour}$$

Total Chromium Emission Rate, lb/hr

$$\text{ER}_{\text{TCr}} = T_{\text{Cr}} (\mu\text{g/dscf}) \times Q_{\text{sd}} (\text{dscf/min}) \times 1.323 \times 10^{-7} [ (\text{lb} \times \text{min}) / (\mu\text{g} \times \text{hour}) ]$$

$$\text{ER}_{\text{TCr}} = (5.10) \times (16,400) \times (1.323 \times 10^{-7}) = 0.011 \text{ lb/hour}$$

Hexavalent Chromium Emission Rate, mg/ampere-hour

$$E = ER_{Cr6} \text{ (lb/hour)} \times (64 \text{ minute} / \text{x-ampere-hours}) \times (1 \text{ hour}/60 \text{ min}) \times (453.6 \text{ grams/lb}) \times (1000 \text{ mg/gram})$$

$$E = ER_{Cr6} \text{ (lb/hour)} \times (64 \text{ minutes} / \text{x-ampere-hours}) \times 7,560$$

$$E = (0.0092) \times (64 / 38,074) \times (7,560)$$

$$E = 0.117 \text{ mg/ampere-hour}$$

Total Chromium Emission Rate, mg/ampere-hour

$$E = ER_{TCr} \text{ (lb/hour)} \times (64 \text{ minutes} / \text{x-ampere-hours}) \times (7,560)$$

$$E = (0.011) \times (64 / 38,074) \times (7,560)$$

$$E = 0.140 \text{ mg/ampere-hour}$$



**APPENDIX B**

**CHROMIUM FIELD DATA AND RECOVERY SHEETS**





## HEXAVALENT CHROMIUM EPA 0013 SAMPLE RECOVERY AND INTEGRITY DATA SHEET

Plant U.S. CHROME/JMC-USA Sample Date 10/2/91  
 Sample Location BATAVIA, NY Recovery Date 10/2/91  
 Run No. INLET RUN #1 Recovered by BR  
 Filter Number(s) \_\_\_\_\_

### MOISTURE

Impingers	1	2	3	4	Silica Gel	
Final Weight	300	X	X	X	674.7	grams
Initial Weight	300	X	X	X	666.2	grams
Net Weight	0	X	X	X	8.5	grams

pH 13.0

Description of impinger water CLEAR 10 % spent  
BLUE Sil gel color

Total moisture = 8.5 grams

### RECOVERED SAMPLE

Nitrogen Purge 10 L/min for 30 Minutes  
 Filter container number(s) JMC-1-PF-TOTAL Cr Sealed   
 Description of particulate on filter NONE

<u>Impinger contents</u>	
container no. <u>JMC-1-IMP-Cr<sup>+6</sup> (Imp 1-3)</u>	Liquid level marked/sealed <input checked="" type="checkbox"/>
* <u>JMC-BLANK-KOH-Cr<sup>+6</sup> 0.1N KOH blank</u>	Liquid level marked/sealed <input checked="" type="checkbox"/>
container no. <u>JMC-1-HNO<sub>3</sub>-TOTAL Cr</u>	Liquid level marked/sealed <input checked="" type="checkbox"/>
* <u>JMC-BLANK-H<sub>2</sub>O-Cr<sup>+6</sup> DI Water blank</u>	Liquid level marked/sealed <input checked="" type="checkbox"/>
container no. <u>* JMC-BLANK-HNO<sub>3</sub>-TOTAL Cr</u>	
* <u>JMC-BLANK-PF-TOTAL Cr</u>	
Samples stored and locked <u>BR</u>	

Remarks \* ALL BLANKS LISTED ON THIS SHEET, COLLECTED BETWEEN RUNS 1 AND  
 Relinquished by \_\_\_\_\_ Date \_\_\_\_\_





## HEXAVALENT CHROMIUM EPA 0013 SAMPLE RECOVERY AND INTEGRITY DATA SHEET

Plant U.S. CHROME / JMC-USA Sample Date 10/2/91  
 Sample Location BATAVIA, NY Recovery Date 10/2/91  
 Run No. INLET #2 Recovered by BR  
 Filter Number(s) N/A

### MOISTURE

Impingers	1	2	3	4	Silica Gel	
Final Weight	300	X	X	X	698.2	grams
Initial Weight	300	X	X	X	686.7	grams
Net Weight	0	X	X	X	11.5	grams

pH 13.0

Description of impinger water CLEAR 10 % spent  
BLUE Sil gel color

Total moisture = 11.5 grams

### RECOVERED SAMPLE

Nitrogen Purge 10 L/min for 30 Minutes  
 Filter container number(s) JMC-2-PF-TOTAL Cr Sealed   
 Description of particulate on filter NONE

#### Impinger contents

container no. <u>JMC-2-IMP-Cr<sup>+6</sup>(Imp 1-3)</u>	Liquid level marked/sealed	<input checked="" type="checkbox"/>
container no. <u>0.1N KOH blank</u>	Liquid level marked/sealed	<input checked="" type="checkbox"/>
container no. <u>DI Water blank</u>	Liquid level marked/sealed	<u>N/A</u>

Samples stored and locked  BR

Remarks DIFFICULTY FILTERING FOR TOTAL Cr RECOVERY EXTREME ON THIS  
 Relinquished by \_\_\_\_\_ Date SAMA

EMISSION TESTING I.D. DATA

PLANT AND CITY	JMC / USA CHROME BATH	DATE	10-2-91	SAMPLING LOCATION	Scrubber Inlet Exhaust	SAMPLE TYPE	Hexchr/Totalchr	RUN NUMBER	JMC-R3-SILE
OPERATORS	CD BR	AMBIENT TEMP (°F)	92	STACK (D) (in)	31.25	PITOT TUBE (in)	0.84	PROBE NUMBER	CR-3
ASSUMED MOIST. (%)	7M	STATIC PRESS (in. H <sub>2</sub> O)	-5.6	FILTER TYPE & FILTER NUMBER(S)	JMC-3-PF	ORISAT NO.	N/A	NOZZLE ID.	L.20
		DGM CAL FACTOR (G)	1.008	STACK PITOT NO.	3T1	IMPINGER THERM NO.	3T1	TRAIN LEAK CHECK (INITIAL)	
		DGM H@	1.92	VELOCITY HEAD (in. H <sub>2</sub> O)	0.63	ORIFICE (in. H <sub>2</sub> O)	1.00	TRAIN LEAK CHECK (FINAL)	
		DGM H@	1.92	%O <sub>2</sub>	20.0	RUN 1	20.0	AVG	20.0
				%CO <sub>2</sub>	0	RUN 2	20.0	AMBIENT	21.0
						RUN 3	20.0	PITOT SYSTEM LEAK CHECK	
								INIT	0
								FINAL	0.001
									1.64
									0.195
									1.64

TRAV. POINT NO.	ELAPSED TEST TIME (min)	CLOCK TIME (24-hr)	GAS METER READING (m. (ft))	VELOCITY HEAD (in. H <sub>2</sub> O)	ORIFICE (in. H <sub>2</sub> O)	STACK TEMP (°F)	PROBE TEMP (°F)	FILTER OVEN TEMP (°F)	SIL GEL IMPINGER TEMP (°F)	DGM IN/OUT TEMP (°F)	AUX TEMP (°F)	SAMPLE TRAIN ACQU (in. Hg)
8	0	1257	191.933	0.61	1.00	84			49	92		6
7	4		194.23	0.61	1.00	85			49	92		6
	8		196.59	0.54	0.89	85	N/A		49	93		5
	12		198.78	0.54	0.89	84			51	93		5
	16		200.92	0.53	0.87	84			52	93		5
	20		203.05	0.53	0.87	85			52	94		5
	24		205.18	0.70	1.15	84			52	94		6
	28		207.52	0.71	1.16	85			52	94		7
	32		209.89	1.3	2.13	85			52	95		13
	36		212.71	1.2	1.97	85			50	95		12
	40		215.91	1.2	1.97	85			51	95		11
	44		218.97	1.2	1.97	84			52	95		11
TOTAL TIME												
Page Totals												

AVG DGM F

AVG STK F

AVG H

21.838  
P545

Sheet Checked By: *[Signature]*

Date 10-31-91

EMISSION TESTING FI. DATA

PLANT AND CITY	DATE	SAMPLING LOCATION	SAMPLE TYPE	RUN NUMBER									
SMC / USA CHROME	10-29	Scribble Tank Exhaust	Hex Chr	SMC - R3 - SIE									
TRAV. POINT NO.	ELAPSED TEST TIME (min)	CLOCK TIME (24-HR)	GAS METER READING (m <sup>3</sup> /15)	VELOCITY (ft. H <sub>2</sub> O)	P	H ORIFICE (in. H <sub>2</sub> O)	STACK TEMP (°F)	PROBE TEMP (°F)	FILTER OVEN TEMP (°F)	SIL GEL IMPINGER TEMP (°F)	DGM IN/OUT TEMP (°F)	AUX. TEMP. (°F)	SAMPLE TRAIN ACUU (in. Hg)
2	48		222.05	1.1		1.80	84			53	96		10
1	52		225.00	1.1		1.80	85	N/A		53	95		10
	56		228.53	1.3		2.13	85			53	96		12
	60		230.07	1.3		2.13	85			53	96		
	64	12/01	234.289										
TOTAL TIME		42.356		0.936		1.483		84.6		94.3			
Page Totals		64		10.915									

## HEXAVALENT CHROMIUM EPA 0013 SAMPLE RECOVERY AND INTEGRITY DATA SHEET

Plant V.S. CHROME/JMC-USA Sample Date 10/2/91  
 Sample Location BAT Recovery Date 10/2/91  
 Run No. INLET # 3 Recovered by BR  
 Filter Number(s) N/A

### MOISTURE

Impingers	1	2	3	4	Silica Gel	
Final Weight	300	<del>  </del>	<del>  </del>	<del>  </del>	683.7	grams
Initial Weight	300	<del>  </del>	<del>  </del>	<del>  </del>	674.7	grams
Net Weight	0	<del>  </del>	<del>  </del>	<del>  </del>	9.0	grams

pH 13.0

Description of impinger water CLEAR 25 % spent  
BLUE/WHITE Sil gel color  
 Total moisture = 9.0 grams

### RECOVERED SAMPLE

Nitrogen Purge 10 L/min for 30 Minutes  
 Filter container number(s) JMC-3-PF-TOTAL Cr Sealed   
 Description of particulate on filter NONE

