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June 7, 2021

Freedom of Information Act Officer
U.S Environmental Protection Agency
Region 5
77 W. Jackson Blvd.
Chicago, IL 60604
Via FOIAOnline

Re: Freedom of Information Act Request

Pursuant to the Freedom of Information Act (FOIA), 5 U.S.C. § 552, I request copies of specific records within the possession of the U.S. Environmental Protection Agency ("USEPA") relating to a September 2009 Final Report by USEPA Region 5 entitled *PFOS Chromium Electroplater Study* (attached to this request).

According to the study, a Cleveland-area chromium electroplating company (identified in the report as "Facility 3") used a mist suppressant (identified as "Mist Suppressant F") in its operations. I request copies of the following materials collected by USEPA during the preparation of this study:

- Any and all documents generated or compiled for the study that identify or discuss "Facility 3," which is suspected to be MPC Plating Inc. (possibly listed as "Manufacturers Plating Company"), located at 9921 Clinton Road, Brooklyn, Ohio, 44144
- Any and all documents generated or compiled for the study that identify or discuss "Mist Suppressant F," including, but not limited to, its manufacturer and brand name.

I also request any documents in USEPA's possession concerning the potential presence of perfluorononanoic acid ("PFNA") in mist suppressants used by chromium electroplating companies, including information on the manufacturer(s) and brand name(s) of the PFNA-containing mist suppressants.

I will pay any reasonable fee provided for in the FOIA and associated with reproducing the requested records; however, if the costs exceed two hundred fifty dollars ($250.00) please contact me with an estimate prior to incurring such costs.

If for any reason any portion of this request is denied, please inform me of the reasons for the denial, in writing, together with the name and address of the person to whom to direct an appeal.

Thank you in advance for your courtesies in this regard. Should you have any questions or concerns related to this request, please contact me at (202) 394-9473 or via email at andrew.simpson@fticonsulting.com.

Sincerely,

Andrew Simpson

PFOS CHROMIUM ELECTROPLATER STUDY

U.S. ENVIRONMENTAL PROTECTION AGENCY-REGION 5

September 2009

Final Report

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Cover: View of Chromium Electroplating Line

Photo: Mark Conti & David Barna
U.S. EPA-Region 5-Cleveland Office

Acronyms

3M	Minnesota Mining, and Manufacturing Corporation
ANPRM	Advance Notice of Proposed Rulemaking
CAA	Clean Air Act
CARB	California Air Resources Board
Cr(VI)	hexavalent chromium
DQA	data quality assessment
dynes/cm	dynes/centimeter
LC/MS/MS	liquid chromatography/mass spectrometry
LCS	laboratory control samples
MACT	Maximum Achievable Control Technology
MDH	Minnesota Department of Health
MDL	method detection limit
MPCA	Minnesota Pollution Control Agency
MS	matrix spike
MSD	matrix spike duplicates
NEORSD	Northeast Ohio Regional Sewer District
ng/l	nanograms per liter
OAQPS	U.S. EPA - Office of Air Quality Planning and Standards
OSHA	Occupational Safety and Health Administration
PFBA	perfluorobutanoate
PFBS	perfluorobutane sulfonate
PFCs	perfluorinated chemicals
PFDA	perfluorodecanoate
PFDoA	perfluorododecanoate
PFHpA	perfluoroheptanoate
PFHxA	perfluorohexanoate
PFHxS	perfluorohexane sulfonate
PFOA	perfluorooctanoate
PFOS	perfluorooctane sulfonate
PFOSA	perfluorooctane sulfonamide
PFNA	perfluorononanoate
PFPeA	perfluoropentanoate
PFUnA	perfluoroundecanoate
POTW	publicly Owned Treatment Works
ppt	parts per trillion
PQL	practical quantification limit
QAPP	Quality Asssurance Project Plan
R5	U.S. EPA - Region 5 office
RPD	relative percent difference
U.S. EPA	United States Environmental Protection Agency
WWTP	wastewater treatment plant

EXECUTIVE SUMMARY

In June 2008, R5 conducted a study to examine whether detectable levels of PFOS may be in the effluent of decorative chromium electroplating facilities that discharged to WWTPs. A year earlier, the State of Minnesota found high levels of PFOS at the Brainerd, Minnesota WWTP, and identified a chromium electroplating facility (Keystone Automotive) as the source [1]. Based on the State of Minnesota's findings, R5 initiated this study to investigate whether releases from chromium electroplating facilities could be a widespread source of PFOS in the environment. Along with other data, R5's study will be considered by the OAQPS to evaluate the use of PFOS in suppressing Cr(VI) emissions under air standards for this industry.

Samples were taken from seven Chicago, Illinois (Chicago), and four Cleveland, Ohio (Cleveland) facilities. R5 tested for thirteen PFCs, including PFOS, and data showed the following:

- PFCs were discharged from all eleven facilities' waste streams at quantifiable levels above background.

- "Background" was defined by the rinse water measurements. All eleven facilities used municipal tap water for their rinse water. Therefore, one rinse water (background) sample was taken in each city as a measurement of background PFC levels. The background PFOS level for Cleveland was 5.75 ppt. The background PFOS level for Chicago was 2.52 ppt.

- Ten out of the eleven facilities had PFOS detected in their wastewater in concentrations ranging from 31.4-39,000 ppt.

- Of the ten facilities with PFOS detections, none had effluent levels higher than those found at Keystone Automotive facility located near Brainerd, Minnesota.

INTRODUCTION

In 2007, the MPCA tested the influent, effluent, and sludge at WWTPs across the State for PFCs, including PFOS. This investigation was done in order to determine if PFCs were present in these waste streams, and could therefore be a source of PFCs to the broader environment [1]. PFCs had been manufactured in Minnesota by 3M since the 1950s. 3M phased out the manufacturing of PFOS-related products in 2002 because of the growing research findings that PFOS was toxic to animals, persistent in humans, and widespread in the environment. In 2004, PFCs were detected in drinking water supplies in several eastern Twin Cities communities and traced to the legal disposal of 3M waste [2]. Through broader investigations, MPCA found widespread PFC contamination in various environmental media, including places with no known PFC sources.

Through testing at WWTPs, MPCA found relatively high levels of PFOS at the WWTP in Brainerd, Minnesota. The city of Brainerd is located about 135 miles northwest of St. Paul, along the Mississippi River. The initial 2007 sampling results at Brainerd were:

- Influent: 811 ppt PFOS;
- Effluent: 1500 ppt PFOS;
- Sludge: 861,000 ppt PFOS [1].

WWTP effluent may be a significant entry of PFCs to the environment [3], and several studies have concluded that conventional wastewater treatment may not be effective in removing these compounds [4] [5].

MPCA traced the PFOS in Brainerd's WWTP to a local chromium electroplating facility, Keystone Automotive (Keystone). Keystone was reportedly one of the largest chrome bumper repair and plating facilities in the United States. Since 1995, Keystone had been applying a commonly used PFOS-containing mist suppressant (Fumetrol 140®) in order to comply with the CAA's Cr(VI) MACT standard. As a result of MPCA's findings, the company switched to an alternate non-PFOS containing mist suppressant in early September 2007 [1].

