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Background Report Reference

AP-42 Section Number: 12.20

Background Chapter: 4

Reference Number: 38

Title: State of California Air Resources
Board Test of Standard Nickel-
Chromium Plating Company

April 1987

State of California AIR RESOURCES BOARD test of ~~Standard Nickel-Chromium~~
~~Plating Company~~ - Test Report C-86-085

UNACCEPTABLE

On the first page of this report, the first sentence of the first paragraph says, "This Report presents an estimate of emissions from Standard Nickel-Chromium Plating Company." The report provides an estimate and that is all. Testing was not done in accordance with procedures that would have been accepted by the Emission Measurement Branch for a source test whose data might be used by ISB.

In looking at the velocity traverse sheets, the inlet duct diameter is given at 19 inches. The text of the report gives 18 inches. The duct length for the inlet is too short to meet Reference Method 1 requirements. We have had this problem with our testing as well. On the outlet, a 5-foot extension was added. The drawing in the report does not make it possible to determine precisely where the outlet ports are placed; however, if the correct location was chosen, the total number of sampling points would have been 25. Only 16 points were actually sampled. The number of sample points at the inlet was also insufficient, 16 total points being sample instead of 24.

When sampling for chromium emissions, experience by the EMB has shown that the distribution of chromic acid is not necessarily uniform throughout the stack gas. It is important, then, to sample the correct number of points in order to obtain accurate data. Since this report was merely to provide an "estimate" of emissions, this is a possible reason for not enough sample points being chosen.

EMISSIONS STACK TESTING AT MCDONNELL DOUGLAS,
TORRANCE, CALIFORNIA

Chromium Emissions Tests for Tank 193, Bldg. 2 (Etching Tank Scrubber) and Tank 195, Bldg 2 (Anodizing Tank Scrubber) at Douglas Aircraft Company.

UNACCEPTABLE

This report has discrepancies between the field data sheets and the computer printouts and it appears that a minimal effort was made for the entire project.

From looking at the report, it is possible to conclude that single point sampling was done in order to obtain the data. Both ducts were rectangular, but information on duct length and port location along the duct is missing. Only two ports were placed into each duct which would not allow for the proper matrix of sampling points as specified by Reference Method 1. Although a velocity traverse was made, field data sheets for the sample runs show constant stack and meter box parameters, and the absence of a sampling nozzle diameter in the report indicates that only a piece of Teflon tubing was used to extract the sample. Although the text at one point refers to a "Method 5" train, sampling was not done in accordance with Method 5 procedures.

The text says that sampling was done proportionally. The correct size nozzle for Run 1, Scrubber 1, Sparger On is 0.25. Using the data given in the report and performing isokinetic calculations using the three most frequently used nozzle sizes of 0.1875, 0.25, and 0.3125, the isokinetic rates for this run are 153.3, 86.2, and 55.2 percent respectively. Although the report says that proportional sampling was done, the data sheets show that the sampling rate was constant and there is no indication of traversing.

This report was prepared for two attorneys. The report was probably used in a court case and data were needed, but no one involved knew enough to determine if the data gathered were accurate or properly obtained. The report provides a "quick and dirty and not too accurate estimate" of the emissions from a single point. Previous testing of chromium sources by the EMB has shown that chromium emissions from a source cannot be accurately determined by sampling a single point. Thus, the report does not provide accurate emission data for the source tested.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Air Quality Planning and Standards
Research Triangle Park, North Carolina 27711

MEMORANDUM

SUBJECT: Acceptability of Test Reports

FROM: Frank R. Clay *FRC*
Field Testing Section, EMB, TSD (MD-14)

TO: Andrew Smith
Industrial Studies Branch, ESD (MD-13)

Enclosed are three test reports that have been reviewed. One report is acceptable and the other two are not. The reasons for accepting or rejecting each report follow:

Enclosures

ENVIRONMENTAL SOURCE SAMPLERS, INC. TEST OF THE NAVAL AIR REWORK
FACILITY AT THE NORFOLK NAVAL AIR STATION (CHROME LINE 1 DEMISTER) PERFORMED
ON NOVEMBER 22, 1986.

ACCEPTABLE

This report is acceptable, however, only total chromium analysis was performed.

At the outlet, the last two runs had moisture contents that were greater than saturation (though not by much). The same data were run again for run #2 using correct moisture content and a comparison of the emission numbers can be found in the enclosed Summary of Emissions Sheet which is on the opposite side of the Sampling Summary Sheet. The red numbers give the emission rates for incorrect moisture and the difference is slight. If the difference is significant to ISB, the report can be returned and the correct numbers calculated.

State of California
AIR RESOURCES BOARD

STANDARD NICKEL-CHROMIUM PLATING COMPANY
TOTAL AND HEXAVALENT CHROMIUM EMISSIONS

Engineering Evaluation Branch
Test Report

C-86-085

Report Date: April 6, 1987

APPROVED:

David F. Todd Project Engr.,
Testing Section

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Neen C. Linquist Chief,
Engineering Evaluation Branch

STANDARD NICKEL-CHROMIUM PLATING COMPANY
TOTAL AND HEXAVALENT CHROMIUM EMISSIONS

This report presents an estimate of emissions from Standard Nickel-Chromium Plating Company. These emission estimates are based on tests performed by the Air Resources Board (ARB) staff using a draft ARB test method. The results have been reviewed by the staff and are believed to be accurate within the limits of the method. However, data are usually affected by the test method and process variables which are sometimes not apparent during the tests. The data should not, therefore, be necessarily considered typical of a specific source or industry until the effects of such variables are known and taken into account.

ACKNOWLEDGEMENTS

The project engineer was David Jodd and the field engineer was Angus MacPherson of ARB. Instrument technicians were Jack LaBrue and Dwight Warner of ARB. The chemists were Paul Wong and Ed Jeung of the California Department of Health, Air and Industrial Hygiene Laboratory (AIHL). Assistance was provided by Doug Dulgarian, George Dulgarian, and Dean Dulgarian of Standard Nickel-Chromium Plating Company. Additional assistance was provided by Frances Cameron of the ARB Toxic Pollutants Branch. Chemical analyses were performed by AIHL.

Standard Nickel-Chromium Plating Company
Total and Hexavalent Chromium Emissions

SUMMARY

The Air Resources Board (ARB) has identified hexavalent chromium as a toxic air contaminant. Because of this, sampling was requested by the ARB Toxic Pollutants Branch to determine chromium emissions from a "hard" chromium plater. Samples were collected at the Standard Nickel-Chromium Plating Co. on November 14 through 16, 1986, by the staff of the Engineering Evaluation Branch (EEB) of the ARB. The samples were analyzed by the Department of Health Services (DOHS) Air and Industrial Hygiene Laboratory (AIHL) for total chromium and hexavalent chromium.

Chromium is applied by electroplating from a plating solution in which it is present as hexavalent chromium, an anion in a high oxidation state. Electroplating is accomplished by applying a high amperage, low voltage (4,000 amps, 4.5 volts) direct current (dc) through the plating solution. The material to be plated is hung from the negative, or cathode, bus bars of the plating tank. Anodes are hung from the positive bus bars. Chromium plating is only 8-20% efficient. A major reason for the inefficiency is that some of the applied electric current converts some of the water in the plating solution to hydrogen and oxygen gases, resulting in the release of hydrogen gas at the cathode and oxygen at the anode. A portion of the plating solution is entrained with these released gases, which are vented for safety reasons. At Standard Plating, these gases are passed through a scrubber and then vented to the atmosphere. The scrubber is used to recover some of the vented plating solution and return it to the plating tank.

Sampling and analysis for hexavalent and total chromium was conducted in accordance with the ARB's Draft Test Method 425 (Determination of Total Chromium and Hexavalent Chromium Emissions from Stationary Sources). The results of the sampling and analysis and other data are shown in the Summary Table. Emissions from the plating tank ranged between 0.42×10^{-3} lb/hr and 0.85×10^{-3} lb/hr for total chromium and 0.035×10^{-3} and 0.64×10^{-3} lb/hr for hexavalent chromium. Emissions after the scrubber ranged between 0.20×10^{-3} lb/hr and 0.58×10^{-3} lb/hr for total chromium and 0.18×10^{-3} lb/hr and 0.54×10^{-3} lb/hr for hexavalent chromium. Scrubber efficiency for preventing the release of chromium ranged between 28 and 67 percent for total chromium and 15 to 62 percent for hexavalent chromium.

