

**RECYCLING NICKEL ELECTROPLATING RINSE WATERS BY LOW TEMPERATURE  
EVAPORATION AND REVERSE OSMOSIS**

**BY**

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**Contract Number  
CR-815829**

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## FOREWORD

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The Risk Reduction Laboratory is responsible for planning, implementing, and managing research, development, and demonstration programs to provide an authoritative, defensible engineering basis in support of the policies, programs, and regulations, of the EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. This publication is one of the products of that research and provides a vital communication link between the researcher and the use community.

This document presents the results of experiments conducted to evaluate two technologies currently available to minimize environmental problems associated with rinse waters generated during electroplating operations. The objective was to concentrate nickel to the same level as the electroplating bath so that the nickel could be reused. The high cost of nickel and of treating and disposing of electroplating wastes is an incentive to develop these recovery technologies to reduce wastes at the source.

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## ABSTRACT

Electroplating operations generate rinse water wastes that are classified as hazardous due to the presence of heavy metals. Disposal of the rinse water waste without prior treatment is cost prohibitive and environmentally unacceptable. End-of-pipe treatments that utilize chemical precipitation and destruction of the plating chemicals waste valuable materials that could be recycled. This project examined two alternative technologies for reducing the waste generated and recovering heavy metals such that they can be recycled in the electroplating process. Specifically, low temperature evaporation and reverse osmosis systems were evaluated on a pilot scale to process rinse water collected from a nickel electroplating operation.

The low temperature evaporation system exhibited consistent and predictable results throughout the tests. It was capable of concentrating the rinse water feed solution to contain nickel levels well above the eight percent required for replacement into the plating bath. The cleaned rinse water was high quality and essentially metal free and could be either reused for rinse water or discharged to a POTW. Disadvantages associated with the evaporation system include its relatively high capital costs and high energy requirements.

The reverse osmosis system exhibited superior productivity at the beginning of the tests while productivity dropped off dramatically after about 60 percent of the feed solution had been processed. The decline commenced when nickel levels in the feed solution reached about 4,000 to 5,000 mg/L. The decline continued until the productivity of the reverse osmosis equipment was reduced to almost nothing. At this point the reverse osmosis system had concentrated the feed solution to nickel concentrations of 12,560 to 18,200 mg/L which are well below the eight percent nickel concentration required for the plating bath. The cleaned rinse water produced by the reverse osmosis equipment was directly related to the quality of the feed solution pumped into the unit. The quality of the cleaned rinse water from the reverse osmosis test was acceptable for reuse as rinse water but unacceptable for discharge to the POTW. The reverse osmosis system offers relatively low capital cost and energy requirements when compared to an evaporation system. However, its application is limited by the extent to which it concentrates solutions and the quality of the cleaned solution it produces.

Both systems offer advantages under specific operating conditions. The low temperature evaporation system appears to be best suited to processing solutions with relatively high nickel concentrations. The reverse osmosis system is best adapted to conditions where the feed solution is of relatively low (less than 4,000 to 5,000 mg/L) nickel concentration. In electroplating operations where relatively dilute rinse water solutions must be concentrated to levels acceptable for replacement in the plating bath, a combination of the two technologies might provide the best

process alternative. The reverse osmosis system could be used to initially concentrate the feed solution followed by low temperature evaporation processing to concentrate the solution to levels acceptable for replacement in the plating bath.

This report was submitted in partial fulfillment of Contract No. CR-815829 under the sponsorship of the U.S. Environmental Protection Agency. This report covers a period from November, 1991 to August, 1992 and work was completed as of December, 1992.

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## ACKNOWLEDGEMENTS

This project was performed jointly by the US EPA and the Hazardous Waste Research and Information Center, Champaign, Illinois. The cooperative efforts of Graham Plating, Chicago, Illinois; Licon, Inc. Pensacola, Florida; and Osmonics, Minnetonka, Minnesota; facilitated successful completion of this project. A list of individuals who made significant contributions to the management and execution of this project is provided below.

Mr. Paul Randall	USEPA
Mr. Tim Lindsey	HWRIC
Mr. Clayton Graham	Graham Plating
Mr. Ken Majewski	Graham Plating
Mr. John Campbell	Licon, Inc.
Mr. Curt Weitnauer	Osmonics

## SECTION 1

### INTRODUCTION

This is the final report for one of five projects constituting the Illinois/EPA WRITE (Waste Reduction Innovative Technology Evaluation) Program. The project was a joint effort of Graham Plating, Chicago, Illinois, an electroplating firm; the Hazardous Waste Research and Information Center (HWRIC) which is a division of the Illinois Department of Energy and Natural Resources, Champaign, Illinois; and the Pollution Prevention Research Branch of the U.S. Environmental Protection Agency's Risk Reduction Research Lab, Office of Research and Development, Cincinnati, Ohio. Assistance and direction was also provided by Licon, Inc. of Pensacola, Florida and Osmonics of Minnetonka, Minnesota.

The purpose of this project was to evaluate, compare, and document the effectiveness of two technologies for recovery and reuse of water and plating bath chemicals associated with electroplating rinse waters. The recovery technologies examined in this project include 1) low temperature evaporation and 2) reverse osmosis. These treatment systems were evaluated based on their effectiveness with respect to the following considerations:

- O Recovery efficiency and purity of the treated rinse water
- O Recovery efficiency and purity of the rinse water concentrate
- O Anticipated reduction in water use and chemical utilization due to recycling
- O Anticipated reduction in waste volume associated with installation of the treatment systems, and
- O Economic analysis of installation and operation of the treatment and reuse systems

### INDUSTRY BACKGROUND

During electroplating operations, metal parts are immersed in a bath containing dissolved plating metals and chemicals. Typical metals and alloys used for plating include cadmium, copper, iron, lead, nickel, gold, silver, platinum, brass and bronze. When an electrical potential is applied, an electrochemical cell is created and the dissolved metal deposits on the part surface. Parts are then conveyed to a series of rinse tanks to remove residual plating solution. Rinsing prevents spotting, uneven metal deposition and cross-contamination of the plating chemicals with later operations. This source of rinse water contamination is called dragout. When the dragout in the rinse tanks reaches high enough concentrations, rinsing is no longer effective and the rinse water must be replaced (Hunt, 1988). Some electroplating operations deal with this problem by replacing the entire rinse water volume on an as needed basis while others replace the rinse

water with a continuous inflow of fresh water. In addition to the dissolved metals utilized in the plating solutions, some baths contain organic compounds that modify the growth of the metal deposit to produce bright, semi-bright or satin finished surfaces.

Electroplating rinse water wastes are classified as hazardous due to the presence of heavy metals and, in some cases, cyanide in these solutions. Direct discharge of rinse waters can pollute natural resources, inhibit or destroy biological activity and sewage treatment processes, and corrode sewer lines and structures. Fortunately, treatment technologies exist which can minimize the harmful effects of rinse waters to human health and the environment. These processes render inactive or remove the hazardous components from the rinse waters.

Electroplating rinse waters can be treated by either end-of-pipe or in-plant recovery techniques. End-of-pipe treatments rely on chemical reactions such as pH adjustment to precipitate metal and other plating chemicals. These methods provide an effective means to remove the metal and cyanide species from the rinse water thus enabling reuse or discharge of the water. However, in most plant treatment systems, waste streams from the various plating lines are combined prior to treatment. Consequently, the resulting sludge must either be disposed of or treated with a high temperature metals recovery system to recover the metals. These options tend to be wasteful and expensive to implement. In-line treatments that make it possible to recover and return lost plating chemicals to the electroplating bath do exist. These recovery techniques are superior to the end-of-pipe treatments which merely reduce the waste volume. Additionally, in-line techniques require less processing than end-of-pipe recovery techniques. Examples of effective in-line recovery techniques include; low temperature evaporation, ion exchange, electrowinning, electrodialysis and reverse osmosis. Low-temperature evaporation and reverse osmosis techniques were selected for examination in this project because officials from the participating company, Graham Plating, believed that they offered the most promise for their facility.

## GRAHAM PLATING

Graham Plating is a large "job-shop" which has been located for many years on the north west side of the city of Chicago. A new modern building has recently been completed in Arlington Heights, Illinois and Graham Plating plans to relocate the plating operations to the new facility. Incorporated into the design of this new structure and in the new plating line layouts are special features which promote waste reduction. Examples of the waste reduction features are large underground rinse water collection tanks which provide the means to segregate the rinse waters by principal metal component and to store these waters for later processing and reuse.

The plating lines at the new Graham Plating facility will be like those at most plating operations in that they contain multiple bays for the different phases of the plating process. Objects to be plated are processed through a number of cleaning, pretreatment, and rinsing steps before the final metal plate is deposited. Part of the pretreatment may include plating with several other metals before the final desired finishing metal is applied. Figure 1 depicts plating

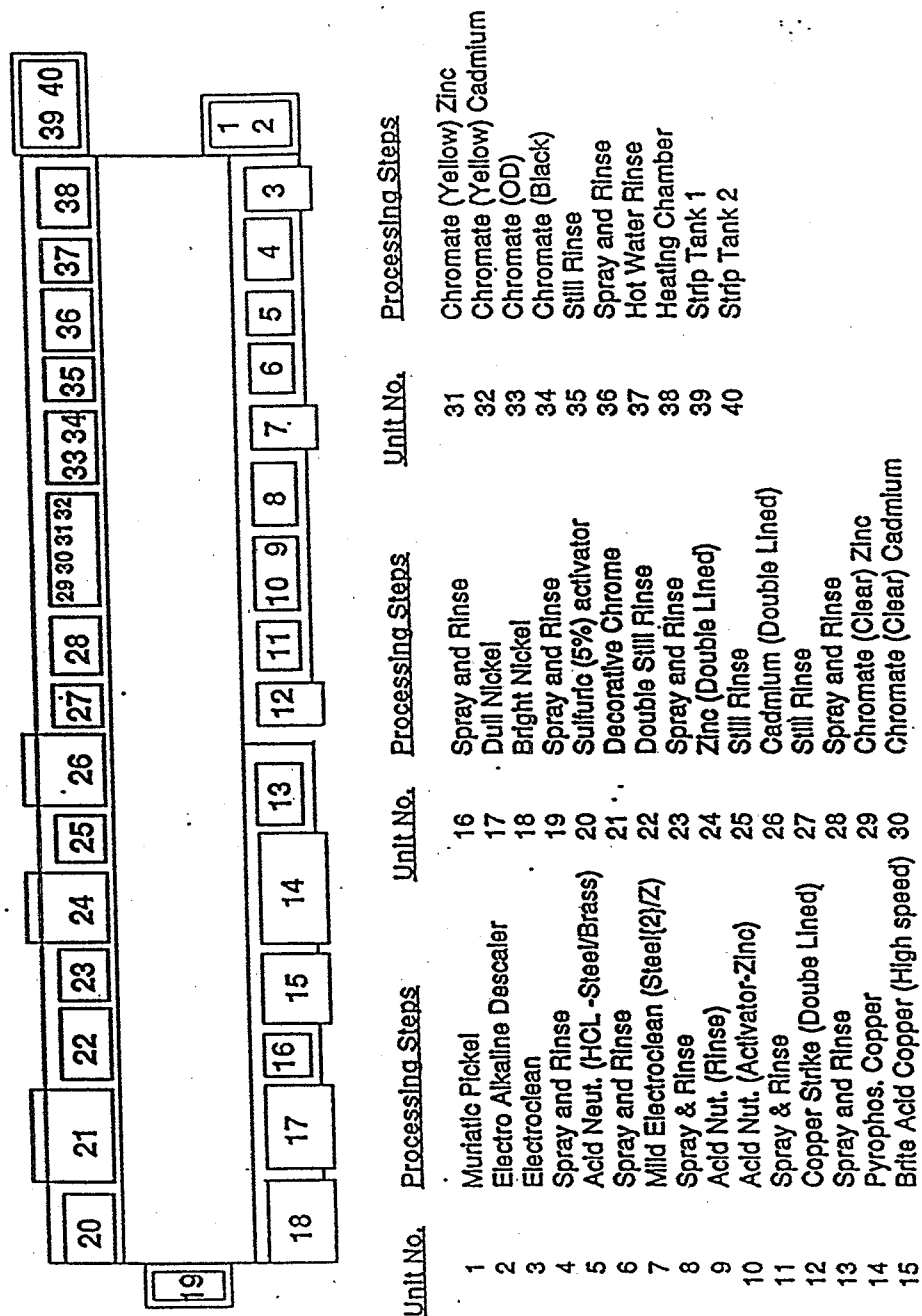


Figure 1.

Processing Line 2 at new Graham Plating facility

line 2 at the new Graham Plating facility which will be used for processing a variety of metals.

Once the final metal has been plated, the object will be rinsed using both spray and counter flow rinses. Water from the rinses will be collected in tanks for subsequent treatment through a vacuum evaporation unit. This unit is capable of processing 7,200 gallons of rinse water per day. If additional processing capacity is required, the low temperature evaporation unit may be supplemented with a reverse osmosis unit to expand the ability of the facility to process rinse water. The unique design of the new Graham Plating facility is such that these rinse waters can be collected in large underground storage containers allowing complete segregation of the rinse waters by their major metal component. The rinse water can then be collected in storage tanks and treated in batches.

## LOW TEMPERATURE EVAPORATION

Rinse water from the plating operation will be pumped into a low temperature evaporation unit where it will be treated such that the water can be reused for rinsing operations in the plating lines and the concentrate solution can be replaced in the plating bath. This type of operation has been successfully used in some plating operations because it allows the recovery of not only the water but also the plating bath chemicals. The economics associated with purchase of the equipment are usually favorable and utilization of the equipment can facilitate compliance with environmental regulations (Making Pollution Prevention Pay, 1983).

Evaporation units can be classified as either atmospheric or vacuum. In both cases the water is heated to produce vapor which is later condensed resulting in distilled water. Impurities in the untreated water remain as a concentrated slurry or solution of chemicals, in this case, the latter. In vacuum evaporation, the process takes place at pressures lower than atmospheric pressure. The pressure reduction lowers the boiling point of the water which permits the use of lower temperatures to create the steam and finally, the distilled water (Kushner and Kushner, 1981). Commercial systems may be constructed of a combination of glass, titanium, fiberglass, PVC, and stainless steel. Although the capital and operating costs of these systems are expensive when compared to other waste treatment options, their corrosion resistance makes them ideally suited to use with electroplating wastes.

In addition to facilitating recovery of the plating rinse waters, the lower boiling temperatures of the vacuum type unit enable recovery of the plating chemicals which might decompose during a standard evaporation process (Electroplating Engineering Handbook, 1984). This is accomplished by concentrating the metal salts and organic compounds present in the rinse water to a point that is acceptable for replacement in the plating bath. At the new Graham plating facility the recovery process will begin with the collection and storage of the rinse waters from both spray and counter flow rinses. Rinse water from nickel plating will collect in the designated storage tank until it is filled. Processing will begin by pumping water into the vacuum evaporator condenser. Here the water is heated under low pressure to produce steam which is condensed. At this point, inorganic contaminants will have been removed from the rinse water but organic additives are likely to remain since they tend to volatilize with the steam. To remove the organics, the water can be passed through a carbon filter. The recovered water,

which will then be suitable for reuse in the plating line rinses, will continue through a closed loop system to a reservoir for storage until it is needed. The inorganic salts and the non-volatile organic compounds will remain in the condenser as a concentrated solution or slurry. Prior to replacement of the concentrate into the plating bath, the concentrate will be removed from the condenser for characterization and adjustment by the laboratory. A schematic drawing of materials flow through a vacuum evaporator is provided in Figure 2.

Characterization of the concentrate is perhaps the most important step in the chemical recycle process. Changes in plating quality will result if the integrity of the plating bath is not maintained. To do this one must be certain of the composition of everything that is being added to the bath including the recycled rinse water chemicals. It is almost certain that the concentrate will be somewhat different from the original plating bath solutions. The anticipated inorganic components will be metallic nickel, nickel sulfate, nickel chloride, nickel sulfamate, boric acid, and hydrochloric acid. But other inorganic components, such as metal from the object being plated, may also be present (Metal Finishing, 1987).

The amount of organic compounds in the concentrate and plating solutions is important. Since these organic compounds are critical in obtaining the desired finishes, knowledge of their concentration in the concentrate solution is critical. Volatile organic compounds can be removed by a carbon filter but there will be non-volatile organics present in the concentrate as well. Knowing the concentration of all of the inorganic and organic constituents of the concentrate is vital in determining if the solution can be recycled in the plating bath and what supplements (if any) need to be added to the concentrate before recycling. Additionally, chemical characterization of the concentrate would help determine if contaminants were concentrated during rinse water processing.