Background

Cr(VI) electroplating is the electrical application of a coating of chromium onto a surface for decoration, corrosion protection, or durability. An electrical charge is applied to a tank (bath) containing an electrolytic salt solution. The electrical charge causes the chromium metal in the bath to fall out of solution and deposit onto objects placed into the plating bath. In an anodizing process, an oxide film is formed on the surface of the part. These electrolytic processes cause mist and bubbles containing Cr(VI) to be ejected from the bath, released into the work place, and eventually dispersed into outdoor ambient air unless controlled with add-on air pollution control equipment or chemical fume suppressants.

Chemical fume suppressants reduce surface tension and thereby, control Cr(VI) emissions. Surface tension is the force that keeps a fluid together at the air/fluid interface, and typically is expressed in force per unit of width, such as dynes/cm. By reducing surface tension in the plating/anodizing bath, gas bubbles become smaller, and rise more slowly than larger bubbles. Slower bubbles have reduced kinetic energy so that when the bubbles do burst at the surface, the

Cr(VI) is less likely to be emitted into the air, and the droplets fall back onto the surface of the bath [6]. Ideally, chromium plating baths should have surface tension values between 45-55 dynes/cm [7].

Cr(VI) is a human carcinogen. Therefore, the U.S. EPA regulates Cr(VI) electroplating or Cr(VI) anodizing tank operations by applying the CAA MACT limits. The MACT limits require control of Cr(VI) emissions to the atmosphere by either limiting the amount of Cr(VI) through use of add-on air pollution control devices or utilizing a chemical fume suppressant [8]. These facilities are also regulated by OSHA under 29 CFR Part 1910.1026 to protect workers from occupational Cr(VI) exposure. Employers are required to use engineering and work practice controls to reduce and maintain employee exposure to Cr(VI) at or below the permissible exposure level of 5 micrograms per cubic meter of air, calculated as an 8 hour time weighted average [6] [8].

Purpose of Study

After the release of MPCA's findings, R5 examined whether the release of PFOS through normal electroplating operations to WWTPs was a widespread or isolated event. Conversations between R5 staff and the Metal Refinishers Association indicated that PFOS use had become the industry standard as the most economic method of complying with the MACT rule [9]. Additionally, a 2003 survey conducted by the CARB, found that 190 of the 222 Cr(VI) electroplating operations in California used a fume suppressant, either in part or solely, to control Cr(VI) emissions. Almost all of the 190 operations used a chemical fume suppressant with PFOS as the active ingredient, and 124 reported using the same suppressant (Fumetrol 140®) that Keystone used. [6].

R5 provided this information to OAQPS. OAQPS was preparing to conduct a residual risk assessment for Cr(VI) electroplating, and to collect data through the ANPRM. Typically, releases of PFOS compounds would not be considered during a residual risk review since it is not one of the listed 188 hazardous air pollutants as defined by the Clean Air Act. However, OAQPS agreed to use the ANPRM to review data on the extent of PFOS mist suppressant use in Cr(VI) electroplating facilities and the potential release to WWTPs.

Because available data were likely to be limited, R5 also decided to gather data for OAQPS through a study to evaluate whether detectable levels of PFOS were present in the effluent of decorative Cr(VI) electroplating facilities that discharged to publicly owned WWTPs.

PROJECT DESCRIPTION

During June 2008, R5 collected samples of discharged process wastewaters at eleven decorative Cr(VI) electroplating facilities. The study was confined to facilities in the Chicago and Cleveland areas. Only decorative, rather than hard, chromium electroplaters were selected because of their higher likelihood to generate wastewaters that would then be discharged to a WWTP [10].

Site Selection

Prior to sampling, field investigators conducted telephone surveys of chromium electroplating and chromic acid anodizing facilities to determine the best candidates. The Cleveland area list of potential study candidates was assembled by identifying facilities with chromium emissions reported in the Aerometric Information Retrieval System, and facilities that were subject to the Electroplating Point Source Category at 40 CFR 413. The latter group of facilities was provided by the NEORSD, which operates the three area POTWs, and is the control authority for indirect dischargers in the Cleveland area.

The combined list contained sixty-four potential study candidates. Twenty-two candidates were contacted by telephone and asked if they: (1) performed Cr(VI) electroplating or anodizing; (2) discharged process wastewater; and (3) used chemical fume suppressants. If a company met these criteria, it was given a brief description of the project and told that the Cleveland team may sample their wastewater discharge as part of the project. Seven of the twenty-two facilities screened by telephone met all three criteria, and due to funding limitations, only four facilities that were furthest along in arrangements were selected (see **Figure 1**).

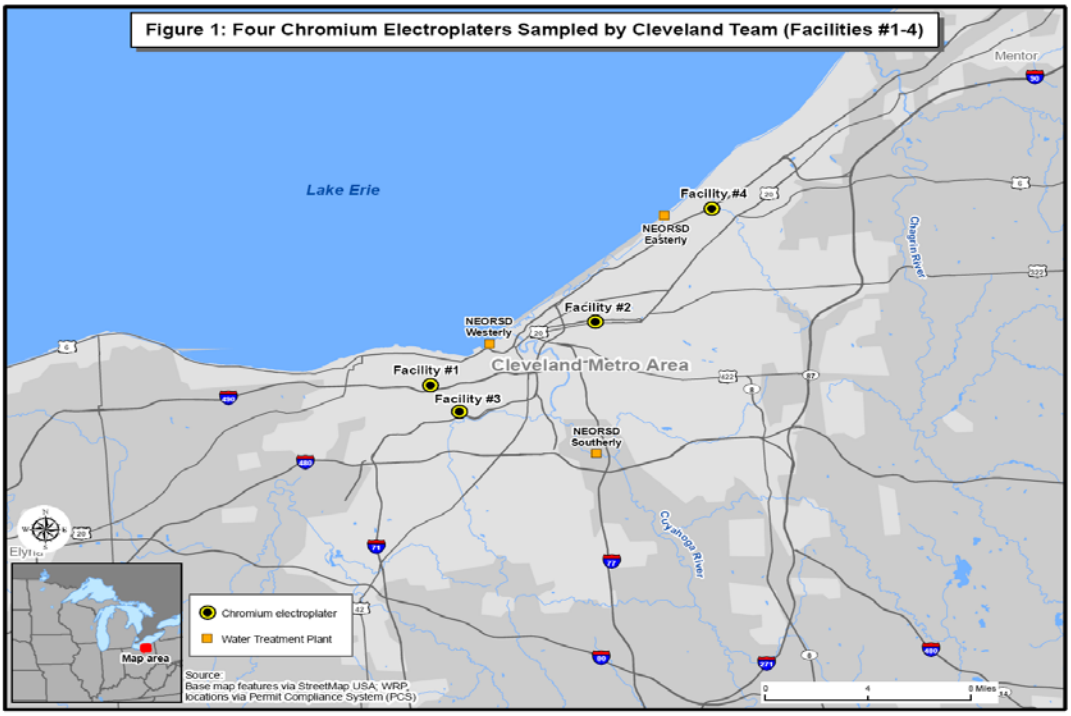


Figure 1: Location of Electroplating Facilities in Cleveland, Ohio

The Chicago area list of potential candidates was assembled by identifying facilities with existing air permits issued by the State of Illinois for operation of Cr(VI) electroplating tanks. The initial list contained twenty-six potential study candidates. Each of the twenty-six facilities were telephoned and asked the same questions as the Cleveland facilities. In addition, pre-sampling site visits were conducted following the telephone surveys. Seven Chicago facilities were picked as final candidates (see **Figure 2)**.