Summary Table
Test Data For
Standard Nickel-Chromium Plating Co.

1. Run Number	1		2		3		4	
2. Chromium Emissions	a/ Total	b/ Hex.	Total	Hex.	Total	Hex.	Total	Hex.
a. Before Scrubber, 10 ⁻³ lb/hr	0.81	0.64	0.42	0.35	0.85	0.55	0.60	0.47
b. After Scrubber, 10 ⁻³ lb/hr	0.58	0.54	0.28	0.20	7.14 ^{c/}	0.47	0.20	0.18
c. Scrubber Efficiency, percent	28	16	33	43	d/ NC	15	67	62
3. Chromium in Scrubber Water								
a. Total, ug/ml	7383		43.6		16.0		9.9	
b. Hexavalent, ug/ml	21.1		33.8		17.0		17.0	
4. Plating Tank Data								
a. Tank Temperature, degrees F	140		140		140		140	
b. Tank Surface Area, sq. ft.	10.6		10.6		10.6		10.6	
c. Electroplating Amps	4000		4000		4000		4000	
d. Chromic Acid Conc., oz/gal	32		32		32		32	

a/ Total - Total chromium including hexavalent chromium

b/ Hex. - Hexavalent chromium

c/ For this sample, the laboratory reported 900 ug total chromium in the impinger catch and that is 91% of the total reported sample. For all other samples, the impinger catch was less than 5 ug and represented between 0.3 and 11 % of the sample totals.

d/ NC - Not Calculated

Standard Nickel-Chromium Plating Company
Total and Hexavalent Chromium Emissions

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I. INTRODUCTION

The Air Resources Board (ARB) has identified hexavalent chromium as a toxic air contaminant. Subsequently, the Toxic Pollutants Branch of the ARB requested the Engineering Evaluation Branch (EEB) of the ARB conduct an emissions test to evaluate hexavalent chromium emissions from a "hard" chromium plating operation. The Standard Nickel-Chromium Plating Company in Los Angeles was selected for testing and testing was conducted on November 14 through 16, 1986. The samples were analyzed for total and hexavalent chromium by the Air and Industrial Hygiene Laboratory (AIHL) of the Department of Health Services. The objectives of this test were to:

1. Determine total and hexavalent chromium emissions from a hard chromium plater.
2. Determine the efficiency of the wet scrubber used by Standard Plating for controlling chromium emissions.
3. Determine if it is necessary to stabilize the sample filters immediately after sampling as part of the test method development.

At different times during the ARB evaluation test, parallel samples were collected by the South Coast Air Quality Management District (AQMD) and Truesdail Laboratories. Truesdail Laboratories is a contract laboratory commissioned for the Metal Finishing Association of Southern California Inc. Matt Dunn of Dames and Moore, Santa Barbara, observed the evaluation test for the Metal Finishing Association.

II. Conclusions

Emissions of total chromium (including hexavalent chromium at Standard Plating ranged from 0.42×10^{-3} lb/hr to 0.85×10^{-3} lb/hr before the scrubber and 0.20×10^{-3} lb/hr to 0.58×10^{-3} lb/hr after the scrubber. Emissions of hexavalent chromium at Standard Plating ranged from 0.35×10^{-3} lb/hr to 0.64×10^{-3} lb/hr before the scrubber and 0.18×10^{-3} lb/hr to

0.54×10^{-3} lb/hr after the scrubber. Scrubber efficiency ranged from 28 to 67 percent in removing total chromium and 15 to 62 percent in removing hexavalent chromium from the exhaust.

The number of samples was too limited to determine if treating the sample filters on site had any effect on the hexavalent chromium analytical results.

III. PROCESS DESCRIPTION

Standard Plating is classified as a "hard" chromium plater. In "hard" chromium plating, chromium is applied to steel surfaces in thicknesses of 0.0001-0.010 inches to build up worn parts and provide abrasion resistant surfaces such as those seen on hydraulic rams and cylinders. "Decorative" chromium plating is the other classification where a much thinner layer (0.00001-0.00002 inches) of chromium is applied to produce a bright and decorative, as well as corrosion and abrasion resistant surface.

The chromium is deposited onto the surface of the base material by electroplating. Electroplating operations, including cleaning, rinsing, plating, and post plating treatments, can be manual or automatic. Standard Plating's, operation is basically manual to accommodate the wide variation in the parts they plate.

The part to be plated is suspended by wires or racks before being submerged into a tank containing the plating solution. At Standard Plating, the parts are suspended by wires. The chromium is deposited onto the material surface from a solution in which it is present as hexavalent chromium, an anion in a high oxidation state. Along with chromium acid, the aqueous solution usually contains a small and carefully controlled amount of catalyst

ion, such as sulfate. The necessary direct current (dc) power for electroplating is supplied by rectifiers and applied to the plating tank by bus bars. Anodes are hung from positive bus bars and the item to be plated is hung from the negative or cathode bus bars. Tank voltage and current are monitored with voltmeters and ammeters.

Chromium plating is one of the most difficult electroplating operations; temperature, current density, and bulk composition are kept within a relatively narrow range. With respect to electric current, chromium plating is only 8-20% efficient. Most of the electric current is used converting water in the plating solution into hydrogen and oxygen gases. This results in the off gassing of hydrogen at the cathode and oxygen at the anode. The release of these gases from the plating tank causes a spray that carries some of the plating solution with the gases. For safety reasons the spray must be vented. For economic reasons, the spray is usually vented through a scrubber to recover some of the plating solution. Part of the scrubber water is then used as makeup to replace some of the lost plating solution.

During the three days of emissions sampling, Standard Plating tried to maintain consistent plating operations by plating similar parts under similar conditions over the three test days. Table 1 shows the operating parameters and conditions during the 3 days of testing.

IV. SAMPLING LOCATIONS AND METHODS

Hexavalent and total chromium samples were collected according to a draft of ARB's Stationary Source Test Method 425 (Determination of Total Chromium and Hexavalent Chromium Emissions from Stationary Sources). A type S pitot tube was used to determine stack gas velocity. Moisture content was

Table 1

Operation Data During Emissions Testing
At Standard Nickel-Chromium Plating, Inc.

1.	Tank Number	50
2.	Plating Tank Data	
	a. Tank Temp	140°F
	b. Surface Area,	10.6 sq. ft.
	c. Dimensions	44 inches, diameter 38 feet, deep
3.	Plating Solution Data	
	a. Chromic Acid	32-35 oz./gallon solution
	b. Sulfuric Acid, total	0.32-0.35 oz./gallon solution
	c. Solution Volume	2700 gallons
4.	Power Data	
	a. Voltage	4.5 volts, dc
	b. Current	4,000 amperes
5.	Parts Data	
	a. Number	4
	b. Dimensions, each part	5/8 inch diameter 20 feet long
6.	Depth of Chrome Deposited	.003 inches total in 5 hours

determined as specified by ARB Method 4 (Determination of Moisture Content in Stack Gases). Dry molecular weight (CO , CO_2 , O_2 , N_2) was estimated based upon atmospheric concentrations. Figure 1 shows the sampling locations before and after the scrubber.

Draft Method 425 is similar to ARB Test Method 5 but with special requirements. Those special requirements for sampling chromium at Standard Plating included the use of glass-lined stainless steel sampling probes, special teflon coated glass-fiber filters, and specially prepared acetone and distilled water. Hexavalent chromium is very reactive and the purpose of these special requirements was to prevent the loss of hexavalent chromium after it was collected. Also, the acetone and deionized distilled water used for sample recovery were analyzed before and after sampling to document their chromium content and the presence of impurities that would react with the hexavalent chromium. Appendix A contains a copy of Draft Method 425.

For analysis, all samples were split as described in Method 425. One half was analyzed for hexavalent chromium and the other half for total chromium. For two of the sample runs (a sample run includes two sample trains, one before the scrubber and one after the scrubber), the hexavalent half of the filters were placed in an extraction solution (sodium hydroxide and sodium carbonate in water) immediately after they were collected. The hexavalent halves of the other two runs were not placed in the extraction solution until they arrived at AIHL in Berkeley. This was done to determine if the delay affected the hexavalent chromium results.

