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Testing Nickel Emissions from Metal Finishing Operations (Phase 2)

Final Report

National Risk Management Research Laboratory
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

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FOREWORD

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Sally Gutierrez, Director
National Risk Management Research Laboratory

PREFACE

This report provides an overview of nickel plating air emissions and waste release issues within the nickel plating industry. It is the objective of this report to assist the metal finishing community and specifically, those involved with nickel plating operations, with the management of environmental challenges that result from wastes that are potentially generated by nickel plating. Both the electrodeposition and electroless deposition processes for nickel plating have been profiled to examine resultant waste streams and potential releases.

Nickel plating practitioners are challenged to making a high-quality product that meets the needs of the customer while being competitive within the market. Furthermore, nickel plating practitioners must deal with environmental, regulatory, and technical, requirements to protect human health and the environment. This report serves as an advisory to nickel plating practitioners by providing technical information to reduce environmental impacts and lower the liabilities associated with environmental releases.

NOTICE

This document has been reviewed in accordance with the U.S. Environmental Protection Agency's peer and administrative review policies and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

ACKNOWLEDGMENTS

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ABSTRACT:

Nickel emissions from electroplating operations are of concern to EPA**.** Therefore, EPA has determined that it would be useful and important to obtain **actual** nickel emission data from nickel plating operations using EPA-Approved testing methods (both Method 29 and Method 306A). The five nickel plating processes tested in this project were: Electroless Nickel, Rack Watts Nickel, Barrel Watts Nickel, Nickel Sulfamate, and Wood's Nickel Strike Plating. In addition, by modifying some of the operating parameters (i.e. surface tension, agitation methods, etc.), EPA can determine the impact of these operational changes on nickel emissions.

The stack testing was conducted over a two year span from June of 2004 through January of 2006. Based on the stack testing results, it was concluded that:

- Nickel emissions from typically-operated Rack Watts Nickel and Rack Nickel Sulfamate tanks (wetting agent, no mesh pad, air agitation) are not very significant (<0.05 mg/dscm).

- Nickel emissions from <u>uncontrolled and vented</u> Electroless Nickel, Barrel Nickel, and Wood's Nickel Strike plating are significant (greater then 0.1 mg/dscm), but can be significantly reduced (49% to 92%) by employing eductors, wetting agents, or simple mesh pads.

- Wetting agents, simple mesh pads, and eductors can be used to reduce nickel emissions from these processes with varying degrees of success. Simple mesh pads were found to be the most favorable because they outperformed wetting agents and eductors, are less expensive, and are totally external to the plating process (no impact on the plating chemistry).

Although the results of the study indicate that USEPA Method 306A and USEPA Method 29 provide similar results when measuring nickel emissions from Electroless Nickel, Watts Nickel, and Wood's nickel strike, further testing needs to be conducted on a controlled process or conducted simultaneously to determine whether Method 306A truly is an acceptable alternative to Method 29 for measuring nickel emissions from nickel plating processes in general.

Section 1.0 PROJECT DESCRIPTION AND OBJECTIVES

Purpose and Background

The nickel plating industry is a large part of the metal finishing community in the United States. It consists of job shops (independently owned plating businesses) and captive shops (metal finishing operations contained in larger manufacturing facilities). There are over 3,000 U.S. job shops that average fewer than 50 employees each with annual sales of, approximately $5 million. Captive shops that support larger manufacturing facilities can vary in size depending on their role within the company. The metal finishing industry is regulated for environmental protection and occupational health and safety due to the nature of the processes and materials required to satisfy industrial and public consumer demand. Nearly all manufactured products require some type of surface finishing. Consumers demand products that have aesthetic appeal, will not deteriorate, and have durability. The nickel plating industry provides a product that improves appearance, slows or prevents corrosion, changes magnetic properties, and increases strength and resistance to wear for manufactured parts and products.

Most of the nickel plating industry is located in or near major metropolitan areas and may generate air emissions, water discharges, and solid wastes that add to overall pollution concerns. Pollution abatement costs and expenditures for the metal finishing industry comprise nearly 20% of its budget. Industry representatives are working together with government, trade associations, and professional organizations to encourage technological advances that lead to more efficient, cleaner production while reducing waste generation and control costs.

Nickel plating is most commonly applied though the utilization of aqueous chemical reagents by means of electroplating or via a chemical reducing agent (a process that is referred to as electroless plating). Typical constituents of nickel electroplating solutions include nickel sulfate (or nickel sulfamate) nickel chloride, and boric acid, along with inorganic or organic additives that modify the crystal structure of the nickel deposit. While a variety of formulations for nickel plating exists, the Watts, Woods, and Sulfamate processes comprise the majority of the formulations used by the metal finishing industry. Also, electroless nickel is used throughout the industry and these process solutions are based on nickel sulfate plus a reducing agent containing boron or phosphorus. The phosphorus-based solutions produce nickel alloy deposits ranging from about 4% to about 12% phosphorus.

Nickel emissions from electroplating operations are of concern to EPA. Air emission estimation for electroplating is described in EPA’s AP42 Manual. As part of this manual, the AP42 states: “Emissions from plating operations other than chromium electroplating can be estimated using the emission factors and operating parameters for chromium electroplating.” Based on this theory, EPA developed a computer based Metal Finishing Facility Risk Screening Tool (MFFRST) which estimates emissions from electroplating operations. The tool is believed to be very accurate for chromium emissions, however, there was a need to validate air emissions for other metals such as nickel.

In Phase I of this project, it was determined that the MFFRST Model that used the AP42 methodology was not very accurate in estimating nickel emissions from nickel plating operations. Therefore, EPA has determined that it would be useful and important to obtain more **actual** nickel emission data from nickel plating operations to improve AP42 estimates using EPA-Approved testing methods (both Method 29 and Method 306A).

In addition, by modifying some of the operating parameters from one test to another within the plating process (i.e. surface tension, agitation methods, etc.) and then comparing the test results, EPA can determine the effects of these operating parameters on nickel emissions.

Process, Site, Facilities Tested

Actual data was collected by stack testing various types of nickel plating processes, operating under several different conditions, using EPA-Approved procedures. Similar testing was also conducted in Phase 1 of this project in 2004.

For Phase 2, the exhausted air from five different types of nickel plating processes was sampled and analyzed for nickel in order to obtain the nickel emissions from each process. These nickel plating processes were all production lines located in actual plating facilities. Each nickel plating process was tested several times under various conditions in order to observe the effects of that condition on nickel emissions. There were a total of twenty-three (23) actual stack tests conducted during this phase. The nickel plating processes that were tested were: Electroless Nickel Plating, Rack Watts Nickel Plating, Barrel Watts Nickel Plating, Rack Nickel Sulfamate Plating, and Rack Woods Nickel Strike Plating. The various conditions under which each of these processes was tested are summarized in the Table 1.

Some nickel plating processes were tested more (varying more operating conditions) than other nickel plating processes (i.e. Rack Watts Nickel is tested under more operating conditions than Woods Nickel Strike). The total number of stack tests (23) was based solely on the budget of the project. The decision on which plating processes were tested more than others was based on the popularity of that process in the industry. In other words, Rack Watts Nickel is far more common in the industry than Woods Nickel Strike so more emphasis (i.e. more conditions were varied) was given to Rack Watts Nickel.

The testing sites for each of these nickel plating processes were as follows:

1. Electroless Nickel Plating

Facility: Reliable Plating Company
Site: 1538 West Lake Street
Chicago, IL 60607

2. Rack Watts Nickel Plating

Facility: America’s Best Quality Coatings

Site: 1602 S. First Street
Milwaukee, WI 53204

3. Barrel Watts Nickel Plating

Facility: Artistic Plating Company
Site: 405 W. Cherry Street
Milwaukee, WI 53212

4. Rack Nickel Sulfamate Plating

Facility: Elite Finishing, LLC
Site: 3270 S. 3rd Street
Milwaukee, WI 53207

5. Rack Woods Nickel Strike Plating

Facility: Artistic Plating Company
Site: 405 W. Cherry Street
Milwaukee, WI 53212

The six (6) stack tests that were completed on nickel plating processes as Phase 1 of this project are also included in Table 1 (identified as "Completed - Phase 1").

The actual nickel emission results from Phase 1 and Phase 2 were also used to estimate the effects of several plating operating parameters on nickel emissions. Based on the operating conditions selected and summarized in Table 1, we were able to estimate the effects of the following operating conditions/parameters on nickel emissions:

- air agitation versus pump (eductor) agitation
- the use of wetting agents to control the surface tension of the plating solution
- the use of simple mesh pads on the exhaust hood to control splashing
- the ratio of nickel emissions attributed to electrolysis (electroplating) versus attributed to barrel transfer (entrainment from splashing) in barrel plating
- the ratio of nickel emissions attributed to plating versus attributed to agitation and evaporation in electroless nickel plating
- the type of stack testing used (Method 29 versus Method 306A)

Table 2 summarizes these selected process parameters and which tests were compared to each other when estimating the effects of the selected parameter.

The data collected in Phase 1 was also used in Phase 2 to compare nickel emissions from various nickel processes and from same nickel processes operated under different conditions. The results from Phase 1 and Phase 2 are comparable because identical sampling and testing methodologies was used in both phases (i.e. Method 29, three individual test runs per stack test, etc.) and because, in many cases, the same nickel plating site was tested in both Phase 1 and Phase 2 (except under different operating conditions).