## REVERSE OSMOSIS

Reverse osmosis is a proven technology for concentrating dilute solutions. In a closed-loop electroplating operation, reverse osmosis can be used to recycle purified water to the rinse tanks and return plating chemicals to the bath. Reverse osmosis systems can be used in combination with other treatment systems, such as low temperature evaporation and ion exchange, or it can be used alone. The higher metal ion concentrations of the processed rinse water produced with reverse osmosis systems can improve the efficiency of the other treatment systems. When reverse osmosis is used alone, it can purify rinse water for rinsing and reclaim lost metals and plating chemicals for reuse (Rozelle, 1973).

Reverse osmosis is a pressure-driven membrane separation process in which a feed stream under pressure (200-800 psig) is separated into a purified "permeate" stream and a "concentrate" stream by selective permeation of solution through a semi-permeable membrane. The pressure required to force the permeate through the membrane is dictated by the osmotic pressure of the feed stream (Rousseau, 1987). Three important parameters describe the performance of the reverse osmosis process: recovery, flux, and rejection. Recovery is defined as the percentage of the feed that is converted to permeate. Flux is the rate at which the permeate passes through the membrane per unit of membrane surface area. Rejection is the

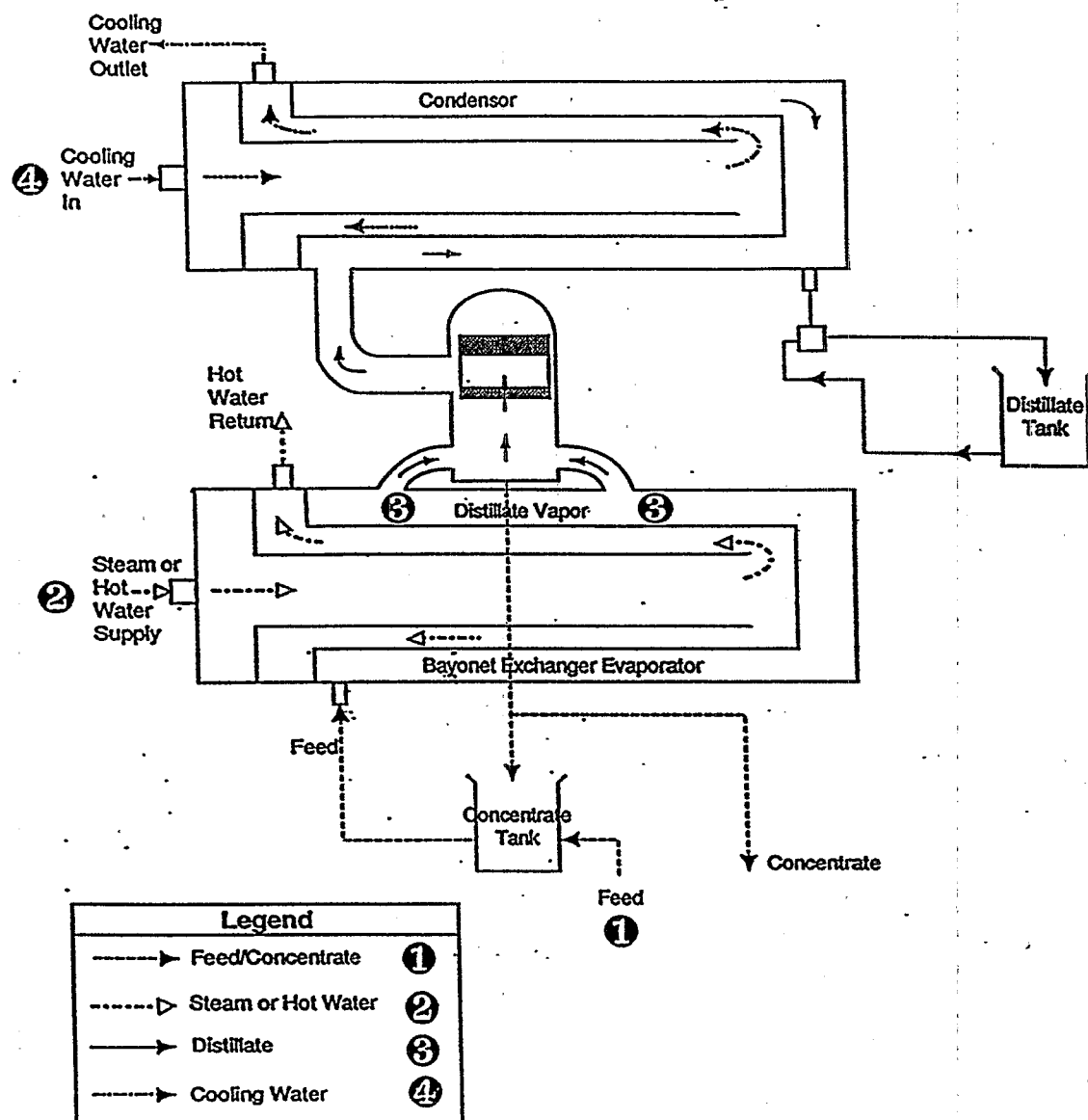


Figure 2.

Basic flow diagram for single effect evaporator.



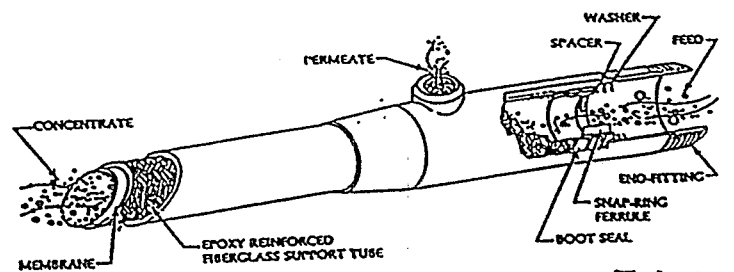
ability of the membrane to restrict the passage of dissolved salts into the permeate, and is related to particular salt species (Cushnie, 1985).

One of the most significant operating problems common to all membrane types is gradual reduction in performance associated with plugging by suspended solids. Another potential problem is caused by the precipitation of dissolved solids in the feed solution as it is concentrated in the reverse osmosis unit. With proper pretreatment (suspended solids filtration, oxidation, pH adjustment, and so forth), performance can be trouble-free and the equipment easily maintained.

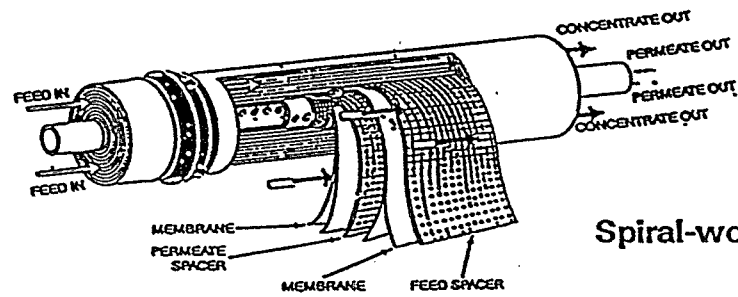
Three major types of membrane modules are available for commercial use. These include; tubular, spiral-wound, and hollow fiber (Figure 3). Tubular membranes are not susceptible to plugging by suspended solids and can be operated at high pressures. However, they are expensive to operate and require large amounts of space. Spiral-wound and hollow-fiber modules cost considerably less to operate. The hollow-fiber modules require less space to operate while the spiral-wound modules are less susceptible to plugging by suspended solids. All three types of membranes can be constructed from a variety of materials, such as: aromatic polyamide, cellulose acetate, and polyether/amide.

Reverse osmosis systems offer advantages over evaporation and ion exchange systems for treating rinse waters in that they are cheaper and require less energy for operation. However, the application of reverse osmosis technology is limited due to the modest degree of metal salt concentration it can achieve compared to other technologies. Additionally, the permeate purity resulting from treating concentrated feed streams may not be acceptable for reuse as rinse water.

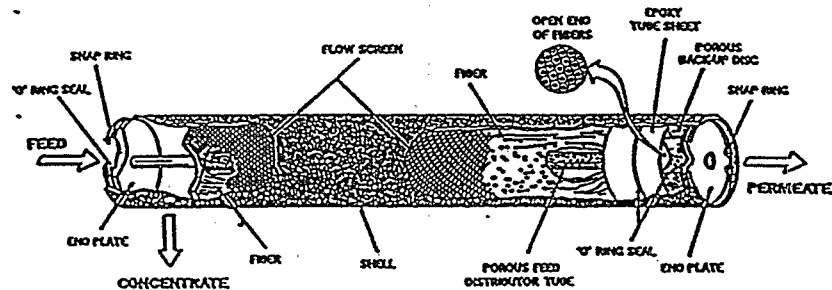
Currently, no plans exist to install a reverse osmosis system at the Graham Plating facility. However, if the low temperature evaporation system is not capable of processing the quantities of rinse water they will be generating at their new facility, Graham Plating will consider supplementing the evaporation system with reverse osmosis. If the decision is made to install a reverse osmosis unit at the new Graham plating facility, results obtained from tests conducted during this project will be utilized to determine the type, size, and operating parameters of the system.



Tubular



Spiral-wound



Hollow Fiber

Figure 3. Components of tubular, spiral-wound, and hollow fiber membranes.

## SECTION 2

### MATERIALS AND METHODS

#### LOW TEMPERATURE EVAPORATION TESTING

The Low Temperature Evaporation unit utilized in this study was manufactured by Licon, Inc. of Pensacola, Florida. This unit is a model C-3, single effect, pilot scale evaporator specially designed for conducting pilot scale tests on a variety of feed solutions. The full scale unit to be installed at the new Graham Plating facility was also manufactured by Licon, Inc.; however, it is a double effect type evaporator. Double effect evaporators differ from single effect evaporators in that heat generated in a first evaporation step is conserved and used in a second step. The primary advantage of utilizing a double effect system is that only half of the energy is required to process solutions as is required with a single effect evaporator. The quality of the concentrate and distillate produced by the two types of evaporators is virtually the same. Consequently, with the exception of energy usage, the pilot scale unit provided an accurate representation of performance that might be expected from a full scale system.

The Low Temperature Evaporation testing was conducted at HWRIC's pilot laboratory using the pilot scale unit described above. A brief discussion of the low temperature evaporation process that takes place in this unit is provided below and shown on Figure 2. Feed solution is pumped from a reservoir (in this case, 2 - 55 gallon sample drums were used) to a 6 gallon concentrate tank on the unit where it is stored for processing. Float limit switches maintain the concentrate tank at a relatively constant level. Solution is pumped from the concentrate tank into a bayonet exchanger evaporator cell which is equipped with heat exchangers that provide the heat necessary to separate water vapor from the feed solution. The bayonet exchanger evaporator cell maintains a vacuum of approximately 23 to 25 inches which enables the solution to boil at a temperature of approximately 150 to 160° F. The vapor rises through a separator cell until it collects on a water cooled condenser which transforms the water vapor into liquid water. The liquid water is then collected in a 6 gallon capacity distillate tank and ultimately discharged to a drum or holding tank where it can be recycled for use as rinse water or any number of other purposes.

#### REVERSE OSMOSIS TESTING

The reverse osmosis unit used in this project was manufactured by Osmonics of Minnetonka, Minnesota. The unit is a model PES/OSMO-19T-80SSXXC Reverse Osmosis machine for process evaluation. It is capable of operating at pressures ranging from 100 to 800

psi and can accommodate one membrane cartridge 2-1/2 inches in diameter x 39 inches long. The feed inlet to this unit is equipped with a prefilter which was supplied with a 5 micron cartridge filter for the duration of this project. Prefiltering the solution helps prolong the life of the membrane by removing dirt and other abrasives that might damage the membrane. This unit is capable of utilizing a variety of membrane cartridges. For the purposes of this project, an Osmonics model number 192T-MSO5 thin film composite membrane cartridge was used to process the rinse water solution. This is a spiral wound type membrane cartridge and is constructed of a polyamide film cast over a polysulfone backing. The membrane cartridge contains approximately 19 square feet of membrane and has a molecular weight cutoff of 100 - 150. This membrane was selected because Osmonics representatives indicated that it has been used extensively for processing nickel rinse waters with good results.

Two 55 gallon drums of Graham Plating rinse water solution were processed individually through the reverse osmosis system at HWRIC's pilot lab facility. The solution was pumped through a 5 micron filter prior to reverse osmosis processing. The filter had to be changed once during the prefiltering stage due to buildup of a waxy film on the filter cartridge which restricted flow. Based on preliminary electrical conductivity analysis performed on the rinse water (10230 umhos for Drum C and 6110 umhos for Drum D), it was assumed that the solution had an osmotic pressure of approximately 10 psi. Reverse osmosis systems can be effective on solutions with osmotic potentials as high as 200 psi. Therefore, it was estimated that the equipment could concentrate each drum of the solution approximately 20 fold to about 2 3/4 gallons per drum. Based on these calculations, it was estimated that primary pressures of 250 to 380 psi would have to be maintained in the system during solution processing. The pressure was increased periodically as the volume of feed solution decreased and became more concentrated. Temperatures were maintained at 74 to 80 degrees fahrenheit through use of a heat exchanger which is mounted on the unit. The concentrate flow rate was maintained at approximately 3 gallons per minute for the duration of the testing while the permeate rate started at approximately 15 to 25 gallons per hour and decreased steadily as the solution became more concentrated until a final rate of less than 3 gallons per hour was attained.

## CHEMICAL ANALYSIS

In an effort to determine the effectiveness of both the low temperature evaporation and reverse osmosis systems with respect to treating the rinse water, samples were collected during the tests and analyzed for key chemical constituents. Concentrate and distillate samples comprised the bulk of samples collected during the low temperature evaporation testing while concentrate and permeate samples were the predominant sample types collected in the reverse osmosis testing. Analyses conducted on the samples included electrical conductivity, pH, total organic carbon, and nickel.

As the testing progressed for both technologies, samples were analyzed for pH and electrical conductivity immediately after collection in the pilot lab. A Beckman model number 32 pH meter was used to measure the sample pH. Using method number 9040 from SW846 (EPA 1986). An Orion model number 140 Conductivity/Salinity meter was used to determine

electrical conductivity using method number 9050 from SW846 (EPA 1986). Both instruments were operated according to procedures provided in the instruction manuals.

Analysis for total organic carbon (TOC) was conducted to provide an indication of the fate of the organic constituents present in the rinse water. The TOC analysis was conducted on a Rosemount Analytical Dohrmann model DC-190 TOC Analyzer according to method 9060 from SW846 (EPA 1986). Samples were diluted according to strength to ensure the instrument was working within the instrument working range.

Nickel analyses were performed to determine the efficiency of both the low temperature evaporator and reverse osmosis systems with respect to removing this metal from the rinse water and concentrating it for potential recycling. Samples were digested according to method 3010 and analyzed according to method number 7520 from EPA SW846 (EPA 1986). A Varian Spectra-10 Atomic Absorption Spectrophotometer was utilized to perform the analyses. Most analyses were run at 352.4 nm, the wavelength most appropriate for the high concentrations in many of the samples. Instrument settings were in accordance with the manufacturer's AAS operating/procedures manual. Because of the large range in concentrations in these samples, alternative wavelengths were also used. Samples were diluted as necessary to ensure the concentration was within a range that would provide optimum accuracy.

## SECTION 3

### EXPERIMENTAL PROCEDURE

#### FIELD SAMPLE COLLECTION

Since Graham Plating has not yet relocated to their new facility in Arlington Heights, Illinois, field samples of the rinse water had to be collected from their Chicago facility. Rinsing of nickel plated parts is accomplished at the Chicago facility by manually dipping baskets of plated parts into two separate (primary and secondary) rinse tanks which contain about 150 gallons of rinse water each. Four - 55 gallon drums of electroplating rinse water (henceforward referred to as Drums A,B,C, and D) were collected from a secondary nickel rinse water tank at the Graham Plating facility in Chicago and shipped to the HWRIC pilot lab facility by a DOT licensed hazardous materials transporter. The samples were collected by pumping the solution intermittently into the drums over a 2 day period. Upon their arrival at the pilot lab, 2 subsamples were collected from each drum and placed in 250 ML. nalgene bottles. These samples were analyzed to determine baseline nickel concentrations present in the drums. Results of these analyses are summarized below:

O	Drum A	4,820 to 4,940 mg/L
O	Drum B	2,680 to 2,750 mg/L
O	Drum C	2,740 to 2,770 mg/L
O	Drum D	1,580 to 1,590 mg/L

Based on these data, calculations were performed to estimate the volume reduction necessary to produce a concentrate solution of approximately eight percent nickel (the target nickel level maintained in the Graham Plating nickel bath). In addition to the bulk samples collected at the Graham Plating facility, a sample of the nickel plating bath solution was collected and analyzed to verify that the bath contained eight percent nickel.