V. TEST RESULTS

The analytical results and other test data are shown in Table 2. The sample analytical results are presented according to the parts of the sampling train that were analyzed separately. In general, the larger particles are

Figure 1
Standard Plating Co.
Sample Port Location
(before and after Scrubber)

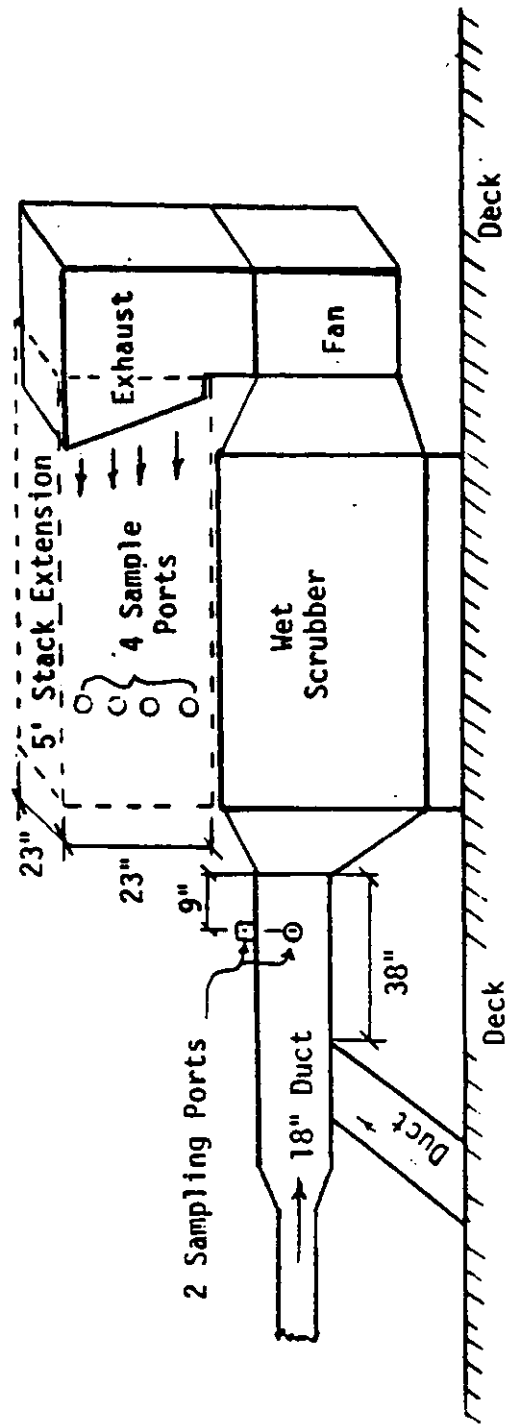


Table 2
Emissions Data For
Standard Nickel-Chromium Plating Co.

1 Run Number	1 11/4/86 PM				2 11/5/86 AM				3 11/5/86 PM				4 11/6/86 AM			
	Total		Hexavalent		Total		Hexavalent		Total		Hexavalent		Total		Hexavalent	
	BS	AS	BS	AS	BS	AS	BS	AS	BS	AS	BS	AS	BS	AS	BS	AS
3 Chromium Sample																
4 Sample Location																
5 Concentration, 10 ug/DSCF																
a. Before Filter Sample	35.39	9.42	21.78	2.32	26.56	5.31	5.88	0.12	22.46	6.93	12.70	0.54	83.35	4.50	19.50	1.01
b. Filter Sample	138.60	87.30	114.87	87.30	54.00	37.80	63.60	35.37	150.44	93.03	101.15	70.21	92.16	23.34	75.09	26.27
c. After Filter Sample	0.44	0.41	<1.08	<1.01	2.83	5.55	<1.20	<1.22	1.52	1010.62	<1.18	2.48	0.50	2.38	<1.21	<1.13
Total	176.43	97.13	136.65	89.62	83.39	48.66	69.48	35.49	174.42	1110.58	113.85	73.23	176.01	30.22	94.59	27.28
5 Stack Flow Rate, DSCFM	3517	4531	3517	4531	3800	4303	3800	4303	3653	4862	3653	4862	3747	4947	3747	4947
6 Emission Rate, 10 lb/hr	0.81	0.58	0.64	0.54	0.42	0.28	0.35	0.20	0.85	7.14	0.55	0.47	0.60	0.20	0.47	0.10
7 Scrubber Data																
a. Efficiency, %	28		16		33		43		NC		15		67		62	
b. Chromium Conc., ug/ml	7383		21.1		43.6		33.8		16.0		17.0		9.9		17.0	

a/ BS-Before Scrubber AS-After Scrubber

b/ For this sample, the laboratory reported 900 ug total chromium in the impinger catch and that is 91% of the total reported sample. For all other samples, the impinger catch was less than 5 ug and represented between 0.3 and 11 % of the sample totals.

c/ NC-Not Calculated

collected in the sampling probe before the sample filter. Finer particles pass through the probe to be collected by the filter. And still finer particles can pass through the filter to be collected in the impingers and sample lines after the filter.

Based upon the test results, overall scrubber collection efficiency for total chromium ranges between 28 and 67 percent and for hexavalent chromium, between 15 to 62 percent. The data indicates the scrubber is more efficient in collecting the larger chromium containing particles than the finer chromium containing particles. The data also indicates the use of recirculated scrubber water may be a contributing factor to the scrubber's marginal performance in controlling the finer chromium containing particles. This may be due to the presence of chromium in the water recirculated to the scrubber's sprayers. Samples of the recirculated scrubber water used in the spray contained between 9.9 to 7,383 ug of total chromium and 17.0 to 33.3 ug of hexavalent chromium per milliliter of scrubber solution (0.0013 to 0.986 ounces of total chromium and 0.0023 to 0.0044 ounces of hexavalent chromium per gallon of scrubber solution).

Results of the study to determine if treating the filters on site is effective in reducing hexavalent chromium loss are presented Table 3. Table 3 compares the concentration (ug/dscf) of hexavalent chromium collected on the treated and untreated filters. The filters with the "before scrubber" samples do not indicate there is a difference between the treated and untreated filters. The "after scrubber" filter samples indicate that the untreated filters may be losing hexavalent chromium. But because of the limited number of samples, no specific conclusions can be made.

Table 3

**Comparison of Filter Results For
Hexavalent Chromium**

RUN #	1	2	3	4	Avg.
I. Concentration, ug/DSCF					
A. Before Scrubber					
1. Treated Filter ^{a/}	1.15	0.64			0.90
2. Untreated Filter ^{a/}			1.01	0.75	0.88
B. After Scrubber					
1. Treated Filter	0.87	0.35			0.61
2. Untreated Filter			0.70	0.26	0.48

^{a/} In the field, after sampling, the hexavalent chromium half of the treated filters was treated with the sodium hydroxide and sodium carbonate extract solution. The untreated filters were not treated until later in the test week at the laboratory.

VI. QUALITY CONTROL

Hexavalent chromium is very reactive and collected in very low concentrations. The emphasis of the various quality control techniques is to prevent the loss or contamination of hexavalent chromium samples. For that reason all sample containers both in the field and the laboratory were made of glass or teflon and prewashed with specially prepared detergents, acids, and deionized distilled water. Other quality control techniques are discussed below.

A. SAMPLING

When collecting samples the only recommended probes are quartz or glass-lined probes. Any other probe material, including stainless steel, could contaminate the collected sample. Teflon coated glass-fiber filters are the recommended filter media. During laboratory sample recovery and preparation, the uncoated glass-fiber filter can release silicate from the filter material which, when acidified, becomes silicic acid. The silicic acid exists in the recovered sample as a suspended particulate that interferes with the colormetric analysis of the sample. The teflon coating greatly reduces the amount of silicate that is released from the glass fibers.

The deionized, distilled water used for impinger catches and the acetone used to rinse samples from the probes and impingers were provided by AIHL. Both were pre-analyzed for hexavalent chromium and other impurities by AIHL and stored in glass or teflon containers.

B. SAMPLE RECOVERY AND ANALYSIS

The laboratory method for recovering and analyzing the samples for hexavalent and total chromium is similar to that for ARB Method 5 samples. However, AIHL determined that parts of the Method 5 sample recovery and

analysis will cause some of the hexavalent chromium to be lost from the sample because of the reactivity of hexavalent chromium. To prevent this, the following changes were made. All solvents containing samples are reduced in volume at room temperature rather than in an oven at 105°C because the reactivity of hexavalent chromium increases with temperature. Some of the analytical reagents were treated with potassium permanganate to remove the reducing agents that would react with hexavalent chromium during sample recovery and analysis.