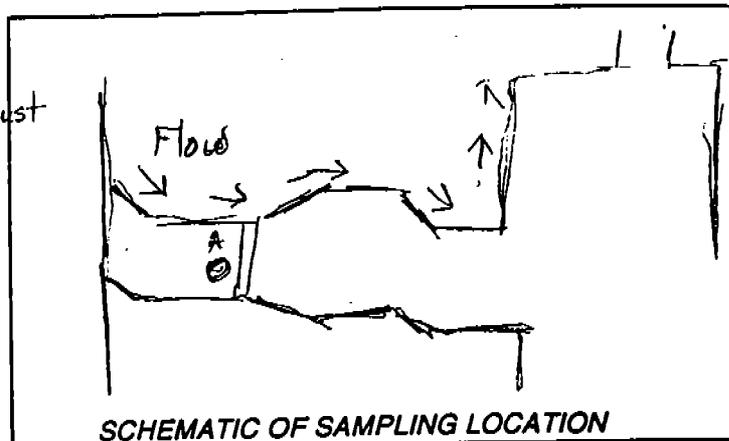
<u>Impinger contents</u>			
container no.	<u>JMC-3-MP-Cr<sup>6+</sup> Imp 1-3</u>	Liquid level marked/sealed	<input checked="" type="checkbox"/>
container no.	<u>0.1N KOH blank</u> <u>JMC-3-HNO<sub>3</sub>-TOTAL Cr</u>	Liquid level marked/sealed	<input checked="" type="checkbox"/>
container no.	<u>DI Water blank</u> <u>N/A</u>	Liquid level marked/sealed	<u>N/A</u>

Samples stored and locked  BR  
 Remarks \_\_\_\_\_  
 Relinquished by \_\_\_\_\_ Date \_\_\_\_\_



## EPA METHOD 1 TRAVERSE POINT LOCATION FOR CIRCULAR DUCTS

PLANT JMC USA CHROME  
 CITY BATAVIA STATE NY  
 SAMPLING LOCATION Scrubber Inlet Plating Exhaust  
 INSIDE OF FAR WALL TO OUTSIDE OF NIPPLE, (DISTANCE A) 31.5 31.25  
 INSIDE OF NEAR WALL TO OUTSIDE OF NIPPLE, (DISTANCE B) .25  
 NEAREST UPSTREAM DISTURBANCE 17.5  
 DISTURBANCE 0.8  
 NEAREST DOWNSTREAM DISTURBANCE 8.0  
 DISTURBANCE 0.3  
 SAMPLER GS BR DATE 10-1-91

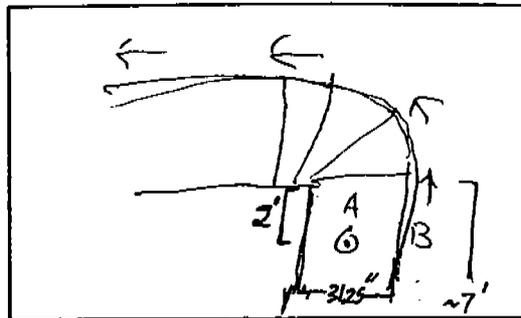


TRAVERSE POINT NUMBER	FRACTION OF STACK I.D.	STACK I.D.	PRODUCT OF COLUMNS 2 AND 3 (TO NEAREST 1/8-INCH)	DISTANCE B	TRAVERSE DISTANCE FROM OUTSIDE OF NIPPLE (SUM OF COLUMNS 4 & 5)
1	.032	31.25	1.0	.25	1.25
2	.105		3.28		3.5
3	.194		6.1		6.4
4	.323		10.1		10.4
5	.677		21.2		21.5
6	.806		25.2		25.5
7	.895		28.0		28.3
8	.968	√	30.1	√	30.4

**PRELIMINARY VELOCITY TRAVERSE  
EPA METHOD 2**



PLANT/CLIENT JMC / USA CHROME  
 SOURCE SCRUBBER EXIT  
 DATE 10-1-91  
 STACK I.D. (inches) 31.25  
 BAROMETRIC PRESSURE (in. Hg) 30.0  
 STACK GAUGE PRESSURE (in. H2O) ~~0.0~~ 7.5  
 OPERATORS CJ BR  
 PITOT NO. P410 TYPE S COEFF 0.84



TRAVERSE POINT LAYOUT SCHEMATIC

TRAVERSE POINT NUMBER	VELOCITY HEAD (in. H2O)	STACK TEMP (°F)
A- 1	0.98	82
2	0.67	
3	0.77	
4	0.71	
5	0.69	
6	0.70	
7	0.68	
8	0.62	
B- 1	0.77	
2	0.45	
3	0.42	
4	0.40	
5	0.67	
6	0.69	
7	0.70	
8	0.72	
<b>AVERAGE</b>	<b>SQRT 0.809</b>	<b>82</b>

0.77  
0.48  
0.42  
0.40  
0.63  
0.69  
0.71  
0.69

TRAVERSE POINT NUMBER	VELOCITY HEAD (in. H2O)	STACK TEMP (°F)
<b>AVERAGE</b>	<b>SQRT</b>	

POST PITOT LEAK CHECK PASSED  FAILED

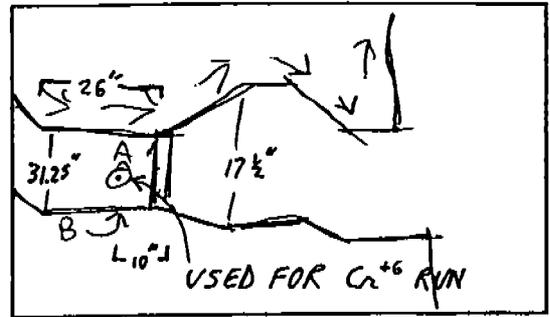
MEASUREMENT DEVICE  
 MICROMETER   
 0-10" MANOMETER   
 MAGNEHELIC   
 OTHER

EXPLAIN:

# PRELIMINARY VELOCITY TRAVERSE EPA METHOD 2



PLANT/CLIENT JMC / USA CHROME  
 SOURCE Scrubber Inlet Plating Exhaust  
 DATE 10-2-91  
 STACK I.D. (inches) 31.25  
 BAROMETRIC PRESSURE (in. Hg) 30.0  
 STACK GAUGE PRESSURE (in. H<sub>2</sub>O) - 5.6  
 OPERATORS CJ/BR  
 PITOT NO. C23 TYPE S COEFF 0.84



TRAVERSE POINT LAYOUT SCHEMATIC

TRAVERSE POINT NUMBER	VELOCITY HEAD (in. H <sub>2</sub> O)	STACK TEMP (°F)
A-8	0.70	81
7	0.63	80
6	0.61	80
5	0.74	80
4	1.3	79
3	1.2	79
2	1.2	79
1	0.95	79
B-8	1.2	80
7	0.98	80
6	0.73	80
5	0.77	81
4	0.82	81
3	0.99	82
2	1.0	82
1	1.2	82
<b>AVERAGE</b>	<b>SQRT 0.964</b>	<b>80.4</b>

SQRT = 0.890

TRAVERSE POINT NUMBER	VELOCITY HEAD (in. H <sub>2</sub> O)	STACK TEMP (°F)
<b>AVERAGE</b>	<b>SQRT</b>	

POST PITOT LEAK CHECK PASSED  FAILED

MEASUREMENT DEVICE

- MICROMETER
- 0-10" MANOMETER
- MAGNEHELIC
- OTHER

EXPLAIN:



EPA Method 3  
Dry Molecular Weight Determination

PLANT SAC / USA CHROME

SAMPLING LOCATION SCRUBBER INLET PLATING EXHAUST

DATE 10-19-1

RUN NO. PRELIMINARY

OPERATOR C.S. BR

AMBIENT TEMPERATURE 74

ANALYTICAL METHOD FYRITE

SAMPLE TYPE  Single-Point Grab  
 Single-Point Integrated  
 Multi-Point Integrated (fill out Sampling Rate Data)

\* % dev = (Q-Qavg)/Qavg x 100%  
Must be <10%

SAMPLING SYSTEM LEAK-CHECKED

GAS	Time:			Average Volume %	Multiplier	Molecular Weight of Stack Gas (Dry Basis) Md, lb/lb-mole
	Run 1 %	Run 2 %	Run 3 %			
CO2	0	0	0	0	0.44	0
O2	21.0	21.0	21.0	21.0	0.32	6.72
CO	N/A	N/A	N/A	N/A	0.28	0
N2	79.0	79.0	79.0	79.0	0.28	22.12
TOTAL						28.84



**APPENDIX C**  
**LABORATORY AND CHAIN-OF-CUSTODY DATA**

# DRAFT

RTI Project No. : 4848-02J

Samples : Impinger Samples

Company : IEA (P.O. # 0002755 & 2998)

Analyte : Cr(VI)

Method of Analysis : Ion Chromatography / Post Column Reaction

Samples Received : 10-7-91

Report Date : 11-8-91

Unreliable

Sample	Total Volume mL	Cr(VI) ug/mL	Cr(VI) Total, ug	Vol. (mL) Taken for Digestion	Total Cr ug/mL	Total Cr ug
JMC-1-IMP-Cr+6	281	0.600	169	172	0.564	46.1 *
JMC-2-IMP-Cr+6	273	0.244	66.6	182	0.412	30.9 *
JMC-3-IMP-Cr+6	298	0.399	119	190	0.278	21.8 *
JMC-Blank-DIW	312	ND	ND	150	--	--
JMC-Blank-KOH	305	0.00584	1.78	200	0.0107	0.816
JMC-Blk-HNO3-Cr+6	314	--	--	200	0.0142	1.11
JMC-Blk-PF-Tot.Cr	--	--	--	--	0.00330	0.165
JMC-1-PF-Tot.Cr	--	--	--	--	0.00970	0.485
JMC-2-PF-Tot.Cr	--	--	--	--	0.00320	0.160
JMC-3-PF-Tot.Cr	--	--	--	--	0.00420	0.210
JMC-1-HNO3-Tot.Cr	310	--	--	150	0.323	33.4
JMC-2-HNO3-Tot.Cr	204	--	--	150	0.222	15.1
JMC-3-HNO3-Tot.Cr	222	--	--	150	5.43	402

Detection Limit

0.0015

0.002

ND : Non-detectable; less than detection limit

Total Cr was determined by GFAA

\* : Results of Total Cr, ug for impinger samples were unreliable. There was problems in digestion. Gelatin precipitates that were formed during digestion were difficult to dissolve and filtered.