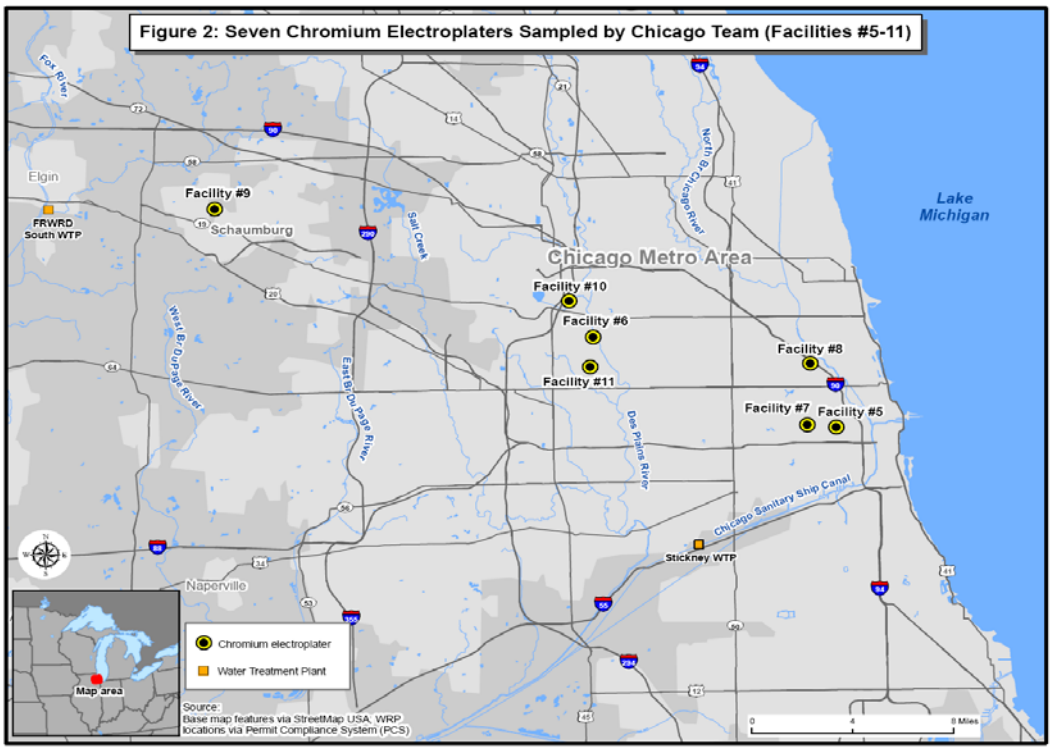


Figure 2: Location of Electroplaters Sampled in Chicago, Illinois.

All of the Chicago and Cleveland facilities selected performed decorative Cr(VI) electroplating on metal and/or plastic. Each facility used chemical fume suppressants (wetting agent and/or foam blanket) in its chromic acid bath tank to comply with the Cr(VI) electroplating MACT. In addition to chemical fume suppressants, two Cleveland facilities also employed add-on air pollution control devices. It was noted that facilities plating on plastic also used wetting agents in their chrome etch tanks for process control (i.e. to prevent voids in corners and creases of parts).

Facility Inspections

Both field investigation teams conducted cursory inspections at the facilities in conjunction with the sampling. During the inspections, the plating process, wastewater treatment, water usage, and usage of chemical fume suppressants were reviewed. Details of the inspections are summarized in **Appendix B-D.**

Sampling

The Cleveland field investigators collected samples between June 9 and 12, 2008, and the Chicago field investigator collected samples on June 9, 2008. All samples were taken during normal plating operations. At least one sample of discharged process wastewater from each facility was collected immediately prior to entry into the public sewerage system. Discharged process wastewater was comprised of the treated rinse waters from the plating operations, not including sanitary wastewater. In addition to the single effluent sample collected at each facility, the field investigators collected additional quality control samples at one select facility. These additional samples included the rinse water (background sample), field blank, and effluent duplicate. Samples were collected directly into laboratory-provided containers using standard operating procedures. The field blank was obtained by pouring reagent grade water into a laboratory-provided container while adjacent to the facility's discharge location. Samples were placed into iced coolers, refrigerated under custody until shipment to the laboratory, and cooled with blue ice packs during shipment. The samples were subsequently analyzed by AXYS Analytical Services Ltd. of Sidney, British Columbia, Canada, for thirteen PFCs, including PFOS (see **Figure 3** for results). The analytical method used was solid phase extraction with High Performance Liquid Chromatography, tandem mass spectrometry (LC/MS/MS).

RESULTS

Figure 3: PFC Concentrations at Sampled Electroplater Facilities (reported in ppt)

Facility ID#: Fume Suppressant(s)	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnA	PFDoA	PFBS	PFHxS	PFOS	PFOSA	Sum of PFCs	PFOS/ PFC
Facility #1: Mist Suppressant A, B, C	9.06	42.6	90.7	56.2	83.3	ND	ND	ND	ND	9,160	67.8	31,100	ND	40,610	77%
Facility #2: Mist Suppressant D, E	48.3	30.9	ND	ND	ND	ND	ND	ND	ND	41,800	306	708	ND	42,893	2%
Facility #3: Mist Suppressant B, F	ND	ND	177	175	650	13,100	27.1	44.1	ND	75.5	ND	ND	ND	14,249	<0.26%
Facility #4: Mist Suppressant D	ND	ND	ND	ND	ND	ND	ND	ND	ND	15,600	ND	39,000	ND	54,600	71%
Facility #5: Mist Suppressant G	ND	ND	ND	ND	ND	ND	ND	ND	ND	1,010	ND	2,320	ND	3,330	70%
Facility #6: Mist Suppressant Unknown	ND	ND	ND	ND	4.02	ND	ND	ND	ND	1,570	16.3	1,380	ND	2,970	46%
Facility #7: Mist Suppressant H	ND	1.08	ND	ND	3.11	ND	ND	ND	ND	ND	ND	301	ND	305	99%
Facility #8: Mist Suppressant H	ND	ND	2.3	1.17	3.17	ND	ND	ND	ND	311	993	1,770	ND	3,081	57%
Facility #9: Mist Suppressant Unknown	ND	ND	ND	ND	1.73	ND	ND	ND	ND	2,250	163	4,460	ND	6,875	65%
Facility #10: Mist Suppressant Unknown	1.54	1.29	1.82	ND	3.32	ND	ND	ND	ND	ND	3.53	31.4	ND	42.9	73%
Facility #11: Mist Suppressant Unknown	14.3	ND	ND	ND	ND	ND	ND	ND	ND	1,510	9,430	1,260	ND	12,214	10%
Number of Detects	4	4	4	3	7	1	1	1	0	9	7	10	0	11	
Minimum	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	31.4	ND	42.9	
Maximum	48.3	42.6	177	175	650	13,100	27.1	44.1	ND	41,800	9,430	39,000	ND	54,600	
Cleveland Background Sample	1.42	1.58	ND	1.74	2.19	ND	ND	ND	ND	ND	ND	5.75	ND		
Chicago Background Sample	ND	ND	ND	ND	1.37	ND	ND	ND	ND	ND	ND	2.52	ND		

Notes

ND means that the analyte was not detected at the method detection limit.
ND ranged from <1.00 to < 45.3 ppt depending on analyte.