APPENDIX A

ARB DRAFT TEST METHOD 425

METHOD 425: DETERMINATION OF TOTAL CHROMIUM AND
HEXAVALENT CHROMIUM EMISSIONS FROM STATIONARY SOURCES

1. APPLICABILITY AND PRINCIPLE

1.1 Applicability.

This method applies to the determination of hexavalent chromium (Cr^{+6}) and total chromium emissions from stationary sources.

1.2 Principle.

Particulate emissions are collected from the source by use of CARB METHOD 5. The collected sample is divided into two equal portions with one portion used for total chromium analysis and the other portion for hexavalent chromium analysis.

1.2.1 For the hexavalent chromium analysis the collected sample portions are extracted in an alkaline solution and analyzed by the diphenylcarbazide colorimetric method.

1.2.2 For the total chromium analysis the collected samples must be prepared in order to convert organic forms of chromium to inorganic forms, to minimize organic interferences, and to convert the sample to a suitable solution for analysis. Samples are then subjected to an acid digestion procedure. Following the appropriate dissolution of the sample, a representative aliquot is placed manually or by means of an automatic sampler into a graphite tube furnace. The sample aliquot is then slowly evaporated to dryness, charred (ashed), and atomized. The absorption of hollow cathode radiation during atomization will be proportional to the chromium concentration.

2. RANGE, SENSITIVITY, PRECISION, AND INTERFERENCES

2.1 Range.

2.1.1. Hexavalent chromium. A straight line response curve was obtained in the range $1 \text{ ug Cr}^{+6}/100 \text{ mL}$ to $20 \text{ ug Cr}^{+6}/100 \text{ mL}$. For a minimum analytical accuracy of ± 10 percent, the lower limit of the range is $2 \text{ ug}/100\text{mL}$. The upper limit can be extended by appropriate dilution.

2.2 Sensitivity.

2.2.1. Hexavalent Chromium. A minimum detection limit of 1 ug Cr^{+6} /100 mL has been observed (9.1).

2.3 Precision for hexavalent chromium. The overall precision for sample collection and analysis for Cr^{+6} was determined at a ferrochrome smelter, a chemical plant, and a refractory brick plant. Replicate Method 5 filters with both high and low particulate loadings were analyzed. The relative standard deviations were less than 10 percent in all cases.

2.4 Interferences.

2.4.1. Interferences of hexavalent chromium. Molybdenum, mercury and vanadium react with diphenylcarbazide to form a color; however, approximately 20 mg of elements can be present in a sample without creating a problem. Iron produces a yellow color, but this effect is not measured photometrically at 540 nm. No interference was observed at the sources listed in Section 2.3.

2.4.2 Interferences for total chromium.

2.4.2.1. The long residence time and high concentrations of the atomized sample in the optical path of the graphite furnace can result in severe physical and chemical interferences. Furnace parameters must be optimized to minimize these effects. If the analyte is not completely volatilized and removed from the furnace during atomization, memory effects will occur. If this situation is detected, the tube should be cleaned by operating the furnace at higher atomization temperatures.

2.4.2.2. Nitrogen should not be used as the purge gas because of a possible CN band interference.

2.4.2.3. Low concentrations of calcium may cause interferences; at concentrations above 200 mg/L calcium's effect is constant. Calcium nitrate is therefore added to ensure a known constant effect.

2.5 If other techniques or conditions are used in the analysis of total chromium and/or hexavalent chromium then the tester is required to substantiate the data through an adequate quality assurance program approved by the Executive Officer.

3. APPARATUS

Any other sampling apparatus which, after review by the Executive Officer, is deemed equivalent for the purposes of this test method, may be used.

3.1 Sampling Train. Same as CARB Method 5, Section 2.1. except use a glass lined stainless steel probe.

3.2 Sample Recovery. Same as CARB Method 5, Section 2.2.

3.3 Analysis. The following apparatus and materials are needed.

3.3.1. Analysis of hexavalent chromium.

3.3.1.1. Philips Beakers. Borosilicate, 125mL, with watchglass covers.

3.3.1.2. Filtration Apparatus. Vacuum unit constructed of glass, to accommodate sintered glass funnels.

3.3.1.3. Volumetric Flasks. 100-mL and other appropriate volumes.

3.3.1.4. Hot Plate.

3.3.1.5. Pipettes. Assorted sizes, as needed.

3.3.1.6. Spectrophotometer. To measure absorbance at 540nm.

3.3.2. Analysis of total chromium.

3.3.2.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm and provisions for simultaneous background correction and interfacing with a strip chart recorder.

3.3.2.2. Chromium hollow cathode lamp or electrodeless discharge lamp.

3.3.2.3. Graphite furnace: Any graphite furnace device with the appropriate temperature and timing controls.

3.3.2.4. Strip chart recorder: A recorder is strongly recommended for furnace work so that there will be a permanent record and so that any problems with the analysis such as drift, incomplete atomization, losses during charring,

changes in sensitivity, etc., can easily be recognized.

4. REAGENTS

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

- 4.1 Sampling. Same as CARB method 5, Section 3.1. except Teflon-coated glass fiber filters are used. Any other sampling method which, after review of the Executive Officer, is deemed equivalent for the purposes of this test method, may be used.
- 4.2 Sample Recovery. Same as CARB Method 5, Section 3.2.
- 4.3 Reagents for hexavalent chromium.
 - 4.3.1 Water. Deionized distilled, meeting American Society for Testing and Materials (ASTM) specification for type reagent - ASTM Test Method D 1193-77. The water should be monitored for impurities.
 - 4.3.2. Extraction solution. Dissolve 20.0 g NaOH and 30.0 g anhydrous Na_2CO_3 in water in a 1-liter volumetric flask, and dilute to the mark. Store the solution in a tightly capped polyethylene bottle. Prepare fresh monthly.
 - 4.3.3. Potassium Dichromate Stock Solution. Dissolve 2.829 g of analytical reagent grade $\text{K}_2\text{Cr}_2\text{O}_7$ in water, and dilute to 1 liter (1 mL = 1000 ug Cr^{+6}).
 - 4.3.4. Potassium Dichromate Standard Solution. Dilute 10.00mL potassium dichromate stock solution to 100 mL (1 mL = 100 ug Hexavalent Chromium) with water.
 - 4.3.5. Sulfuric Acid, 6N. Dilute 166 mL sulfuric acid to 1000 mL in water.
 - 4.3.6. Acetone. Same as Method 5, Section 3.2.
 - 4.3.7 Diphenylcarbazide Solution. Dissolve 0.5 mg of 1, 5-diphenylcarbazide in 100 mL acetone. Store in a brown bottle. Discard when the solution becomes discolored.
 - 4.3.8 0.1% Potassium Permanganate Solution
 - 4.3.9 0.01% Potassium Permanganate Solution
- 4.4. Reagents for total chromium

- 4.4.1. ASTM Type II water (ASTM D1193): refer to section 4.3.1.
- 4.4.2. Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.
- 4.4.3. Hydrogen peroxide (30%) optional.
- 4.4.4. Calcium nitrate solution: Dissolve 11.8 g of calcium nitrate, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (analytical reagent grade), in Type II water and dilute to 1 liter. If the instrument manufacturer recommends a different matrix modifier then use that reagent.
- 4.4.5. Chromium standard stock solution (1000mg/L): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve 2.829 g of Potassium Dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$, analytical reagent grade) in Type II water and dilute to 1 liter.
- 4.4.6. Chromium working standards: These standards should be prepared to contain 1.0% (v/v) HNO_3 ; 1mL of 30% H_2O_2 and 1mL of the calcium nitrate solution may be added to lessen interferences.

5. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

- 5.1 Sample collection. All samples are collected from the source by use of CARB Method 5.
- 5.2 Sample handling and preservation. All sample containers must be prewashed with detergents, acids and Type II water. Glass containers are suitable.

6. PROCEDURES

6.1 SAMPLE PREPARATION

- 6.1.1. The sample is collected in the probe, impingers and filters. The filter is placed in container number 1. The probe is rinsed with acetone solvent and the rinse is placed in container number 2. Place half the contents of Container Number 2 (the acetone probe rinse) in a 125-mL Philips beaker A. Place the other half of the contents of Container Number 2 in a second 125 mL Philips beaker B. Rinse Container Number 2 with acetone and transfer half the rinse to beaker A and the other half of the rinse to beaker B. Evaporate both beakers to dryness. Place half the contents of the impingers in

beaker C and the other half in beaker D. Rinse the impingers and place half of the rinse into beaker C and the other half into beaker D. Evaporate to dryness. Take the filter from Container Number 1 and divide it into quarters. Take the quarters that are diagonal to each other and place them into beaker E. Then place the other two quarters of the filter from Container Number 1 into beaker F. Cut each filter quarter into small pieces. Save beaker A, C and E for hexavalent chromium analysis. Save beaker B, D and F for total chromium analysis.