Impingers:

Total Cr, ug = Total Cr, ug/mL \* Diluted Vol. (50 mL) / Vol.(mL) taken for Digestion \* Total Volume, mL

Filters ( -PF- ) :

Total Cr, ug = Total Cr, ug/mL \* Diluted Vol. (50mL)

RTI Project No. : 4848-02J

Samples : QC for Impinger Samples

Company : IEA (P.O. # 0002755 & 2998)

Analyte : Cr(VI)

Method of Analysis : Ion Chromatography / Post Column Reaction

Samples Received : 10-7-91

Report Date : 11-8-91

Calibration Check Sample, ug/mL

Sample	Cr(VI)	Cr(VI)	Total	Total
	ug/mL	ug/mL	Cr	Cr
	Measured	Expected	Measured	Expected
QC	0.00933	0.0100	0.0196	0.0186
QC	--	--	0.0203	0.0186

Results of Blank, Duplicate, and Spike Analysis

Sample	Cr(VI)	Spike	Spike	Spike	Total	Total	Total
	ug/mL	Cr(VI)	Cr(VI)	Cr(VI)	Cr	Cr	Cr
	Measured	Measured	Expected	Recovery	Measured	Expected	Recovery
RTI DIW	--	--	--	--	--	--	--
JMP-1-IMP-Cr+6 Dup	0.610	--	--	--	--	--	--
JMP-2-IMP-Cr+6 Spk	--	0.00970	0.0100	97.0	--	--	--
Reagent Blank	--	--	--	--	ND	--	--
SRM (WP481)	--	--	--	--	0.0519	0.0522	99.4
SRM (WP481) Dup	--	--	--	--	0.0549	0.0522	105





**APPENDIX D**  
**PROCESS RATE**



RECORD OF AMPERE-HOUR METER READINGS

PLANT U.S. CHROME/JMC-USA OPERATOR CJ/BR  
 CITY BATAVIA, NY PROJECT # 1331-001  
 DATE 10/2/91 CHECKED BY BHR

	TIME	UNIT 1 (Amp-hours)	UNIT 2 (Amp-hours)	UNIT 3 (Amp-hours)	UNIT 4 (Amp-hours)	TOTAL
RUN 1 FINAL	10:29	860765	389745	995999	527779	
INITIAL	9:25	850600	379824	986720	518420	
TOTAL	64 MIN.	10,165	9,321	9,279	9,309	38,074
RUN 2 FINAL	12:29	884005	410000*	017682	549005	
INITIAL	11:25	874048	400906	008419	539765	
TOTAL	64 MIN.	9957	9,094	9,263	9,240	37,554
RUN 3 FINAL	2:01	894610	419780	027510	558940	
INITIAL	12:57	884594	410600	018213	549684	
TOTAL	64 MIN.	10,016	9,180	9,297	9,256	37,749

\* METER STUCK AT 410,000 AT TIME OF READING; IMMEDIATELY CORRECTED. BR

SHEET CHECKED BY: Chris Johnson  
10/21/91



**APPENDIX E**

**EQUIPMENT CALIBRATION AND CERTIFICATION DATA**



## DRY GAS METER POST-TEST CALIBRATION USING REFERENCE METER

DATE: 8-7-91 METER BOX NO. M-7  
 CALIBRATOR: G.C. BOWSER BAROMETRIC PRESSURE (Pb) 30.24 in. Hg  
 INITIAL CALIBRATION Y: 1.008 ΔH@: 1.92  
 PLANT: S.D. WARR MAINE PROJECT NO.: 1112-005  
 AVERAGE ΔH DURING TESTING: 1.8 MAXIMUM VACUUM 10 in. Hg

Orifice manometer setting* ΔH in. H2O	Volume reference meter Vw ft3	Volume dry gas meter Vd ft3	Temperatures				Test duration θ min	Vacuum setting ** in. Hg	Calibr Factor Y	ΔH@ in. H2O
			Ref Meter		Dry gas meter					
			Tw °F	Avg Tw °F	Td °F	Avg Td °F				
1.8	start 363.746	835.601	start 79		start 71		15	10	0.954	
	stop 374.260	847.438	stop 79	79	stop 73	72				
	diff 10.514	10.827	79		73					
1.8	start 374.260	847.438	start 79		start 73		15	10	0.958	
	stop 384.788	858.246	stop 79	79	stop 74	73.5				
	diff 10.528	10.808	79		74					
1.8	start 384.788	858.246	start 79		start 73		15	10	0.958	
	stop 395.299	869.032	stop 79	79	stop 74	73.5				
	diff 10.511	10.786	79		74					
Post-test Average***									0.956	

- \* To be the average ΔH used during test series.
- \*\* To be the highest vacuum used during test series.
- \*\*\* Post-test Y must be within the range, pre-test Y +/- 5% OK?
- Post-test ΔH@ should be within the range of the initial or pre-test ΔH@ +/- 0.20 in. H2O. OK?

$$Y = \frac{(Vw)(Pbar)(Td + 460)}{(Vd)(Pbar + \Delta H / 13.6)(Tw + 460)}$$

$$\Delta H@ = \frac{(0.0317)(\Delta H)}{(Pbar)(Td + 460)} \left[ \frac{(Tw + 460)(\theta)}{(Vw)} \right]^2$$

$$\text{Percent difference} = \frac{(\text{Avg initial Y}) - (\text{Post-test Y})}{(\text{Avg initial Y})}$$



## DRY GAS METER POST-TEST CALIBRATION USING REFERENCE METER

DATE: 10-4-91 METER BOX NO. 7M  
 CALIBRATOR: CJ BAROMETRIC PRESSURE (Pb) 29.73 in. Hg  
 INITIAL CALIBRATION Y: 1.008 ΔH@: 1.92  
 PLANT: JMC USA CHROME PROJECT NO.: \_\_\_\_\_  
 AVERAGE ΔH DURING TESTING: 1.0 MAXIMUM VACUUM 10 in. Hg

Orifice manometer setting* ΔH in. H2O	Volume reference meter Vw ft3	Volume dry gas meter Vd ft3	Temperatures				Test duration θ min	Vacuum setting ** in. Hg	Calibr Factor Y	ΔH@ in. H2O
			Ref Meter		Dry gas meter					
			Tw °F	Avg Tw °F	Td °F	Avg Td °F				
1.0	start 319.390	277.724	start 74	74	start 75	75.5	10	10	1.004	1.86
	stop 324.983	283.234	stop 74		stop 76					
	diff 5.493	5.510								
1.0	start 324.793	283.234	start 74	74.5	start 76	76.5	20	10	0.999	1.90
	stop 335.661	294.217	stop 75		stop 77					
	diff 10.878	10.977								
1.0	start 335.661	294.211	start 75	75	start 77	77	10	10	0.993	1.93
	stop 341.073	299.704	stop 75		stop 77					
	diff 5.412	5.493								
Post-test Average***									0.999	1.93

\* To be the average ΔH used during test series.

\*\* To be the highest vacuum used during test series.

\*\*\* Post-test Y must be within the range, pre-test Y +/- 5% OK?

Post-test ΔH@ should be within the range of the initial or pre-test ΔH@ +/- 0.20 in. H2O. OK?

$$Y = \frac{(Vw)(Pbar)(Td + 460)}{(Vd)(Pbar + \Delta H / 13.6)(Tw + 460)}$$

$$\Delta H@ = \frac{(0.0317)(\Delta H)}{(Pbar)(Td + 460)} \left[ \frac{(Tw + 460)(\theta)}{(Vw)} \right]^2$$

$$\text{Percent difference} = \frac{(\text{Avg initial Y}) - (\text{Post-test Y})}{(\text{Avg initial Y})}$$

0.008  
 $\Delta H = 1.92$

DRY GAS METER AND ORIFICE SYSTEM CALIBRATION

DATE 8-23-71 OPERATOR CF Metering System Identification M-7

Orifice Manometer Setting $\Delta H$ " H <sub>2</sub> O	Minimum Volume of Gas ft <sup>3</sup>	NET TEST METER GAS VOLUMES AND TEMPERATURES				DRY TEST METER GAS VOLUMES AND TEMPERATURES				Barometer Reading, Inches Hg P <sub>b</sub>					
		Initial ft <sup>3</sup> V <sub>w1</sub>	Final ft <sup>3</sup> V <sub>w2</sub>	Temp. °F T <sub>w1</sub>	Net Volume ft <sup>3</sup> V <sub>w</sub>	Avg. Temp. °F T <sub>w</sub>	Initial ft <sup>3</sup> V <sub>d1</sub>	Final ft <sup>3</sup> V <sub>d2</sub>	Temp. °F T <sub>d1</sub>		Net Volume ft <sup>3</sup> V <sub>d</sub>	Avg. Temp. °F T <sub>d</sub>	Total Test Time Minutes $\theta$		
0.5	5	715.003	70	722.949	70	7.946	70	61.029	72	62.028	75	7.999	73.5	21	29.58
1.00	5	723.258	70	728.746	70	5.488	70	69.338	75	74.888	77	5.560	76	10	29.58
2.00	5	729.084	70	732.19	70	8.083	70	75.235	76	83.420	79	8.185	77.5	11	29.58
3.00	5	744.870	70	752.120	70	7.260	70	91.240	80	98.611	81	7.371	80.5	8	29.58

Check at least every 2 minutes and adjust, if needed.

Vacuum System Leak Check ✓  
 Pressure Leak Check of Meter Box ✓  
 System Operated 15 min. prior to Calibration ✓

CALCULATIONS

$$\gamma = \frac{(V_w)(V_w)(P_b)(T_d + 460)}{(V_d)(P + \frac{\Delta H}{13.6})(T_w + 460)}$$

$$\Delta H = \frac{0.0317 (\Delta H)}{(P_b)(T_d + 460)} \left[ \frac{(T_w + 460) \theta}{(V_d)(V_w)} \right]^2$$

Calc. By:  
 Checked By:  
 Date:

$\Delta H$	GAMMA CALCULATIONS	$\gamma$	$\Delta H\theta$ CALCULATIONS	$\Delta H\theta$
0.5	$(\gamma) (7.946) (29.58) (73.5 + 460)$ $(7.999) (2.958 + \frac{0.5}{13.6}) (70 + 460)$	1.006	$\frac{0.0317 (0.5) (21)^2}{(29.58) (73.5 + 460)} (70 + 460) (21)^2$ $(0.007) (7.946)$	1.93
1.00	$(\gamma) (5.488) (29.58) (76 + 460)$ $(5.560) (2.958 + \frac{1.0}{13.6}) (70 + 460)$	1.003	$\frac{0.0317 (1.0) (10)^2}{(29.58) (76 + 460)} (70 + 460) (10)^2$ $(5.488)$	1.87
2.00	$(\gamma) (8.083) (29.58) (77.5 + 460)$ $(8.185) (2.958 + \frac{2.0}{13.6}) (70 + 460)$	1.004	$\frac{0.0317 (2.0) (11)^2}{(29.58) (77.5 + 460)} (70 + 460) (11)^2$ $(8.083)$	2.08
3.00	$(\gamma) (7.260) (29.58) (80.5 + 460)$ $(7.371) (2.958 + \frac{3.0}{13.6}) (70 + 460)$	1.004	$\frac{0.0317 (3.0) (8)^2}{(29.58) (80.5 + 460)} (70 + 460) (8)^2$ $(7.260)$	2.04
	AVERAGE 1.004		AVERAGE	1.98

( $\gamma$ ) Gamma = ratio of reading of wet test meter to dry test meter.