#### LOW TEMPERATURE EVAPORATION PROCEDURE

Drums A and B were processed separately through the low temperature evaporation unit. The unit processed the solution at a rate of approximately 3 gallons per hour. A total of 14 operating hours were required to process drum A (approximately 42 gallons) while 16 hours were required to process drum B (52 gallons). Numerous operating parameters associated with various pump and system pressures and temperatures were monitored throughout the testing to ensure that optimum conditions were maintained.

Processing continued until the rinse water volume was reduced to a point where its viscosity limited the ability of the unit's feed pump to take in the solution. At this point, virtually all of the remaining unprocessed solution was contained in the unit's plumbing (approximately 1 gallon). Samples of concentrate and distillate were collected at one hour intervals in 250 ML. nalgene bottles and immediately tested for pH and electrical conductivity. The samples were then diluted with 2% nitric acid and stored in a 4 degrees celsius cold storage room until the other chemical analyses could be performed. Nickel analysis was performed on all samples collected during these tests. Additionally, total organic carbon (TOC) was determined on samples collected at the beginning of the testing, and approximately every 4 hours thereafter until concentrations above 3,500 mg/L of TOC were attained. Beyond that point, TOC levels increased dramatically as the volume of the feed solution was reduced. Therefore, TOC measurements were performed on samples collected on an hourly basis for the duration of the testing.

## REVERSE OSMOSIS PROCEDURE

In order to determine baseline flux characteristics of the membrane used in this test, clean tap water was processed through the reverse osmosis unit for a 30 minute period prior to and after testing with the rinse water solution. These "clean water flux" measurements were determined utilizing gauges mounted directly on the reverse osmosis machine. Following determination of the clean water flux measurements, Drums C and D were processed separately through the reverse osmosis unit. Flux measurements were collected at 15 minute intervals during the tests. Results of these tests indicated that, at the beginning of the testing, the reverse osmosis system processed solution at rates of 15 to 25 gallons per hour for drums C and D, respectively. However, as the testing progressed and the feed solution became more concentrated, the flux rate associated with both drums fell off to about 3 gallons per hour.

A primary operating pressure of approximately 250 to 300 psi was maintained for the Drum C test. The Drum D test was conducted at a primary operating pressure of approximately 350 to 380 psi. The higher pressures implemented in the Drum D test were used to determine the impacts of increased operating pressure on the equipment's productivity and the quality of the solutions processed. Overall, the two drums (approximately 52 gallons each) were processed in 6.25 and 5 hours, respectively. However, the solution could only be concentrated down to a final volume of about 6 gallons (as opposed to the 1 gallon volume reached with the low temperature evaporation system) before the flux rate plunged to the 3 gallon per hour level.

Samples of concentrate and permeate were collected at 15 minute intervals during these tests and placed in 250 ML. nalgene bottles. These samples were immediately analyzed for pH and electrical conductivity. The samples were then diluted with 2% nitric acid and placed in a 4 degrees celsius storage room until analysis for the other chemical parameters could be accomplished. Nickel analysis was performed on all samples collected during the reverse osmosis testing. Additionally, total organic carbon was determined in samples collected at the beginning of testing and approximately every 75 minutes during the duration of the reverse osmosis testing.

## SECTION 4

### RESULTS AND DISCUSSION

Results of the pilot scale testing and laboratory analysis were utilized to determine the effectiveness of the two technologies tested in this project with respect to 1) the success in concentrating the nickel levels for reuse in the electroplating process, 2) cleaning the electroplating rinse waters for possible reuse or discharge to the POTW, and 3) the relative productivity and economic feasibility associated with installing these systems in an electroplating facility. The nickel concentrations of the various solutions monitored were determined throughout this project to determine its fate with respect to the processes studied. Total organic carbon concentrations were also monitored during the course of the project to provide an indication of the fate of the organic additives that accumulate in the rinse water concentrate. Electrical conductivity was monitored to determine if relationships exist between this parameter and the other parameters tested. Electrical Conductivity is a relatively quick and inexpensive parameter to monitor during actual operating conditions. Therefore, identification of relationships between electrical conductivity and the other parameters could prove to be important when low temperature evaporation and reverse osmosis technologies are actually implemented in the electroplating process. Additionally, pH was monitored in all samples collected in this project. Distillate and permeate samples tended to exhibit pH levels from 0.5 to as much as 2 units higher than the concentrate pH measurements. Some fluctuations occurred with respect to solution pH as the tests progressed although the pH variations did not play an integral part in determining quality of either distillate, permeate, or concentrate. Therefore, the pH data are not presented in this report.

#### EQUIPMENT PRODUCTIVITY

The low temperature evaporator processed the solutions at a relatively constant rate of 3.4 gallons per hour for drum A and 2.8 gallons per hour for drum B. As shown in figure 4, the productivity rate varied little during the course of these tests. The evaporator processed the solution down to a final volume of approximately 1 gallon which corresponds to a volume reduction of nearly 98 percent.

The reverse osmosis unit processed the solutions at a relatively constant rate until approximately 60 percent of the drum volume was treated. Productivity beyond this point began to decrease steadily until about 80 percent of the solution was treated. Beyond the 80 percent level, the productivity decreased dramatically (see Figure 5). The reverse osmosis equipment



processed the solution from Drum D faster than the Drum C solution. This occurred due to the higher operating pressures that were utilized in the Drum D test.

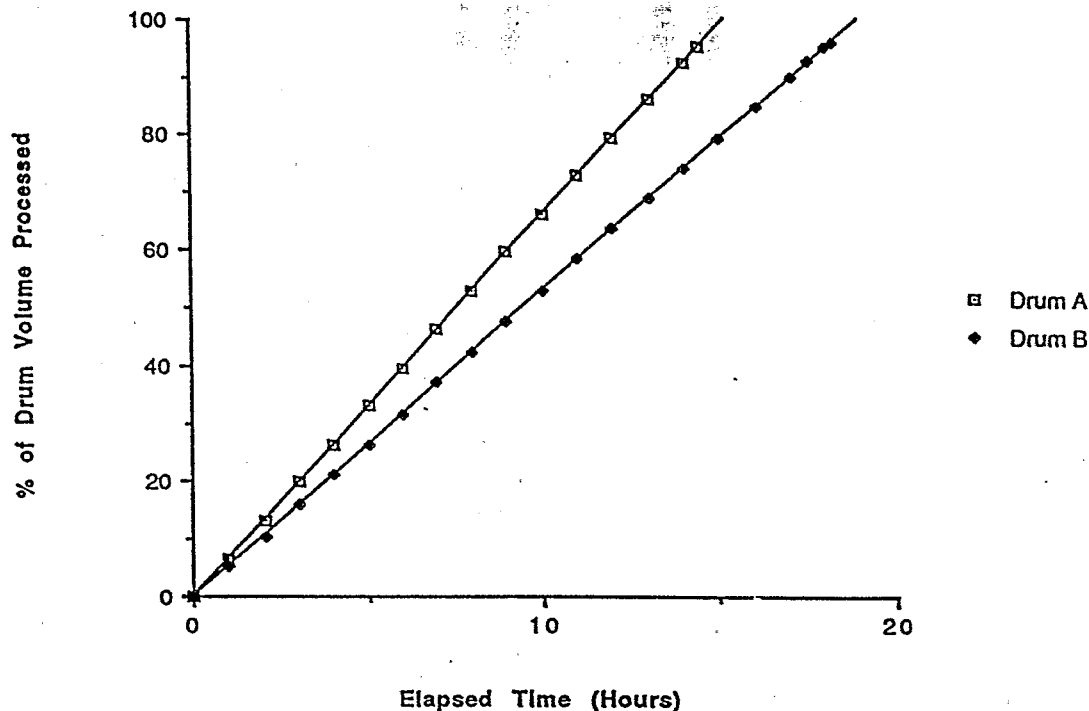


Figure 4. Percent of drum volume processed versus time; low temperature evaporation tests.

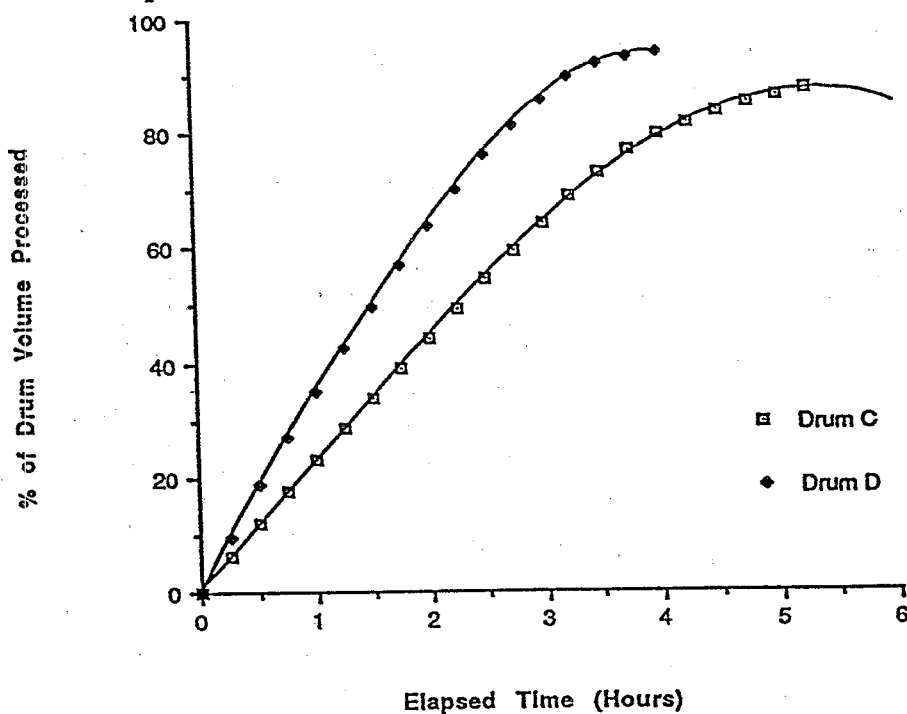


Figure 5. Percent of drum volume processed versus time; reverse osmosis tests.

Figure 6 shows how the permeate flux rate changed over time in the two reverse osmosis tests. Due to the higher operating pressures used in the Drum D test (350 to 380 psi), the unit produced permeate at an initial rate of about 20 gallons per hour which corresponds to a flux rate of about 0.0031 gallons/hour/sq.ft. of membrane/psi. This rate was substantially higher than the initial productivity observed in the Drum C test (operating pressures of 250 to 300 psi) which started at about 13 gallons per hour (0.0027 gallons/hour/sq.ft. of membrane/psi). However, the Drum D flux rates decreased more rapidly over time than those observed in the Drum C test such that the Drum C flux rates were actually higher after about 2 hours of operation. These data indicate that increases in operating pressure will result in improved initial productivity. However, the increased pressure also causes the membrane to foul at a faster rate resulting in a more rapid decline in productivity.

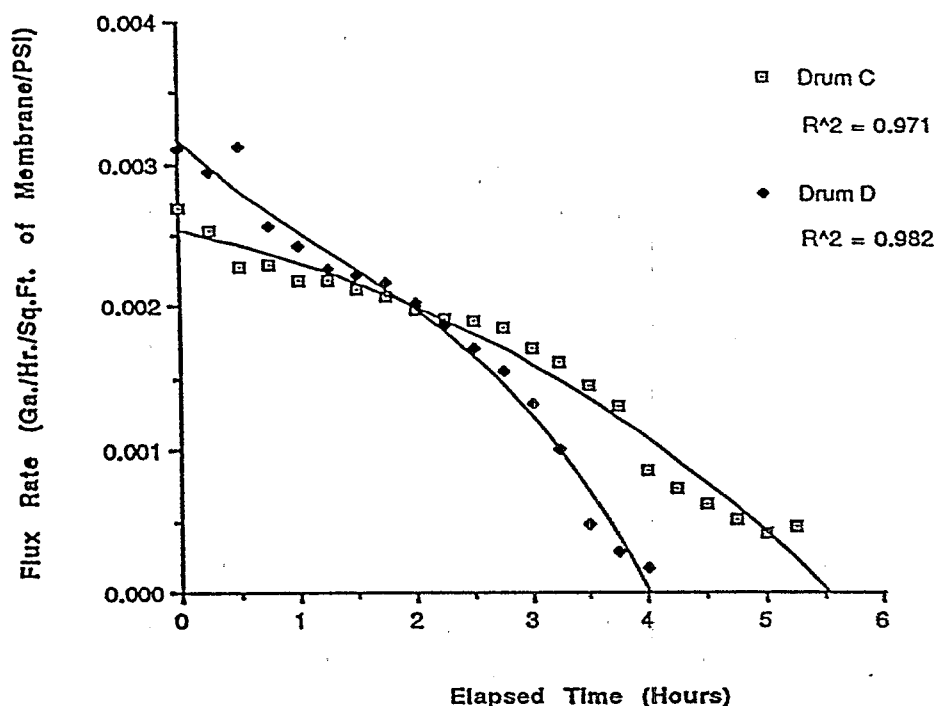


Figure 6. Permeate flux rate versus time; reverse osmosis tests.

Figure 7 shows how the permeate flux rate changed as the relative volume of the two drums was processed through the reverse osmosis equipment. The flux rate for both tests dropped initially as the first 10% of the drum volume was processed. The flux rates then leveled off until approximately 60% of the volume had been processed. Beyond this point, the flux rates dropped off dramatically for both of the drums tested. It is noteworthy that in this comparison the flux rates associated with the Drum D test were consistently higher than the Drum C flux rates. Consequently, it took about 1.5 hours longer to process Drum C than Drum D. These differences in flux rates can be attributed to the higher operating pressure utilized in the Drum D test.

The Drum C solution was processed to a final volume of about 6 gallons (an 88% volume reduction) while the Drum D solution was processed to a final volume of 3 gallons (a 94%

volume reduction). Reverse osmosis processing was terminated at these endpoints because of the drastically reduced production rates (approximately 1 to 3 gallons per hour). Clean water flux measurements taken before and after treatment of each of the two drums indicated that the membranes recovered fully after the tests even though no attempt was made to clean the membranes.

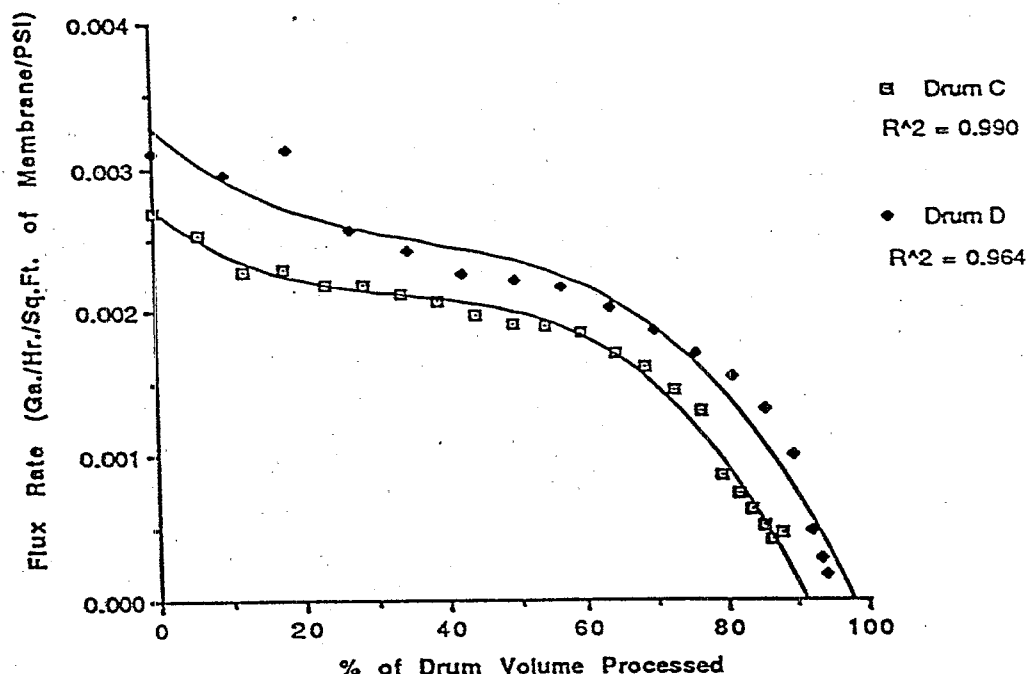


Figure 7. Permeate flux rate versus percent of drum volume processed; reverse osmosis tests.