TABLE 1

USEPA Nickel Project Process Summary

#	Process		Surface Tension/ Wetting Agent		Mesh Pad		Air Agitation/ Eductor		Tank Loading	Status	Site	Method
1	Electroless Nickel		NA		No				Low	**Completed- Phase 1**	Reliable	Method 29
2	Electroless Nickel		NA		No				Low	**Completed- Phase 1**	Reliable	Method 306A
3	Electroless Nickel								High	Phase 2 (USEPA-A)	Reliable	Method 29
4	Electroless Nickel						Eductor		High	Phase 2 (USEPA-B)	Reliable	Method 29
5	Electroless Nickel					Air	Eductor		High	Phase 2 (SCL 3)	Reliable	Method 29
6	Electroless Nickel		NA		No	Air	Air		High	Phase 2 (SCL 4)	Reliable	Method 29
		NA		Yes		Air						
8	Rack Watts (bright)	NA	Yes (30s)	Yes						**Completed- Phase 1**	Site B	Method 29
9	Rack Watts (bright)	NA	Yes (30s)	No			Eductor		High	**Completed- Phase 1**	Site B	Method 29
10	Rack Watts (bright)		No (45-60)		Yes		Eductor		High	Phase 2 (USEPA- C)	ABQC	Method 29
11	Rack Watts (bright)		No (45-60)		Yes		Air		High	Phase 2 (SCL 1)	ABQC	Method 29
12	Rack Watts (bright)		No (45-60)	No	No	Air	Air	High	High	Phase 2 (SCL 10)	ABQC	Method 29
13	Rack Watts (bright)		Yes (30s)	No			Eductor		High	Phase 2 (USEPA- D)	ABQC	Method 29
14	Rack Watts (bright)		Yes							Phase 2 (SCL 2)	ABQC	Method 29
15	Rack Watts (bright)		Yes							Phase 2 (SCL 2)	ABQC	Method 306A
16	Barrel Watts (dull)		Yes (33)	Yes						**Completed- Phase 1**	Artistic	Method 29
17	Barrel Watts (bright)		No (50s)	Yes		Air	NA	High	High	**Completed- Phase 1**	Artistic	Method 29
18	Barrel Watts (dull)		No (45-60)	Yes	Yes	Air		High		Phase 2 (USEPA-E)	Artistic	Method 29
19	Barrel Watts (dull)		No (45-60)		No				High	Phase 2 (SCL-9)***	Artistic	Method 29
20	Barrel Watts (dull)		Yes (30s)	No		NA		High		Phase 2 (USEPA-F)	Artistic	Method 29
21	Barrel Watts (dull)		Yes (30s)	No					High	Phase 2 (SCL-8)***	Artistic	Method 29
						NA		High				
22	Rack Sulfamate		Yes (30s)			NA				Phase 2 (SCL-5)	Elite	Method 29
23	Rack Sulfamate		Yes (30s)	Yes		NA	Eductor	High		Phase 2 (SCL-7)	Elite	Method 29
25	Rack Sulfamate		Yes (30s)	No		NA	Eductor			Phase 2 (USEPA-G)	Elite	Method 29
26	Rack Sulfamate		Yes (30s)							Phase 2 (USEPA-G)	Elite	Method 306A
				No		Air		High				
27	Woods Strike (Rack)		No (50-60)	No	No			High		Phase 2 (USEPA-I)	Artistic	Method 29
28	Woods Strike (Rack)		Yes (30s)	Yes	No		NA	High	High	Phase 2 (USEPA-H)	Artistic	Method 29
7	Woods Strike (Rack)		No (50-60)	No	Yes	Air		High		Phase 2 (SCL-6)	Artistic	Method 29
24	Woods Strike (Rack)		Yes (30s)							Phase 2 (SCL-11)	Artistic	Method 29
29	Woods Strike (Rack)		Yes (30s)		No	NA	NA	High		Phase 2 (USEPA-H)	Artistic	Method 306A
						NA		High				
				Yes		NA		High				
								High				

*** Absolutely no barrel transfer during testing

TABLE 2

Nickel Plating Process Operating Condition/Parameter	Tests Compared to Evaluate the Affect of the Condition/Parameter on Nickel Emissions	
Air Agitation versus Pump (Eductor) Agitation	3-4, 5-6 8-9, 10-11, 13-14 22-23	(Electroless Nickel) (Rack Watts) (Rack Sulfamate)
The Use of Wetting agents	8-12, 10-13, 11-14, 16-17, 18-20, 19-21, 27-28, 7-24	(Rack Watts) (Barrel Watts) (Rack Woods Strike)
The Use of Simple Mesh Pads in Exhaust Hood	4-5, 3-6 11-12, 9-13, 8-14 16-20 17-18 23-25 7-27, 24-28	(Electroless Nickel) (Rack Watts) (Barrel Watts) (Rack Sulfamate) (Woods Nickel)
The Ratio of Nickel Emissions Attributed to Electrolysis (electroplating) Versus Attributed to Barrel Transfer (entrainment from splashing) in Barrel Plating	17-19, 16-21	(Barrel Watts)
The Ratio of Nickel Emissions Attributed to Plating Versus Attributed to Agitation/Evaporation in Electroless Nickel Plating	1-6,	(Electroless Nickel)
The Type of Stack Testing Method Used (Method 29 versus Method 306A)	1-2 14-15 22-26 28-29	(Electroless Nickel) (Rack Watts) (Rack Sulfamate) (Rack Woods Strike)

Project Objectives

The objectives of this project were to 1) develop actual nickel air emission data using EPA-Approved Methods 29 and 306A that may also be used in the future to establish regulations for nickel emissions and/or revise the AP-42 Methodology and consequently the MFFRST Model, 2) compare the total nickel emissions obtained using EPA Method 29 and EPA Method 306A to determine whether Method 306A is a viable alternative to Method 29 for determining nickel emissions, and 3) evaluate the factors that affect nickel emissions such as surface tension, product loading, type of process solution and type of agitation used in the process.

Section 2.0 PROJECT ORGANIZATION

Contacts

Organization	Contact	
USEPA	David Ferguson	(513-569-7518)
Scientific Control Laboratories, Inc.	Frank Altmayer/Jeff Zak	(773-254-2406)
Artistic Plating Company	John Lindstedt	(414-271-8138)
ABQC Company	Matthew Kirchner	(414-649-4900)
Elite Finishing	Jaime Maliszewski	(414-489-9710)
Reliable Plating Company	Jim Greenwell	(312-421-4747)
ETE	Michael Huenink	(262-784-2434)

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Project Participants and Responsibilities

Name	**Responsibility**
David Ferguson, USEPA	USEPA Project Manager
Frank Altmayer, SCL	Project Manager

Name	**Responsibility**
Jeff Zak, P.E., SCL	Planning Coordination, Sample Collection, Sample Custody, Physical Measurement, Process Measurements, Data Validation, Data Reduction, Report Preparation
Linda Kenny, SCL	Analytical Measurements, QA Manager
Michael Huenink, ETE	Sample Collection, Physical Measurements, Process Measurements
John Lindstedt, Artistic	Testing Site Contact
Jaime Maliszewski, Elite	Testing Site Contact
Matthew Kirchner, ABQC	Testing Site Contact
Jim Greenwell, Reliable	Testing Site Contact
Chicago Plastics	Mesh Pad Supplier
Serfilco	Eductor Supplier

Section 3.0 PROCEDURE

General Approach and Testing Methodology

Three (3) test runs were conducted on each of the twenty-three (23) different nickel plating operations/conditions listed in Table 1 in order to measure the nickel concentration in the air emitted from each of these processes. Twenty (20) of the tests were conducted in accordance with EPA-Approved Method 29 of 40 CFR Part 60 Appendix A-8 (A copy of Method 29 is included in Appendix A). All applicable operating parameters were observed and recorded throughout the tests (i.e current density, temperature of plating solution, amperage, plating solution concentration, tank dimensions, etc.).

The nickel emissions from the remaining three (3) processes/conditions were measured in accordance with Method 306A of 40 CFR Part 63, Appendix A (with a couple of minor deviations detailed in Section 4.0). A copy of Method 306A of 40 CFR Part 63 Appendix A is included in Appendix B of this QAPP. The purpose of these tests was to compare the results obtained from Method 29 and Method 306A.

The stack sampling was conducted during normal plating operations at the selected facilities. Since normal plating operations at job-shop electroplating facilities are inherently **non**-steady state, the stack sampling did not rely on steady state conditions of the plating operations.

In order to ensure that the ventilation systems on each of the nickel plating processes were operating efficiently (capturing and exhausting all of the nickel emissions from the process), the ambient air near each process was also monitored/tested during the test runs. The ambient air was tested with two (2) OSHA-approved internal air monitors at each of the processes. The arithmetic mean of the two results was compared to OSHA worker exposure limits for nickel in order to grade the efficiency of the ventilation systems. If the average nickel concentration in the ambient air was below OSHA worker exposure limits for nickel, the ventilation system for that specific process was deemed efficient.

Sampling/Monitoring Points

1) Sampling Port Locations for Nickel Emissions Testing

The locations of the sampling ports in the exhaust stacks of each of the five individual nickel plating processes were determined in accordance with Section 8.1.2 of Method 29 of 40 CFR Part 60 Appendix A-8. Section 8.1.2 of Method 29 references Section 8.2.1 of Method 5 of 40 CFR Part 60 Appendix A which further references Method 1 of 40 CFR Part 60 Appendix A. Section 11.0 of Method 1 states that the sampling ports must be positioned at least two (2) stack or duct diameters downstream and a half diameter upstream from any flow disturbance. The exhaust systems from all five nickel plating testing sites were constructed so that they met this sampling port location requirement.

The sampling point locations were recorded into the field log sheets included with Appendix D of this report.