$\Delta H\theta$  = orifice pressure differential that gives 0.75 cfm of air at 68°F and 29.92" Hg. Average  $\Delta H\theta$  value between 1.59-2.09 is acceptable. In addition, the  $\Delta H\theta$  must not vary by more than 0.05.

**THERMOCOUPLE DIGITAL INDICATOR  
CALIBRATION DATA FORM**

② 70°F

#7 NUTECH

Date 7/18/21 Calibrator Name MR  
 Indicator No. 883A-4600-00AB Serial No. 007  
 Calibration Device No. KF-Z Manufacturer OMEGA

Test Point No.	Millivolt Signal	Equivalent Temperature deg. F	Digital Indicator Temperature deg. F	Percent difference %
1	-0.76	000	-001	0.22
2	3.9	200	200	0.0
3	8.3	400	398	0.23
4	12.9	600	601	0.09
5	17.6	800	801	0.08
6	22.3	1000	1001	0.07
7	27.0	1200	1200	0.0
8	31.7	1400	1400	0.0
9	36.2	1600	1602	0.10
10	40.6	1800	1799	0.04

Percent difference must be less than or equal to 0.5%

Percent Difference:

$$\frac{(\text{Ref temp} + 460) - (\text{Test temp} + 460)}{(\text{Ref temp} + 460)} \times 100\% \text{ should be } < 0.5\%$$

#7 FUSI'S

### THERMOCOUPLE DIGITAL INDICATOR CALIBRATION DATA FORM

@ 70°F

Date 7/19/91

Calibrator Name MCR

Indicator No. PYZ4RCY1-4V / same

Serial No. 122711T / 131894T

Calibration Device No. KF-2

Manufacturer OMEGA

Test Point No.	Millivolt Signal	Equivalent Temperature deg. F	Digital Indicator Temperature deg. F		Percent difference	
			TOP	BOTTOM	T	B
1	-0.76	000	-001	-002	0.22	0.43
2	1.4	100	099	099	0.18	0.18
3	3.7	200	202	200	0.30	0.0
4	6.0	300	300	299	0.0	0.13
5	8.2	400	400	397	0.0	0.35
6	10.4	500	X	X		
7	12.8	600	X	X		

Percent difference must be less than or equal to 0.5%

Percent Difference:

$$\frac{(\text{Ref temp} + 460) - (\text{Test temp} + 460)}{(\text{Ref temp} + 460)} \times 100\% \text{ should be } < 0.5\%$$



# TEMPERATURE CALIBRATION DATA FORM

Date 9/20/91

Calibrator Name MCR

Ambient Temp 71.8

Barometric Pressure 29.92 in. Hg

Reference: Mercury-in-glass

Other PRINCO 453 dno

Thermocouple No.	Source (specify)	Reference thermometer temperature deg F	Thermocouple indicator temperature deg F	Percent error %
CR-5 Chrom 6 PROBE	Ambient	71.8	71.2	0.11
	Ice	35.6	36.0	0.04
	Boiling	211.6	211.3	0.04
CR-3 Chr. 6 Prob.	Ambient	71.8	71.4	0.08
	Ice	40.6	41.4	0.09
	Boil	211.3	211.5	0.02
CR-4 Chr. 6-Prob.	Ambient	71.8	71.2	0.11
	Ice	43.2	43.5	0.04
	Boil	210.6	211.5	0.09
4B	Amb.	72.5	72.1	0.08
	Ice	32.4	32.7	0.03
	Boil	213.44	211.5	0.29
5A	Amb	69.8	71.2	0.26
	Ice	32.9	32.7	0.04
	Boil	210.9	212.0	0.16
6-A	Amb.	71.6	72.7	0.21
	Ice	32.5	32.7	0.04
	Boil	212.2	214.2	0.30

Source: ice bath, ambient air, boiling H2O, hot oil

$$\% \text{ error} = \frac{(\text{Ref temp} + 460) - (\text{Test temp} + 460)}{(\text{Ref temp} + 460)} \times 100\% \text{ should be } < 1.5\%$$

## NOZZLE CALIBRATION

Date 10-2-91

Calibrated by CJ

Nozzle identification number	D1, in.	D2, in.	D3, in.	D, in.	Davg
K-20	0.195	0.196	0.195	0.001	0.195

where:

D1, D2, D3 = nozzle diameter measured on a different diameter, in.  
Tolerance = measure within 0.001 in.

D = maximum difference in any two measurements, in.  
Tolerance = 0.004 in.

Davg = average of D1, D2, and D3.

# NOZZLE CALIBRATION

Date 10-2-91

Calibrated by [Signature]

Nozzle identification number	D1, in.	D2, in.	D3, in.	D, in.	Davg
L-20	0.195	0.195	0.195	0.000	0.195

where:

- D1, D2, D3 = nozzle diameter measured on a different diameter, in.  
Tolerance = measure within 0.001 in.
- D = maximum difference in any two measurements, in.  
Tolerance = 0.004 in.
- Davg = average of D1, D2, and D3.

Chrome Probe #3

TYPE S PITOT TUBE INSPECTION DATA FORM

Pitot tube assembly level?  yes  no

Pitot tube openings damaged?  yes (explain below)  no

$\alpha_1 = 0^\circ (<10^\circ)$ ,  $\alpha_2 = 0^\circ (<10^\circ)$ ,  $\beta_1 = 0^\circ (<5^\circ)$ ,  
 $\beta_2 = 0^\circ (<5^\circ)$

$\gamma = 0^\circ$ ,  $\theta = 0^\circ$ ,  $A = 0.652$  (in.)

$z = A \sin \gamma = 0$  cm (in.);  $<0.32$  cm ( $<1/8$  in.),

$w = A \sin \theta = 0$  cm (in.);  $<.08$  cm ( $<1/32$  in.)

$P_A = 0.326$  (in.)  $P_b = 0.326$  (in.)

$D_t = 0.25$  (in.)

Comments:

10/10/91

Checked by JH Schenk

CR #6 - #3

Calibration required?  yes  no

Chrome Probe CR 4

TYPE S PITOT TUBE INSPECTION DATA FORM

Pitot tube assembly level?  yes  no

Pitot tube openings damaged?  yes (explain below)  no

$\alpha_1 = 0^\circ (<10^\circ)$ ,  $\alpha_2 = 0^\circ (<10^\circ)$ ,  $\beta_1 = 0^\circ (<5^\circ)$ ,  
 $\beta_2 = 0^\circ (<5^\circ)$

$\gamma = 0^\circ$ ,  $\theta = 0^\circ$ ,  $A = 0.726$  (in.)

$z = A \sin \gamma = 0$  (in.);  $< 0.32$  ( $< 1/8$  in.),

$w = A \sin \theta = 0$  (in.);  $< .08$  ( $< 1/32$  in.)

$P_A = 0.363$  (in.)  $P_b = 0.363$  (in.)

$D_t = 0.25$  (in.)

Comments: 10/10/91 - checked By JHShank

Calibration required?  yes  no



**APPENDIX F**

**PROJECT PARTICIPANTS AND PROJECT SUMMARY LOG**

**JMC Hi-Tech Metals, Inc.**

Hideaki Honoki, Senior Vice President  
Kenichi Sakamachi, Executive Vice President

**U.S. Chrome Corporation**

Jim Pendergast, Plant Manager

**Industrial & Environmental Analysts, Inc.**

Jeffrey Burdette, Project Manager  
Barry Rayfield, Air Sampling Team Leader  
Chris Johnson, Technician





**APPENDIX G**

**"DETERMINATION OF HEXAVALENT CHROMIUM EMISSIONS  
FROM STATIONARY SOURCES (METHOD Cr<sup>+6</sup>)"**

3.2 Determination of Hexavalent Chromium Emissions from Stationary Sources (Method Cr<sup>+6</sup>)

3.2.1 Applicability and Principle

3.2.1.1 Applicability. This method applies to the determination of hexavalent chromium (Cr<sup>+6</sup>) emissions from hazardous waste incinerators, municipal waste combustors, sewage sludge incinerators, and boilers and industrial furnaces. With the approval of the Administrator, this method may also be used to measure total chromium. The sampling train, constructed of Teflon components, has only been evaluated at temperatures less than 300°F. Trains constructed of other materials, for testing at higher temperatures, are currently being evaluated.

3.2.1.2 Principle. For incinerators and combustors, the Cr<sup>+6</sup> emissions are collected isokinetically from the source. To eliminate the possibility of Cr<sup>+6</sup> reduction between the nozzle and impinger, the emission samples are collected with a recirculatory train where the impinger reagent is continuously recirculated to the nozzle. Recovery procedures include a post-sampling purge and filtration. The impinger train samples are analyzed for Cr<sup>+6</sup> by an ion chromatograph equipped with a post-column reactor and a visible wavelength detector. The IC/PCR separates the Cr<sup>+6</sup> as chromate (CrO<sub>4</sub><sup>-</sup>) from other components in the sample matrices that may interfere with the Cr<sup>+6</sup>-specific diphenylcarbazide reaction that occurs in the post-column reactor. To increase sensitivity for trace levels of chromium, a preconcentration system is also used in conjunction with the IC/PCR.

3.2.2 Range, Sensitivity, Precision, and Interference

3.2.2.1 Range. Employing a preconcentration procedure, the lower limit of the detection range can be extended to 16 nanograms per dry standard cubic meter (ng/dscm) with a 3 dscm gas sample (0.1 ppb in solution). With sample dilution, there is no upper limit.

3.2.2.2 Sensitivity. A minimum detection limit of 8 ng/dscm with a 3 dscm gas sample can be achieved by preconcentration (0.05 ppb in solution).