## NICKEL CONCENTRATIONS

In the low temperature evaporation tests, nickel concentrations started at 4140 mg/L (Drum A) and 2540 mg/L (Drum B) respectively, (Table 1) and increased at a steady rate until concentrations of approximately 25,000 to 30,000 mg/L were reached. As shown in Figure 8, this level corresponds to a point where approximately 80 to 85 % of the rinse water volume had been processed. Beyond this point, nickel concentrations increased dramatically to concentrations of 179,000 mg/L (Drum A) and 128,000 mg/L (Drum B), respectively. In the Drum A test, the concentrate solidified shortly after the final concentrate sample was collected. A sample of this solid concentrate was collected from the evaporator's plumbing and was found to contain a concentration of 18.6% of nickel. Table 1 shows the nickel concentrations of the concentrate solutions at the beginning and end of both the low temperature evaporation and reverse osmosis tests.

TABLE 1. COMPARISON OF NICKEL CONCENTRATIONS IN CONCENTRATE, DISTILLATE, AND PERMEATE  
NICKEL CONCENTRATIONS

Product	Low Temp. Evap.		Reverse Osmosis	
	Drum A	Drum B	Drum C	Drum D
Concentrations at beginning of test (mg/L):				
Concentrate	4,140	2,540	2,580	1,425
Distillate	2.5	2.2	—	—
Permeate	—	—	44.5	14.5
Concentrations at end of test:				
Concentrate	179,000	128,000	12,560	18,200
Distillate	1	0.3	—	—
Permeate	—	—	210	790
Ratio of distillate permeate to concentrate:				
Distillate	0.02%	0.01%	—	—
Permeate	—	—	1.49%	1.54%

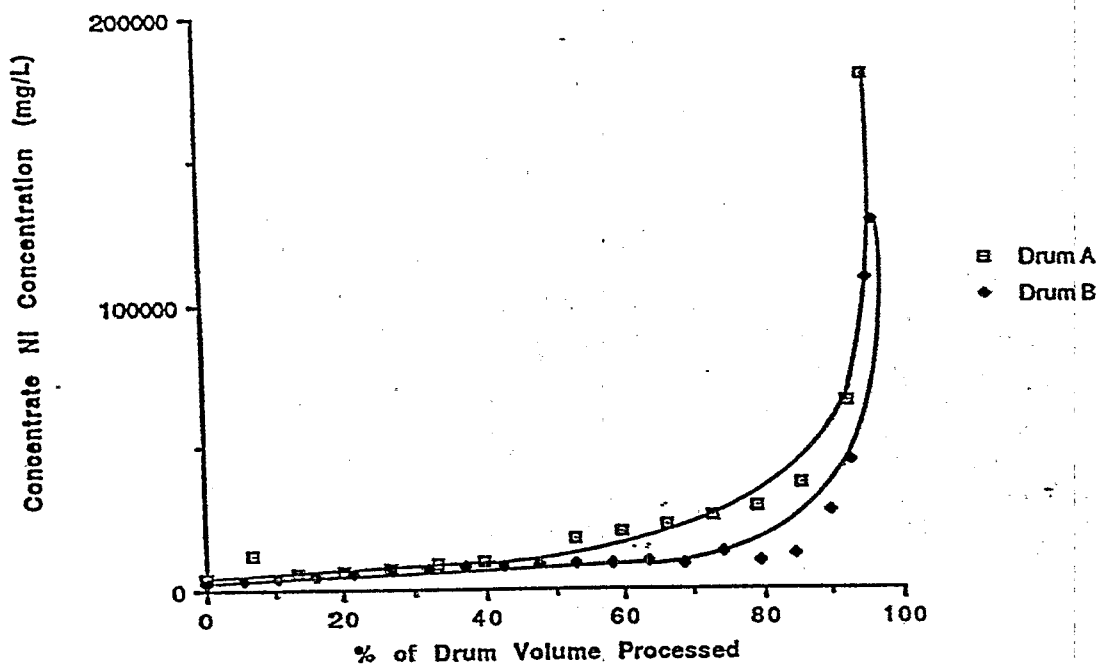


Figure 8. Concentrate nickel concentration versus percent of drum volume processed; low temperature evaporation tests.

Nickel levels used in the reverse osmosis tests started at 2580 mg/L and 1425 mg/L respectively (Table 1). Figure 9 depicts how nickel concentrations in these samples changed as the solutions were processed. Nickel concentrations increased steadily until about 60% of the rinse water volume was processed. At this point, nickel concentrations were about 4,000 to 5,000 mg/L in the two drums. Beyond this point, nickel concentrations increased more rapidly until final concentrations of 12,560 mg/L (Drum C) and 18,200 mg/L (Drum D) were reached. The final concentrations attained in the reverse osmosis tests correspond to about 7 to 14% of the nickel concentrations achieved with the low temperature evaporation tests.

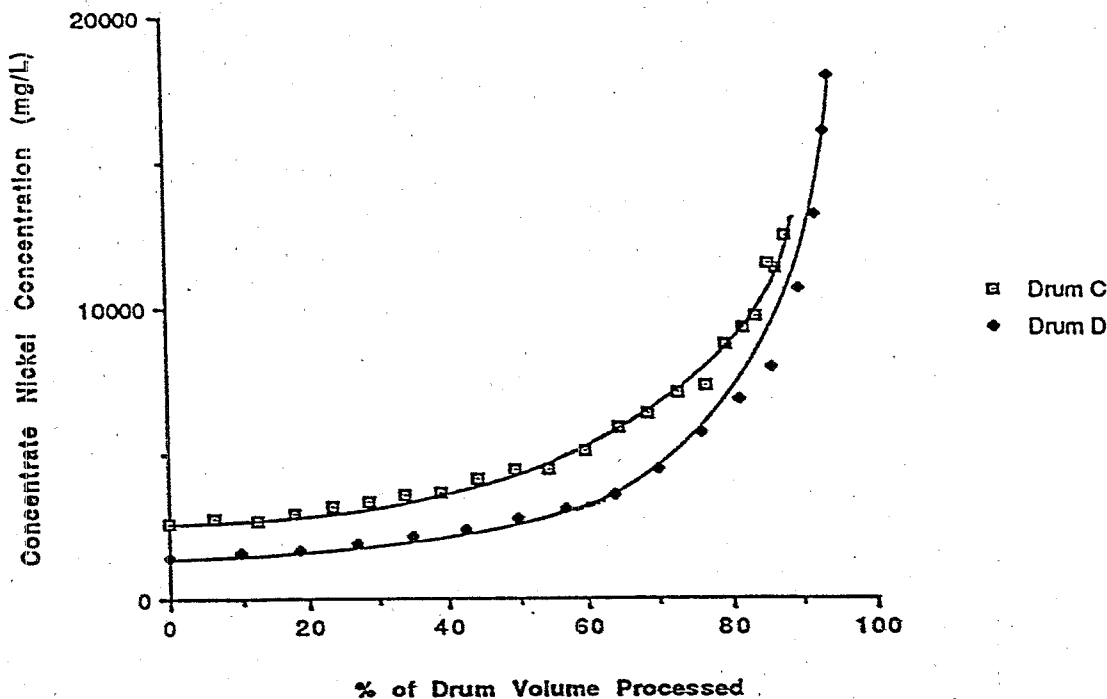


Figure 9. Concentrate nickel concentration versus percent of drum volume processed; reverse osmosis tests.

The increased nickel concentrations observed in concentrate samples from both the low temperature evaporation and reverse osmosis tests were relatively consistent with the volume reductions that were accomplished in these tests. This phenomenon, along with the data presented above, suggests that the equipment effectively concentrated the nickel.

Distillate nickel concentrations determined in the low temperature evaporation tests ranged from 2.5 to 0.1 mg/L during the course of testing. Distillate samples collected from the Drum A test averaged 0.71 mg/L while Drum B samples averaged 0.37 mg/L. Table 2 provides a summary of the average nickel concentrations in the distillate samples. The ratios of distillate to concentrate nickel concentration averaged 0.01% (Drum B) to 0.02% (Drum A) of the concentrate concentrations during the course of the testing. The nickel concentrations of the distillate did not appear to be affected by the rising nickel concentrations in the concentrate as the test progressed. Due to the fact that nickel salts are not volatile, distillate samples collected at the beginning of the tests contained nickel concentrations similar to those collected at the end

of the test. The levels of nickel in the rinse water are low enough in the distillate such that it could be both reused as rinse water or discharged to the POTW without additional treatment.

TABLE 2. AVERAGE NICKEL CONCENTRATIONS IN DISTILLATE AND PERMEATE

	Distillate Ni Concentration		Permeate Ni Concentration	
	Mean (mg/L)	STD (mg/L)	Mean (mg/L)	STD (mg/L)
A (n=13)	0.71	0.63	—	—
B (n=16)	0.37	0.52	—	—
C (n=22)	—	—	89.55	49.22
D (n=17)	—	—	134.38	202.19

The low temperature evaporation test data presented in Tables 1 and 2 do not include several samples which contained elevated nickel concentrations as a consequence of the evaporator malfunctioning. On several occasions, the evaporator feed solution overflowed and contaminated the distillate. Representatives from Licon have indicated that this phenomenon tends to occur in the pilot-scale evaporator due to its sensitivity to operating conditions and the necessity to calibrate all operating parameters manually. Full-scale production evaporators are not as susceptible to overflowing because the operating parameters are more fully automated. Based on this information, it was decided not to include the samples contaminated by the boil over in the Table 1 and 2 analysis.

Nickel concentrations in permeate samples (Table 2) averaged 89.55 mg/L in the Drum C test and 134.38 mg/L in the Drum D test. The difference in these tests can be attributed primarily to the higher pressures associated with the Drum D test which caused more of the nickel salts to permeate through the membrane. The ratio of permeate to concentrate nickel concentration averaged 1.49% (Drum C) and 1.54% (Drum D), respectively, during the course of the testing. Figure 10 shows the relationship between concentrate and permeate nickel concentrations during the reverse osmosis tests. This graph indicates that the relationship between concentrate and permeate nickel concentrations is relatively linear until the concentrate reaches a level of about 12,000 mg/L. Beyond this point, additional increases in concentrate nickel result in dramatically higher nickel concentrations in the permeate. The nickel levels in the permeate samples indicate that initial treatment with reverse osmosis would not produce water of adequate quality for discharge to a POTW. However, the processed water would be of sufficient quality for reuse as rinse water. Further, the quality of this solution could be further improved through additional reverse osmosis processing to remove additional nickel.

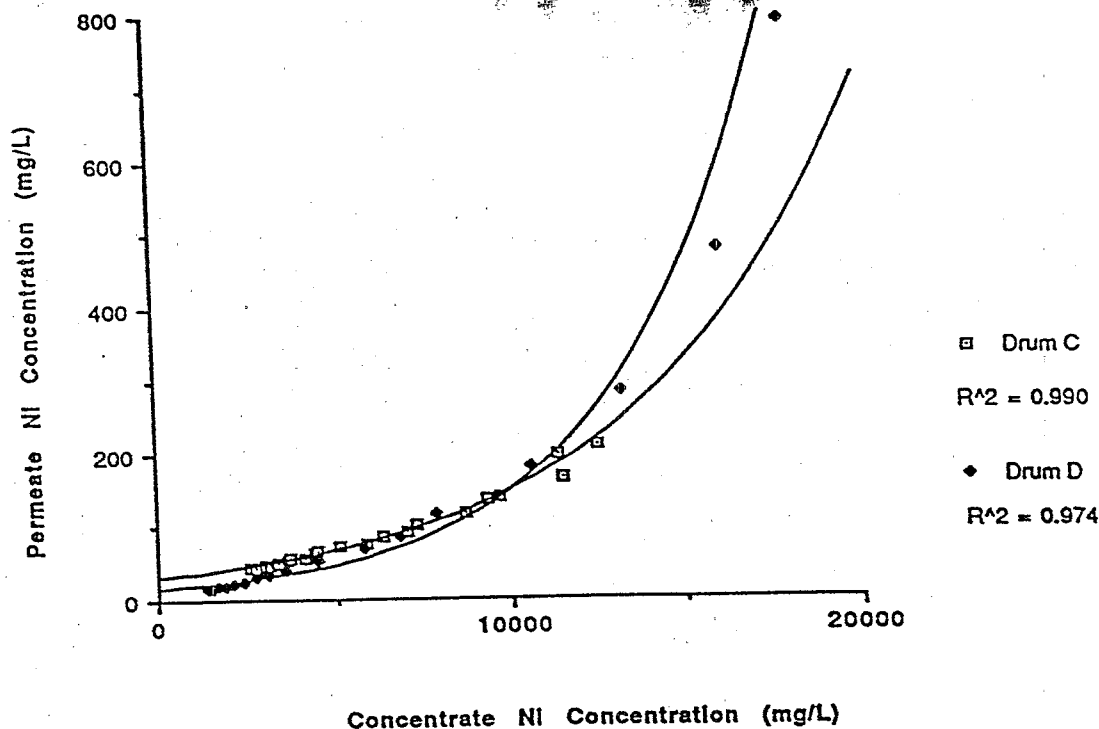


Figure 10. Permeate nickel concentration versus concentrate nickel concentration; reverse osmosis tests.

#### TOTAL ORGANIC CARBON CONCENTRATIONS

Total organic carbon (TOC) concentrations in the Graham Plating nickel plating baths are maintained at around 14,000 mg/L. TOC concentrations in rinse water samples collected during the low temperature evaporation tests started at 990 mg/L (Drum A) and 550 mg/L (Drum B), respectively. TOC levels in the concentrate increased slowly until a concentration of 4,000 to 5,000 mg/L was reached. These levels were reached when approximately 80 to 90% of the drum volume had been processed. Beyond this point, TOC concentrations increased dramatically until final concentrations of 26,000 mg/L A to 25,000 mg/L (Drum B) were attained. These concentration increases were relatively consistent with the volume reduction that was achieved. Figure 11 shows how levels of TOC increased in the concentrate solution as the rinse water was processed through the evaporator. Table 3 includes a summary of the TOC levels at the start and finish of these tests.

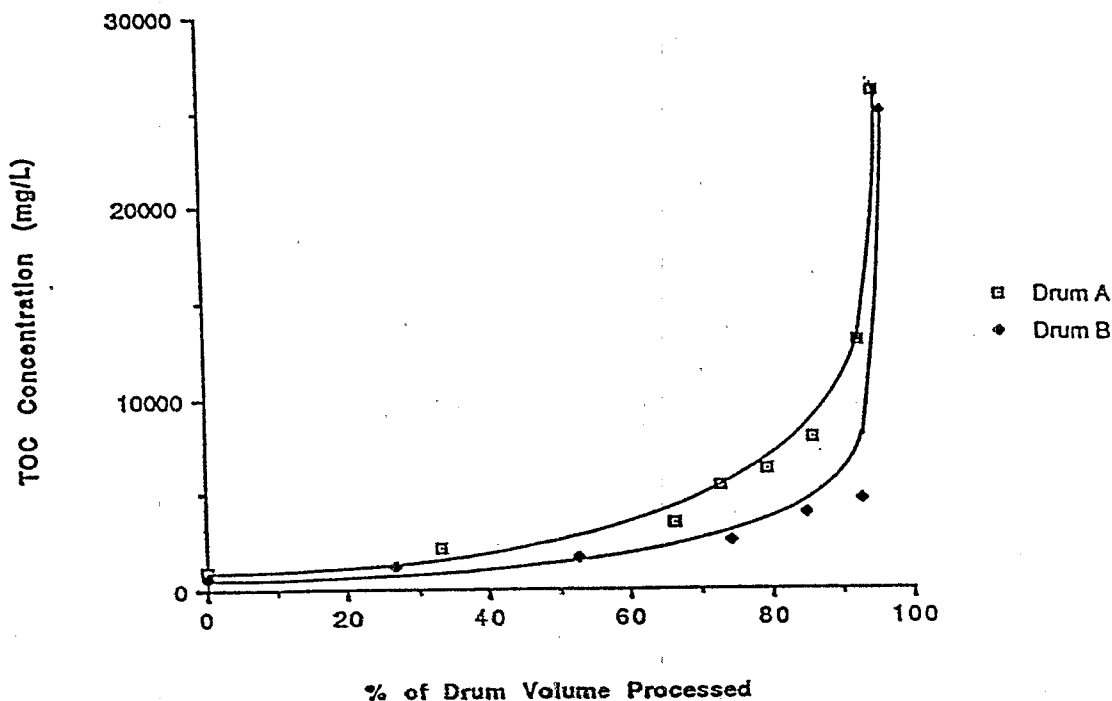


Figure 11. Concentrate TOC concentration versus percent of drum processed; low temperature evaporation tests.