2) Traverse Point Locations for Nickel Emissions Testing

The location of the sampling (traverse) points within the exhaust stacks of each of the five individual nickel plating processes were determined in accordance with Section 11.0 of Methods 1 and 1A of 40 CFR Part 60 Appendix A. Method 1 is to be used for ducts with diameters greater than 12 inches. Method 1A is to be used for round ducts with a diameter less than 12 inches. The traverse point locations were documented and recorded on the field log sheets located in Appendix D of this report.

3) Location of Ambient Air Monitors

The ambient air monitors used to determine the effectiveness of the ventilation systems of each of the five nickel plating processes were positioned on stands or taped to columns at the perimeter of the nickel plating tanks.

4) Frequency of the Sampling Events:

Three (3) stack tests were conducted at each of the 29 testing conditions listed in Table 1 with the exception of Condition 13 in which processing problems limited the testing to one (1) stack test (See page 32 for a detailed explanation). Otherwise, all three test runs were conducted within the same day under similar operating conditions.

Section 4.0 DISCUSSION OF SAMPLING/ANALYTICAL PROCEDURES

Sampling Procedures

The sampling procedures described in Section 8.0 of EPA Method 29 of 40 CFR Part 60 Appendix A-8 (which further references Section 8.5 of Method 5 of 40 CFR Part 60 Appendix A-3) were followed when sampling the air emitted from twenty (20) of the twenty three nickel plating process scenarios listed in Table 1 for Phase 2. A copy of Method 5 of 40 CFR Part 60 Appendix A-3 was included in Appendix G of the approved QAPP.

The following parameters were recorded from each nickel plating processes throughout the testing/sampling:

- Temperature of Plating Solution
- Current Density (where applicable)
- Plating Bath Concentration, including surface tension
- Tank Dimensions
- Amperage/Voltage (where applicable)
- Types of Parts being Plated
- Plating Time
- Freeboard Height
- Air Agitation Rate (where applicable)
- Solution Circulation Rate

After the samples were collected from each test run, each sample container was marked with the following data:

- Nickel Plating Process Type
- Site Location
- Date
- Run #

Scientific Control Laboratories was responsible for completing the chain of custodies for all the samples taken at the site(s). The samples were in the custody of Scientific Control personnel at all times from each site to the Scientific Control Laboratories where they were analyzed for nickel. There was no splitting of the samples. Scientific Control Laboratories was responsible for all sampling and analysis. Environmental Technology and Engineering was subcontracted to supply the sampling equipment and calibration of sampling equipment.

Stack Testing Using USEPA Method 306A

The sampling procedure described in Section 8.0 of EPA Method 306A of 40 CFR Part 63 Appendix A (Included in Appendix C of approved QAPP) was followed when sampling the air emitted from three of the twenty three nickel plating process scenarios listed in Table 1 (with the exception of the minor deviations listed below)..

The following factors were recorded from the nickel plating processes throughout the Method 306A testing/sampling:

- Temperature of Plating Solution
- Plating Bath Concentration
- Tank Dimensions
- Types of Parts being Plated
- Freeboard
- Air Agitation Rate (where applicable)
- Solution Circulation Rate

After the samples were collected from each test run, each sample container was marked with the following data:

- Nickel Plating Process Type
- Site Location
- Date
- Run #

Scientific Control Laboratories, Inc. (SCL) was responsible for completing the chain of custodies for all the 306A samples taken at the site. The samples were in the custody of SCL personnel at all times from the site to the SCL laboratory where they were analyzed for nickel. There will be no splitting of the samples. SCL was responsible for all sampling and analysis; there were no subcontractors involved in this part of the project.

Modifications to Sampling Procedures in Method 306A

There were a few deviations to the EPA-Approved Method 306 A Procedure for this project. These modifications were discussed in the approved Quality Assurance Project Plan (QAPP) and include the following:

1) Section 8.1.1.9.1 B Instead of using 0.1N NaOH or 0.1N NaHCO3 as absorbing solution in the first impinger of the sampling train, a mixture of 5% HNO_3 and 10% H_2O_2 was used. This absorbing solution is the recommended absorbing solution for nickel as stipulated in EPA-Approved Method 29 of Appendix A of 40 CFR Part 60.

2) The current Method 306A (dated October 17, 2000) is a revision of the original version of Method 306A (dated January 25, 1995). The author of the original method (Frank

Clay of the USEPA) has been very critical of the new method. Mr. Clay has written an article, A Revised Method 306A- Second Review and Critique, that details the errors, omissions, and superfluous material that are found in the new 306A Method and offers some recommendations on how to improve the current method. After reading Mr. Clay's article and based on our experience of performing Method 306A, Scientific Control Laboratories concurred with Mr. Clay's opinion and included the following applicable recommendations (minor deviations) when using Method 306A.

1. A rocker switch was used to start and stop the sampling train instead of a bypass valve.
2. All runs were performed in a single day.
3. The cotton ball in the third impinger was omitted from the sampling train.
4. When recovering the sample, the tubing and nozzle (and the tubing between the first and second impinger) was rinsed three times in each direction with the absorbing solution instead of two.

A copy of Mr. Clay's article that justifies these recommendations is located in Appendix D of the approved QAPP. It is SCL's opinion that these proposed modifications are minor deviations that, in no way, affected the accuracy of the sampling results.

Ambient Air Monitoring

The sampling procedures as detailed in Section 5 (specifically Method 5.4) of OSHA-Approved Method ID-121 were followed when monitoring the internal air near the five (5) nickel plating processes. This sampling was conducted during all twenty-three (23) tests. A copy of OSHA Method ID-121 is included in Appendix F of the QAPP. Scientific Control Laboratories calibrated the air sampling pumps prior to each sampling.

Method 29 and Method 306A Field Blanks

Field Blanks were used to determine the quantity, if any, of nickel contamination in the virgin scrubbant (HNO3/H2O2) and rinse (HNO3) materials. The scrubbant material is used to capture the nickel emitted from the various nickel plating processes. The rinse material is used to rinse the probe and scrubbers after the testing. The nickel concentration present in the virgin scrubbant and rinse materials, if any, is subtracted from the nickel concentrations obtained from the actual testing.

Before testing, batches of virgin scrubbant and rinse materials were formulated at Scientific Control Laboratories.

Laboratory Analyses

The samples obtained from the stack testing were analyzed for nickel in accordance with the methods detailed in Section 11.0 of Method 29 of 40 CFR Part 60 Appendix A or Section 11 of Method 306A of 40 CFR Part 63 Appendix A. The analyses were conducted by Atomic Absorption-AA (Section 11.1.2 of Method 29). Only total nickel emissions were measured from the various nickel plating processes using Method 29. Particulates were not measured as part of this project.

The samples obtained from the OSHA internal air monitoring were analyzed for nickel in accordance with Section 6 of OSHA-Approved Method ID-121. The analyses were performed by ICAP-AES.

The QA/QC program(s) described in Sections 9.0 and 10.0 of Method 29 of 40 CFR Part 60 Appendix A-8 and Method 306A of 40 CFR Part 63 Appendix A were followed when sampling the nickel plating system stacks. The QA/QC program described in Sections 9.0 and 10.0 of Method 29 of 40 CFR Part 60 Appendix A were followed when analyzing the resultant samples for nickel.

The laboratory QA/QC data for the stack testing samples is included in Appendix E of this report.

The QA/QC program described in Section 6 of OSHA-Approved Method ID-121 was followed when analyzing the filter samples from the internal air monitoring for nickel. The laboratory QA/QC data for the ambient air samples is included in Appendix F of this report.

Section 5.0 SUMMARY OF RESULTS

The results of the project are presented in the following tables:

Table 3.	Summary of Nickel Emissions Obtained from Phase 1 and Phase 2 Stack Testing
Table 4.	Comparison of Nickel Emissions (Air Agitation versus Eductors)
Table 5.	Effects of Wetting Agents on Nickel Emissions
Table 6.	Effects of Mesh Pads on Nickel Emissions
Table 7.	Effects of Barrel Transfer on Nickel Emissions from Watts Nickel Barrel Plating
Table 8.	Effects of Product Loading on Nickel Emissions from Electroless Nickel Plating
Table 9.	Nickel Emissions Method 29 Compared to Method 306A
Table 10A-10E	Operating Parameters Observed During Stack Testing for Nickel Emissions
Table 11.	Ambient Air Nickel Concentrations Observed During Stack Testing

The data presented in Table 3 is the actual nickel emission concentrations from each of the three (3) individual test runs from all twenty-nine conditions (29) that were tested (including the six conditions tested as part of Phase 1). The nickel emission concentrations were calculated using the analytical data obtained in accordance with the Method 29 Stack Testing procedure (or the equivalent Method 306A Stack Testing Procedure) and were performed in accordance with Section 12 of Method 29 of 40 CFR Part 60 Appendix A-8 (or the equivalent Section 12 of Method 306A depending on which method was used for the sampling). The data used for these calculations for the twenty-three Phase 2 testing conditions is included in Appendix A of this report. The data used for the calculations for the six Phase 1 testing conditions is included in Appendix A of the Final Phase 1 Report. The average of the three (3) test runs for each of the twenty-nine (29) conditions is also reported in Table 3.

The data presented in Table 4 summarizes the effects of air agitation versus eductors on nickel emissions in electroless nickel, Watts nickel, and nickel sulfamate solutions based on the testing comparison protocol listed in Table 2.

The data presented in Table 5 summarizes the effects of wetting agents on nickel emissions in Watts nickel, nickel sulfamate, and Woods nickel strike solutions based on the testing comparison protocol listed in Table 2.

The data presented in Table 6 summarizes the effects of mesh pads on nickel emissions in electroless nickel, Watts nickel, nickel sulfamate, and Woods nickel strike solutions based on the testing comparison protocol listed in Table 2.