3.2.2.3 Precision. The precision of the IC/PCR with sample preconcentration is 5 to 10 percent. The overall precision for sewage sludge incinerators emitting 120 ng/dscm of  $\text{Cr}^{+6}$  and 3.5  $\mu\text{g}/\text{dscm}$  of total chromium is 25% and 9% for  $\text{Cr}^{+6}$  and total chromium, respectively; for hazardous waste incinerators emitting 300 ng/dscm of  $\text{Cr}^{+6}$  it is 20 percent.

3.2.2.4 Interference. Components in the sample matrix may cause  $\text{Cr}^{+6}$  to convert to trivalent chromium ( $\text{Cr}^{+3}$ ) or cause  $\text{Cr}^{+3}$  to convert to  $\text{Cr}^{+6}$ . A post-sampling nitrogen purge and sample filtration are included to eliminate many of these interferences. The chromatographic separation of  $\text{Cr}^{+6}$  using ion chromatography reduces the potential for other metals to interfere with the post-column reaction. For the IC/PCR analysis, only compounds that coelute with  $\text{Cr}^{+6}$  and affect the diphenylcarbazide reaction will cause interference. Periodic analysis of deionized (DI) water blanks is used to demonstrate that the analytical system is essentially free from contamination. Sample cross-contamination that can occur when high-level and low-level samples or standards are analyzed alternately is eliminated by thorough purging of the sample loop. Purging can easily be achieved by increasing the injection volume of the samples to ten times the size of the sample loop.

### 3.2.3 Apparatus

3.2.3.1 Sampling Train. Schematics of the recirculating sampling trains employed in this method are shown in Figures 3.2-1 and 3.2-2. The recirculatory train is readily assembled from commercially available components. All portions of the train in contact with the sample are either glass, quartz, Tygon, or Teflon, and are to be cleaned as per subsection 3.2.5.1.1.

The metering system is identical to that specified by Method 5 (see Section 3.8.1); the sampling train consists of the following components:

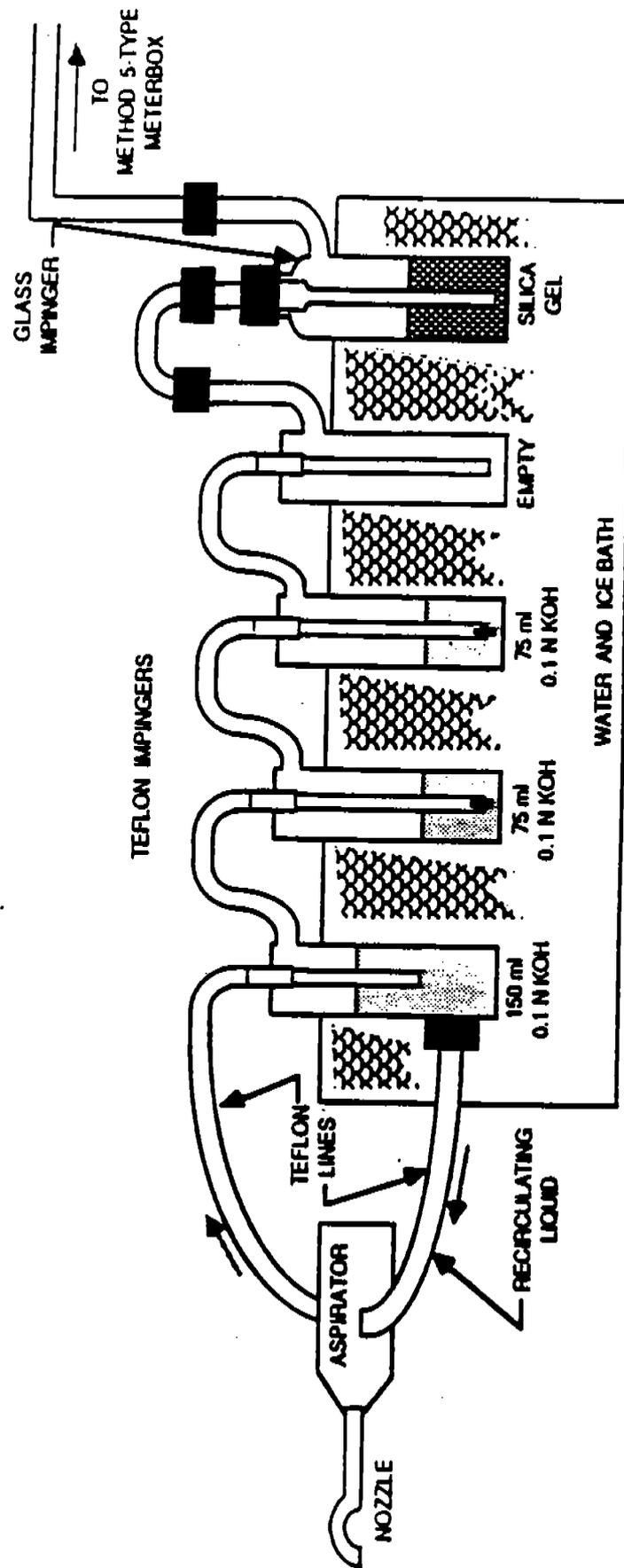


Figure 3.2-1  
Schematic of recirculatory impinger train with aspirator assembly

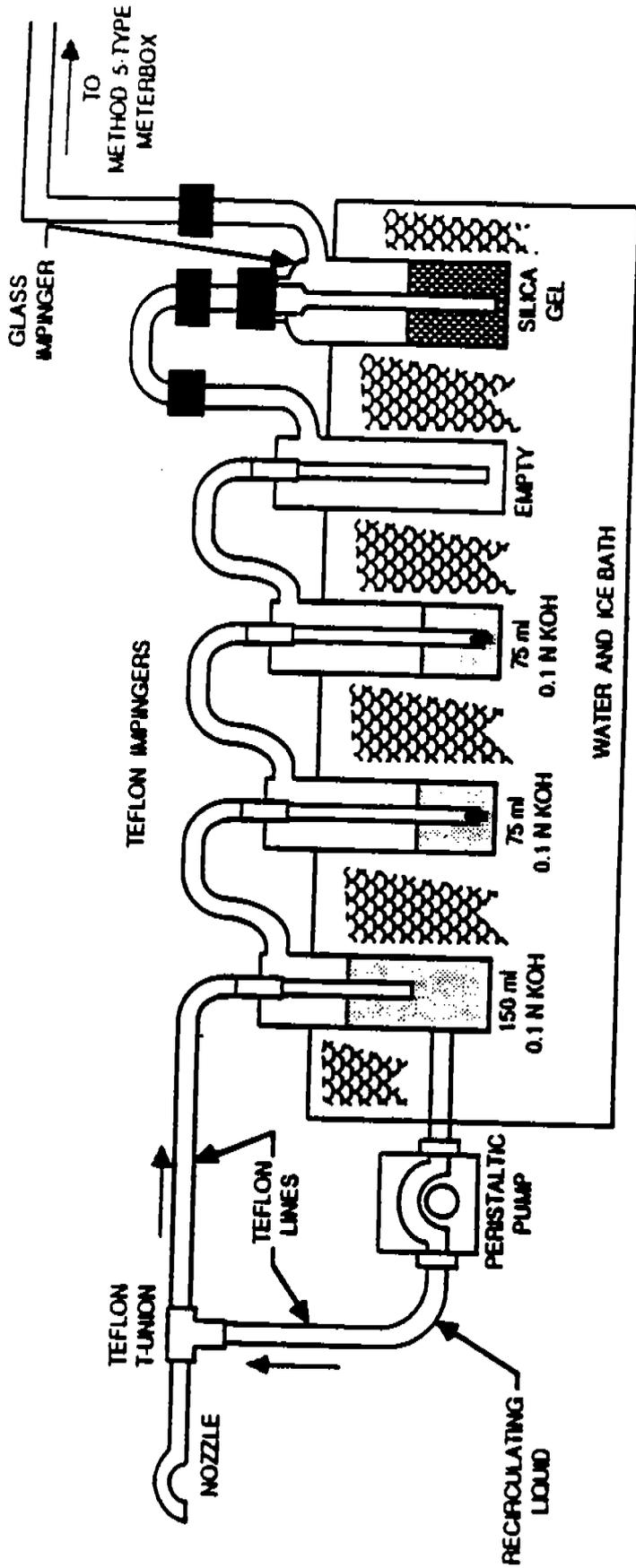


Figure 3.2-2

Schematic of recirculating impinger system with empty section for efficiency

3.2.3.1.1 Probe Nozzle. Glass or Teflon with a sharp, tapered leading edge. The angle of taper shall be  $\leq 30^\circ$  and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator.

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (1/8 to 1/2 in) (or larger if higher volume sample trains are used) inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in). Each nozzle shall be calibrated according to the procedures outlined in Section 3.2.6.

3.2.3.1.2 Teflon Aspirator or Pump/Sprayer Assembly. Teflon aspirator capable of recirculating absorbing reagent at 50 ml/min while operating at 0.75 cfm. Alternatively, a pump/sprayer assembly may be used instead of the Teflon aspirator. A Teflon union-T is connected behind the nozzle to provide the absorbing reagent/sample gas mix; a peristaltic pump is used to recirculate the absorbing reagent at a flow rate of at least 50 ml/min. Teflon fittings, Teflon ferrules, and Teflon nuts are used to connect a glass or Teflon nozzle, recirculating line, and sample line to the Teflon aspirator or union-T. Tygon, C-flex\*\* or other suitable inert tubing for use with peristaltic pump.

3.2.3.1.3 Teflon Sample Line. Teflon, 3/8" outside diameter (OD) and 1/4" inside diameter (ID), or 1/2" OD x 3/8" ID, of suitable length to connect aspirator (or T-union) to first Teflon impinger.

3.2.3.1.4 Teflon Recirculation Line. Teflon, 1/4" O.D. and 1/8" I.D., of suitable length to connect first impinger to aspirator (or T-union).

3.2.3.1.5 Teflon Impingers. Four Teflon Impingers; Teflon tubes and fittings, such as made by Savillex\*\*, can be used to construct impingers

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\*\*NOTE: Mention of trade names or specific product does not constitute endorsement by the Environmental Protection Agency.

.2" diameter by 12" long, with vacuum-tight 3/8" O.D. Teflon compression fittings. Alternatively, standard glass impingers that have been Teflon-lined, with Teflon stems and U-tubes, may be used. Inlet fittings on impinger top to be bored through to accept 3/8" O.D. tubing as impinger stem. The second and third 3/8" OD Teflon stem has a 1/4" OD Teflon tube, 2" long, inserted at its end to duplicate the effects of the Greenburg-Smith impinger stem. The first impinger stem should extend 2" from impinger bottom, high enough in the impinger reagent to prevent air from entering recirculating line; the second and third impinger stems should extent to 1/2" from impinger bottom. The first impinger should include a 1/4" O.D. Teflon compression fitting for recirculation line. The fourth impinger serves as a knockout impinger.