TABLE 3. COMPARISON OF CONCENTRATE, DISTILLATE, AND PERMEATE TOC CONCENTRATIONS

	Low Temp. Evap.		Reverse Osmosis	
	Drum A	Drum B	Drum C	Drum D
Concentrations at Beginning of Test (mg/L)				
Concentrate	990	550	590	340
Distillate	2.6	6.3	—	—
Permeate	—	—	9.1	1.9
Concentrations at End of Test (mg/L)				
Concentrate	26,000	25,000	2,800	3,500
Distillate	6.3	1	—	—
Permeate	—	—	16	12

Figure 12 depicts changes in concentrate TOC levels that occurred as the volume of rinse water was processed through the reverse osmosis equipment. This information is summarized further in Table 3. As shown, concentrate TOC levels started at 590 mg/L (Drum C) and 340



mg/L (Drum D), respectively. TOC levels increased steadily until concentrations of approximately 1,000 mg/L were reached. This level was reached when about 60% of the drum volume was processed. Beyond this point, TOC levels increased more rapidly until final concentrations of 2,800 mg/L (drum C) and 3,500 mg/L (Drum D) were reached.

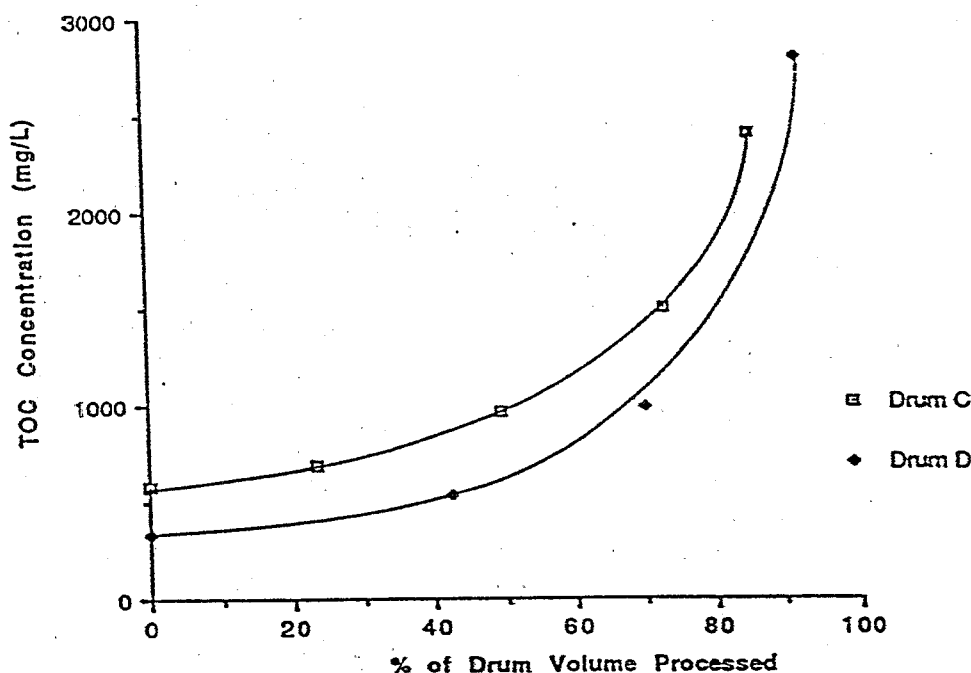


Figure 12. Concentrate TOC concentration versus percent of drum volume processed; reverse osmosis test.

The increased TOC concentrations observed in the concentrate samples from both the low temperature evaporation and reverse osmosis tests were fairly consistent with the volume reductions that were accomplished in these tests. This phenomenon suggests that the organic compounds present in the rinse water were effectively concentrated by the equipment and, in general, were not allowed to pass through the processes to the distillate and permeate solutions. While this information illustrates the fate of the quantity of organic compounds, it does not take into account changes in the quality of these compounds which may have occurred as a result of the processes. Evaluation of the individual organic compounds processed through the equipment would be costly and time consuming. While this information would be useful, it is beyond the scope of this study.

Permeate TOC levels were closely related to TOC levels present in the concentrate (Figure 12). Figure 13 provides information regarding changes in permeate TOC concentrations as the volumes of rinse water were processed through the reverse osmosis system. Comparison of this information to Figure 12 suggests that permeate TOC levels are closely related to TOC levels present in the concentrate. Permeate TOC concentrations increased relatively slowly at first but increased dramatically after the first 70 percent of the solution volume had been processed.

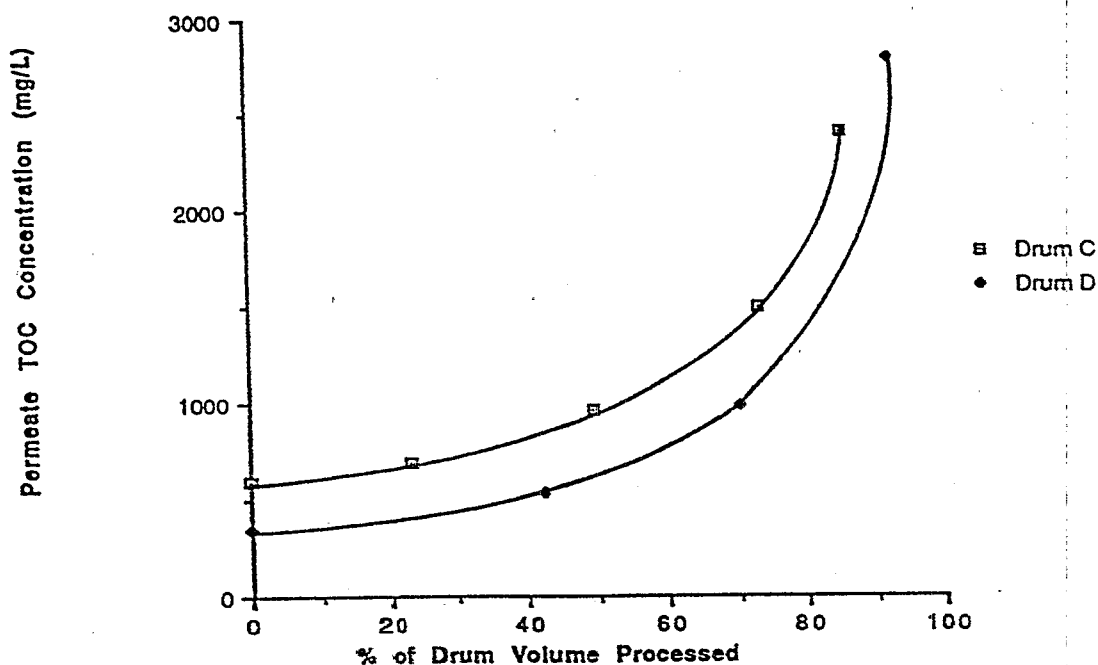


Figure 13. Permeate TOC concentration versus percent of drum volume processed; reverse osmosis tests.

Table 4 provides a summary of TOC levels in distillate and permeate solutions. As shown, the distillate samples averaged 3.5 (Drum A) and 3.03 (Drum B) mg/L, respectively. The permeate samples contained substantially more TOC, averaging 19.46 (Drum C) and 21.98 mg/L (Drum D), respectively. The permeate samples contained 5 to 7 times as much TOC as the distillate samples. TOC levels observed in the distillate samples averaged only 0.13 to 0.31% as much as the concentrate TOC levels. TOC was present in the permeate at levels which averaged 1.24 to 1.48% of the corresponding concentrate levels. This data suggest that the organic constituents (brighteners, etc.) associated with the rinse water are able to penetrate the reverse osmosis membrane but are not distilled in the low temperature evaporation process.

Table 4. Average TOC Concentrations in Distillate and Permeate

Drum	Distillate TOC Concentration		Permeate TOC Concentration	
	Mean (mg/L)	STD (mg/L)	Mean (mg/L)	STD (mg/L)
A (n=3)	3.50	2.48	—	—
B (n=4)	3.03	2.55	—	—
C (n=5)	—	—	19.46	15.48
D (n=4)	—	—	21.98	32.91

## ELECTRICAL CONDUCTIVITY

In the low temperature evaporation tests, electrical conductivity values in concentrate samples started at 16,600 (Drum A) and 10,150 (Drum B) umhos, respectively. As shown in Figure 14, conductivities increased steadily as the first 80 percent of the drum volume was processed to about 40,000 umhos (Drum B) and 65,000 umhos (Drum A). As the final few gallons of rinse water were processed, electrical conductivities increased dramatically to 98,700 umhos (Drum C) and 108,800 umhos (Drum B). Table 5 summarizes changes in concentrate ECs as the tests progressed.

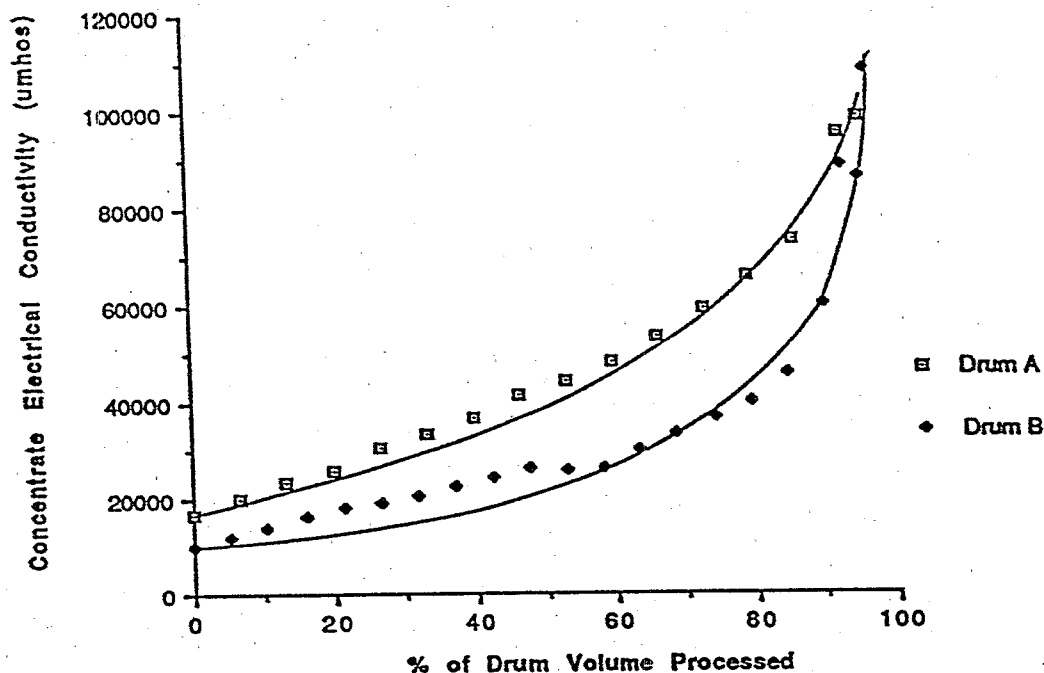


Figure 14. Concentrate electrical conductivity versus percent of drum volume processed; low temperature evaporation tests.

The increases in concentrate electrical conductivity values observed in the reverse osmosis tests were fairly consistent with the volume reductions that were achieved in these tests. This phenomenon combined with the data presented above suggests that the equipment was effective at keeping most of the salts from crossing the membrane into the permeate solution. Electrical conductivity values in concentrate samples collected from the first 80 percent of the low temperature evaporation tests were also reasonably consistent with the volume reduction that occurred. However, electrical conductivity values associated with concentrate samples collected from the final 20 percent of the low temperature evaporation tests are well below what might be expected based on the reduction of feed solution volume that occurred. These data suggest that at conductivities above about 60,000 umhos, salts begin precipitating out of solution and, consequently, are not measured in the electrical conductivity analysis.

Table 5. Comparison of Concentrate, Distillate, and Permeate Electrical Conductivities

	Low Temp. Evap.		Reverse Osmosis	
	Drum A	Drum B	Drum C	Drum D
Electrical Conductivities at Beginning of Test (umhos)				
Concentrate	16,600	10,150	10,300	6,270
Distillate	35.8	30.5	—	—
Permeate	—	—	500	242
Electrical Conductivities at End of Test (umhos)				
Concentrate	98,700	108,800	37,300	47,900
Distillate	27.3	21.6	—	—
Permeate	—	—	3,720	9,190

As shown in Figure 15 and Table 5, concentrate electrical conductivity values in the reverse osmosis tests started at 10,300 and 6,270 umhos for Drums C and D, respectively. As the test progressed, electrical conductivity increased steadily until approximately 60 percent of the drum volume had been processed. At this point, Drum D displayed an electrical conductivity of about 10,000 umhos while Drum C's electrical conductivity was about 18,000 umhos. As the next 25 to 35 percent of the solution was processed, electrical conductivity increased dramatically until final conductivities of 37,000 umhos (Drum C) and 48,300 umhos (Drum D) were attained. The higher conductivity reached during the Drum D test was most likely a consequence of the higher pressures utilized in this test. The higher pressures enabled the reverse osmosis equipment to force more permeate across the membrane and concentrate the feed solution further.

Changes in electrical conductivity values associated with permeate samples collected in the reverse osmosis tests were very similar to those which occurred in the concentrate samples. Figure 16 and Table 5 indicate that permeate electrical conductivities started at 500 umhos (Drum C) and 242 umhos (Drum D), respectively. Permeate electrical conductivities increased steadily until about 70 percent of the drum volume was processed. Approximately 70 percent of the test volume had been processed at this point. Permeate conductivity increased dramatically as the next 15 to 25 percent of the rinse water volume was processed until final conductivities of 3720 umhos (Drum C) and 9190 umhos (Drum D) were reached. The higher conductivities associated with the Drum D test may again be attributed to the higher pressures utilized in this test. The higher pressures forced more salts to pass through the membrane into the permeate.

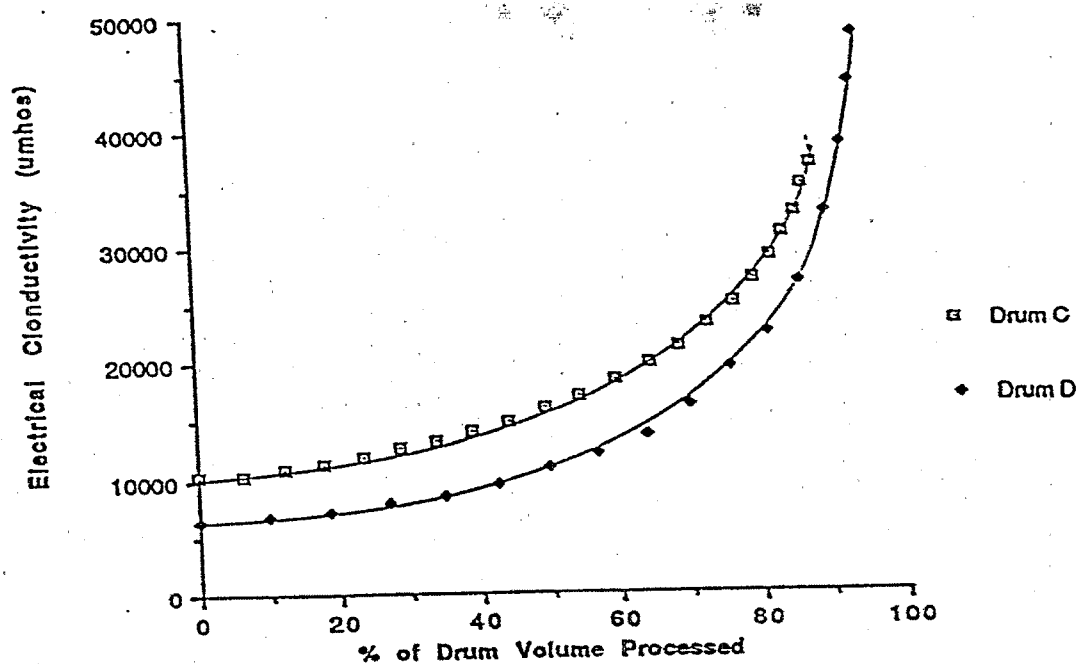


Figure 15. Concentrate electrical conductivity versus percent of drum volume processed; reverse osmosis tests.