For total PFCs, a value of zero was used in the sum of PFCs calculation.

DISCUSSION

Data results summarized in **Figure 3** showed the following.

- PFCs were discharged from all eleven facilities' waste streams at quantifiable levels above background. Ten out of the eleven facilities had PFOS above background detected in their waste discharge streams (**Figure 4)**.

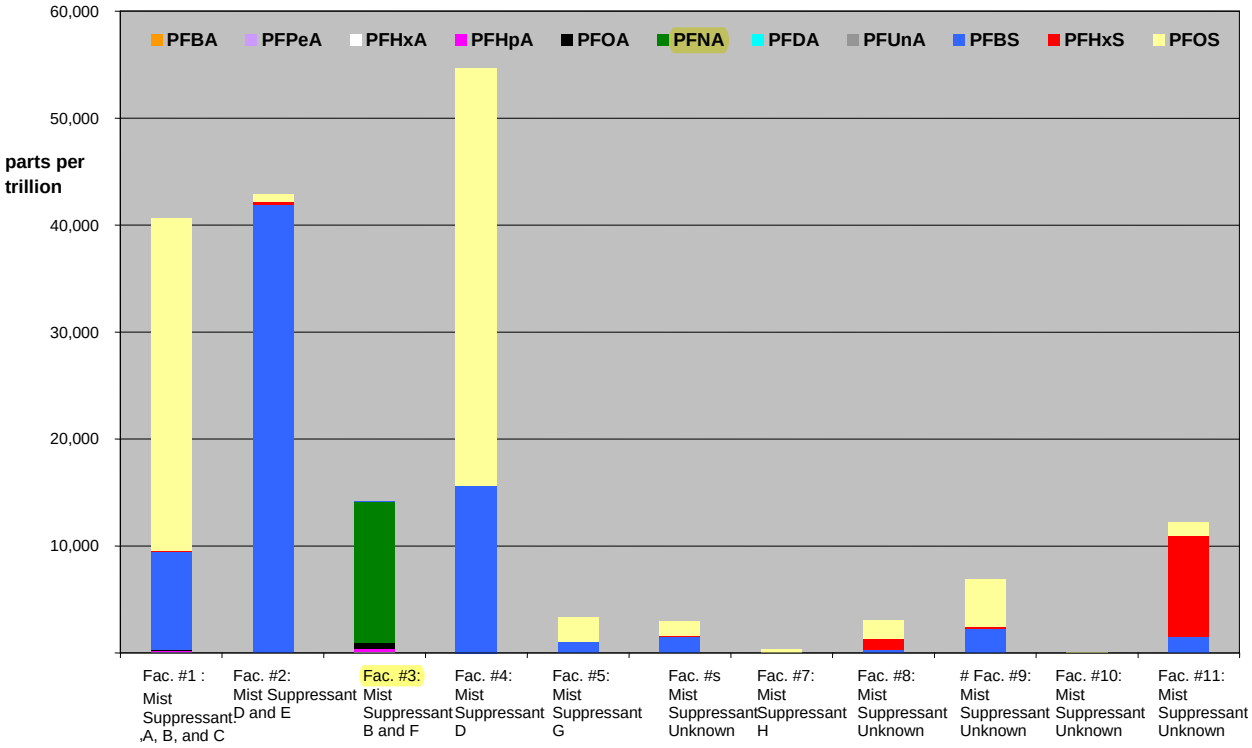


Figure 4: PFC Concentrations in Effluent of Electroplaters Using Chemical Fume Suppressants

- "Background" was defined by the rinse water measurements. All eleven facilities used municipal tap water for their rinse water. Therefore, one rinse water (background) sample was taken in each city as a measurement of background PFC levels. The background PFOS level for Cleveland was 5.75 ppt. The background PFOS level for Chicago was 2.52 ppt. In addition to PFOS, four other PFCs were detected in the Cleveland background sample, and one other PFC was detected in the Chicago background sample.

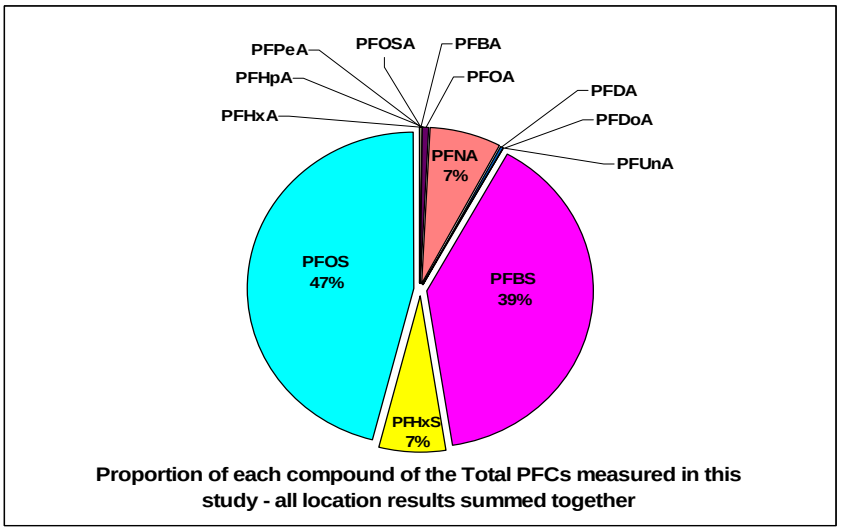
- At least eight different mist suppressants or mixture of suppressants were used at the various facilities and are as followed (several facilities did not provide information).

  Benchmark Benchbrite STX AB (custom-made)
  Benchmark Benchbrite STX
  Benchmark CFS
  MacDermid Proquel B
  MacDermid Macuplex STR
  Plating Process Systems PMS-R
  Fumetrol-140
  Brite Guard AF-1 fume control.

- Of the ten facilities with PFOS detections, none had effluent levels higher than those found at Keystone. In a sample dated December 2007, Keystone had a PFOS result of 278,000 ppt. [1]. The highest effluent PFOS result in this study was 39,000 ppt.