The data presented in Table 7 summarizes the effects of barrel transfer on nickel emissions from Watts nickel barrel plating based on the testing comparison protocol listed in Table 2.

The data presented in Table 8 summarizes the effects of product loading on nickel emissions from Electroless Nickel Plating based on the testing comparison protocol listed in Table 2.

The data presented in Table 9 are the stack test results obtained from testing the same process (electroless nickel, Watts nickel, nickel sulfamate, and Woods nickel strike) using two different sampling procedures (EPA Method 29 and EPA Method 306A).

The data presented in Table 10A through 10E are the individual nickel plating process operating parameters that were recorded during the stack tests.

The data presented in Table 11 are the ambient air nickel concentrations as calculated using the analytical data obtained in accordance with OSHA-Approved Method ID-121. These calculations were performed in accordance with Section 7 of OSHA-Approved Method ID-121. The OSHA eight (8) hour, time-weighted average PEL for nickel is also included in the table for comparison purposes. The data used for these calculations are included in Appendix B of this report.

TABLE 3

Summary of Nickel Emissions Obtained from Phase 1 and Phase 2 Stack Testing

												Results (mg/dscm)	
#	Process		Surface Tension/ Wetting Agent**		Mesh Pad		Air Agitation/ Eductor		Tank Loading	Run #1	Run #2	Run #3	Average
1*	Electroless Nickel								Low	0.478	0.126	0.220	**0.275**
2*	Electroless Nickel (306A)		NA						Low	0.060	0.311	0.381	**0.251**
3	Electroless Nickel									0.011	0.006	0.005	**0.007**
4	Electroless Nickel						Eductor		High	0.024	0.012	0.004	**0.013**
5	Electroless Nickel	NA		No		Air	Eductor		High	0.142	0.192	0.111	**0.148**
6	Electroless Nickel		NA	No	No	Air	Air		High	0.124	0.313	0.505	**0.314**
		NA		Yes		Air		High					
8*	Rack Watts (bright)	NA	Yes (38)	Yes						0.048	0.044	0.032	**0.041**
9*	Rack Watts (bright)	NA	Yes (38)	No			Eductor		High	0.016	0.027	0.028	**0.024**
10	Rack Watts (bright)		No (58)		Yes		Eductor		High	0.043	0.003	0.016	**0.021**
11	Rack Watts (bright)		No (57)		Yes					0.057	0.012	0.005	**0.025**
12	Rack Watts (bright)		No (57)	No	No	Air		High		0.051	0.133	0.056	**0.080**
13	Rack Watts (bright)		Yes (42)	No			Eductor		High	0.015	NA	NA	**0.015**
14	Rack Watts (bright)		Yes (37)							0.003	0.004	0.003/0.037/0.008	**0.011**
15	Rack Watts (bright) (306A)		Yes (37)		Yes	Air	Air	High	High	0.014	0.008	0.012	**0.011**
						Air		High					
16*	Barrel Watts (dull)		Yes (34)	Yes						0.080	0.061	0.118	**0.086**
17*	Barrel Watts (bright)		No (57)	Yes	No	Air		High		0.975	1.495	0.921	**1.130**
18	Barrel Watts (dull)		No (68)		Yes					0.009	<0.005	0.023	**0.012**
19	Barrel Watts (dull)***		No (68)		No					0.056	0.019	0.228	**0.101**
20	Barrel Watts (dull)		Yes (31)	No		NA		High		0.009	0.004	0.012	**0.008**
21	Barrel Watts (dull)***		Yes (31)			NA		High		0.023	0.007	0.009	**0.013**
						NA		High					
22	Rack Sulfamate		Yes (39)			NA		High		0.022	0.011	0.010	**0.014**
23	Rack Sulfamate		Yes (44)	Yes		NA	Eductor	High	High	0.018	0.013	0.020	**0.017**
25	Rack Sulfamate		Yes (44)	No		NA	Eductor	High	High	0.004	0.006	0.006	**0.005**
26	Rack Sulfamate (306A)		Yes (35)							0.031	0.061	0.053	**0.048**
				No		Air		High					
27	Woods Strike (Rack)		No (73)	No	No					0.614	0.282	0.515	**0.470**
28	Woods Strike (Rack)		Yes (30s)	Yes						0.129	0.162	0.252	**0.181**
7	Woods Strike (Rack)		No (61)	No	Yes	Air		High		0.063	0.081	0.236	**0.127**
24	Woods Strike (Rack)		Yes (30s)							0.048	0.077	0.071	**0.065**
29	Woods Strike (Rack) (306A)		Yes (40-50s)		No	NA	NA	High	High	0.400	0.165	0.218	**0.261**
				No		NA		High					
						NA		High					
				Yes		NA		High					

* Phase 1

** The units for the surface tension value in the parenthesis is dynes/cm

*** Absolutely no barrel transfer during testing

Table 4
Comparison of Nickel Emissions (Air Agitation versus Eductors)

Process:	Air Agitation	Eductors
Electroless Nickel	0.124	0.142
(No Mesh Pad)	0.313	0.192
6 vs 5	0.505	0.111
	0.314	**0.148**
Electroless Nickel	0.011	0.024
(Mesh Pad)	0.006	0.012
3 vs 4	0.005	0.004
	0.007	**0.013**
Rack Watts	0.048	0.016
(No Mesh Pad, Wetting Agent)	0.044	0.027
8 vs 9	0.032	0.028
	0.041	**0.024**
Rack Watts	0.057	0.043
(Mesh Pad, No Wetting Agent)	0.012	0.003
11 vs 10	0.005	0.016
	0.025	**0.021**
Rack Watts	0.003	0.015
(Mesh Pad, Wetting Agent)	0.004	NA
14 vs 13	0.003/0.037/0.008	NA
	0.011	**0.015**
Rack Sulfamate	0.022	0.043
(No Mesh Pad, Wetting Agent)	0.011	0.003
22 vs 23	0.010	0.016
	0.014	**0.017**

Results reported in mg/dscm

Table 5
Effects of Wetting Agents on Nickel Emissions

Process:	**No Wetter**	**Wetter**
Rack Watts	0.051	0.048
(No Mesh Pad, Air)	0.133	0.044
12 vs 8	0.056	0.032
	0.080	**0.041**
Rack Watts	0.043	0.015
(Mesh Pad, Eductor)	0.003	NA
10 vs 13	0.016	NA
	0.021	**0.015**
Rack Watts	0.057	0.003
(Mesh Pad, Air)	0.012	0.004
11 vs 14	0.005	0.003/0.037/0.008
	0.025	**0.011**
Barrel Watts	0.975	0.080
(No Mesh Pad)	1.495	0.061
17 vs 16	0.921	0.118
	1.130	**0.086**
Barrel Watts	0.009	0.009
(Mesh Pad)	<0.005	0.004
18 vs 20	0.023	0.012
	0.012	**0.008**
Barrel Watts	0.056	0.023
(No Mesh Pad, No Transfer)	0.019	0.007
19 vs 21	0.228	0.009
	0.101	**0.013**
Wood Strike	0.614	0.129
(No Mesh Pad)	0.252	0.162
27 vs 28	0.515	0.252
	0.470	**0.181**
Wood Strike	0.063 (0.089)*	0.048
(Mesh Pad)	0.081 (0.159)*	0.047
7 vs 24	0.236	0.071
	0.127 (0.161)*	**0.065**

Results reported in mg/dscm

* Results adjusted to compensate for cfm difference between Condition 7 and 24 (See Table 10E)

Table 6
Effects of Mesh Pads on Nickel Emissions

Process:	No Mesh Pad	Mesh Pads
Electroless Nickel	0.142	0.024
(Eductor)	0.192	0.012
5 vs 4	0.111	0.004
	0.148	**0.013**
Electroless Nickel	0.124	0.011
(Air)	0.313	0.006
6 vs 3	0.505	0.005
	0.314	**0.007**
Rack Watts	0.051	0.057
(No Wetting Agent, Air)	0.133	0.012
12 vs 11	0.056	0.005
	0.080	**0.025**
Rack Watts	0.016	0.015
(Wetting Agent, Eductor)	0.027	NA
9 vs 13	0.028	NA
	0.024	**0.015**
Rack Watts	0.048	0.003
(Wetting Agent, Air)	0.046	0.004
8 vs 14	0.032	0.003/0.037/0.008
	0.041	**0.011**
Barrel Watts	0.080	0.009
(Wetting Agent)	0.061	0.004
16 vs 20	0.118	0.012
	0.086	**0.008**
Barrel Watts	0.975	0.009
(No Wetting Agent)	1.495	<0.005
17 vs 18	0.921	0.023
	1.130	**0.012**
Nickel Sulfamate	0.018	0.004
(Wetting Agent, Eductor)	0.013	0.006
23 vs 25	0.020	0.006
	0.017	**0.005**
Wood Strike	0.614	0.063 (0.089)*
(No Wetting Agent)	0.282	0.081 (0.159)*
27 vs 7	0.515	0.236
	0.470	**0.127 (0.161)***
Wood Strike	0.129	0.048
(Wetting Agent)	0.162	0.077
28 vs 24	0.252	0.071
	0.181	**0.065**

Results reported in mg/dscm

* Results adjusted to compensate for cfm difference between Condition 7 and 27 (See Table 10E)

Table 7

Effects of Barrel Transfer on Nickel Emissions from Watts Nickel Barrel Plating

Process:	**Barrel Transfer**	**No Barrel Transfer**
Barrel Watts	0.975	0.056
(No Wetting Agent)	1.495	0.019
17 vs 19	0.921	0.228
	1.130	**0.101**
Barrel Watts	0.080	0.023
(Wetting Agent)	0.061	0.007
16 vs 21	0.118	0.009
	0.086	**0.013**