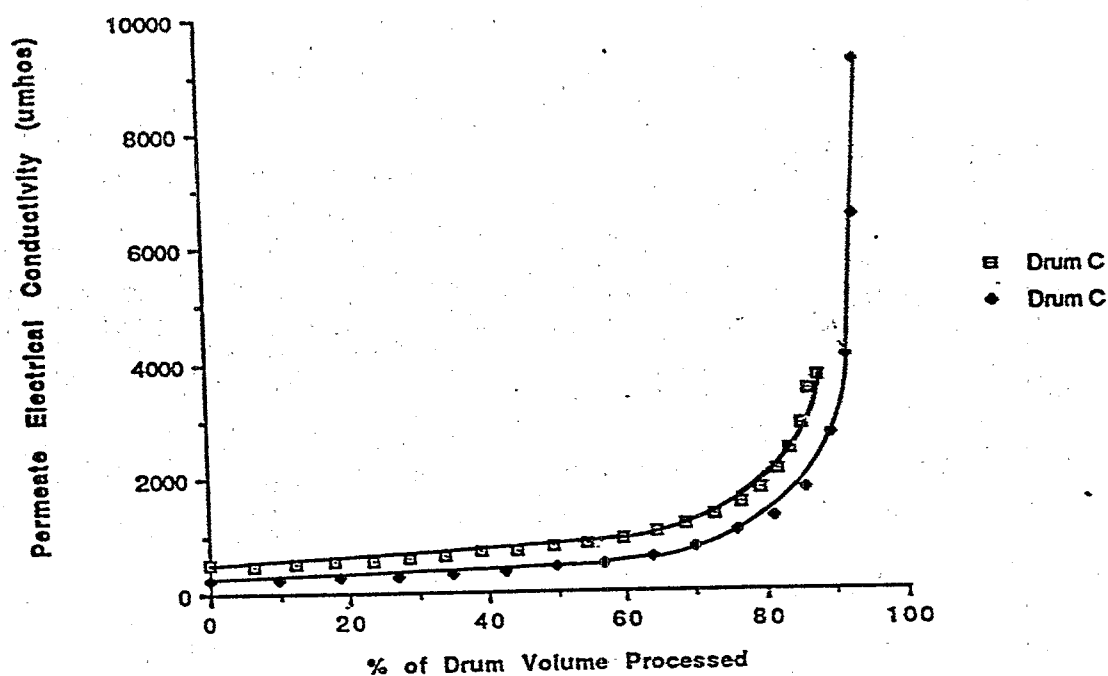


Figure 16. Permeate electrical conductivity versus percent of drum volume processed; reverse osmosis tests.

Table 6 provides a summary of electrical conductivities present in permeate and distillate samples. As shown, the low temperature evaporation tests produced distillate samples that averaged 22.94 (Drum A) and 25.24 (Drum B) umhos, respectively. The reverse osmosis tests produced permeate samples which averaged 1331 (Drum C) and 1794 (Drum D) umhos. Distillate samples collected during the low temperature evaporation tests exhibited electrical conductivities which averaged only 0.09 to 0.15 percent as high as the concentrate conductivities. Permeate samples from the reverse osmosis tests exhibited electrical conductivities which averaged 5.97 to 6.45 percent as high as the conductivities displayed in the concentrate samples suggesting that more salts can pass through the reverse osmosis membrane than the evaporation process.

Table 6. Average Electrical Conductivity Values in Distillate and Permeate

Drum	Distillate EC		Permeate EC	
	Mean (umhos)	STD (umhos)	Mean (umhos)	STD (umhos)
A (n=13)	22.94	5.69	—	—
B (n=16)	25.24	5.60	—	—
C (n=22)	—	—	1331	989
D (n=17)	—	—	1794	2539

## PARAMETER RELATIONSHIPS

In addition to examining the equipment productivity, nickel concentration, total organic carbon concentration, and electrical conductivity data on an individual basis, efforts were made to define relationships which exist between these parameters. These relationships can be utilized to monitor equipment performance such that adjustments can be made to operating procedures that will optimize efficiency. A summary of the relationships defined in this project is provided below.

In the reverse osmosis tests, permeate flux rates appeared to be directly impacted by nickel concentrations in both the concentrate and permeate streams. Figure 17 shows the relationship between permeate flux rate and concentrate nickel concentration while Figure 18 shows the relationship between permeate flux rate and permeate nickel concentration. As indicated on both graphs, flux rates fell relatively quickly in the beginning stages of the tests as nickel concentrations in concentrate and permeate increased. However, the rate of decline tapered off as higher concentrations were reached in the concentrate and permeate solutions.

Permeate flux rates also appear to be related to electrical conductivities of both the concentrate and permeate solutions. Figure 19 shows the relationship between permeate flux rates and concentrate electrical conductivity while Figure 20 shows the relationship between permeate flux rates and permeate electrical conductivity. These graphs show that flux rates decrease exponentially as the electrical conductivity of both the concentrate and permeate solutions increase.

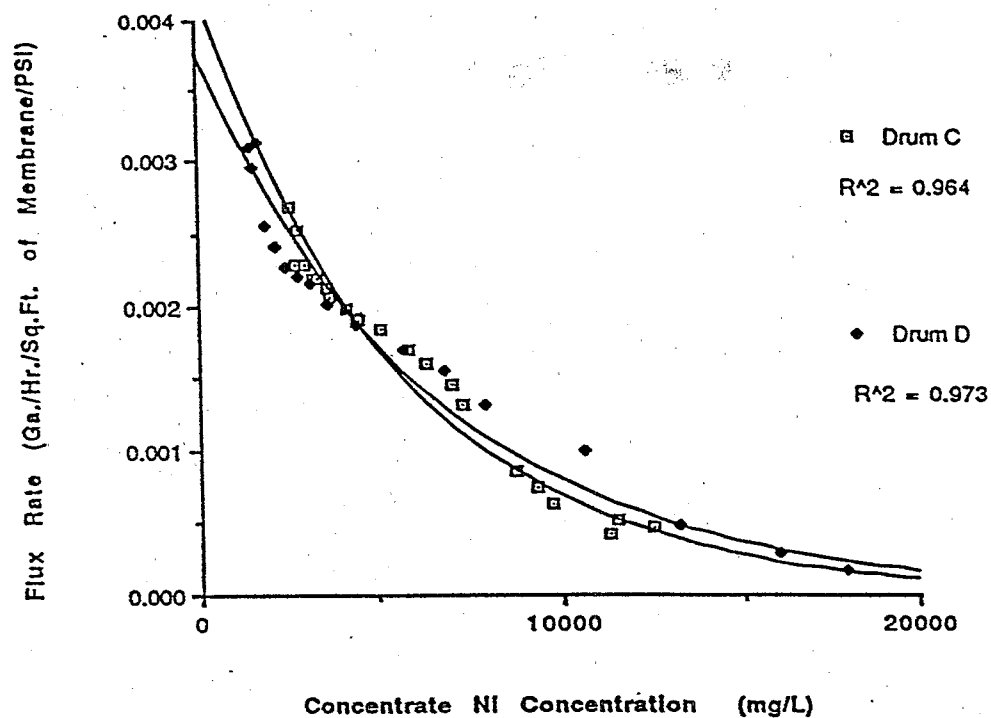


Figure 17. Permeate flux rate versus concentrate nickel concentration; reverse osmosis tests.

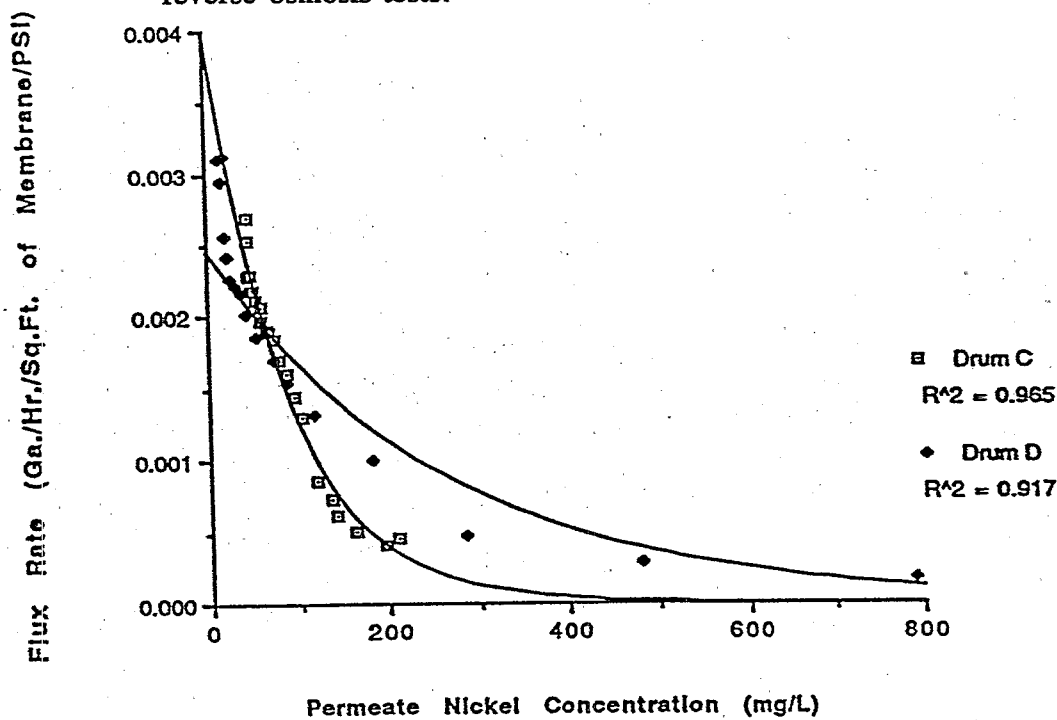


Figure 18. Permeate flux rate versus permeate nickel concentration; reverse osmosis tests.

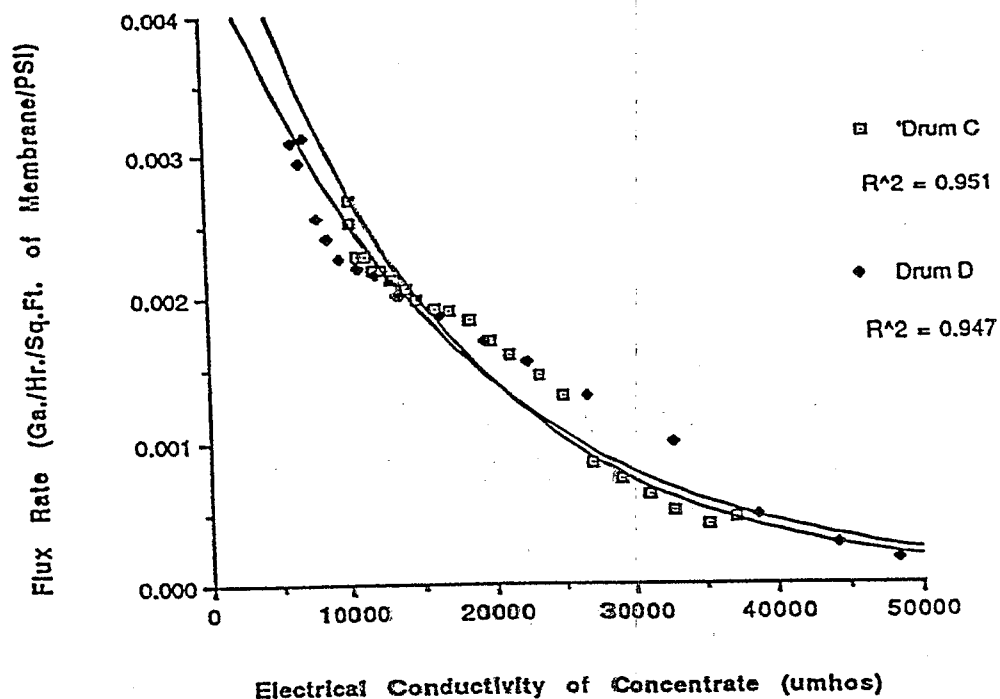


Figure 19. Permeate flux rate versus concentrate electrical conductivity; reverse osmosis tests.

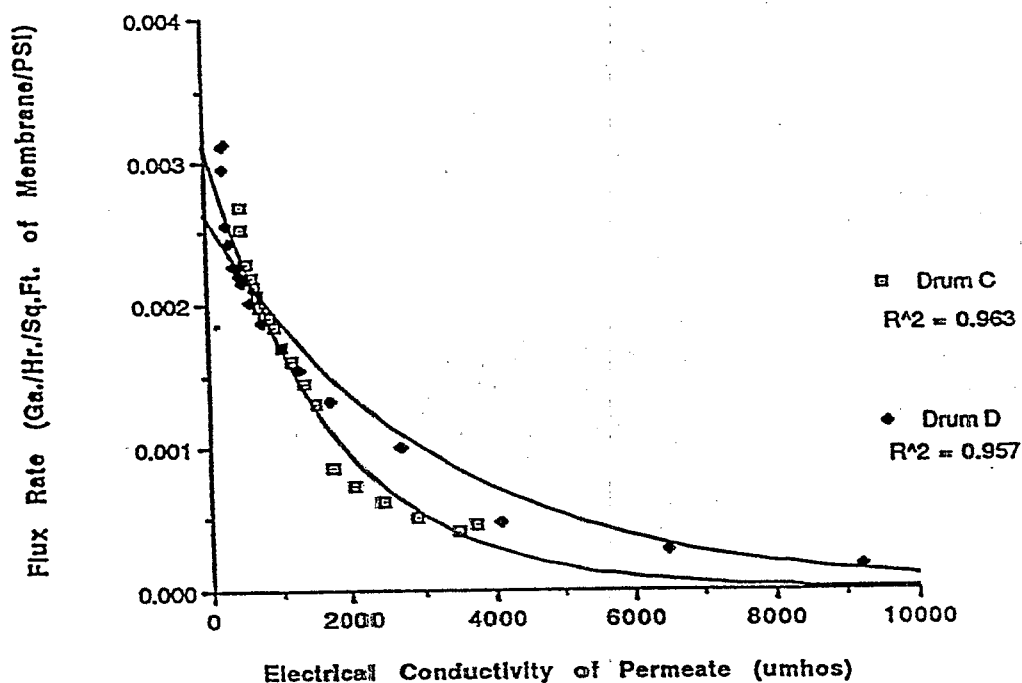


Figure 20. Permeate flux rate versus permeate electrical conductivity; reverse osmosis tests.



Electrical conductivity also proved to be a good indicator of the nickel concentrations present in the concentrate solutions produced by both the low temperature evaporation and reverse osmosis tests. Figure 21 shows that an exponential relationship existed between concentrate electrical conductivity and nickel concentrations. This relationship appears to be linear until concentrate electrical conductivities reach about 70,000 umhos. Then nickel concentrations increased exponentially as increases in electrical conductivity occurred. Figure 22 shows the relationship between the electrical conductivity of concentrate samples and their respective nickel concentrations for the reverse osmosis tests. As shown, this relationship appears to be very linear. However, it should be noted that the highest conductivities observed in these tests were in the 40,000 to 50,000 umhos range which is well below the point (70,000 umhos) where the concentrate electrical conductivities began increasing exponentially. Figure 23 shows the relationship between electrical conductivities associated with permeate samples and their respective nickel concentrations. This relationship appears to be similar to the one defined in Figure 22 for the concentrate samples. Permeate nickel concentrations increased linearly as the electrical conductivity levels increased.

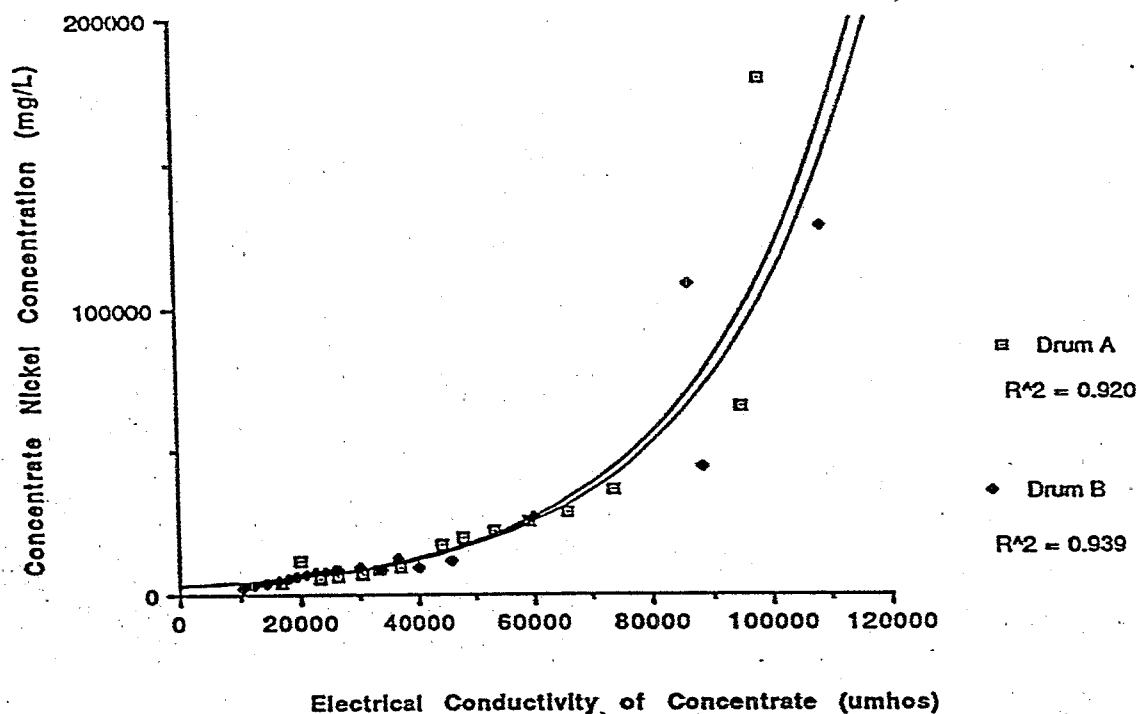


Figure 21. Concentrate nickel concentration versus electrical conductivity; low temperature evaporation tests.