- The averages of the four highest concentrated compounds were: PFOS at 7680 ppt; PFBS at 6580 ppb; PFNA at 1190 ppt; and PFHxS at 1100 ppt (these averages were calculated using zero for the nondetects). These four chemicals made up over 99% of all compounds (**Figure 5)**. PFOS, PFBS, PFHxS, and PFOA were the most commonly detected PFCs.

Figure 5: Proportion of Total PFCs



- Another PFC compound of general interest, PFOA, was detected at seven of the eleven facilities, ranging from 1.73 - 650 ppt.

- PFCs were found in one field blank and in the background samples. Field blanks consisted of reagent grade bottled water exposed to the atmosphere at the designated facility. The field blank with PFHxS detection was exposed at facility #11 whose effluent samples contained PFHxS concentrations at the highest levels detected in this study. We attributed PFCs in the background samples to trace background levels found

in municipal tap water. Lake Michigan is the source water for the Chicago Municipal Utilities, and Lake Erie is the source water for the Cleveland Municipal Utilities. During the time of this study, both utilities were in compliance with all federal and state drinking water standards.

- Although not applicable to this industry, we compared our results to state and federal PFC guidance levels. Nine of the ten facilities tested above the U.S. EPA provisional health advisory for PFOS in drinking water set at 200 ppt. Ten of the eleven facilities tested above the Minnesota water quality criteria for PFOS in the Mississippi River (6 ppt).

Conclusions

The data clearly indicated that decorative chromium electroplaters discharge PFOS and other PFCs to WWTPs in concentrations higher than background levels. Data also indicated that mist suppressants have very specific PFC mixtures, which may be found in the resulting electroplater effluent. The concentrations vary widely which is most likely due to the inherit design of study. Therefore, care should be taken when comparing results from one facility to another, as the study included facilities of different operational sizes and production schedules. Facilities also varied widely in the brand of mist suppressant used, and amount added to the plating baths.

We would like to emphasize the nexus between the PFOS emissions and the Chromium MACT rule. To comply with the MACT rule, many facilities have chosen PFOS-containing mist suppressants as the best available technology to achieve Cr(VI) risk reduction in lieu of adding control technology. EPA believes that the PFOS emissions (as well as other PFC emissions reported in this survey) should provide target areas for improved pollution prevention performance including: (1) the development of alternative PFC free mist suppressants; (2) the improved procedures to reduce and capture downstream PFC levels in the wastewater prior to release into the waste water treatment facility; and (3) enhancing operating processes that limit the amount of PFC added to plating baths to efficaciously promote plating while reducing PFC total consumption.

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APPENDIXES

Appendix A - Data Quality Assessment Report

INTRODUCTION

This report was developed as a scientific evaluation designed to determine if the PFOS data obtained from the 2008 R5 Electroplater PFOS study were appropriate to meet the study objectives, and were of the right type, quality, and quantity to support the intended use. This assessment also estimated the level of confidence attributable to the data set. In brief, our analyses showed that some decisions and conclusions associated with these data could be made with a high degree of confidence, while other decisions had significant limitations associated with them.

The data were used to evaluate wastewater contamination associated with average industrial chromium decorative electroplaters. This PFOS study did not include a statistical sample design, and as such, rigorous statistical evaluations were not used. The data were assessed using the following criteria.

1. Review the data quality objectives
2. Conduct a preliminary data review
3. Perform an analysis of the data
4. Verify the assumption of the analysis
5. Draw conclusion from the data

1. REVIEW OF DATA QUALITY OBJECTIVES

Statement of the problem: The overall objective of this study was to evaluate potential wastewater PFOS release associated with decorative chromium electroplaters.

Study question:

1) Was there PFOS in the wastewater discharge to WWTPs from decorative chromium electroplaters?
2) Were these discharges quantifiable?

Identification of the decision:
Decision statement -
If PFOS discharges from decorative chrome electroplaters are present at facilities using an approved MACT standard technology for suppressing Cr(VI) emissions, then these results may be useful in informing the OAQPS rulemaking process.

Identification of inputs to the decision:
Facilities were selected based on:

- Use of PFOS containing surfactant; and
- Potential for rinse stream/waste water contamination.

Definitions of the boundaries for the study:
This study was confined to chromium electroplating facilities in the Chicago and Cleveland areas. Samples were taken during normal plant operating conditions, and sampling locations were representative of discharged wastewater to POTWs.

Documented decision rules:

- PFOS was present when the analyzed concentration was above the laboratory MDL.
- PFOS discharges were quantifiable when effluent concentrations observed were above the laboratory PQL.
- PFOS discharges were believed attributable to the use of MACT complaint Cr(VI) suppressants, when effluent concentrations were above background PFOS levels.

Optimize the design for obtaining data:
This analysis may be useful in addressing future studies of PFOS and other PFCs as related to wastewater discharges.

2 PRELIMINARY DATA REVIEW

Completeness
All samples identified in the QAPP were collected and analyzed.

Holding Time
All samples were analyzed within the required holding times.

Sample Preservation
All samples were collected and iced for shipment to lab.

Sample Receipt
All samples were received on ice within 24 hours of shipping. The samples were all refrigerated at 4 degree C prior to extraction and analysis.

Sample Extraction and Analysis
Samples were analyzed in three batches. Sample extraction, instrumental analysis, and analyte quantification procedures were in accordance with the lab's standard operating procedures. Samples were spiked with13C-labelled quantification standards and extracted and cleaned up using SPE cartridges. Extracts were instrumentally analyzed using liquid chromatography/mass spectrometry (LC/MS/MS). Analyte concentrations were determined by isotope dilution/ internal standard quantification. Reporting limits were defined as the concentration equivalent to the lowest calibration standard or the sample specific detection limit, whichever was greater.

Instrument Calibration
All initial calibrations specifications were reported as met. Initial calibration percent recoveries and retention times demonstrate ongoing precision and accuracy.

Continuing Calibration
All continuing calibration and verification specification were met.

Internal standards
All ongoing precision and recovery specifications were met.

Target Compound Results
No apparent matrix interferences were noted in the analysis of the target compounds. Sample analyte concentrations were not blank corrected and results should be evaluated with consideration of the procedural blank results.

3. Data Analysis

In this study, the MDL was used to determine if an analyte was present in a sample and the PQL was used to make a quantitative determination of the amount of analyte in the sample. The U.S. EPA uses the term MDL and PQL to describe the specific approaches of estimating the detection and quantification limits, respectively. If comparing concentration directly to a standard, it must be greater than the quantification limit in order to provide a reliable estimate whether or not the standard has actually been exceeded. To determine whether or not an analyte is present or absent in a sample, a result will be above the detection limit. Measurements above the quantification limit can be used directly. Measurements below the quantification limit are considered censored and must be appropriately adjusted.

The blank data from this experiment showed rather conclusively that PFOS concentrations found in the electroplater effluents were tied to PFOS-containing suppressants. It is also unlikely that PFOS or other PFCs were introduced at significant levels through other means. Laboratory blank samples were free of contamination. Field blanks, consisting of commercially available reagent water, were also free of significant contamination. Facility source waters were analyzed to evaluate potential contamination coming into the facilities. These samples were also free of significant PFOS concentrations. Surrogate recoveries for the lab, field, and source water blanks ranged from 69 to 148% and averaged 105%, demonstrating that sample preparation and analyses were free of contamination.