3.2.3.1.6 Glass Impinger. Silica gel impinger. Vacuum-tight impingers, capable of containing 400 g of silica gel, with compatible fittings. The silica gel impinger will have a modified stem (1/2" ID at tip of stem).

3.2.3.1.7 Thermometer, (identical to that specified by Method 5) at the outlet of the silica gel impinger, to monitor the exit temperature of the gas.

3.2.3.1.8 Metering System, Barometer, and Gas Density Determinations Equipment. Same as Method 5, Sections 2.1.8 through 2.1.10, respectively.

3.2.3.2 Sample Recovery. Clean all items for sample handling or storage with 10% nitric acid solution by soaking, where possible, and rinse thoroughly with DI water before use.

3.2.3.2.1 Nitrogen Purge Line. Inert tubing and fittings capable of delivering 0 to 1 scf/min (continuously adjustable) of nitrogen gas to the impinger train from a standard gas cylinder (see Figure ~~C-3~~<sup>3.2-3</sup>). Standard 3/8-inch Teflon tubing and compression fittings in conjunction with an adjustable pressure regulator and needle valve may be used.

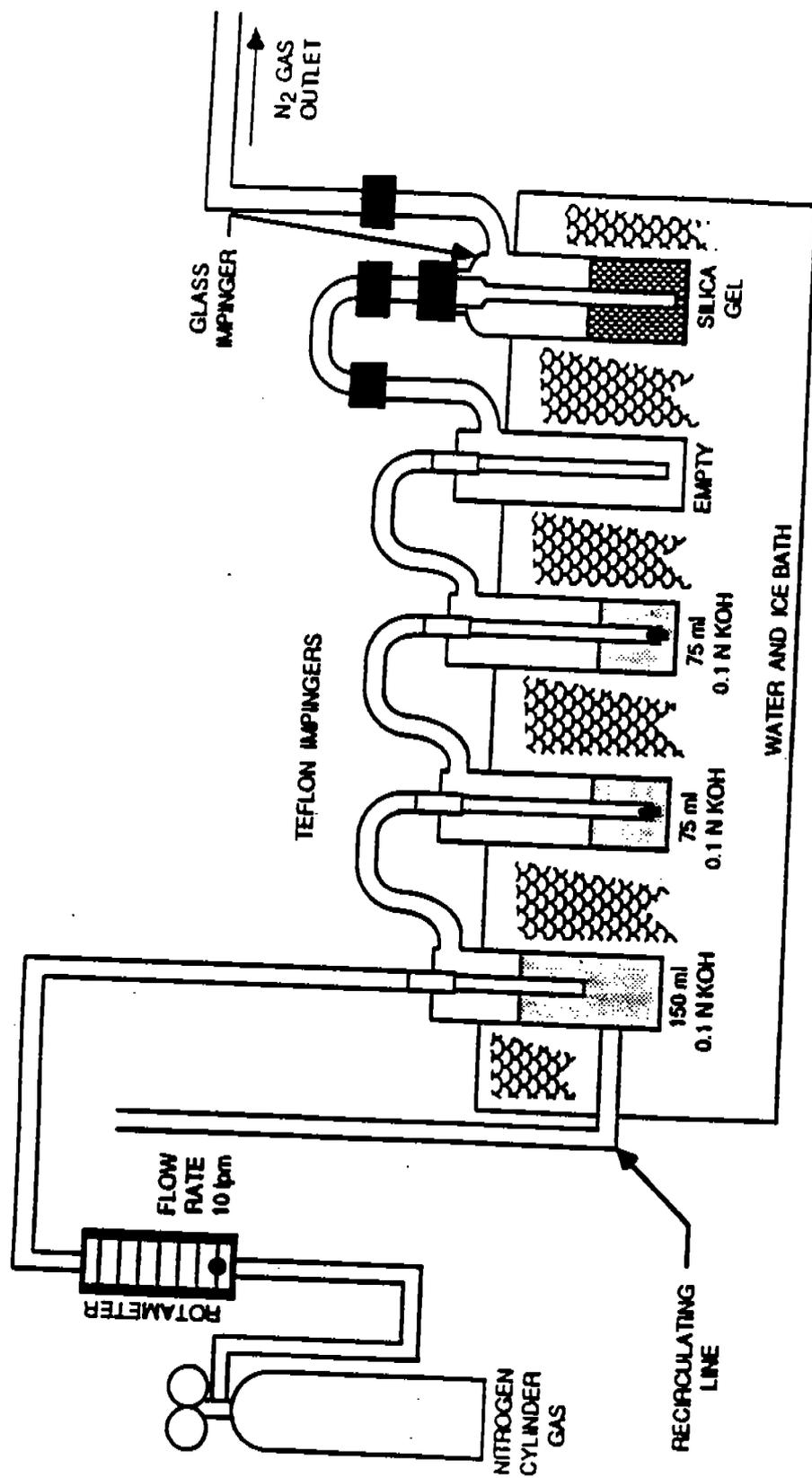


Figure 3.2-3  
Schematic of post test microwave reactor

3.2.3.2.2 Wash Bottles. Two polyethylene wash bottles, for DI water and nitric rinse solution.

3.2.3.2.3 Sample Storage Containers. Polyethylene, with leak-free screw cap, 500-ml or 1000-ml.

3.2.3.2.4 1000-ml Graduated Cylinder.

3.2.3.2.5 Plastic Storage Containers. Air tight containers to store silica gel.

3.2.3.2.6 Funnel and Rubber Policeman. To aid in transfer of silica gel from impinger to storage container; not necessary if silica gel is weighed directly in the impinger.

3.2.3.2.7 Balance

3.2.3.3 Sample Preparation for Analysis. Sample preparation prior to analysis includes purging the sample train immediately following the sample run, and filtering the recovered sample to remove particulate matter immediately following recovery.

3.2.3.3.1 Beakers, Funnels, Volumetric Flasks, Volumetric Pipets, and Graduated Cylinders. Assorted sizes, Teflon or glass, for preparation of samples, sample dilution, and preparation of calibration standards. Prepare initially following procedure described in Section 3.2.5.1.3 and rinse between use with 0.1 N HNO<sub>3</sub> and DI water.

3.2.3.3.2 Filtration Apparatus. Teflon, or equivalent, for filtering samples, and Teflon filter holder. Teflon impinger components have been found to be satisfactory as a sample reservoir for pressure filtration using nitrogen.

#### 3.2.3.4 Analysis.

3.2.3.4.1 IC/PCR System. High performance liquid chromatograph pump, sample injection valve, post-column reagent delivery and mixing system, and a visible detector, capable of operating at 520 nm, all with a non-metallic (or inert) flow path. An electronic recording integrator operating in the peak area mode is recommended, but other recording devices and integration techniques are acceptable provided the repeatability criteria and the linearity criteria for the calibration curve described in Section 3.2.5.5 can be satisfied. A sample loading system will be required if preconcentration is employed.

3.2.3.4.2 Analytical Column. A high performance ion chromatograph (HPIC) non-metallic column with anion separation characteristics and a high loading capacity designed for separation of metal chelating compounds to prevent metal interference. Resolution described in Section 3.2.5.4 must be obtained. A non-metallic guard column with the same ion-exchange material is recommended.

3.2.3.4.3 Preconcentration Column. An HPIC non-metallic column with acceptable anion retention characteristics and sample loading rates as described in Section 3.2.5.5.

3.2.3.4.4 0.45  $\mu\text{m}$  filter cartridge. For the removal of insoluble material. To be used just prior to sample injection/analysis.

#### 3.2.4 Reagents

All reagents should, at a minimum, conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. All prepared reagents should be checked by IC/PCR analysis for  $\text{Cr}^{+6}$  to ensure that contamination is below the analytical detection limit for direct injection or, if selected, preconcentration. If total chromium is also to be determined, the reagents

should also be checked by the analytical technique selected to ensure that contamination is below the analytical detection limit.

#### 3.2.4.1 Sampling.

3.2.4.1.1 Water. Deionized water. It is recommended that water blanks be checked prior to preparing sampling reagents to ensure that the  $\text{Cr}^{+6}$  content is less than the analytical detection limit.

3.2.4.1.2 Potassium Hydroxide, 0.1 N. Add 5.6 gm of  $\text{KOH}(s)$  to approximately 900 ml of DI water and let dissolve. Dilute to 1000 ml with DI water. NOTE: At sources with high concentrations of acids and/or  $\text{SO}_2$ , the concentration of  $\text{KOH}$  should be increased to 0.5 N to ensure that the pH of the solution is above 8.5 after sampling.

3.2.4.1.3 Silica Gel and Crushed Ice. Same as Method 5, Sections 3.1.2 and 3.1.4, respectively.

3.2.4.2 Sample Recovery. The reagents used in sample recovery are as follows:

3.2.4.2.1 Water. Same as Subsection 3.2.4.1.1.

3.2.4.2.2 Nitric Acid, 0.1 N. Add 6.3 ml of concentrated  $\text{HNO}_3$  (70 percent) to a graduated cylinder containing approximately 900 ml of DI water. Dilute to 1000 ml with DI water, and mix well.

3.2.4.2.3 pH Indicator Strip. pH indicator capable of determining pH of solution between the pH range of 7 and 12, at 0.5 pH intervals.

#### 3.2.4.3 Sample Preparation

3.2.4.3.1 Water. Same as Subsection 3.2.4.1.1.

3.2.4.3.2 Nitric Acid, 0.1 N. Same as Subsection 3.2.4.2.2.

U.S. Environmental Protection Agency  
Atmospheric Research and Exposure Assessment Laboratory  
Quality Assurance Division  
Source Branch, Mail Drop 77-A  
Research Triangle Park, North Carolina 27711

The audit sample should be prepared in a suitable sample matrix at a concentration similar to the actual field samples.

### 3.2.5 Procedure

SAFETY FIRST - WEAR SAFETY GLASSES AT ALL TIMES DURING THIS TEST METHOD.

3.2.5.1 Sampling. The complexity of this method is such that to obtain reliable results, testers should be trained and experienced with test procedures.

3.2.5.1.1 Pretest Preparation. All components shall be maintained and calibrated according to the procedures described in APTD-0576, unless otherwise specified herein.