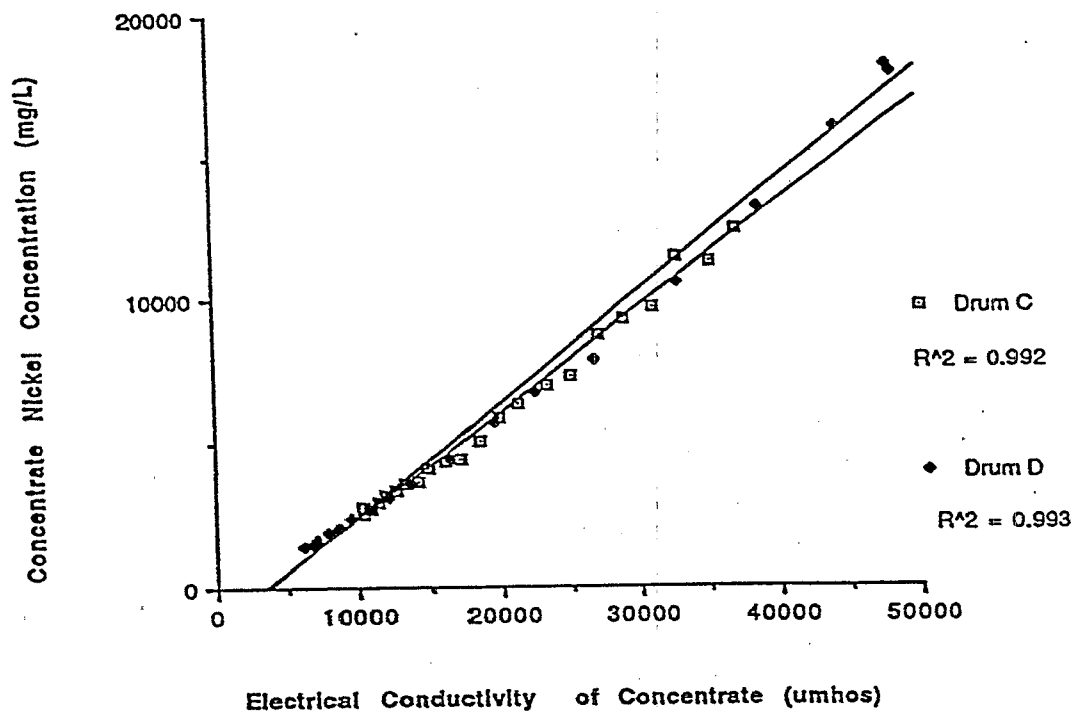


Figure 22.

Concentrate nickel concentration versus electrical conductivity;  
reverse osmosis tests.

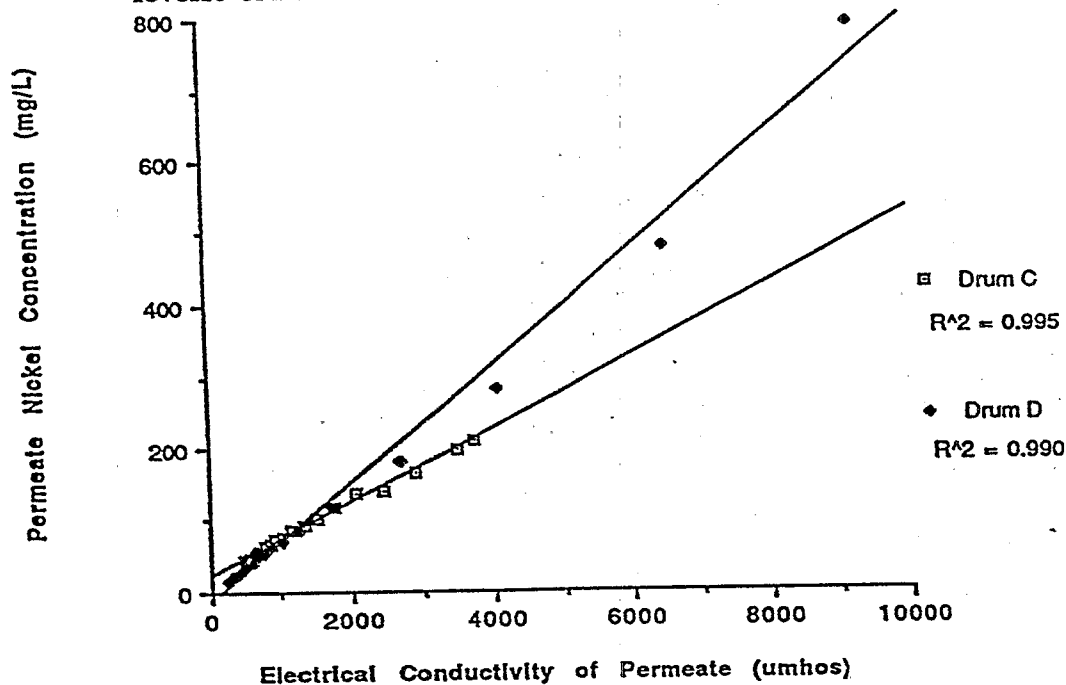


Figure 23.

Permeate nickel concentration versus permeate electrical  
conductivity; reverse osmosis tests.

Electrical conductivity data was also well correlated with total organic carbon concentrations in both the low temperature evaporation and reverse osmosis tests. Figure 24 shows the relationship between electrical conductivity of concentrate samples and corresponding total organic carbon levels. This relationship appears to be linear through electrical conductivity levels of approximately 50,000 umhos. At higher electrical conductivity levels, total organic carbon levels increased exponentially. Figure 25 shows a linear relationship between concentrate electrical conductivity and total organic carbon results in the reverse osmosis tests. It should be noted that the highest electrical conductivity values attained in the reverse osmosis tests were below the 50,000 umhos level where nickel concentrations began increasing exponentially in the low temperature evaporation tests.

The relationships described above will be very useful in developing operating standards for the low temperature evaporation and reverse osmosis systems. It is fortunate that electrical conductivity data are well correlated with the other parameters because conductivity measurements are relatively quick, simple, and inexpensive to obtain. Further, electrical conductivity instruments can be incorporated into operating systems to help provide process control.

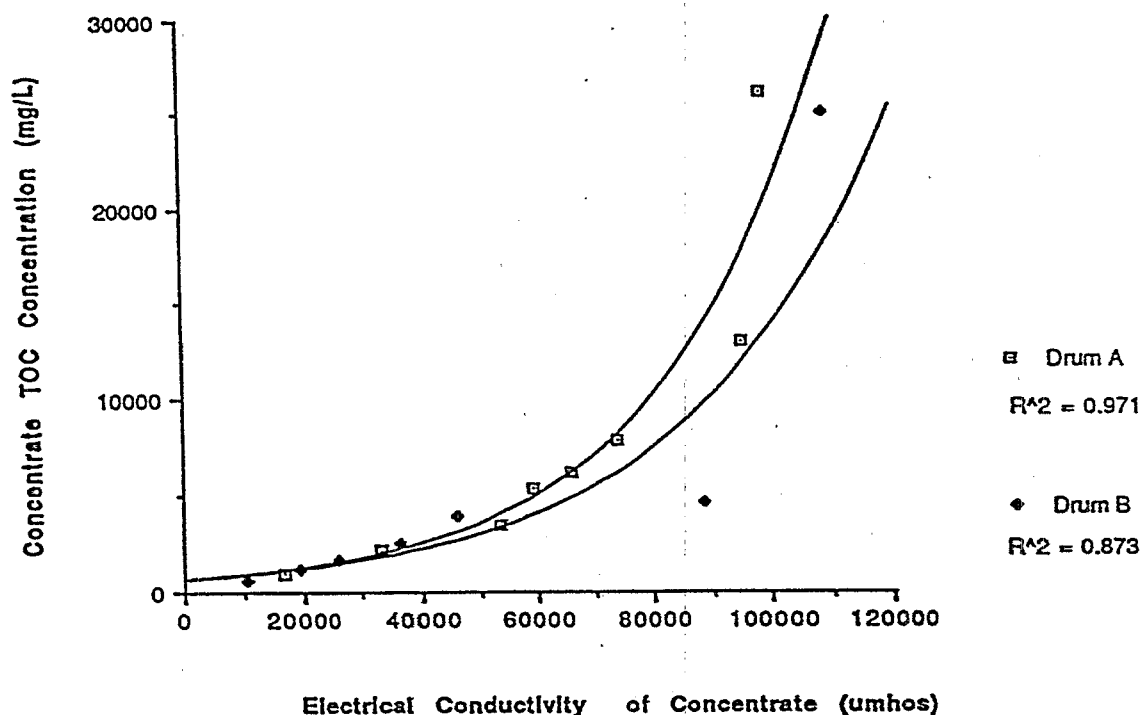


Figure 24. Concentrate TOC concentration versus electrical conductivity; low temperature evaporation tests.

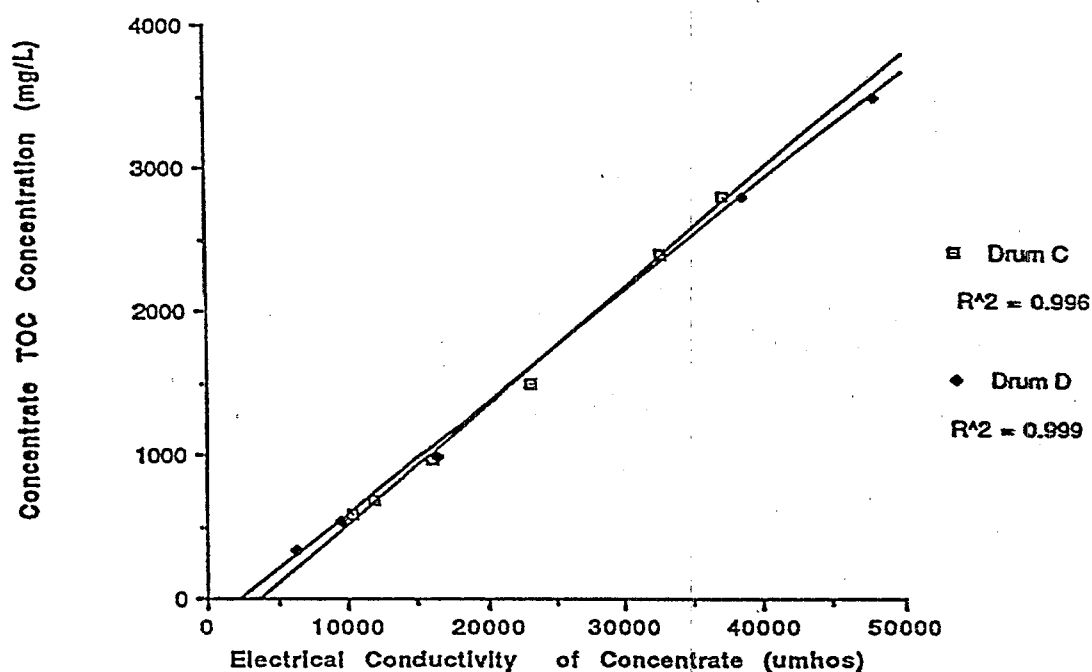


Figure 25. Concentrate TOC concentration versus electrical conductivity; reverse osmosis tests.

## SECTION 5

### ECONOMIC ANALYSIS

The costs and benefits associated with installing low temperature evaporation and/or reverse osmosis systems at the new Graham Plating facility were analyzed to determine the economic feasibility of these technologies. Assumptions regarding inflation rate, discount rate, federal tax rate, depreciation schedule, project life and various operating expenses were entered into a LOTUS spreadsheet (General Electric, 1987) which calculates a number of economic indices. The assumptions utilized in these calculations and their sources are presented in Table 7. Projected costs of future liabilities associated with hazardous waste generated from the Graham Plating facility were not included in this assessment due to the lack of an accurate means to assess these liabilities. Therefore, the economic indices presented below are most likely a conservative estimate of the monetary benefits that might be expected due to implementation of these technologies.

TABLE 7. ASSUMPTIONS FOR ECONOMIC CALCULATIONS

Item	Factor	Source
Inflation Rate	4%	Consumer Price Index
Discount Rate	7.72%	10 year treasury bill rate +0.5%
Federal Tax Rate	34%	General Electric, (1987)
Depreciation Schedule	7 years	General Electric, (1987)
Project Life	10 years	Osmonics and Licon
Power Costs	\$.10/kilowatt	Commonwealth Edison
Thermal Unit Costs	\$.40/100,000 BTU	Commonwealth Edison
Labor Costs	\$15/hour	Graham Plating
Salvage Value	10% of Capital Cost	Osmonics and Licon
Sludge Disposal Costs	\$7.27/gallon	Graham Plating
Water Costs	\$3/1,000 gallons	Arlington Heights Public Works
Reclaimed Ni Value	\$3.75/pound	Stutz Metal Finishing Products

In addition to the assumptions presented in Table 7, it was further assumed that all of the rinse water treated with the systems would be produced from a nickel electroplating line. Under actual plant conditions, rinse water from other electroplating lines would be processed with the equipment. However, since nickel rinse water was the only feed solution tested in this project

it was not appropriate to make assumptions regarding equipment performance with respect to other metals. It was also assumed that all equipment evaluated in the economic assessments would operate 24 hours per day, 5 days per week at only 80% of its capacity (5,760 gallons per day). This estimate takes into account down time associated with maintenance activities and production fluctuations.

These assumptions along with information collected in the low temperature evaporation and reverse osmosis tests were used to prepare the economic analysis on three alternative process systems that could be implemented at the new Graham Plating facility. The alternatives examined include: 1) a low temperature evaporator system, 2) a reverse osmosis system, and 3) a combined system which would utilize both technologies. Detailed cost/benefit assessments of these alternatives are provided below.

The low temperature evaporation economic assessment was based on the anticipated utilization of the Licon evaporator purchased for use at the new Graham Plating facility. This unit is capable of processing up to 7,200 gallons of electroplating rinse water per day. Table 8 provides a cash flow summary associated with implementation of the evaporator system. As shown, this unit requires a significant capital investment of \$140,000. Additionally, the evaporator requires considerable energy input (5,000 BTU per gallon of solution processed) which would result in energy costs of just under \$30,000 per year (based on 1992 costs) or \$20 per 1,000 gallons. Replacement of pump seals, miscellaneous repairs and equipment monitoring activities would cost about \$3,060 per year. Replacement of the various pumps utilized on this equipment would take place twice during the project life at the projected costs displayed in Table 8.

Considerable savings could be expected through utilization of the evaporation system to reclaim nickel salts from the rinse water. It was estimated that over 12 thousand pounds of nickel could be salvaged from the nickel rinse water in a year's time through utilization of the evaporation system. Based on 1992 dollars, these salts would have an approximate value of \$36,660 per year as compared to the cost of buying an equivalent amount of new plating chips. Additional savings would be realized through reduction in water utilization (\$4492 per year) and sludge generation and disposal (\$19,200 per year).

Based on the parameters described above, the LOTUS spreadsheet was utilized to calculate the economic indices presented in Table 9. As shown, the economics associated with installing a Low Temperature Evaporation system at the Graham Plating facility are relatively favorable. It is estimated that the payback period associated with this technology would require about 6.9 years. An implied rate of return of about 10.6% would be realized while the net present value would be \$54,017.