Analysis of Quality Control Background PFOS Sampling (in ppt)

Blanks	Analyses	Min	Max
Laboratory	3	0	0
Field	2	0	0
Source Water	2	2.52	5.75

Note: Lab blanks were free of contamination.
One field blank contained trace level of PFHxS.
Source water samples contained trace levels of PFOS and other PFCs.

Laboratory Control Samples

Three laboratory control samples were analyzed for the Chicago and Cleveland batch of samples as well as the re-analysis of selected samples. PFOS recoveries ranged from 89 to 105%, and had an average recovery of 96% (see below).

Sample Name	**A-Ohio**	**B-Illinois**	**C-Illinois**		
Analyte	% Recovery	% Recovery	% Recovery	AVG	%RPD
PFBA	77.3	107	85.3	90	33
PFPeA	87.1	109	94	97	23
PFHxA	84	119	101	101	35
PFHpA	76.8	117	102	99	41
PFOA	84.4	112	78.1	92	37
PFNA	89.9	96.6	99.2	95	10
PFDA	104	98.8	118	107	18
PFUnA	107	94.2	118	106	22
PFDoA	86.1	119	95.5	100	33
PFBS	102	110	113	108	10
PFHxS	85	119	101	102	33
PFOS	89.2	105	94.2	96	16
PFOSA	94.7	100	109	101	14

Matrix Spike Duplicates

Matrix spike and spike duplicate analyses were performed to evaluate the potential for sample interferences. Matrix interferences are also referred to as matrix effects. Matrix spike interferences are those chemical and/or physical interferences that impede the analytical instrumentation in detecting the true value concentration of a target analyte within a sample. One possible source of matrix interferences may be caused by contaminants that are co-extracted from the sample and result in a positive or negative bias. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the sample matrix.

	MATRIX SPIKE	MATRIX SPIKE DUPLICATE		
	Illinois	Illinois		
ID	Matrix Spike I	Matrix Spike Duplicate I		
Sample Size	0.0349 L	0.0318 L		
Analyte	% Recovery	% Recovery	AVG	%RPD
PFBA	101	89.6	95	12
PFPeA	92.4	120	106	26
PFHxA	101	114	108	12
PFHpA	99	109	104	10
PFOA	81.8	122	102	39
PFNA	116	112	114	4
PFDA	99.1	90.1	95	9
PFUnA	76.9	88.7	83	14
PFDoA	93.5	87	90	7
PFBS	104	109	107	5
PFHxS	98.2	101	100	3
PFOS	95.8	105	100	9
PFOSA	105	114	110	8

Spike recoveries for PFOS ranged from 96 to 105 averaging 100% with a 9% RPD. While these recoveries were within laboratory specifications, the spiking concentrations were well above the sample concentration. This practice did not allow an appropriate assessment of the impacts from the sample [see comparison table of laboratory control samples (LCS) and matrix spike(MS) /matrix spike duplicates (MSD) %RPDs below].

Comparison of Precision between LCS and MS/MSD

	MS/MSD	LCS
Analyte	%RPD	%RPD
PFBA	12	33
PFPeA	26	23
PFHxA	12	35
PFHpA	10	41
PFOA	39	37
PFNA	4	10
PFDA	9	18
PFUnA	14	22
PFDoA	7	33
PFBS	5	10
PFHxS	3	33
PFOS	9	16
PFOSA	8	14

Field Duplicates

A field duplicate is a duplicate sample collected by the same team or by another sampler or team at the same place, at the same time. It is used to estimate sampling and laboratory analysis precision. PFOS duplicate analyses ranged from 40 to 96% RPD and demonstrated variable

precision for the selected samples. Values for RPD appeared dependent on the concentrations found in the sample. Analysis of sample with low concentration of PFOS appeared more precise (i.e., lower RPD). Samples with higher concentration of PFOS appear less precise (i.e., higher RPD). The laboratory narrative report from AYXS attributes these variable recoveries to the presence of particulate matter in the subject samples. Given the limited number of samples collected and the general expectation that PFOS will attach to particulate matter, future analyses should ensure greater homogenization of samples or collection of sufficient samples such that statistical evaluations may be conducted. Based on the particulate-free LCS results and their acceptable precision and accuracy, we believe the variability in wastewater sample recoveries do not impinge our study conclusions.

Field Duplicate Results

		Cleveland	**&**	**Chicago**	**Dup**	**Sampling**	
ID							
Sample size	**0.0163 L**	**0.0154 L**		**0.0658 L**	**0.0727 L**	**0.0718 L**	
Analytes	**ppt**	**ppt**	**%RPD**	**ppt**	**ppt**	**ppt**	**%RPD**
PFBA	48.3	45.6	6	< 7.60	8.6	17.1	66
PFPeA	30.9	33.4	8	8.29	9.93	7.4	30
PFHxA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFHpA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFOA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFNA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFDA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFUnA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFDoA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFBS	41800	39900	5	1410	1580	1820	26
PFHxS	306	227	30	8900	11400	12600	34
PFOS	708	470	40	2040	6180	4680	96
PFOSA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0

Surrogate Spikes

A surrogate is a pure analyte that is extremely unlikely to be found in any sample. It is added to a sample aliquot in known amounts before extraction and is measured with the same procedure used to measure other sample components. A surrogate behaves similarly to the target analyte and most often used with organic analytical procedures. The purpose of a surrogate analyte is to monitor method performance with each sample. This study used 7- C 13 substituted isotopes.

For all samples collected, surrogates recoveries ranged from 25 to 148% and averaged 91%. These recoveries were within historical laboratory specifications and analyses were generally within control. For the analyte specific surrogate, 13-C4 PFOS recoveries ranged from 53.1 to 132% and averaged 90%. These recoveries demonstrate acceptable precision and accuracies for evaluating the target compound.

		Surrogate	Percent	Recoveries							
Sample ID	A	B	C	D	E	F	G	H	I	J	K
LABELED COMPOUND											
13C4-PFBA	115	119	118	79.6	24.7	28	84.6	47	78.2	46.9	105
13C2-PFHxA	107	124	114	99.2	94.2	40	95.7	80.5	91.4	61.5	115
13C2-PFOA	138	113	116	101	109	106	85.9	109	113	93.4	104
13C5-PFNA	77.8	147	66	137	126	66.4	96.6	78.2	85.7	69.9	111
13C2-PFDA	75.4	124	108	69.5	90.3	43.7	84.9	80.9	66	82.2	78.8
13C2-PFDoA	111	148	83.7	84.7	114	69.7	60.2	89.2	74.5	73.4	86.8
13C4-PFOS (80)	100	132	132	83	82.7	53.1	93.8	70.6	55.7	72.6	103

PFOS/PFC Suppressant Analysis

Eleven facilities were sampled and analyzed for the presence of PFOS, and other PFCs. A review of facility records showed that at least eight different suppressants or mixtures were used at the various facilities. Several facilities did not provide information on the suppressant used. Ten facilities had PFOS wastewater sample results above the MDL. For this study, the PQL was defined as 5 times the MDL. The positive PFOS results ranged from 231 to 2976 % of the calculated sample PQLs.