Results reported in mg/dscm

Table 8
Effects of Product Loading on Nickel Emissions from Electroless Nickel Plating

Process:	**Low Tank Loading**	**High Tank Loading**
Electroless Nickel	0.478 (0.290)*	0.124
(No Mesh Pad)	0.126 (0.078)*	0.313
1 vs 6	0.220 (0.135)*	0.505
	0.277 (0.167)*	**0.314**

Results reported in mg/dscm
* Results adjusted to compensate for cfm difference between Condition 1 and 6 (See Table 10A)

Table 9
Nickel Emissions Method 29 Compared to Method 306A

Process:	**Method 29**	**Method 306A**
Electroless Nickel	0.478	0.080
(No Mesh Pad)	0.126	0.311
1 vs 2	0.220	0.381
	0.277	**0.251**
Rack Watts	0.003	0.014
(Wetting Agent, Mesh Pad, Air)	0.004	0.008
14 vs 15	0.003/0.037/0.008	0.012
	0.011	**0.011**
Rack Sulfamate	0.022	0.031
(Wetting Agent, No Mesh Pad)	0.011	0.061
22 vs 26	0.010	0.053
	0.014	**0.048**
Wood's Strike	0.129	0.400
(Wetting Agent, No Mesh Pad)	0.162	0.165
28 vs 29	0.252	0.218
	0.181	**0.216**

Results reported in mg/dscm

Table 10A. Operating Parameters Observed During Stack Testing of Electroless Nickel Process

	Surface Tension	Nickel Content (oz/gal)	Time Actual Plating (min)*	# of Parts	Freeboard Height (in)**	Average Surface Area Plated (ft^2)	Ventilation (cfm)	Emissions (mg/m^3)
Condition 1, Run #1***	35.0	0.77	30	88	4	25.7	1,214	0.478
Condition 1, Run #2***	35.0	0.77	12	40	3	5.56	1,240	0.126
Condition 1, Run #3***	35.0	0.77	12	166	3	23.0	1,227	0.220
Condition 2, Run #1***	39.1	0.85	90	3	3	0.52	NA	0.060
Condition 2, Run #2***	39.1	0.85	60	1080	4	13.0	NA	0.311
Condition 2, Run #3***	39.1	0.85	0	0	4	0	NA	0.381
Condition 3, Run #1	68.7	0.81	115	72-416	8	5.8-22.3	1,776	0.011
Condition 3, Run #2	68.7	0.81	115	108-126	8	27-31.5	1,860	0.006
Condition 3, Run #3	68.7	0.81	105	1-126	8	12-31.5	1,860	0.005
Condition 4, Run #1	68.7	0.81	120	2,400 (barrel)	8	17.7	1,999	0.024
Condition 4, Run #2	68.7	0.81	120	2,400 (barrel)	8	17.7	2,023	0.012
Condition 4, Run #3	68.7	0.81	120	2,400 (barrel)	8	17.7	2,037	0.004
Condition 5, Run #1	68.7	0.81	120	1****	8	12.0	2,003	0.142
Condition 5, Run #2	68.7	0.81	120	1****	8	12.0	2,003	0.192
Condition 5, Run #3	68.7	0.81	120	1****	8	12.0	2,027	0.111
Condition 6, Run #1	68.7	0.81	114	15-100	8	3-16	2,039	0.124
Condition 6, Run #2	68.7	0.81	120	1****	8	12.0	1,984	0.313
Condition 6, Run #3	68.7	0.81	120	1****	8	12.0	1,929	0.505

* Out of 120 minute testing period
** Measured from vent to solution level (or foam blanket level, if applicable)
*** Phase 1 Testing
****1 Dummy Part

Table 10B. Operating Parameters Observed During Stack Testing of Rack Watts Nickel Process

	Amps	Current Density (Amps/ft^2)	Time Actual Plating (min)*	Surface Tension***	Freeboard Height (in)**	Ave Surface Area Plated (ft^2)	Nickel Content	(cfm) Ventilation	Emissions (mg/m^3)
Condition 8, Run #1****	295	42.9	100	38	9-10.5	7.0	10.9	261	0.048
Condition 8, Run #2****	300	42.9	100	38	9.5-10.5	7.0	10.9	271	0.044
Condition 8, Run #3****	300	42.1	100	38	9.5	7.0	10.9	264	0.032
Condition 9, Run #1****	288	41.1	100	38	9.5	7.0	10.9	272	0.016
Condition 9, Run #2****	280	39.4	100	38	9.5	7.0	10.9	275	0.027
Condition 9, Run #3****	276	40.0	100	38	9.5	7.0	10.9	262	0.028
Condition 10, Run #1	210	20-26	88	58	9	8.0-10.5	9.4	2,190	0.043
Condition 10, Run #2	210	20-45	84	58	9	4.5-10.5	9.4	2,200	0.003
Condition 10, Run #3	210	30	86	58	9	7.0	9.4	2,171	0.016
Condition 11, Run #1	230	28.8	100	57	9	8.0	8.2	2,168	0.057
Condition 11, Run #2	210	20.0	100	57	9	10.5	8.2	2,184	0.012
Condition 11, Run #3	210	20-26	88	57	6	8.0-10.5	8.2	2,193	0.005
Condition 12, Run #1	240	25.7	109	57	9	9.3	8.2	2,350	0.051
Condition 12, Run #2	240	25.7	105	57	9	9.3	8.2	2,362	0.133
Condition 12, Run #3	180		95	57	9		8.2	2,355	0.056
Condition 13, Run #1	130	18.6	114	42	9	7.0	9.6	2,158	0.015
Condition 14, Run #4	130	18.6	101	42	9	7.0	9.6	2,161	0.037
Condition 14, Run #5	130-180		100	42	9		9.6	2,159	0.008
Condition 14, Run #1	125-200	17-20	105	37	9	7.4-11.7	11.4	2,114	0.003
Condition 14, Run #2	120-200	17-20	96	37	9	6-11.7	11.4	2,112	0.004
Condition 14, Run #3	0	0	0	37	9	0	11.4	2,129	0.003
Condition 15, Run #1	200	17	98	37	9	11.7	11.4	NA	0.014
Condition 15, Run #2	200-240	17-20	92	37	9	11.7-12.0	11.4	NA	0.008
Condition 15, Run #3	120-240	20-30	96	37	9	4.0-12.0	11.4	NA	0.012

* Out of 120 minute testing period

** Measured from vent to solution level (or foam blanket level, if applicable)

*** dynes/cm measured with tensiometer.

**** Phase 1 Testing

Table 10C. Operating Parameters Observed During Stack Testing of Barrel Watts Nickel Process

	Surface Tension	Current Density (Amps/ft^2)	Amps	Time Actual Plating (min)*	# of Barrels***	Freeboard Height (in)**	Ave Surface Area Plated (ft^2)	Nickel Content	Ventilation (cfm)	Emissions (mg/m^3)
Condition 16, Run #1****	34	3.35-3.78	480-540	110	3	5	143	8.4	1,738	0.080
Condition 16, Run #2****	34	3.50-3.72	500-560	105	3	5	143	8.4	1,748	0.061
Condition 16, Run #3****	34	3.50-3.72	500-560	107	2	5	143	8.4	1,782	0.118
Condition 17, Run #1****	57	1.82	700	117	2	8.0	384	9.8	1,752	0.975
Condition 17, Run #2****	57	1.82	700	114	3	8.5	384	9.8	1,603	1.495
Condition 17, Run #3****	57	1.82-2.46	600-700	113	3	9	244-384	9.8	1,608	0.721
Condition 18, Run #1	68	5.28-7.46	300-400	110	4	8	40-76	8.3	1,081	0.009
Condition 18, Run #2	68	5.28	400	118	3	8	76	8.3	1,545	<0.005
Condition 18, Run #3	68	5.28	400	118	3	8	76	8.3	1,642	0.023
Condition 19, Run #1	68	5.28	400	120	1	8	76	8.3	1,778	0.056
Condition 19, Run #2	68	5.28	400	120	1	8	76	8.3	1,674	0.019
Condition 19, Run #3	68	5.28	400	120	1	8	76	8.3	1,542	0.228
Condition 20, Run #1	31	1.56-16.9	100-600	88	5	8	36-322	8.4	1,463	0.009
Condition 20, Run #2	31	5.28	400	106	3	8	76	8.4	1,521	0.004
Condition 20, Run #3	31	5.28	400	116	3	8	76	8.4	1,462	0.012
Condition 21, Run #1	31	5.28	400	120	1	8	76	8.4	1,621	0.023
Condition 21, Run #2	31	5.28	400	120	1	8	76	8.4	1,661	0.007
Condition 21, Run #3	31	5.28	400	120	1	8	76	8.4	1,597	0.009

* Out of 120 minute testing period
** Measured from vent to solution level (or foam blanket level, if applicable)
*** Number of barrels plated during testing time period.
**** Phase 1 Testing

Table 10D. Operating Parameters Observed During Stack Testing for Rack Nickel Sulfamate Process