6.2 Removal of reducing agents in the reagents. The extraction solution (4.3.2) and the 6N sulfuric acid solution may contain small amounts of reducing agents that can react with the hexavalent chromium. Potassium permanganate is added to these reagents in order to neutralize these reducing agents. Pipette 3 mL of the extraction solution into cuvettes A and B. Use cuvette A as a sample cell and cuvette B as a reference cell. Zero the instrument at 528 nm with both cuvettes. Wait 10 minutes. Add an adequate amount (uL) of 0.01% potassium permanganate solution to (4.3.9) cuvette A. Enough should be added so that after 10 minutes a slight change in absorbance is observed. This step may have to be repeated a number of times in order to determine the required amount of potassium permanganate that is required. From the change in absorbance, calculate the amount of potassium permanganate that is needed to neutralize the reducing agents found in the reagents. Then pipette the proper volume of higher concentration 0.1% potassium permanganate solution into the reagents. This is done by assuming that the number of milliequivalents of reducing agents in the reagents are equal to the number of milliequivalents of 0.1% potassium permanganate pipetted. This procedure is repeated with the 6N sulfuric acid solution.

6.3 Hexavalent Chromium sample preparation. Sample digestion and preparation. To beaker (A) add 25 mL of alkaline digestion solution (Section 4.3.2). Shake solution at room temperature for 30 minutes. Quantitatively transfer the solution to the filtration apparatus with water. Filter the solution through the sintered glass funnel into a 150mL receiving beaker. Transfer the filtrate from the filter flask quantitatively to a 100mL volumetric flask. Fill to a volume of approximately 75mL to 85mL.

Adjust the pH to 1 ± 0.2 with 6N percent sulfuric acid. Shake carefully to release carbon dioxide. Add 2.0 ml of diphenylcarbazide solution, and dilute to volume with water. Allow the solution to stand about 10 minutes for color development. For each set of samples analyzed, treat an identical aliquot of reagent blank solution in

the same way. Transfer a portion of the sample to a 5-cm absorption cell, and measure the absorbance at the optimum wavelength of 540 nm. Measure and subtract the reagent blank absorbance reading, if any, to obtain a net reading. If the absorbance of the sample exceeds the absorbance of the 100 ug Cr⁺⁶ standard as determined in Section 7.2.2, dilute the sample and the reagent blank with equal volumes of water. Repeat this procedure for beakers C and E.

6.3.1 Reagent Blank Preparation. Take a representative amount of acetone and a blank Method 5 filter and proceed as in Section 6.3.

6.3.2 Silica Gel Weighing. Weigh the spent silica gel (Container Number 3) or silica gel plus impinger to the nearest 0.5 g using a balance. This step may be conducted in the field.

6.3.3 Analysis.

6.3.4 Check for Matrix Effects on the Cr⁺⁶ Results. Since the analysis for Cr⁺⁶ by colorimetry is sensitive to the chemical composition of the sample (matrix effects), the analyst shall check at least one sample from each source using the method of additions as follows: Obtain two equal volume aliquots of the same sample solution. The aliquots should each contain between 6 and 10 ug of Cr⁺⁶ (less if not possible). Spike one of the aliquots with an aliquot of standard solution that contains between 6 and 10 ug of Cr⁺⁶. Now treat both the spiked and unspiked sample aliquots as described in Section 6.3. Next, calculate the Cr⁺⁶ mass Cs, in ug in the aliquot of the unspiked sample solution by using the following equation:

$$Cs = Ca \frac{As}{At - As} \quad \text{Eq. 1}$$

where:

Ca = Cr⁺⁶ in the standard solution, ug.

As = Absorbance of the unspiked sample solution.

At = Absorbance of the spiked sample solution.

Volume corrections will not be required since the solutions as analyzed have been made to the same final volume. If the results of the method of standard additions procedure used on the single source sample do not agree to within 10 percent of the value obtained by the routine

spectrophotometric analysis, then reanalyze all samples from the source using this method of standard additions procedure.

6.4 Total Chromium Sample Preparation.

6.4.1 Add 10ml of conc. nitric acid to the sample in the beaker(B). Cover the beaker with a watch glass. Place the beaker on a hot plate and reflux the sample down to 2-3mL. Add another 5mL nitric acid to complete digestion. Reflux the sample volume down to 1mL.

6.4.2. Wash down the beaker walls and watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the nebulizer. Filtration should be done only if there is concern that insoluble materials may clog the nebulizer. Adjust the volume to 50 mL or a predetermined value based on the expected metal concentrations. The sample is now ready for analysis. The applicability of a sample preparation technique must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

6.4.3 The 357.9-nm wavelength line shall be used.

6.4.4 Follow the manufacturer's operating instructions for all other spectrophotometer parameters.

6.4.5 Furnace parameters suggested by the manufacturer should be employed as guidelines. Since temperature-sensing mechanisms and temperature controllers can vary between instruments or with time, the validity of the furnace parameters must be periodically confirmed by systematically altering the furnace parameters while analyzing a standard. In this manner, losses of analyte due to higher than necessary temperature settings or losses in sensitivity due to less than optimum settings can be minimized. Similar verification of furnace parameters may be required for complex sample matrices.

6.4.6 Inject a measured uL aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

6.4.7 Repeat procedure (6.4) for beakers D and F.

7. CALIBRATION

7.1 Sampling Train. Perform all of the calibrations described in CARB Method 5, Section 5.

7.2 Calibration Hexavalent Chromium

7.2.1 Optimum Wavelength Determination. Calibrate the wavelength scale of the spectrophotometer every 6 months. The calibration may be accomplished by using an energy source with an intense line emission such as a mercury lamp, or by using a series of glass filters spanning the measuring range of the spectrophotometer. Calibration materials are available commercially and from the National Bureau of Standards. Specific details on the use of such materials should be supplied by the vendor; general information about calibration techniques can be obtained from general reference books on analytical chemistry. The wavelength scale of the spectrophotometer must read correctly within ± 5 nm at all calibration points; otherwise, the spectrophotometer shall be repaired and recalibrated. Once the wavelength scale of the spectrophotometer is in proper calibration, use 540 nm as the optimum wavelength for the measurement of the absorbance of the standards and samples.

Alternatively, a scanning procedure may be employed to determine the proper measuring wavelength. If the instrument is a double-beam spectrophotometer, scan the spectrum between 530 and 550 nm using a 50 ug Cr^{+6} standard solution in the sample cell and a blank solution in the reference cell. If a peak does not occur, the spectrophotometer is malfunctioning and should be repaired. When a peak is obtained within the 530 to 550 nm range, the wavelength at which this peak occurs shall be the optimum wavelength for the measurement of absorbance of both the standards and the samples. For a single-beam spectrophotometer, follow the scanning procedure described above, except that the blank and standard solutions shall be scanned separately. The optimum wavelength shall be the wavelength at which the maximum differences in absorbance between the standard and the blank occurs.

7.2.2 Spectrophotometer Calibration. Either (1) run a series of chromium standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. See section 6.3.4 for the use of the method of standard additions.

7.2.2.1 Spectrophotometer Calibration Quality Control. Multiply the absorbance value obtained for each standard by the Kc factor (least squares slope) to determine the distance each calibration point lies from the theoretical calibration line. These calculated concentration values should not differ from the actual concentrations (i.e., 5, 10, 25, 50, 75, and 100 ug Cr⁺⁶) by more than _ percent (to be determined) for five of the six standards.

7.3 Calibration of Total Chromium

- 7.3.1. Either (1) run a series of chromium standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.
- 7.3.2. Run a check standard after approximately every 10 sample injections. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced. -
- 7.3.3. Duplicates, spiked samples, and check standards should be routinely analyzed.
- 7.3.4. Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 ug/g dry weight).
- 7.3.5. Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.
- 7.3.6. Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.
- 7.3.7. Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

7.3.8. Analyze check standards after approximately every 15 samples.

7.3.9. Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation.