Rinse all sample train components from the glass nozzle up to the silica gel impinger and sample containers with hot tap water followed by washing with hot soapy water. Next, rinse the train components and sample containers three times with tap water followed by three rinses with DI water. All the components and containers should then be soaked overnight, or a minimum of 4 hours, in a 10 percent (v/v) nitric acid solution, then rinsed three times with DI water. Allow the components to air dry prior to covering all openings with Parafilm, or equivalent.

3.2.5.1.2 Preliminary Determinations. Same as Method 5, Section 4.1.2.

3.2.5.1.3 Preparation of Sampling Train. Measure 300 ml of 0.1 N KOH into a graduated cylinder (or tare-weighed precleaned polyethylene

container). Place approximately 150 ml of the 0.1 N KOH reagent in the first Teflon impinger. Split the rest of the 0.1 N KOH between the second and third Teflon impingers. The next Teflon impinger is left dry. Place a preweighed 200- to 400-g portion of indicating silica gel in the final glass impinger. (For sampling periods in excess of two hours, or for high moisture sites, 400-g of silica gel is recommended.)

Retain reagent blanks of the 0.1 N KOH equal to the volumes used with the field samples.

3.2.5.1.4 Leak-Check Procedures. Follow the leak-check procedures given in Method 5, Section 4.1.4.1 (Pretest Leak-Check), Section 4.1.4.2 (Leak-Checks During the Sample Run), and Section 4.1.4.3 (Post-Test Leak-Checks).

3.2.5.1.5 Sampling Train Operation. Follow the procedures given in Method 5, Section 4.1.5. The sampling train should be iced down with water and ice to ensure heat transfer with the Teflon impingers.

NOTE: If the gas to be sampled is above 200°F, it may be necessary to wrap three or four feet of the Teflon sample and recirculating lines inside the ice bath to keep the recirculated reagent cool enough so it does not turn to steam.

For each run, record the data required on a data sheet such as the one shown in Figure 5-2 of Method 5.

At the end of the sampling run, determine the pH of the reagent in the first impinger using a pH indicator strip. The pH of the solution shall be greater than 8.5.

3.2.5.1.6 Calculation of Percent Isokinetic. Same as Method 5, Section 4.1.6.

3.2.5.2 Post-Test Nitrogen Purge. The nitrogen purge is used as a safeguard against the conversion of hexavalent chromium to the trivalent oxidation state. The purge is effective in the removal of SO<sub>2</sub> from the impinger contents.

Attach the nitrogen purge line to the input of the impinger train. Check to ensure the output of the impinger train is open, and that the recirculating line is capped off. Open the nitrogen gas flow slowly and adjust the delivery rate to 10 L/min. Check the recirculating line to ensure that the pressure is not forcing the impinger reagent out through this line. Continue the purge under these conditions for one-half hour, periodically checking the flow rate.

3.2.5.3 Sample Recovery. Begin cleanup procedures as soon as the train assembly has been purged at the end of the sampling run. The probe assembly may be disconnected from the sample train prior to sample purging.

The probe assembly should be allowed to cool prior to sample recovery. Disconnect the umbilical cord from the sample train. When the probe assembly can be safely handled, wipe off all external particulate matter near the tip of the nozzle, and cap the nozzle prior to transporting the sample train to a cleanup area that is clean and protected from the wind and other potential causes of contamination or loss of sample. Inspect the train before and during disassembly and note any abnormal conditions.

3.2.5.3.1 Container No. 1 (Impingers 1 through 3). Disconnect the first impinger from the second impinger and disconnect the recirculation line from the aspirator or peristaltic pump. Drain the Teflon impingers into a precleaned graduated cylinder or tare-weighted precleaned polyethylene sample container and measure the volume of the liquid to within 1 ml or 1 g. Record the volume of liquid present as this information is required to calculate the moisture content of the flue gas sample. If necessary, transfer the sample from the graduated cylinder to a precleaned polyethylene sample container. With DI water, rinse four times the insides of the glass nozzle, the aspirator, the sample and recirculation lines, the impingers, and the connecting

tubing, and combine the rinses with the impinger solution in the sample container.

3.2.5.3.2 Container No. 2 (HNO<sub>3</sub> rinse optional for total chromium). With 0.1 N HNO<sub>3</sub>, rinse three times the entire train assembly, from the nozzle to the fourth impinger and combine the rinses into a separate precleaned polyethylene sample container for possible total chromium analysis. Repeat the rinse procedure a final time with DI water, and discard the water rinses. Mark the height of the fluid level on the container or, alternatively if a balance is available, weigh the container and record the weight to permit determination of any leakage during transport. Label the container clearly to identify its contents.

3.2.5.3.3 Container No. 3 (Silica Gel). Note the color of the indicating silica gel to determine if it has been completely spent. Quantitatively transfer the silica gel from its impinger to the original container, and seal the container. A funnel and a rubber policeman may be used to aid in the transfer. The small amount of particulate that may adhere to the impinger wall need not be removed. Do not use water or other liquids to transfer the silica gel. Alternatively, if a balance is available in the field, record the weight of the spent silica gel (or the silica gel plus impinger) to the nearest 0.5 g.

3.2.5.3.4 Container No. 4 (0.1 N KOH Blank). Once during each field test, place a volume of reagent equal to the volume placed in the sample train into a precleaned polyethylene sample container, and seal the container. Mark the height of the fluid level on the container or, alternatively if a balance is available, weigh the container and record the weight to permit determination of any leakage during transport. Label the container clearly to identify its contents.

3.2.5.3.5 Container No. 5 (DI Water Blank). Once during each field test, place a volume of DI water equal to the volume employed to rinse the sample train into a precleaned polyethylene sample container, and seal the container. Mark the height of the fluid level on the container or, alterna-

tively if a balance is available, weigh the container and record the weight to permit determination of any leakage during transport. Label the container clearly to identify its contents.

3.2.5.3.6 Container No. 6 (0.1 N HNO<sub>3</sub> Blank). Once during each field test if total chromium is to be determined, place a volume of 0.1 N HNO<sub>3</sub> reagent equal to the volume employed to rinse the sample train into a pre-cleaned polyethylene sample container, and seal the container. Mark the height of the fluid level on the container or, alternatively if a balance is available, weigh the container and record the weight to permit determination of any leakage during transport. Label the container clearly to identify its contents.

3.2.5.4 Sample Preparation. For determination of Cr<sup>+6</sup>, the sample should be filtered immediately following recovery to remove any insoluble matter. Nitrogen gas may be used as a pressure assist to the filtration process (see Figure Cr<sup>+6</sup>-4).

Filter the entire impinger sample through a 0.45-micrometer acetate filter (or equivalent), and collect the filtrate in a 1000-ml graduated cylinder. Rinse the sample container with DI water three separate times, pass these rinses through the filter, and add the rinses to the sample filtrate. Rinse the Teflon reservoir with DI water three separate times, pass these rinses through the filter, and add the rinses to the sample. Determine the final volume of the filtrate and rinses and return them to the rinsed polyethylene sample container. Label the container clearly to identify its contents. Rinse the Teflon reservoir once with 0.1 N HNO<sub>3</sub> and once with DI water and discard these rinses.

If total chromium is to be determined, quantitatively recover the filter and residue and place them in a vial. (The acetate filter may be digested with 5 ml of 70 percent nitric acid; this digestion solution may then be diluted with DI water for total chromium analysis.)

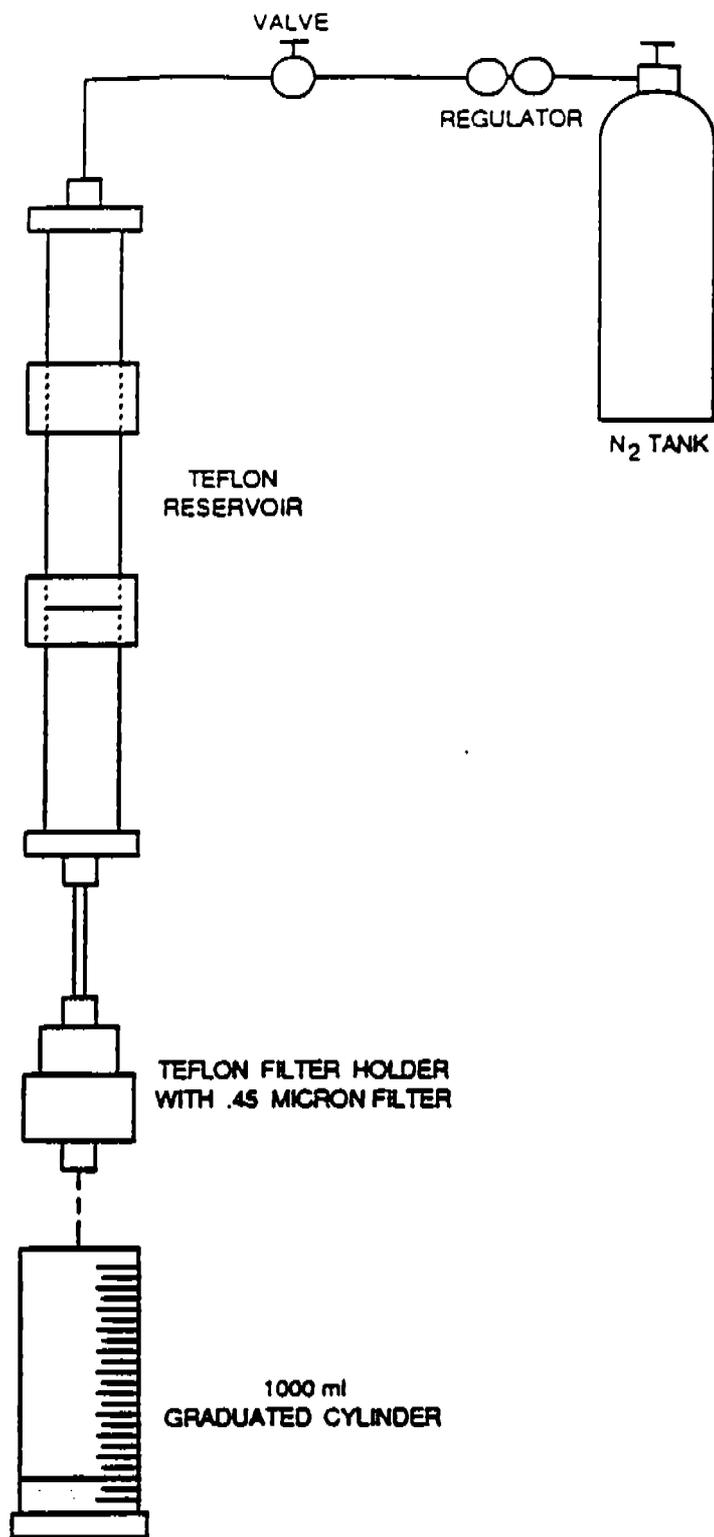


Figure 3.2-4 Schematic of sample filter system.