	Current Density (Amps/ft^2)	Time Actual Plating (min)	Surface Tension***	Freeboard Height (in)**	Average Surface Area Plated (ft^2)	Nickel Content	Ventilation (cfm)	Emissions (mg/m^3)
Condition 22, Run #1	39.6-61.9	96	39	8	14.7-26.6	8.59	3,789	0.022
Condition 22, Run #2	31.9-75.5	96	39	9	5.56-26.6	8.59	3,950	0.011
Condition 22, Run #3	27.5-66.9	96	39	9	14.7-26.4	8.57	3,815	0.010
Condition 23, Run #1	35.1-61.9	98	44	9	9.4-24.0	7.73	4,237	0.018
Condition 23, Run #2	34.8-56.3	90	44	9	17.8-23.0	7.73	4,011	0.013
Condition 23, Run #3	37.2-61.9	64	44	9	9.4-24.50	7.73	4,024	0.020
Condition 25, Run #1	26.4-59.7	75	44	9	9.40-22.7	7.73	3,706	0.004
Condition 25, Run #2	27.7-47.7	45	44	9	9.40-24.0	7.73	3,568	0.006
Condition 25, Run #3	41.7	99	44	9	24.0	7.73	3,639	0.006
Condition 26, Run #1	35-61.9	88	35	9	9.4-26.6	8.51	NA	0.031
Condition 26, Run #2	16.9-49.5	77	35	9	9.4-24.0	8.51	NA	0.061
Condition 26, Run #3	25-61.9	85	35	9	17.8-26.6	8.51	NA	0.053

* Out of 120 minute testing period

** Measured from vent to solution level (or foam blanket level, if applicable)

*** dynes/cm measured with tensiometer.

Table 10E. Operating Parameters Observed During Stack Testing for Wood's Nickel Strike Process

	Current Density (Amps/ft^2)	Time Actual Plating (min)*	Surface Tension***	Freeboard Height (in)**	Ave Surface Area Plated (ft^2)	Nickel Content	Ventilation (cfm)	Emissions (mg/m^3)
Condition 7, Run #1	25.6	120	61	8.5	12.5	3.17	1,155	0.063
Condition 7, Run #2	25.6	120	61	8.5	12.5	3.17	1,610	0.081
Condition 7, Run #3	25.6	120	61	8.5	12.5	3.17	818	0.236
Condition 24, Run #1	25.6	120	37-40	8.5	12.5	3.34	850	0.048
Condition 24, Run #2	25.6	120	37-40	8.5	12.5	3.34	886	0.077
Condition 24, Run #3	25.6	120	37-40	8.5	12.5	3.34	867	0.071
Condition 27, Run #1	4.9-25.6	119	73	8.5	12.5-51.3	2.43	872	0.614
Condition 27, Run #2	25.6	120	73	8.5	12.5	2.43	859	0.282
Condition 27, Run #3	25.6	120	73	8.5	12.5	2.43	879	0.515
Condition 28, Run #1	25.6	120	37-40	8.5	12.5	1.62	860	0.129
Condition 28, Run #2	25.6	120	37-40	8.5	12.5	1.62	887	0.162
Condition 28, Run #3	25.6	120	37-40	8.5	12.5	1.62	892	0.252
Condition 29, Run #1	25.6	120	40-50	8.5	12.5	1.62	NA	0.400
Condition 29, Run #2	25.6	120	40-50	8.5	12.5	1.62	NA	0.165
Condition 29, Run #3	25.6	120	40-50	8.5	12.5	1.61	NA	0.218

* Out of 120 minute testing period
** Measured from vent to solution level (or foam blanket level, if applicable)
*** dynes/cm measured with tensiometer.

Table 11. Ambient Air Nickel Concentrations

	Nickel Concentration Ambient Air (mg/m^3)	OSHA Nickel Concentration Limit (mg/m^3)	Adequate Ventilation
Condition 3 (Reliable – East)	0.0062	1.00	Yes
Condition 3 (Reliable – West)	0.0053	1.00	Yes
Condition 4 (Reliable – East)	0.0060	1.00	Yes
Condition 4 (Reliable – West)	0.0053	1.00	Yes
Condition 5 (Reliable – East)	0.0069	1.00	Yes
Condition 5 (Reliable – West)	0.0063	1.00	Yes
Condition 6 (Reliable – East)	0.0156	1.00	Yes
Condition 6 (Reliable – West)	0.0122	1.00	Yes
Condition 10 (ABQC – East)	0.0065	1.00	Yes
Condition 10 (ABQC – West)	0.0012	1.00	Yes
Condition 11 (ABQC – East)	0.0199	1.00	Yes
Condition 11 (ABQC – West)	0.0029	1.00	Yes
Condition 12 (ABQC – East)	0.0657	1.00	Yes
Condition 12 (ABQC – West)	0.0074	1.00	Yes
Condition 13 (ABQC – East)	0.0694	1.00	Yes
Condition 13 (ABQC – West)	0.0047	1.00	Yes
Condition 14 (ABQC – East)	0.0062	1.00	Yes
Condition 14 (ABQC – West)	0.0043	1.00	Yes
Condition 15 (ABQC – East)	0.0041	1.00	Yes
Condition 15 (ABQC – West)	0.0013	1.00	Yes
Condition 18 (Artistic – North)	0.0025	1.00	Yes
Condition 18 (Artistic – South)	0.0031	1.00	Yes
Condition 19 (Artistic – North)	0.0004	1.00	Yes
Condition 19 (Artistic – South)	0.0004	1.00	Yes
Condition 20 (Artistic – North)	0.0011	1.00	Yes
Condition 20 (Artistic – South)	0.0009	1.00	Yes

Table XI. Ambient Air Nickel Concentrations (Cont.)

	Nickel Concentration Ambient Air (mg/m^3)	OSHA Nickel Concentration Limit (mg/m^3)	Adequate Ventilation
Condition 21 (Artistic – North)	0.0008	1.00	Yes
Condition 21 (Artistic – South)	0.0008	1.00	Yes
Condition 22 (Elite – North)	0.0026	1.00	Yes
Condition 22 (Elite – South)	0.0046	1.00	Yes
Condition 23 (Elite – North)	0.0036	1.00	Yes
Condition 23 (Elite – South)	0.0089	1.00	Yes
Condition 25 (Elite – North)	0.0014	1.00	Yes
Condition 25 (Elite – South)	0.0017	1.00	Yes
Condition 26 (Elite – North)	0.0064	1.00	Yes
Condition 26 (Elite – South)	0.0064	1.00	Yes
Condition 7 (Artistic – East)	0.1259	1.00	Yes
Condition 7 (Artistic – West)	0.0139	1.00	Yes
Condition 24 (Artistic – East)	0.0676	1.00	Yes
Condition 24 (Artistic – West)	0.0120	1.00	Yes
Condition 27 (Artistic – East)	0.3085	1.00	Yes
Condition 27 (Artistic – West)	0.0398	1.00	Yes
Condition 28 (Artistic – East)	0.1831	1.00	Yes
Condition 28 (Artistic – West)	0.0054	1.00	Yes
Condition 29 (Artistic – East)	0.2119	1.00	Yes
Condition 29 (Artistic – West)	0.0133	1.00	Yes

Section 6.0 DISCUSSION OF RESULTS

Table 3

The results presented in Table 1 summarize the nickel emission data obtained from both Phase 1 and Phase 2 of this project. The results from each of the three individual test runs along with the average of the three tests runs from each condition tested are reported in the table. All of the results were obtained using Method 29 stack testing except those identified as Method 306A.

Based on these results, it is apparent that the typically operated Rack Watts Nickel and the Rack Nickel Sulfamate processes (wetting agent, no mesh pads, air agitation) emit very little nickel; less than 0.05 mg/dscm. These concentrations are not much higher than the MACT hexavalent chromium limits that are based on the use of expensive composite mesh pad systems to reduce the chromium emissions. These results were expected due to the high efficiency of the Watts Nickel and Nickel Sulfamate plating process (very little gassing). Furthermore, the Rack Watts Nickel and the Rack Nickel Sulfamate solutions are typically operated with some kind of wetting agent employed in order to maintain the quality of the plating (reduce pitting, etc.). Therefore, platers need to add wetting agents to these solutions for quality purposes; the pollution control effect is a secondary benefit.

Nickel emissions being emitted from uncontrolled Wood's Nickel Strike, Barrel Nickel, and Electroless Nickel plating processes vary from 0.101 mg/dscm to 1.130 mg/dscm. "Uncontrolled" is defined as no wetting agent, eductor system or mesh pads added to the process. None of these solutions require a wetting agent for quality purposes. Therefore, these processes typically operate without one. Other reasons why the uncontrolled emissions are high include:

- The Wood's Nickel Strike process is intentionally very inefficient which causes significant gassing.
- Barrel Nickel plating has many mechanical/physical effects that increase nickel emissions (see Discussion of Table VII).
- The Electroless Nickel plating process is operated at very high temperatures, has a very high air agitation rate, and produces a significant amount of hydrogen gas as a by-product.

Paired Wilcoxon Signed-Rank tests were conducted to evaluate the effect of employing an eductor system, wetting agent or simple mesh pads at the exhaust hood. The paired Wilcoxon Signed Rank Test (WSRT) is a non-parametric test that evaluates the median difference between paired samples.

The results of the eductor system versus air agitation comparison was inconclusive (test statistic value = 0.5256, p-value=0.5992, n=6). There was insufficient information to conclude that nickel emissions were reduced by the addition of an eductor system. The results of the wetting agent versus no wetting agent comparison was statistically significant (test statistic value = 36, p-value = 0.0078, n = 8). Nickel emissions were reduced by the addition of a wetting agent. The results of the mesh pad versus no mesh pad comparison were statistically significant (test statistic value = 55, p-value = 0.002, n = 10. Nickel emissions were reduced by the addition of simple mesh pads at the exhaust hood. The boxplots in Figure 1, 2, and 3, confirm the conclusions of the inferential tests. In each case, employing an operating parameter to reduce nickel emissions consistently reduced between process variability, but only in the case of employing a wetting agent or installing a mesh pad installed at the box hood was median nickel emissions reduced. The investigation did not evaluate the interaction between these operating parameters, therefore no conclusions can be made regarding cumulative effect of employing more than one of these operating parameters.