7.3.10. Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

7.3.11. The method of standard additions shall be used for the analysis of all EP extracts, or whenever a new sample matrix is being analyzed.

7.3.12. The concentration of all calibration standards should be verified against a quality control check sample obtained from an outside source.

7.3.13. All quality control data should be maintained and available for easy reference or inspection.

7.4 Emission Calculations

Carry out the calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculations.

7.4.1 Total Cr^{+6} in Sample. Calculate m_{ch} , the total ug Cr^{+6} in sample. This can be obtained from the calibration curve or from the method of standard additions. Note that m_{ch} is the sum of the masses of hexavalent chromium analyses performed on the contents of beakers B, D and F. Also take into account the dilutions when calculating m_{ch} .

7.4.2 Total Chromium in the Sample. Calculate m_{ct} , the total ug of Chromium in the sample. This can be obtained from the calibration curve or from the method of standard additions. Note that m_{ct} is the sum of the masses of total chromium analyses performed on the contents of beakers A, C and E. Also take into account the necessary dilutions when calculating out m_{ct} .

7.4.3 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop. Same as Method 5, Section 6.2.

7.4.4 Dry Gas Volume, Volume of Water Vapor, Moisture Content. Same as Method 5, Sections 6.3, 6.4,

and 6.5, respectively.

- 7.4.5 Cr^{+6} Emission Concentration. Calculate c_s (g/dscm), the Cr^{+6} concentration in the stack gas, dry basis, corrected to standard conditions, as follows:

$$c_s = (10^{-6} \text{ g/ug})(m_{ch}/V_m(\text{std}))$$

- 7.4.6 Total Chromium Emission Concentration. Calculate c_{st} (g/dscm), the total chromium concentration in the stack gas, dry basis, corrected to standard conditions as follows:

$$c_{st} = (10^{-6} \text{ g/ug})(m_{ct}/V_m(\text{std}))$$

- 7.4.7 Isokinetic Variation, Acceptable Results. Same as Method 5, Sections 6.11 and 6.12, respectively.

8. OTHER METHODS.

- 8.1 Total chromium determination by atomic absorption spectroscopy. This method uses the direct aspiration method.
- 8.2 Hexavalent chromium by flameless atomic absorption spectroscopy. This method complexes hexavalent chromium with ammonium pyrrolidine dithiocarbamate. This chromium complex is then measured by atomic absorption spectrophotometer equipped with a flameless accessory.

9. REFERENCES.

- 9.1 U.S. Environmental Protection Agency/Office of Solid Waste, Washington, D.C., "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, "SW-846 (1980), SW-846 Revision A (August 8, 1980), and SW-846 Revision B (July 1981).
- 9.2 Cox, X.B., R.W., R.W. Linton, and F.E. Butler. Determination of Chromium Speciation in Environmental Particles- A Multitechnique Study of Ferrochrome Smelter Dust. Accepted for publication in Environmental Science and Technology.
- 9.3 Same as in Bibliography of Method 5, Citations 2 to 6 and 7.
- 9.4 California Air Resources Board, Aerometric Data Division. Method ADD1 006.

APPENDIX B

DATA AND CALCULATIONS

PARTICULATE EMISSIONS TEST SUMMARY AND RESULTS

FE-1501-Engr. Lval. Branch

File No. C- 86-085

Project Name: Standard Plating

Remarks: PT-14

SUMMARY OF TEST DATA

	<u>Cr VI</u>	<u>Total</u>
Metered Sample Volume	90.53 cubic feet	
Meter Temperature:	60 deg.F	
Nozzle Diameter:	0.252 inches	
Pitot Tube C-Factor:	0.826	
Sampling Time:	160.00 minutes	
Avg. Delta P Orifice Pressure:	1.38 inches H ₂ O	
Avg. γ (Delta P Pitot Pressure):	0.55 γ (inches H ₂ O)	
H ₂ O in Impingers and Silica Gel:	29.0 milliliters	
Particulate Catch:	126.1 ^{10⁻³} milligrams	161.0 ^{10⁻³}
Stack Diameter:	19.000 inches	
Stack Area:	1.969 square feet	
Stack Temperature:	77 deg.F	
Barometric Pressure:	29.930 inches Hg	
O ₂ In Stack:	20.90 percent	
CO ₂ In Stack:	0.00 percent	
CO In Stack:	0.00 percent	

CALCULATED RESULTS:

	<u>Cr VI</u>	<u>Total</u>
Isokinetic Ratio:	93.3 percent	
Corrected Sample Volume:	92.28 ^{10⁻³} SCF (68 deg.F)	
Particulate Concentration:	0.002108 ^{10⁻³} Grain/SCF (68 deg.F)	0.002692 ^{10⁻³}
Particulate Emissions:	0.00357 ^{10⁻³} lb/hr	0.00451 ^{10⁻³}
Stack Flow:	3517 SCFM (dry, 68 deg.F)	
Stack Velocity:	30.7 feet/second	
H ₂ O Vapor In Stack:	1.5 percent	
(H ₂ O In Stack is BELOW Saturation.)		
Stack Gas Mole Weight (dry):	28.85	
Stack Gas Mole Weight (wet):	28.69	

Verified by: _____

PARTICULATE EMISSIONS TEST SUMMARY AND RESULTS

AFB-SSCD-Engr. Eval. Branch

File No. C- 86-085

Project Name: Standard

Remarks: PT-10

SUMMARY OF TEST DATA

Metered Sample Volume	97.05 cubic feet
Meter Temperature:	60 deg.F
Nozzle Diameter:	0.312 inches
Pitot Tube C-Factor:	0.822
Sampling Time:	160.00 minutes
Avg. delta P Orifice Pressure:	1.57 inches H2O
Avg. $\sqrt{\text{delta P Pitot Pressure}}$:	0.38 $\sqrt{\text{inches H2O}}$
H2O in Impingers and Silica Gel:	37.6 milliliters
Particulate Catch:	88.7 $\times 10^{-3}$ milligrams
Stack Dimensions: I= 23.000 W= 23.000 inches	
Stack Area:	3.674 square feet
Stack Temperature:	70 deg.F
Barometric Pressure:	29.930 inches Hg
O2 In Stack:	20.90 percent
CO2 In Stack:	0.00 percent
CO In Stack:	0.00 percent

Total

$96.1 \times 10^{-3} \text{ mg}$

CALCULATED RESULTS:

Isokinetic Ratio:	94.5 percent
Corrected Sample Volume:	98.97 DSCF (68 deg.F)
Particulate Concentration:	0.01303 $\times 10^{-3}$ grain/DSCF (68 deg.F)
Particulate Emissions:	0.5370 $\times 10^{-3}$ lb/hr
Stack Flow:	4531 SCFH (dry, 68 deg.F)
Stack Velocity:	21.0 feet/second
H2O Vapor In Stack:	1.8 percent
(H2O In Stack is BELOW Saturation)	
Stack Gas Mole Weight(dry):	28.85
Stack Gas Mole Weight(wet):	28.66

Total

0.01498×10^{-3}
 0.5819×10^{-3}

Verified by: _____

PARTICULATE EMISSIONS TEST SUMMARY AND RESULTS

AFB-SSCD-Engr. Eval. Branch

File No. C- 86-085

Project Name: Standard

Remarks: PT-24

SUMMARY OF TEST DATA

Metered Sample Volume	81.72 cubic feet	
Meter Temperature:	60 deg.F	
Nozzle Diameter:	0.252 inches	
Pitot Tube C-Factor:	0.826	
Sampling Time:	128.00 minutes	
Avg. Delta H Orifice Pressure:	1.50 inches H ₂ O	
Avg. $\sqrt{\Delta P}$ (Delta P Pitot Pressure):	0.59 $\sqrt{\text{inches H}_2\text{O}}$	
H ₂ O in Impingers and Silica Gel:	25.3 milliliters	
Particulate Catch:	57.9 ^{mc} g	69.5 x 10 ⁻³
Stack Diameter:	19.000 inches	
Stack Area:	1.969 square feet	
Stack Temperature:	70 deg.F	
Barometric Pressure:	29.930 inches Hg	
O ₂ In Stack:	20.90 percent	
CO ₂ In Stack:	0.00 percent	
CO In Stack:	0.00 percent	

Total Cr

CALCULATED RESULTS:

Isokinetic Ratio:	17.5 percent	
Corrected Sample Volume:	80.33 SCF (68 deg.F)	
Particulate Concentration:	0.01072 ^{mc} g grain/SCF (68 deg.F)	0.1287 x 10 ⁻³
Particulate Emissions:	0.3492 ^{mc} lb/hr	4.191 x 10 ⁻³
Stack Flow:	3800 SCFM (dry, 68 deg.F)	
Stack Velocity:	32.7 feet/second	
H ₂ O Vapor In Stack:	1.4 percent	
(H ₂ O In Stack is BELOW Saturation)		
Stack Gas Mole Weight(dry):	28.85	
Stack Gas Mole Weight(wet):	28.70	

Verified by: _____

PARTICULATE EMISSIONS TEST SUMMARY AND RESULTS

ARE-ESCD-Engr. Eval. Branch

File No. C- 86-085

Project Name: Standard Plotting

Remarks: PT-2D

SUMMARY OF TEST DATA

	<u>Level</u>	<u>Total</u>
Metered Sample Volume	80.72 cubic feet	
Meter Temperature:	60 deg.F	
Nozzle Diameter:	0.312 inches	
Pitot Tube C-Factor:	0.822	
Sampling Time:	128.00 minutes	
Avg. Delta P Orifice Pressure:	1.30 inches H2O	
Avg. $\sqrt{(\Delta P \text{ Pitot Pressure})}$:	0.26 $\sqrt{(\text{inches H2O})}$	
H2O in Impingers and Silica Gel:	29.1 milliliters	
Particulate Catch:	29.2 ^{10⁻³} milligrams	40.0 x 10 ⁻³
Stack Area:	3.674 square feet	
Stack Temperature:	68 deg.F	
Barometric Pressure:	29.930 inches Hg	
CO In Stack:	20.90 percent	
CO2 In Stack:	0.00 percent	
CC In Stack:	0.00 percent	

CALCULATED RESULTS:

		<u>Total</u>
Isokinetic Ratio:	103.4 percent	
Corrected Sample Volume:	12.27 DSCF (68 deg.F)	
Particulate Concentration:	0.00348 ^{10⁻³} grain/DSCF (68 deg.F)	0.0750 x 10 ⁻³
Particulate Emissions:	0.2020 ^{10⁻³} lb/hr	0.2769 x 10 ⁻³
Stack Flow:	4303 SCFH (dry, 68 deg.F)	
Stack Velocity:	19.8 feet/second	
H2O Vapor In Stack:	1.6 percent	
(H2O In Stack is BELOW Saturation)		
Stack Gas Mole Weight(dry):	28.85	
Stack Gas Mole Weight(wet):	28.67	

Verified by: _____

PARTICULATE EMISSIONS TEST SUMMARY AND RESULTS

APE-SSCD-Engr. Eval. Branch

File No. C- 86-085

Project Name: Standard Plating

Remarks: PT-3U

SUMMARY OF TEST DATA

	<u>(100)</u>	<u>Total</u>
Filtered Sample Volume	83.38 cubic feet	
Filter Temperature:	60 deg.F	
Nozzle Diameter:	0.252 inches	
Pitot Tube C-Factor:	0.802	
Sampling Time:	128.00 minutes	
Avg. Delta P Orifice Pressure:	1.50 inches H ₂ O	
Avg. $\sqrt{V}$ (Delta P Pitot Pressure):	0.59 $\sqrt{V}$ (inches H ₂ O)	
H ₂ O in Impingers and Silica Gel:	29.3 milliliters	
Particulate Catch:	98.8 ¹⁰⁰⁻³ milligrams	148.3 x 10 ⁻³
Stack Diameter:	19.000 inches	
Stack Area:	1.969 square feet	
Stack Temperature:	79 deg.F	
Barometric Pressure:	29.930 inches Hg	
O ₂ In Stack:	20.90 percent	
CO ₂ In Stack:	0.00 percent	
CO In Stack:	0.00 percent	

CALCULATED RESULTS:

		<u>Total</u>
Isokinetic Ratio:	101.4 percent	
Corrected Sample Volume:	85.02 SCFH (68 deg.F)	
Particulate Concentration:	0.01757 ¹⁰⁰⁻³ grains/SCFH (68 deg.F)	0.02691 x 10 ⁻³
Particulate Emissions:	0.5500 ¹⁰⁰⁻³ lb/hr	0.7514 x 10 ⁻³
Stack Flow:	3653 SCFH (dry, 68 deg.F)	
Stack Velocity:	32.0 feet/second	
H ₂ O Vapor In Stack:	1.6 percent	
(H ₂ O In Stack is BELOW Saturation)		
Stack Gas Mole Weight(dry):	28.85	
Stack Gas Mole Weight(wet):	28.66	

Verified By: _____

PARTICULATE EMISSIONS TEST SUMMARY AND RESULTS

APL-8101-Logr. Eval. Branch

File No. C- 86-085

Project Name: Standard Plating

Remarks: PT-3D

SUMMARY OF TEST DATA

metered Sample Volume	91.05 cubic feet
meter Temperature:	60 deg.F
Nozzle Diameter:	0.312 inches
Pitot Tube C-Factor:	0.818
Sampling Time:	128.00 minutes
Avg. delta P Orifice Pressure:	1.62 inches H2O
Avg. γ (delta P Pitot Pressure):	0.41 γ (inches H2O)
H2O in Impingers and Silica Gel:	36.8 milliliters
Particulate Catch:	68.0 milligrams
Stack Dimensions: L= 23.000 W= 23.000	inches
Stack Area:	3.674 square feet
Stack Temperature:	70 deg.F
Barometric Pressure:	29.930 inches Hg
O2 In Stack:	20.90 percent
CO2 In Stack:	0.00 percent
CO In Stack:	0.00 percent

Total

1,031.4 $\times 10^{-3}$ mg

CALCULATED RESULTS:

Isokinetic Ratio:	103.3 percent
Corrected Sample Volume:	92.87 DSCF (68 deg.F)
Particulate Concentration:	0.01130 $\times 10^{-3}$ Grain/DSCF (68 deg.F)
Particulate Emissions:	0.4708 $\times 10^{-3}$ lb/hr
Stack Flow:	4862 SCFM (dry, 68 deg.F)
Stack Velocity:	22.5 feet/second
H2O Vapor In Stack:	1.6 percent
(H2O In Stack is BELOW Saturation)	
Stack Gas Mole Weight (dry):	28.85
Stack Gas Mole Weight (wet):	28.65

Total

17137 $\times 10^{-3}$
7.14 $\times 10^{-3}$

Verified by: _____

PARTICULATE EMISSIONS TEST SUMMARY AND RESULTS

AFL-SSCD-Engr. Eval. Branch

File No. C- B6-085

Project Name: Standard Plating

Remarks: PT-44

SUMMARY OF TEST DATA

	(r VI)	Total
Metered Sample Volume	81.28 cubic feet	
Meter Temperature:	60 deg.F	
Nozzle Diameter:	0.253 inches	
Pitot Tube C-Factor:	0.802	
Sampling Time:	128.00 minutes	
Avg. delta H Orifice Pressure:	1.60 inches H2O	
Avg. $\sqrt{(\text{delta P Pitot Pressure})}$:	0.60 $\sqrt{}$ (inches H2O)	
H2O in Impingers and Silica Gel:	23.5 milliliters	
Particulate Catch:	78.1 milligrams ($\times 10^{-3}$)	145.3 $\times 10^{-3}$ mg
Stack Diameter:	19.000 inches	
Stack Area:	1.969 square feet	
Stack Temperature:	70 deg.F	
Barometric Pressure:	29.810 inches Hg	
O2 In Stack:	20.90 percent	
CO2 In Stack:	0.00 percent	
CC In Stack:	0.00 percent	

CALCULATED RESULTS:

	(r VI)	Total
Isokinetic Ratio:	97.1 percent	
Corrected Sample Volume:	82.57 DSCF (68 deg.F)	
Particulate Concentration:	0.0146 $\frac{\text{lb}}{\text{DSCF}}$ (68 deg.F) (0.0146) 0.0146×10^{-3}	
Particulate Emissions:	0.4687 $\frac{\text{lb}}{\text{hr}}$ (0.4687) 0.4687×10^{-3}	
Stack Flow:	3747 SCFH (dry, 68 deg.F)	
Stack Velocity:	32.4 feet/second	
H2O Vapor In Stack:	1.3 percent	
(H2O In Stack is BELOW Saturation)		
Stack Gas Mole Weight(dry):	28.85	
Stack Gas Mole Weight(wet):	28.71	