NOTE: If the source has a large amount of particulate in the effluent stream, testing teams may wish to filter the sample twice, once through a 2 to 5-micrometer filter, and then through the 0.45-micrometer filter.

3.2.5.4.1 Container 2 (HNO<sub>3</sub> rinse, optional for total chromium). This sample shall be analyzed in accordance with the selected procedure for total chromium analysis. At a minimum, the sample should be subjected to a digestion procedure sufficient to solubilize all chromium present.

3.2.5.4.2 Container 3 (Silica Gel). Weigh the spent silica gel to the nearest 0.5 g using a balance. (This step may be conducted in the field.)

3.2.5.5 Sample analysis. The Cr<sup>+6</sup> content of the sample filtrate is determined by ion chromatography coupled with a post-column reactor (IC/PCR). To increase sensitivity for trace levels of chromium, a preconcentration system is also used in conjunction with the IC/PCR.

Prior to preconcentration and/or analysis, all field samples will be filtered through a 0.45- $\mu$  filter. This filtration should be conducted just prior to sample injection/analysis.

The preconcentration is accomplished by selectively retaining the analyte on a solid absorbent (as described in 3.2.3.4.3), followed by removal of the analyte from the absorbent. The sample is injected into a sample loop of the desired size (repeated loadings or larger size loop for greater sensitivity) and the Cr<sup>+6</sup> is collected on the resin bed of the column. When the injection valve is switched, the eluent displaces the concentrated Cr<sup>+6</sup> sample moving it off the preconcentration column and onto the IC anion separation column. After separation from other sample components, Cr<sup>+6</sup> forms a specific complex in the post-column reactor with a diphenylcarbazide reaction solution, and the complex is then detected by visible absorbance at a wavelength of 520 nm. The amount of absorbance measured is proportional to the concentration of the Cr<sup>+6</sup> complex formed. The IC retention time and absorbance of the Cr<sup>+6</sup> complex is compared with known Cr<sup>+6</sup> standards analyzed

under identical conditions to provide both qualitative and quantitative analyses.

Prior to sample analysis, establish a stable baseline with the detector set at the required attenuation by setting the eluent flowrate at approximately 1 ml/min and post-column reagent flowrate at approximately 0.5 ml/min. (Note: As long as the ratio of eluent flowrate to PCR flowrate remains constant, the standard curve should remain linear.) Inject a sample of DI water to ensure that no Cr<sup>+6</sup> appears in the water blank.

First, inject the calibration standards prepared, as described in Section 3.2.4.4.4, to cover the appropriate concentration range, starting with the lowest standard first. Next, inject, in duplicate, the performance audit sample, followed by the 0.1 N KOH field blank and the field samples. Finally, repeat the injection of the calibration standards to allow for compensation of instrument drift. Measure areas or heights of the Cr<sup>+6</sup>/DPC complex chromatogram peak. The response for replicate, consecutive injections of samples must be within 5 percent of the average response, or the injection should be repeated until the 5 percent criterion can be met. Use the average response (peak areas or heights) from the duplicate injections of calibration standards to generate a linear calibration curve. From the calibration curve, determine the concentration of the field samples employing the average response from the duplicate injections.

The results for the analysis of the performance audit sample must be within 10 percent of the reference value for the field sample analysis to be valid.

3.2.6 Calibration. Maintain a written log of all calibration activities.

3.2.6.1 Sample Train Calibration. Calibrate the sample train components according to the indicated sections of Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering System (Section 5.3); Temperature Gauges (Section 5.5); Leak-Check of the Metering System (Section 5.6); and Barometer (Section 5.7).

3.2.6.2 Calibration Curve for the IC/PCR. Prepare working standards from the stock solution described in Section 3.2.4.4.4 by dilution with a DI water solution to approximate the field sample matrix. Prepare at least four standards to cover one order of magnitude that bracket the field sample concentrations. Run the standards with the field samples as described in Section 3.2.5.5. For each standard, determine the peak areas (recommended) or the peak heights, calculate the average response from the duplicate injections, and plot the average response against the  $\text{Cr}^{+6}$  concentration in  $\mu\text{g/L}$ . The individual responses for each calibration standard determined before and after field sample analysis must be within 5 percent of the average response for the analysis to be valid. If the 5 percent criteria is exceeded, excessive drift and/or instrument degradation may have occurred, and must be corrected before further analyses are performed.

Employing linear regression, calculate a predicted value for each calibration standard with the average response for the duplicate injections. Each predicted value must be within 7 percent of the actual value for the calibration curve to be considered acceptable. If not acceptable, remake and/or rerun the calibration standards. If the calibration curve is still unacceptable, reduce the range of the curve.

### 3.2.7 Calculations

3.2.7.1 Dry Gas Volume. Using the data from the test, calculate  $V_{m(\text{std})}$ , the dry gas sample volume at standard conditions as outlined in Section 6.3 of Method 5.

3.2.7.2 Volume of Water Vapor and Moisture Content. Using the data from the test, calculate  $V_{w(\text{std})}$  and  $B_{wg}$ , the volume of water vapor and the moisture content of the stack gas, respectively, using Equations 5-2 and 5-3 of Method 5.

3.2.7.3 Stack Gas Velocity. Using the data from the test and Equation 2-9 of Method 2, calculate the average stack gas velocity.

3.2.7.4 Total  $\mu\text{g Cr}^{+6}$  per Sample. Calculate as described below:

$$m = (S-B) \times V_{1s} \times d$$

where:

- m - Mass of  $\text{Cr}^{+6}$  in the sample,  $\mu\text{g}$ .
- S - Concentration of sample,  $\mu\text{g Cr}^{+6}/\text{ml}$ .
- B - Concentration of blank,  $\mu\text{g Cr}^{+6}/\text{ml}$ .
- $V_{1s}$  - Volume of sample after filtration, ml.
- d - Dilution factor (1 if not diluted).

$$PR \left[ \frac{A \cdot hr}{m} + \frac{1 \text{ min}}{x \text{ hrs}} \right] = \frac{A \cdot hr}{hr}$$

	Hexavalent (Cr <sup>+6</sup> )		Emis Fact (lb/A·hr)
	Emis Rate (lb/hr)	Prod Rate (A·hr/hr)	
1	0.0092	38074 ÷ 1.08 hr → 35254	2.61 × 10 <sup>-7</sup>
2	0.0034	37554 ÷ 1.07 hr → 35097	9.89 × 10 <sup>-8</sup>
3	0.0062	37749 ÷ 1.09 hr → 35279	1.76 × 10 <sup>-7</sup>
Avg	<u>0.0063</u>	<u>35210</u> → 1.79 × 10 <sup>-7</sup>	<u>1.78 × 10<sup>-7</sup></u>

1	65 min	$\frac{hr}{60m}$	1.08 hr
2	64 min		1.07 hrs
3	64		1.07 hrs

NC0005

$$1257 \rightarrow 2.01$$

$$\frac{61}{3} \\ 64$$

	Total Chromium (Cr)		Emis Fact (lb/A·hr)
	Emis Rate (lb/hr)	Prod Rate (A·hr/hr)	
1	0.0110	35254	
2	0.0042	35097	
3	0.0273	35279	
Avg	<u>0.0142</u>	<u>35210</u>	<u>1.17 × 10<sup>-6</sup> lb/A·hr</u>

Source category: Electroplating  
 Plant name : U. S. Chrome Corporation  
 Process : Hard chromium electroplating

Filename: REF\_4-71.WQ1  
 Location: Batavia, NY  
 Test date: 10/2/91

Date: 03/07/96  
 Ref. No.: 4-71  
 Process rate basis: production

Source	Type of control	Pollutant	Run No.	Test Method	Samp. time, min	Isokinetic, %	Gas volume, DSCF	Volum. flow rate, DSCFM	Mass, ug	Concen., gf/DSCF	Emission rate, lb/hr	Process rate, A-hr	Emission factor		
													mg/A-hr	Rat.	
Electroplating tank	PBS	total Cr	1	EPA 13	64	96.2	39.20	16,355	199.82	7.87E-05	0.01103	38,074	0.14	0.0022	
		total Cr	2		64	100.7	40.11	15,980	78.80	3.03E-05	0.0042	37,554	0.054	0.00083	
		total Cr	3		64	100.7	40.91	16,160	518.15	1.95E-04	0.02707	37,749	0.35	0.0054	
											Average		0.18	0.0028	
			Cr+6	1	EPA 13	64	96.2	39.2	16,355	167.20	6.58E-05	0.00923	38,074	0.12	0.0018
			Cr+6	2		64	100.7	40.1	15,980	64.80	2.49E-05	0.00342	37,554	0.044	0.00068
			Cr+6	3		64	100.7	40.9	16,160	117.20	4.42E-05	0.00612	37,749	0.078	0.0012
											Average		0.080	0.0012	
															A

Basis for rating:  
 Problems noted:  
 Other notes:

Report does not specify type of electroplating, but based on emission rate and type of control, it appears to be HARD chromium.  
 Report indicates that sampling location was PBS inlet, but the concentrations and emission factors are consistent with PBS-controlled emissions.

*According to Sherron Vogel*  
*- Hard chromium*  
*- contact at plant*  
*JMC (USA)*  
*John Baker*  
*549-4150*

CONTACT REPORT--MRI Project No. 4603-01-05

From: Richard Marinshaw, Environmental Engineering  
Department

Date of Contact: April 29, 1996

Contacted by: Telephone

Company/Agency: JMC (USA), Inc.  
Post Office Box 12138  
Research Triangle Park, NC 27709

Telephone Number: (919) 549-4150

Person(s) Contacted/Title(s)

John Baker, Vice President of Research and Research Applications

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CONTACT SUMMARY:

Mr. Baker was contacted for additional information on the emission test conducted on the high purity chromium metal production process at the U.S. Chrome Corporation Facility in Batavia, New York, on October 2, 1991. The report was submitted by JMC (USA), Inc. (JMC) as part of their permit application for their facility in Research Triangle Park, North Carolina.

Mr. Baker stated that the high purity chromium metal production process is similar to hard chromium electroplating. However, in the high purity chromium metal process, the cathode is plated to a thickness of 3 to 4 millimeters over a period of 7 days. The high purity chromium metal then is stripped off the cathode and used in the electronics industry. Emissions from the plating tank were controlled with a mesh pad mist eliminator followed by a packed bed scrubber. Emissions were measured at the scrubber inlet.