Facility Results Compared To Quantitative Definitions

Facility	AVG Sample size	PFOS (ppt)	Sample specific MDL	PQL (5XMDL)	% >PQL
#1	0.0619 L	31100	209	1045	2977
#2	0.0163 L	708	61.2	306	231
#3	0.0269 L	U	37.1	185	0
#4	0.0601 L	39000	423	2115	1844
#5	0.200 L	2320	42.9	214.5	1082
#6	0.201 L	1380	11.8	59	2339
#7	0.498 L	301	9.13	45.7	659
#8	0.494 L	1770	32.1	161	1099
#9	0.350 L	4460	97.5	488	914
#10	.497 L	31.4	2.01	10.05	312
#11	.0718 L	4680	33	165	2836

4. VERIFICATION OF ASSUMPTIONS

We have verified the following assumption in evaluating our study question, “Is PFOS discharged from decorative chrome plating operations?”

- Various Cr(VI) control methods are available;
- PFC-containing mist suppressants are in common industry use;

- Composition of suppressants may include various PFOS formulations;
- Active suppressant ingredients contain other PFCs beyond PFOS;
- Suppressant application is monitored;
- Electroplating discharges are amenable to PFOS analysis; and
- PFOS analyses are reasonably precise, accurate, recoverable, and reproducible.

5. SUMMARY OF CONCLUSIONS

Several conclusions can be made from the data collected:

- ✓ The small sample size limits the ability to draw conclusions beyond the observation that PFOS as well as other PFCs appear to be discharged from decorative chromium electroplating facilities through wastewater discharge;
- ✓ These discharges are quantifiable;
- ✓ Composition of PFOS containing mist suppressant vary widely;
- ✓ Variability in wastewater sample recoveries do not impinge our study conclusions; and
- ✓ PFOS data obtained from this study were appropriate to meet the study objective, and were of the right type, quality, and quantity to support the intended use.

Appendix B. Facility Operations

	Facility #1	**Facility #2**	**Facility # 3**	**Facility # 4**
Plating and metal finishing operations	Two lines of decorative chrome electroplating with chromic acid on plastic parts. A third plating line applies a gold-colored finish to nickel-plated parts.	Decorative chrome electroplating with chromic acid on metal and plastic parts. One line uses plastic parts and one uses metal parts.	Decorative chrome electroplating with chromic acid on plastic parts.	Decorative chrome electroplating with chromic acid on metal parts (brass, steel, and stainless steel), cadmium plating, and chromate conversion coating.
Operating schedule	24 hours/day, 5 days/week	Plastic substrate parts line: 24 hours/day, 5 days/week Metal substrate parts line: 8 hours/day, 5 days/week	24 hours/day, 5 days/week	10 hours/day, 4 days/week
Chromium electroplating process description	Parts are prepared by dipping in a chromic acid etch bath, neutralization tank, palladium-tin activator bath, and accelerator to remove tin. The plating process includes copper strike, bright acid copper, electroless nickel (and semi-bright, high sulfur and bright nickel for exterior use parts), and chromium electroplating with chromic acid. Each step is followed with rinsing.	Plastic substrate parts line: Parts are prepared by dipping in a chromic acid etch bath, neutralization tank, activator, and accelerator. The plating process includes electroless copper, copper strike, bright acid copper, nickel, and chromium electroplating with chromic acid. Each step is followed by rinsing. Metal substrate parts line: Parts are prepared by dipping in a cleaner and then an acid tank. The plating process includes copper strike, bright acid copper, nickel, and chromium electroplating with chromic acid. Each step is followed with rinsing.	Parts are prepared by dipping in a chromic acid etch bath, neutralization tank, activator, and accelerator. The plating process includes electroless copper, copper strike, bright acid copper, semi-bright or satin nickel, bright nickel, microporous nickel, and chromium electroplating with chromic acid. Each step is followed with rinsing.	Parts are prepared by dipping in a soak cleaner, electro-cleaner, and then sulfuric acid (with or without current). The plating process includes nickel strike, bright nickel, and chromium electroplating with chromic acid. Each step is followed with rinsing.

	Facility #5	Facility #6	Facility #7	Facility #8	Facility #9	Facility #10	Facility #11
Plating and metal finishing operations	This facility operates two chrome tank lines which are 225-gallons each.		This facility operates a single 350-gallon chrome tank.	This facility operates a single 400-gallon chrome tank.	This facility operates a 500-gallon chrome tank.	This company operates a 500-gallon chrome tank.	This facility operates a large 4000-gallon chrome tank. The facility decorates a variety of metal parts including shopping carts and other pieces.
Operating schedule		This facility has not used its chrome tank for over sixty days and rarely chromes metal pieces.	2 hour/day, maximum of 15 hours/week.		1 day/week.	2-4 hours/day.	8 hours/day, 5 days/week.
Chromium electroplating process description							

Appendix C. Rinsing Practices, Pretreatment, and Wastewater Discharge

	Facility #1	Facility #2	Facility #3	Facility #4
Rinsing practice for chromium electroplated parts	The interior parts line has four city water rinses followed by one deionized water rinse. The exterior parts line has three city water rinses followed by one deionized water rinse. Rinsewaters flow counter-currently.	The metal substrate parts line has three city water rinses. Rinsewaters flow countercurrently. The plastic substrate parts line has four city water rinses followed by one deionized water rinse. Rinsewaters flow countercurrently.	Two city water rinses followed by three deionized water rinses. Rinsewaters flow countercurrently.	One city water static rinse, three countercurrent city water rinses, then one deionized water static rinse. The final deionized water static rinse is emptied daily.
Rinse water pretreatment	Acid, chromium electroplating, electroless nickel, and copper-nickel rinsewaters are received in separate tanks in the pretreatment plant. Nickel is recovered by ion exchange before nickel rinsewaters are pumped to the pretreatment facilities. At the pretreatment plant, acid rinsewaters are combined with chrome-bearing rinsewaters. Chrome is reduced with sodium metabisulfite. Dissolved metals in electroless nickel rinsewaters are precipitated with calcium chloride. Chemically treated chromium and nickel wastewaters are pumped to the copper-nickel tank, where pH is adjusted with acid or caustic. Solids are settled in three clarifiers in series with the addition of a flocculent. Clarified wastewater is pumped to a storage tank with level control. When the tank level reaches a set point, wastewater is pumped to a sand filter. Filtered wastewater flows to a discharge tank. Clarifier sludge is pressed. Filtrate is returned to the copper-nickel tank.	Chrome-bearing rinsewaters are reduced by lowering pH with sulfuric acid and adding sodium metabisulfite. Reduced chrome rinsewaters are combined with other metal-bearing rinsewaters in the acid/alkali tank and neutralized with caustic. Solids are settled in a clarifier with the addition of flocculent polymer. Clarified wastewater flows through a surge tank and equalization tank. Wastewater then flows through a sand filter prior to discharge. Clarifier sludge is pressed. Filtrate is returned to the acid/alkali tank.	Chrome-bearing rinsewaters are reduced in a 4-stage tank by lowering pH to 2.5 s.u. with sulfuric acid and adding sodium metabisulfite. Other rinsewaters and calcium chloride are mixed with reduced chrome rinsewaters to raise the pH. Solids are settled in a clarifier with the addition of polymer. Clarified wastewater discharges from a flow-through final effluent tank. Clarifier sludge is thickened and pressed. Filtrate is returned to the clarifier. Electroless copper rinse water is treated separately by plating on steel wool.	Chrome-bearing rinsewaters (from chrome electroplating and chromate conversion) are reduced by lowering pH with sulfuric acid and adding sodium metabisulfite. Other rinsewaters are combined with reduced chrome rinsewaters and the pH is raised with sodium hydroxide. Solids are settled in a clarifier with the addition of polymer. Clarified wastewater is discharged. Clarifier sludge is dewatered in a filter press. Filtrate is returned to the clarifier. Cyanide plating rinse water is batch-treated with sodium hypochlorite, then combined with other rinsewaters.
Average wastewater discharge for operating days during June 2008[1]	97,000 gal/day	29,400 gal/day	146,000 gal/day	6,700 gallons/day