Verified by: _____

PARTICULATE EMISSIONS TEST SUMMARY AND RESULTS

APB-SSCD-Engr. Eval. Branch

File No. C- 86-085

Project Name: Standard Plating

Remarks: PT-4ED

SUMMARY OF TEST DATA

	<u>(-VI)</u>	<u>Total</u>
Metered Sample Volume	87.30 cubic feet	
Meter Temperature:	60 deg.F	
Nozzle Diameter:	0.313 inches	
Pitot Tube C-Factor:	0.826	
Sampling Time:	128.00 minutes	
Avg. delta P Orifice Pressure:	1.57 inches H2O	
Avg. γ (delta P Pitot Pressure):	0.41 γ (inches H2O)	
H2O in Impingers and Silica Gel:	23.5 milliliters	
Particulate Catch:	24.2 milligrams ($\times 10^{-3}$)	$26.8 \times 10^{-3} \text{ mg}$
Stack Dimensions: L= 23.000 W= 23.000 inches		
Stack Area:	3.674 square feet	
Stack Temperature:	65 deg.F	
Barometric Pressure:	29.810 inches Hg	
O2 In Stack:	20.90 percent	
CO2 In Stack:	0.00 percent	
CO In Stack:	0.00 percent	

CALCULATED RESULTS:

		<u>Total</u>
Isokinetic Ratio:	94.0 percent	
Corrected Sample Volume:	86.08 DSCF (68 deg.F)	
Particulate Concentration:	0.00421 $\frac{\text{grain}}{\text{DSCF}}$ (68 deg.F)	$\times 1.00466 \times 10^{-3}$
Particulate Emissions:	0.1786 $\frac{\text{lb}}{\text{hr}}$	$.1978 \times 10^{-3}$
Stack Flow:	4947 SCFH (dry, 68 deg.F)	
Stack Velocity:	22.7 feet/second	
H2O Vapor In Stack:	1.2 percent	
(H2O In Stack is BELOW Saturation)		
Stack Gas Mole Weight (dry):	26.65	
Stack Gas Mole Weight (wet):	28.72	

Verified by: _____

66-085
Standard Plot
F-105

APPENDIX C

AIHL ANALYTICAL RESULTS

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AIHL/ARB
LABORATORY REPORT

TO: ADD ☐
CD ☐
SSD ☒
OTHER ☐

I.D. No.

STUDY OR CONTROL NUMBER

C 8 6 - 0 8 5

PROJECT ENGINEER David Todd

of Establishment Standard Plating Company

Date this Report:

Mo.	Day	Yr

Los Angeles, CA.

Lab. No. Lab. Use Only	SAMPLE NUMBER	Laboratory Results		
556 B		Chromium (VI), net ug		
		Probe Rinse	Filter	After rinse / impinger water
	1D	2.3	86.4	< 1.0
	1U	20.1	106.0	< 1.0
	2D	0.1	29.1	< 1.0
	2U	4.9	53.0	< 1.0
	3D	0.5	65.2	2.3
	3U	10.8	86.0	< 1.0
	4D	0.9	23.3	< 1.0
	4U	16.1	62.0	< 1.0
	BIK 1	< 1.0	0.1	< 1.0
	BIK 2	-	0.1	< 1.0
	H ₂ O BLK	-	-	-
		detection limit 0.5 ug Cr ⁺⁶ / 50 mL		

Names of Chemists Involved:

Date

Signature of Supervising Chemist

Date

PAUL WONG

12/2/86

▷

TO: ADD ☐ -
CD ☐
SSD ☒
OTHER ☐ -

STUDY ON DOLLAR COUNTER

PROJECT NUMBER: David T. Hall

Standard Plating Company

Date of Report:

1000

Les régimes

Mo.	Day	Yr

Date of Chemicals Received: _____ Date: <u>11/19/56</u>	Signature of Supervising Chemist: _____ Date: <u>11/19/56</u>
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LABORATORY REPORT

TO: ADD ☐
 CD ☐
 SSD ☒
 OTHER ☐

I.D. No.

STUDY OR CONTROL NUMBER

C E 6 - 0 8 5

PROJECT ENGINEER

David Todd

Establishment

Standard Plating Company

Date this Report:

No.	Day	Yr
01	16	87

Lab. No. b. Use Only	SAMPLE NUMBER	Laboratory Results
556 A		Total Chromium, ug per sample
	1D-PR	9.32
	1U-PR	32.66
	2D-PR	4.37
	2U-PR	22.13
	3D-PR	6.44
	3U-PR	19.10
	4D-PR	3.99
	4U-PR	68.82
	1D-AF&IMP	0.41
	1U-AF&IMP	0.41
	2D-AF&IMP	4.57
	2U-AF&IMP	2.36
	3D-AF&IMP	938.56
	3U-AF&IMP	1.29
	4D-AF&IMP	2.11
	4U-AF&IMP	0.41

Names of Chemists Involved:

Date

Signature of Supervising Chemist

Date

VL WONG

1/12/87

E. Young

1/14/87

TO: ADD ☐
CD ☐
SSD ☒
OTHER ☐

STUDY OR CONTROL NUMBER

C	8	6	-	0	8	5
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PROJECT ENGINEER David Todd

of Establishment Standard Plating Company

Date this Report:

Mo.	Day	Yr

[illegible]

arks of Chemists Involved:

Date _____

Signature of Supervising Chemist

Date _____

AUL WONG

1/12/87

A young

1/21/87

LABORATORY SAMPLE ANALYSIS REQUEST

Page 1 of 3

SUBMITTING SAMPLE(S): ☐ TSD ☐ ED ☒ SSCD

Date Submitted

Mo.	Div.	Yr.
11	12	86

Submitter David Todd

Phone No: 8- 422-279

Engineer Approval Michael Todd

I.D. No.

LABORATORY OR CONTROL NUMBER

L	8	6	-	0	8	5
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Laboratory Results Desired:

1	2	C	1	8	6
Mo.	Day	Yr.			

Date Returned:

Mo.	Day	Yr.	

Number of Samples

Submitted:

0	4	0
---	---	---

Information:

Establishment: Standard Plating

82.6 E. 62nd St. Los Angeles

No. Use Only	SAMPLE NUMBER	Type of Sample Air Mat.	Volume	FIELD SAMPLE DESCRIPTION	Analysis Required
✓ 56	✓ S-1 S-1			Scrubber Water	Total Chrome and
	✓ S-2			Scrubber water	Chrome 6
	✓ S-3			Scrubber water	
	✓ PT-1A			Probe Rinse Probe Rinse	
	✓ PT-1A			After-Filter Rinse	
	✓ PT-1A			Impinger water	
	✓ PT-2A			Probe Rinse	
	✓ "			After Filter Rinse	
	✓ "			Impinger water	
	✓ PT-1D			Probe Rinse	
	✓ "			After filter Rinse	
	✓ "			Impinger water	
	✓ PT-2D			Probe Rinse	
	✓ "			A.F. Rinse	
	✓ "			Impinger water	
	✓ PT-3D			Probe Rinse	
	✓ "			A.F. Rinse	

AHL/ARB LABORATORY SAMPLE ANALYSIS REQUEST

Page 2 of 3

Submitting Sample(s): ☐ ASD ☐ ED ☒ SSCD

Date Submitted

Mo.	Day	Yr.
11	12	86

Submitter: David Todd

Phone No: 8-492-8279

Engineer Approval

David Todd

Study or Control Number

186-085

Laboratory Results Desired:

12-01-86
Mo. Day Yr.

Date Returned:

Mo.	Day	Yr.	

Number of Samples

Submitted:

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Information:

of Establishment:

Standard Plating

26 E. 62nd St. Los Angeles

S.No. b. Use Only	SAMPLE NUMBER	Type of Sample		Volume	FIELD SAMPLE DESCRIPTION	Analysis Required
		Air	Mat.			
556	PT-2D				Impinger Water (combined with	Total Chrome and
✓	PT-3U				A.F. Rinse (AF)	Chrome 6
✓	"				Probe Rinse	
✓	"				Impinger Water	
✓	PT-4D				A.F. Rinse	
✓	"				Probe Rinse	
	"				Impinger Water (combined with	
✓	PT-4U				A.F. Rinse (AF) ex	
✓	"				Probe Rinse	
✓	"				Impinger Water	
✓	S-4				Scrubber Water	
✓					Blank Impinger Water	
✓					Blank Acetone	✓