Figure 1. *Box plots of Nickel Emissions for Eductor System versus Air Agitation.*

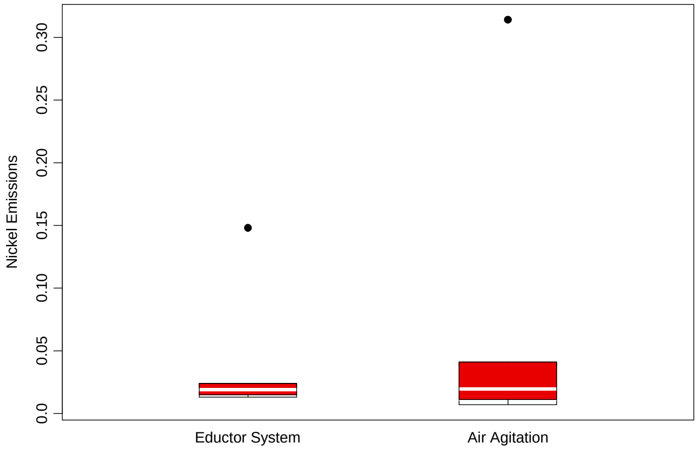
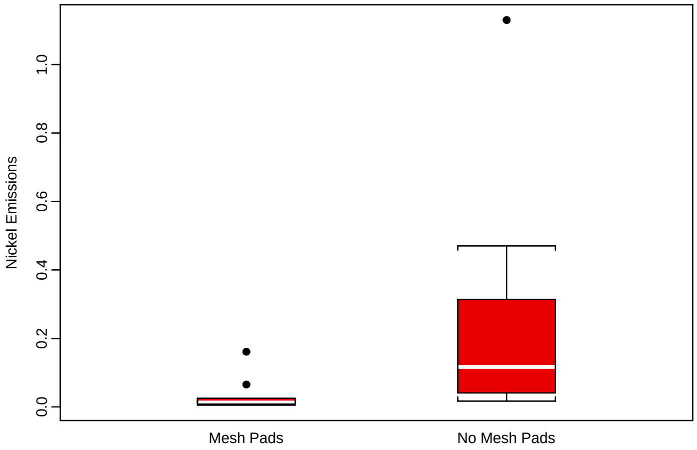


Figure 2. *Box plots of Nickel Emissions for Wetting Agent versus No Wetting Agent.*

Figure 3. *Box plots of Nickel Emissions for Mesh Pads and No Mesh Pads.*



Tables 4 through 9 present the results summarized in Table 3, in a comparison manner that illustrate the effects on nickel emissions of the operating conditions identified by this project (i.e. the effects of wetting agents, the effects of mesh pads, eductors vs. air agitation, etc.).

The nickel emissions from the rack Watts nickel process operated with a wetting agent, a mesh pad, and an eductor system (Condition 13) was only tested for one two-hour test run due to quality issues caused by the eductor system. The eductor system caused dullness and pitting in the nickel finish in many of the parts that were plated in the nickel tank. Since most of the testing conducted in this project was conducted on actual plating processes in operation, the quality of the plated parts took precedence over the stack testing. When modifications to the operating parameters of the nickel process (temp, current density, etc,) and to the eductor system itself would not alleviate the plating problems, the eductor system was decommissioned and replaced with the air agitation system. The two remaining test runs during that day were completed with the air agitation system in operation resulting in a total of five test runs conducted on a rack Watts nickel process operated with a wetting agent, a mesh pad, and an air agitation system (Condition 14).

It was later determined that the plating problems were caused by iron contamination in the nickel solution that was directed at the parts by the eductors and then becoming embedded in the plating finish causing the pitting and the dullness. It is anticipated that once the iron contamination is removed (via filtration), the eductor system can be used in the process without causing quality problems; as others have used eductors on Watt's Nickel very successfully.

As with the Phase 1 testing, there was a comparably large difference in nickel emissions measured within each of the three individual test runs of a condition tested. For example, the nickel emission concentrations for the three tests runs on the Electroless Nickel process operated with air agitation (Condition 6) were measured to be 0.124, 0.313, and 0.505 mg/dscm. The main reason for these differences within individual test runs may be the fact that operating parameters changed slightly from test run to test run. Because these tests were conducted, for the most part, on nickel plating processes plating actual production parts, keeping all the operating parameters constant during all three test runs was not possible. Attempts were made, however, to minimize the operating parameter fluctuations where possible. Because of these differences within the individual test runs, the average of the three were taken and also reported.

Table 4

Based on the results of this study and illustrated in Table 4, when the nickel emissions are uncontrolled, the data shows nickel emissions are reduced by 53% when eductors are used for agitation in place of air (See Condition 5 versus 6). When the nickel emissions are low, whether it is due to the nickel plating process itself having low emissions (as is the case with nickel sulfamate) or the fact that the emissions are reduced by some other means (i.e., mesh pad), the effects of using eductors instead of air agitation are negligible with an average reduction of 8% (See Conditions 3 versus 4, 13 versus 14, and 22 versus 23). In fact, in many of these cases, the nickel emissions measured with the eductors in operation were actually slightly higher than those measured with the air agitation system operating. That said, we do not believe that using the eductors actually increased the emissions; simply that eductors have very little effect on the nickel emission when the emissions are low to begin with. These unexpected results obtained when emissions are low are probably due to the inherent error in the Method 29 procedure or the fact that, in some of the cases, the surface tension of the plating solution was slightly higher when the eductors were tested than when the air agitation system was tested. For example, the surface tension of the Watts Nickel solution when the eductors were tested was 42 dynes/cm while the surface tension of the same solution when the air was operating was 37 dynes/cm. Similarly, the surface tension of the Nickel Sulfamate solution when the eductors were tested was 45 dynes per centimeter while the surface tension of the same solution when the air was operating was 39 dynes per centimeter. These small differences in surface tension could have masked the emission reduction effects of the eductors because it is believed (and validated in this study) that nickel emission of a plating solution are related to its surface tension; the emissions increase as the surface tension increases.

Maintaining a constant surface tension within a plating solution is difficult because only a small amount of wetting agent is needed to reduce the surface tension from about 70 to 40 dynes/cm. To raise a surface tension in a solution to a specified level requires considerable time to allow the surface tension to rise naturally or complicated carbon treatment.

Table 5

Based on the results of this study and illustrated in Table 5, the use of wetting agents to reduce the surface tension of a nickel plating solution significantly reduces the nickel emissions from that process an average of 72% from uncontrolled tanks and an average of 42% for tanks controlled with mesh pads. In all eight comparison studies involving the use of wetting agents and encompassing Rack Watts Nickel, Barrel Watts Nickel, and Wood’s Nickel Strike plating solutions, the nickel emissions measured with the wetting agent were 29% to 92% lower than the nickel emissions measured without the wetting agent.

Several chemical suppliers were contacted to determine the availability of a wetting agent specifically made for a Wood's nickel strike solution. None of these chemical manufacturers could provide one nor did they believe one was developed yet. Therefore, a wetting agent that was typically used in a Watts Nickel solution was used instead. Initial tests proved that this wetting agent was successful in reducing the surface tension of the Wood's nickel strike solution from 70 dynes/cm to 38 dynes/cm. However, it was later discovered that the acidic nature of the Wood's solution destroyed the wetting agent relatively quickly (within two to three days). Moreover, the resultant decomposition products of the wetting agent formed a film on the plating solution which adversely affected plating adhesion.

In Table 5, there are two results reported for some of the test runs of Condition 7. The results in the parenthesis are results that have been modified to account for the air ventilation rate fluctuations that occurred during the Condition 7 testing. For all the other Wood's Nickel Strike test runs, the ventilation rate measured during the testing was 818 to 892 cfm. The ventilation rate for the three Condition 7 tests runs was measured to be 1155 cfm, 1610 cfm, and 818 cfm respectively. The blower for the Wood's Nickel Strike ventilation system is a variable speed blower. It is believed that the speed was switched to "high" sometime during the first test run and then switched back to "low" after the second test run. To compensate for this dilution effect and to provide a viable comparison, the nickel emissions from the first two test runs of Condition 7 were mathematically revised to reflect a ventilation rate of 818 cfm.

Table 6

Based on the results of this study and illustrated in Table 6, the use of simple mesh pads to capture nickel emissions in the vent reduces the nickel emissions from that process. In all ten comparison studies involving the use of mesh pads and encompassing Rack Watts Nickel, Barrel Watts Nickel, Wood's Nickel Strike, Nickel Sulfamate, and Electroless Nickel plating solutions, the nickel emissions measured with the mesh pad in place were significantly lower than the nickel emissions measured without the mesh pad by an average of 88% for both controlled and uncontrolled conditions..

The mesh pad, based on this study, was the most efficient in reducing nickel emissions; outperforming the wetting agents and the eductor system as shown in Table 12.. Besides providing better reduction efficiency, simple mesh pads are less expensive to purchase than the eductor system and less expensive to use than the wetting agents. Moreover, mesh pads are totally external to plating solution system. When contemplating using a wetting agent or an eductor system, one needs to consider the effects of these items on the plating quality because they are placed in the plating tank. The mesh pad does not affect the plating at all since it is installed in the ventilation system.

Table 12. Comparison of Nickel Emission Reductions

Condition	**%Reduction of Nickel Emissions**		
	Mesh Pads	Wetting Agent	Eductors
Uncontrolled Tank	83%	69%	53%
Partially Controlled Tank	69%	45%	8%

The composite mesh pads used in this project were supplied by Chicago Plastics and were easily inserted in the face of the exhaust hoods; although some exhausts hoods had to be modified in order to accommodate the composite mesh pads (See photos in Appendix C). The composite mesh pads are constructed of intertwined polypropylene threads and are approximately three (3) inches thick. As stated above, these composite mesh pads were easily placed in the face of the exhaust hood or directly in the exhaust duct (as was the case with Elite Finishing) and are designed to capture mists and large particulates (greater than 8 microns).