[1] Discharge flow data were provided by the supervisor of enforcement, water quality & industrial surveillance, Northeast Ohio Regional Sewer District. The companies' permits require that self-monitoring data be reported to NEORSD.

	Facility #1	Facility #2	Facility #3	Facility #4
POTW that receives wastewater from facility	Northeast Ohio Regional Sewer District Westerly WWTP	Northeast Ohio Regional Sewer District Easterly WWTP	Northeast Ohio Regional Sewer District Easterly WWTP	Northeast Ohio Regional Sewer District Southerly WWTP
POTW design flow	35 million gal/day	155 million gal/day	155 million gal/day	175 million gal/day

	Facility #5	Facility #6	Facility #7	Facility #8	Facility #9	Facility #10	Facility #11
Rinsing practice for chromium electroplated parts	The chrome pieces are dipped into a single rinse tank. Rinse tank flows are approximately 1-2 gpm.	Discharge flow through chrome rinse tanks are 1-2 gpm.	Rinse waters at the chrome tank flow between 2-4 gpm.	After metal pieces are chromed they pass thru two dead rinse tanks, followed by a running tank at 1-2 gpm.	After metal pieces are chromed they are dipped into two static rinse tanks, followed by three flowing rinse tanks 0.5 gpm.	Rinse waters flow are 5 gpm.	They operate three rinse tanks (one counterflow). The make up rinse rate is approximately 2 gpm.
Rinse water pretreatment	Pretreatment consists of a series of oxidations tank, followed by a flocculation tank, followed by a clarifier, then discharged to a sewer.	The facility's pretreatment system consists of an oxidation tank, flocculation tank, clarifier holding tank, then finally to discharge. Flow thru entire pretreatment system is approximately 20 gpm.	All rinse waters flow to a pretreatment system. Entire flow thru the pretreatment system is 50 -60 gpm.	This facility operates a complete pretreatment system consisting of a pH adjust with flocculation agent, clarifier, final filtration then discharge. Flows thru the pretreatment system are typically 50 -55 gpm.	This facility operates a complete pretreatment system consisting of a pH adjust with flocculation agent, clarifier, final filtration, then discharge. Flow thru the pretreatment system is about 55-55 gpm.	The facility operates a complete pretreatment system consisting of chrome reduction tank, flocculation tank, clarifier, sand filters and pH adjust tank. Flow thru the pretreatment system is about 80 gpm.	The electroplating shop operates a complete pretreatment system. It includes a chrome reduction tank, an equalization tank, a pH adjust tank, a clarifier, and finally, an effluent discharge pipe. Typical flows thru the pretreatment system varies between 50 - 55 gpm
Average wastewater discharge							
POTW that receives wastewater from facility							
POTW design flow							

Appendix D. Hexavalent Chromium Controls

	Facility #1	Facility #2	Facility #3	Facility #4
Chemical fume suppressant and add-on air pollution control devices used at facility	Mist Suppressant A, B and C	Mist Suppressant D and E	Mist Suppressant B and F	Mist Suppressant D
Tensiometer readings	Surface tension not greater than 35 dynes/cm as measured by a tensiometer	Surface tension not greater than 45 dynes/cm as measured by a stalagmometer	Surface tension not greater than 35 dynes/cm as measured by a tensiometer	Surface tension not greater than 45 dynes/cm as measured by a stalagmometer
Amount of chemical fume suppressant used	~2.6 gal/week of Mist Suppressant A, ~1.5 gal/week of suppressant B, and ~ 0.9 gal/week of suppressant C.[2]	1.2 gal/week of mist suppressant D and 3.5 gal/week of mist suppressant E. (Mist suppressant E usage is about 1.1 gal/week for the chromic acid tanks and 2.4 gal/week for the chrome etch tank.)[3]	8-9 gal/week of mist suppressant F.[4] The usage of mist suppressant B was not determined.	0.06 gal/week of mist suppressant D.[5]

[2] The company will cease using mist suppressant C once it depletes its inventory.

[3] These are average values based on the following usage – 16 gallons, 15 gallons, and 15 gallons of mist suppressant D during the third and fourth quarters of 2007 and the first quarter of 2008; 47 gallons, 44 gallons, and 46 gallons of mist suppressant E during the third and fourth quarters of 2007 and the first quarter of 2008. 300 mL/day of mist suppressant E (0.4 gallons/week) are added to the chromic acid tank of the metal substrate line and 500 mL/day of Mist Suppressant E (0.7 gallons/week) are added to the chromic acid tank of the plastic substrate line based on surface tension logs. The remainder of suppressant E's usage is for the plastic substrate line chromic acid etch tank.

[4] This value was given verbally by Facility #3 Director of Engineering during the inspection.

[5] This is an average value calculated by summing the amount of chemical fume suppressant recorded on the company's surface tension log from February 19, 2007 through June 12, 2008 and dividing by the number of calendar weeks during that period.

	Facility #5	Facility #6	Facility #7	Facility #8	Facility #9	Facility #10	Facility #11
Chemical fume suppressant and add-on air pollution control devices used at facility	Mist Suppressant G	Unknown	Mist Suppressant H	Mist Suppressant H	Unknown	Unknown	Unknown
Tensiometer readings	Recent tensiometer value of 44 dynes/cm2 was measured on June 7, 2008.	Latest tensiometer reading at chrome tank was measures at 43dynes/cm2 on April 10, 2008.	Latest tensiometer values of 23.6 dynes/cm2 were measured on June 2, 2008.	Latest tensiometer reading of 41dynes/cm2 was measured on June 9, 2008.	Last tensiometer reading was 27.3 dynes/cm2 as measured on June 3, 2008.	The last tensiometer value recorded at the chrome tank was 33.96 dynes/cm2 on May 29, 2008.	
Amount of chemical fume suppressant used							