Table 7

The stack testing results presented in Table 7 suggests that the majority of the nickel that is emitted from barrel nickel operations is due to mechanical/physical sources such as splashing produced during entry of the barrel into the tank, the large amounts of liquid leaving the barrel during removal from the tank, splashing from filter pumps, etc and not from the electrolysis involved in plating. These results are consistent in what was suggested in the Final Report for the Phase 1 testing.

Table 8

Based on the results presented in Table 8, it appears that the chemical reaction involved in the electroless plating process does increase the nickel emissions from an electroless nickel process. In the Final Report for Phase 1 of this project, it was suggested that the majority of the emissions from an electroless nickel plating process were due to the high temperature of the electroless nickel plating solution and the violent air agitation required in the process and that the amount of parts (or surface area of the parts) had little affect on the nickel emissions. In the Phase 1 testing (Condition 1), parts were being plated for only 12-30 minutes of the test run time (120 minutes). In Phase 2 (Condition 6), however, parts were being plated for 114 to 120 minutes of the 120 minute test run. As illustrated in Table 8, the emissions measured in Phase 2 with the high tank loading were higher than those measured during Phase 1 with the low tank loading. However, in the year that elapsed between Phase 1 and Phase 2, the EN testing site switched EN solution suppliers. The EN solution that was being used during Phase 1 of the study had a wetting agent incorporated into the chemistry while the EN solution that was being used during Phase 2 did not. Consequently, the surface tension of the EN solution in process during Phase 1 was about 30 dynes/cm lower than the surface tension of the EN solution that was in process during Phase 2. Therefore, it is inconclusive as to the cause of the increased emissions observed during

Condition 6 as compared to Condition 1 (increased parts loading or increased surface tension, or a combination of the two).

Part of the Phase 2 testing was to observe the effects of a mesh pad on the nickel emissions from the electroless nickel plating process. In order to accomplish this goal, the ventilation system for the electroless nickel tank had to be modified to accept a mesh pad. The modification to the ventilation system increased the ventilation flow rate. The ventilation rate during Phase 1 was 1214 to 1240 cfm while the ventilation rate for Phase 2 was 1860 to 2039 cfm. Therefore, to compensate for this dilution effect and to provide a viable comparison, the nickel emissions from the test runs of Condition 1 were mathematically revised to reflect a ventilation rate of 2000 cfm. The modified results are listed in parenthesis in Table 8.

Table 9

The results presented in Table 9 indicate that, although the individual test runs were not consistent, the average of the three runs using Method 29 were within 10% of the average of the three runs using Method 306A for the Electroless Nickel, Watts Nickel and Wood’s Nickel Strike processes. For the Nickel Sulfamate process, the emissions measured by Method 306A were actually higher than those measured by Method 29. The inconsistency within the three individual test runs and the test runs on the Nickel Sulfamate process may have been due to the changes in operating parameters during the individual test runs. Based on this testing, we believe that Method 306A is a viable alternative to Method 29 for measuring nickel emissions. However, we believe more testing is required.

In order to validate Method 306A as a viable alternative, a non-production process should be tested in order to have absolute control over all the operating parameters to ensure that they are constant and identical during both the Method 29 and Method 306A testing. When testing production processes, the operating parameters change slightly from test run to test run based on the production needs. Another option would be to test a production process using Method 29 and Method 306A simultaneously.

The emissions from the Woods Nickel Strike process measured with Method 306A were higher than the emissions measured with Method 29. Both of these tests were conducted with wetting agent in the Woods Nickel Strike solution. The wetting agent was added in the morning of the first day. Method 29 was conducted on the first day while Method 306A was conducted on the second day. As discussed earlier, it was discovered that the acidic nature of the Woods Strike solution destroyed the wetting agent relatively quickly. Analysis on the first day using a stalagmometer indicated that the surface tension was approximately 37 dynes/cm. Analysis of the Woods Nickel Strike solution three days later using a tensiometer indicated that the surface tension was 59 dynes/cm. Therefore, the surface tension on the second day (Method 306A) may have been higher than that on the first day (Method 29); possibly in the 40-50 dynes/cm range. This discovery may explain why the Method 306A testing measured higher emissions.

Tables 10A through 10E

The purpose of Tables 10A through 10E is to summarize the working parameters observed and recorded during the testing for all of the conditions tested. As stated before, most of the testing was conducting during actual processing. Therefore, many of the operating parameters did not remain constant during all of the individual test runs within a condition. By comparing the operating parameters from one test run to another, one may be able to explain why there was a difference in nickel emissions measured from one test one to another.

Table 11

The results presented in Table 11 illustrate that the ventilation systems of all the nickel plating processes were operating efficiently because the nickel concentrations in the ambient air near the nickel plating tanks at the times of the testing were significantly below the OSHA eight hour, time weighted average permissible exposure limit for nickel. This criteria was set-up prior to testing in order to ensure that the ventilation systems were capturing the majority of nickel emissions from the plating processes.

QA/QC Summary

The data generated has been evaluated in terms of quality objectives for precision, accuracy, completeness, representativeness, and comparability, in accordance with EPA Methods 29 and 306A and OSHA Method ID-121.

Prior to analyses, demonstration of method performance and analyst capability was verified and documented through various studies as MDL, LDR, QCS, DOC, and Performance Evaluation Samples. The appropriate blanks (CCB and LRB), spikes (LFM and LFM dups), and standards (QCS, CCV, LFB) were incorporated into the analytical runs as is required by EPA Methods 29 and 306A and OSHA Method ID-121. These QC samples were evaluated to meet acceptable criteria limits, as determined by the procedures and control charts. In order to maintain consistency in applying the QC data, the “QA/QC Worksheet” was generated to condense the QC values, limits, and qualifiers, if any. These worksheets are included in Appendices E and F.

All data is considered to be of acceptable quality and validity, as determined by the laboratory QA Coordinator.

Section 7.0 CONCLUSIONS

Based on the stack testing results presented in Table 3, the following conclusions regarding nickel emissions from nickel plating processes can be made:

1) Nickel emissions from typically-operated Rack Watts Nickel and Rack Nickel Sulfamate tanks (wetting agent, no mesh pad, air agitation) are not very significant (<0.05 mg/dscm).

2) Nickel emissions from uncontrolled (no eductors, mesh pads or wetting agents) and vented Electroless Nickel, Barrel Nickel, and Wood's Nickel Strike plating processes are significant (greater then 0.1 mg/dscm), but can be significantly reduced by 49% to 92%. This can be accomplished by employing eductors, wetting agents, or simple mesh pads.

3) The use of wetting agents in nickel plating processes significantly reduced nickel emissions. The use of wetting agents to reduce the surface tension of a nickel plating solution reduces the nickel emissions from that process an average of 72% from uncontrolled tanks and an average of 42% for tanks controlled with mesh pads. At this time, however, there does not seem to be a wetting agent specifically developed for the Wood's nickel strike solution. A Watt's Nickel wetting agent can be successfully used to reduce the surface tension of the Wood's nickel strike solution. However, the acidic nature (at least 10% by volume hydrochloric acid) of the Wood's solution destroys the wetting agent relatively quickly leaving a film on the solution surface that greatly affects the plating adhesion.

4) Nickel emissions were reduced an average of 15% using eductors in lieu of air agitation for the various controlled and uncontrolled conditions…

5) Regardless of type of nickel process, using simple mesh pads to capture splashing and misting provides the best reduction of nickel emissions. These reductions averaged 69% to 89% compared to 8% to 69% for other control methods as shown in Table 12.

6) Most of the nickel emissions from nickel barrel plating operations are generated from mechanical/physical sources such as splashing produced during entry of the barrel into the tank, the large amounts of liquid leaving the barrel during removal from the tank, splashing from filter pumps, etc. and not from the electrolysis involved in plating.

7) The nickel emissions from an electroless nickel process are generated from **both** the chemical reaction involved in the electroless plating process and the high solution temperature and high agitation rate required in the process.

8) Although the results presented in Table 9 indicate that USEPA Method 306A and

> USEPA Method 29 provide similar results when measuring nickel emissions from Electroless Nickel, Watts Nickel, and Wood’s nickel strike, further testing needs to be conducted on a controlled process or conducted simultaneously to determine whether Method 306A truly is an acceptable alternative to Method 29 for measuring nickel emissions from nickel plating processes in general.

Three methods of reducing nickel emissions observed in this study are the use of wetting agents, eductors, and simple mesh pads. The implementation of simple mesh pads provide a better reduction efficiency, they are cheaper to employ and do not affect the quality of the plating.

Due to the extreme number of variables involved in plating (tank dimensions, current density, temperature, types of parts plated, plating times, chemical differences, etc.), the results presented in this report should not be used as absolute for all Watt’s Nickel, Wood’s Nickel Strike, Electroless Nickel, etc. processes “across the board” but rather as a relative guide or estimate for these processes. In the same manner, one cannot assign actual emission reduction efficiencies for wetting agents, eductors, and mesh pads solely on the results of these tests. Instead, it is much more accurate to state that these options reduce nickel emissions but the reduction percentage from process to process will vary. We base these project limitations or constraints on the fact that even though extreme care was given to control the variables within the three tests runs of a test condition, the results for each of these test runs did vary more than expected (see Table 3). We would expect the results to vary even greater when the variables are not controlled or, for example, from one Electroless Nickel tank to another.