**TABLE 8: LOW TEMPERATURE EVAPORATION CASH FLOW SUMMARY**  
**[Dollar Amounts Before Taxes and Depreciation]**

YEAR	1 1993	2 1994	3 1995	4 1996	5 1997	6 1998	7 1999	8 2000	9 2001	10 2002
<b>CAPITAL EXPENDITURES</b>										
Con Double Effect Evaporator	140000									
<b>OPERATING EXPENDITURES</b>										
Energy Consumption (7.5 billion BTU/year)	29952	31150	32396	33692	35040	36441	37899	39415	40991	42631
Equipment Maintenance*										
Replace Concentrate Pump				450				526		
Replace Recirculation Pump				787				921		
Replace Distillate Pump				787				921		
Replace Pump Seals	1000	1040	1082	1125	1170	1217	1265	1316	1369	1423
Miscellaneous Repairs*	500	520	541	562	585	608	633	658	684	712
Monitor Equipment*	1560	1622	1687	1755	1825	1898	1974	2053	2135	2220
<b>TOTAL EXPENDITURES</b>	173012	34332	35706	39159	38619	40164	41771	45810	45179	46986
<b>SAVINGS</b>										
Reclaimed Nickel 9776 lbs./year @ \$3.75 per lb.	36660	38126	39651	41238	42887	44602	46387	48242	50172	52179
Water Consumption 1497000 ga./year @ \$3/1000 ga.	4492	4672	4859	5053	5255	5465	5684	5911	6148	6394
Waste Disposal 2640 ga./yr. sludge @ \$7.27/ga.	19200	19968	20767	21597	22461	23360	24294	25266	26277	27328
Equipment Salvage										14000
<b>TOTAL SAVINGS</b>	60352	62766	65277	67888	70603	73427	76365	79419	82596	99900
<b>CASH FLOW</b>	-112660	28433	29570	28729	31983	33263	34593	33608	37416	52913

\* Prices include labor

TABLE 9. ECONOMIC SUMMARY - LOW TEMPERATURE EVAPORATION OPTION

Capital Invested	\$140,000
Payback Period	6.9 years
Net Present Value (NPV)	\$54,017
Implied Rate of Return (IRR)	10.6%

The economic assessment for the reverse osmosis system was based on the utilization of an Osmonics unit equipped with 12, 4 inch diameter membrane cartridges and a ten horse power motor. Based on results of the reverse osmosis tests and manufacturers recommendations, this unit should be capable of processing rinse water volumes of about seven to eight thousand gallons per day. This estimate takes into account the low processing rates that can be expected as the concentrate solution is condensed to the maximum concentrations attainable with this technology. A cash flow summary explaining the costs and benefits associated with installing and operating this unit is provided in Table 10.

Capital costs required to install this unit would be approximately \$50,000. The membrane cartridges for this unit would have to be replaced every other year at a cost of \$800 each (1992 dollars). The reverse osmosis unit would require about \$3,724 annually (1992 dollars) in electrical power costs or \$2.50 per 1,000 gallons. Other expenses required to operate this unit would include costs associated with prefilters, cleaning the membranes, and monitoring the equipment which would cost approximately \$4608 per year (1992 dollars).

Based on the results of the reverse osmosis tests conducted in this project, the unit could only concentrate the nickel solution to a concentration of about 1.2 to 1.8 percent nickel. This concentration is well below the eight percent nickel concentration normally utilized in the Graham Plating operation's plating baths. Therefore, the concentrate solution produced through the reverse osmosis process could only be placed in the plating baths to replace water losses. It is estimated that about 300 gallons of concentrate containing approximately 1.5% nickel would be produced daily from the reverse osmosis process. This volume would be well beyond that which could be used to replace water in the plating baths. For the purposes of economic calculations, it was assumed that 3/4 of this concentrate (about 58,656 gallons per year) could be used to replace plating bath water losses. This figure probably greatly exceeds that which could reasonably be used in the plating operation but it is used in this economic assessment to provide a "best case" scenario. The remaining 1/4 of the concentrate solution (about 19,552 gallons per year) which could not be utilized to replace plating bath water losses would have to be treated further by precipitation and shipped off site to a facility that conducts metal reclaiming operations at a net cost of just over \$45,000 per year.

Substantial savings would be realized as a result of installing a reverse osmosis system at the Graham Plating facility. Approximately \$27,000 (1992 dollars) per year in savings would be realized through reclamation of nickel salts from the processed rinse water (as compared to having to purchase fresh salts). An additional \$4492 (1992 dollars) per year could be anticipated



**TABLE 10: REVERSE OSMOSIS CASH FLOW SUMMARY**  
**[Dollar Amounts Before Taxes and Depreciation]**

YEAR	1 1993	2 1994	3 1995	4 1996	5 1997	6 1998	7 1999	8 2000	9 2001	10 2002
<b>CAPITAL EXPENDITURES</b>										
Osmonics 12 membrane system	50000									
<b>OPERATING EXPENDITURES</b>										
Energy Consumption (37,240 kw/year)	3724	3873	4028	4189	4357	4531	4712	4901	5097	5300
Equipment Maintenance *										
Replace Membranes		9984		10799		11680		12633		13664
Replace pre-filters	2028	2109	2193	2281	2372	2467	2566	2669	2775	2886
Clean Membranes	1020	1061	1103	1147	1193	1241	1291	1342	1396	1452
Monitor Equipment	1560	1622	1687	1755	1825	1898	1974	2053	2135	2220
Replace Pump						6083				
Waste Disposal** 364 drums/year @ \$110/drum. + \$5000 shipping	45040	46842	48715	50664	52690	54798	56990	59270	61640	64106
<b>TOTAL EXPENDITURES</b>	<b>103372</b>	<b>65491</b>	<b>57727</b>	<b>70835</b>	<b>62438</b>	<b>82698</b>	<b>67533</b>	<b>82867</b>	<b>73043</b>	<b>89629</b>
<b>SAVINGS</b>										
Reclaimed Nickel 7223 lbs./year @ \$3.75 per lb.	27084	28167	29294	30466	31684	32952	34270	35641	37066	38549
Water Consumption 1497000 ga./year @ \$3/1000 ga.	4492	4672	4859	5053	5255	5465	5684	5911	6148	6394
Waste Disposal 2640 ga./yr. sludge @ \$7.27/ga.	19200	19968	20767	21597	22461	23360	24294	25266	26277	27328
Equipment Salvage										5000
<b>TOTAL SAVINGS</b>	<b>50776</b>	<b>52807</b>	<b>54919</b>	<b>57116</b>	<b>59401</b>	<b>61777</b>	<b>64248</b>	<b>66818</b>	<b>69490</b>	<b>77270</b>
<b>CASH FLOW</b>	<b>-52596</b>	<b>-12684</b>	<b>-2808</b>	<b>-13719</b>	<b>-3037</b>	<b>-20922</b>	<b>-3285</b>	<b>-16049</b>	<b>-3553</b>	<b>-12359</b>

\* Prices include labor

\*\* Assumes 3/4 of concentrated nickel solution can be used in plating  
while other 1/4 must be disposed of bath makeup

through reduction in water utilization. Waste disposal costs associated with electroplating sludge riddance would be reduced by about \$19,200 per year.

The economic indices (payback period, net present value, and implied rate of return) associated with the reverse osmosis system (Table 11) show that for this operation, the reverse osmosis system is not an economically viable option. The primary factor contributing to the unfavorable economic conditions associated with this system can be traced directly to the costs required for disposal of the excess nickel concentrate. If all of this solution could be recycled into the electroplating process, the economics associated with the reverse osmosis option would be much more favorable. It is possible that other companies could recycle all of the concentrate solution from the reverse osmosis process. An economic assessment performed on the feasibility of a reverse osmosis system installed at such a facility would probably suggest that the reverse osmosis technology is acceptable and may be the most beneficial of all rinse water recycling options.

TABLE 11. ECONOMIC SUMMARY - REVERSE OSMOSIS OPTION

Capital Invested	\$50,000
Payback Period	Never
Net Present Value (NPV)	(\$78,317)
Implied Rate of Return (IRR)	(9.8%)

The economic assessment for the combined technologies option was based on the utilization of an Osmonics reverse osmosis unit equipped with six membrane cartridges and a Licon double effect evaporator. Based on results from the reverse osmosis tests and manufacturers recommendations, the reverse osmosis unit would be capable of processing 5,500 to 6,000 gallons per day. Although this unit would be equipped with only six membranes, the individual membranes would be more productive than the membranes placed in the unit specified in the reverse osmosis system. The increased productivity could be attributed to the fact that the reverse osmosis system used in the combined technologies option would only be utilized on feed solution containing less than 4,000 mg/L of nickel. At these concentrations, the membranes would not foul as rapidly resulting in higher productivity and less down time for membrane cleaning. The evaporator specified for the combined technology option would be a Licon double effect evaporator capable of processing about 2,000 gallons of rinse water per day.

The reverse osmosis system would be used to concentrate about 80 percent of the rinse water volume from an initial concentration of about 784 mg/L nickel to about 4,000 mg/L nickel. The solution concentrated by the reverse osmosis system would then be transferred to the low temperature evaporation system. The evaporator would concentrate the remaining 20 percent of the rinse water solution to a concentration of about eight percent nickel which could subsequently be replaced in the plating bath. Using the equipment within its optimum operating ranges would augment the ability of the systems to process the rinse water with maximum efficiency while supplying the electroplating operation with high quality concentrate, distillate, and permeate solutions for reuse. Since the equipment would always be functioning within

**TABLE 12: COMBINED SYSTEM CASH FLOW SUMMARY**  
**[Dollar Amounts Before Taxes and Depreciation]**

YEAR	1 1993	2 1994	3 1995	4 1996	5 1997	6 1998	7 1999	8 2000	9 2001	10 2002
<b>CAPITAL EXPENDITURES</b>										
Uicon Double Effect Evaporator	85000									
Osmonics 6 Membrane System	30000									
<b>OPERATING EXPENDITURES</b>										
<b>LOW TEMPERATURE EVAPORATION SYSTEM</b>										
Energy Consumption (1.5 billion BTU/year)	5990	6230	6479	6738	7007	7288	7579	7882	8198	8528
Equipment Maintenance*										
Replace Concentrate Pump				394				481		
Replace Recirculation Pump				675				790		
Replace Distillate Pump				675				790		
Replace Pump Seals	800	832	865	900	936	973	1012	1053	1095	1139
Miscellaneous Repairs	400	416	433	450	468	487	506	526	547	569
<b>REVERSE OSMOSIS SYSTEM</b>										
Energy Consumption [37,240 kw/year]	3724	3873	4028	4189	4357	4531	4712	4901	5097	5300
Equipment Maintenance*										
Replace Membranes		4992		5399		5840		6316		6832
Replace pre-filters	806	838	872	907	943	981	1020	1061	1103	1147
Clean Membranes	460	478	498	517	538	560	582	605	630	655
Replace Pump						4867				
<b>BOTH SYSTEMS</b>										
Monitor Equipment	1560	1622	1687	1755	1825	1898	1974	2053	2135	2220
<b>TOTAL EXPENDITURES</b>	<b>128740</b>	<b>19282</b>	<b>14861</b>	<b>22599</b>	<b>16074</b>	<b>27423</b>	<b>17385</b>	<b>26437</b>	<b>18804</b>	<b>26388</b>
<b>SAVINGS</b>										
Reclaimed Nickel 9651 lbs./year @ \$3.75 per lb.	36191	37639	39144	40710	42338	44032	45793	47625	49530	51511
Water Consumption 1497000 ga./year @ \$3/1000 ga.	4492	4672	4859	5053	5255	5465	5684	5911	6148	6394
Waste Disposal 19200 ga./yr. sludge @ \$1/ga.	19200	19968	20767	21597	22461	23360	24294	25266	26277	27326
Equipment Salvage										11500
<b>TOTAL SAVINGS</b>	<b>59883</b>	<b>62278</b>	<b>64769</b>	<b>67360</b>	<b>70055</b>	<b>72857</b>	<b>75771</b>	<b>78802</b>	<b>81954</b>	<b>96732</b>
<b>CASH FLOW</b>	<b>-68857</b>	<b>42996.72</b>	<b>49908.26</b>	<b>44761.71</b>	<b>53980.78</b>	<b>45433.46</b>	<b>58385.61</b>	<b>52364.87</b>	<b>63149.88</b>	<b>70343.98</b>

\*Prices include labor

optimum concentration ranges, smaller reverse osmosis and low temperature evaporation units could be implemented than if the individual units were used alone.

Table 12 provides a cash flow summary for the combined technologies option. As shown, a capital investment of \$115,000 would be required to install the two systems described above into the new Graham Plating facility. Energy costs to operate the two systems would require about \$9714 (1992 dollars) per year. Costs for replacing pre-filters and cleaning membranes associated with the reverse osmosis unit would be about \$1,266 per year. Membranes for the reverse osmosis unit would have to be replaced every other year at a cost of about \$4,800 (1992 dollars). Pumps for the reverse osmosis and evaporator units would have to be replaced at various times during the projected life of the equipment at the approximate costs projected on Table 12. Additionally, about \$2,760 (1992 dollars) would have to be spent on a yearly basis to replace pump seals, perform miscellaneous repairs and monitor equipment performance.

The economic indices calculated for the combined system are presented in Table 13. As shown, this option offers a net present value of \$177,057, an implied rate of return of 27.6 percent, and would payback the capital investment in about 2.8 years. Based on the assumptions and conditions used in this assessment, the combined option appears to offer the best economic alternative for processing the electroplating rinse water. The superior indices produced by this alternative may be attributed to the fact that the two technologies proposed in this option would be utilized under conditions for which the equipment is best suited. The reverse osmosis equipment is more efficient at processing the dilute (less than 4,000 mg/L) solution while the evaporator is more efficient at processing rinse water containing nickel in concentrations in excess of 4,000 mg/L.

TABLE 13. ECONOMIC SUMMARY - COMBINED TECHNOLOGY OPTION

Capital Invested	\$115,000
Payback Period	2.8 years
Net Present Value (NPV)	\$177,057
Implied Rate of Return (IRR)	27.6%

## SECTION 6

### CONCLUSIONS AND RECOMMENDATIONS

#### SYSTEM EFFICIENCY

The low temperature evaporation system exhibited consistent productivity throughout the tests. This performance feature was unfailing regardless of the chemical concentrations of the feed solution provided to the system. In addition to the steady production rate of the low temperature evaporation system, it was capable of concentrating the rinse water feed solution to nickel levels as high as 13 to 18 percent which are well above the 8 percent required for return to the plating bath. The low temperature evaporation system concentrated the organic constituents of the rinse water to total organic carbon levels of 25,000 to 26,000 mg/L. The concentration rate of the organic components paralleled the nickel concentration rate suggesting that few organic materials were lost to volatilization. Characterization and quantitation of the individual organic constituents was not performed in this project and should be a consideration in future investigations. Prior to reuse of the concentrate in a plating bath, these organics may need to be removed to prevent buildup and adverse affects on plating quality.

The quality of the cleaned rinse water is of great importance to electroplating shops, particularly if it is to be reused in the electroplating process. Distillate produced by the low temperature evaporation system was very low in both nickel concentration (average 0.31 to 0.71 mg/L) and TOC concentration (average 3.04 to 3.50 mg/L). This water is of good enough quality that it could be both reused within the electroplating facility or discharged to a POTW.

Disadvantages of the low temperature evaporation system include its relatively high (\$140,000) capital cost and high energy requirements (\$20 per 1,000 gallons processed). The implied rate of return of 10.6 percent determined in the economic assessment for the low temperature evaporation system suggests that it is a marginal investment opportunity by today's standards. However, these estimates do not take into account the future liabilities that would be minimized as the result of reducing the quantity and type of hazardous waste discharges from the facility.

The reverse osmosis system exhibited superior productivity at the beginning of the tests and productivity dropped off dramatically after about 60 percent of the feed solution had been processed. At this point, feed solution nickel concentrations were approximately 4,000 to 5,000 mg/L. Beyond these levels, the productivity of the reverse osmosis equipment decreased dramatically as solids began to precipitate and foul the membrane. The reverse osmosis system

was capable of concentrating the feed solution to nickel concentrations of 12,560 to 18,200 mg/L. These concentrations are well below the 8 percent nickel concentration required for the plating bath. Some of this solution could be used to replace water losses in the electroplating process. However, it is likely that the reverse osmosis system would produce too much concentrated rinse water containing 1.2 to 1.8 percent nickel. This material would have to be further processed by using an alternative technology such as low temperature evaporation or shipped to a facility that could extract the nickel for utilization in other industrial processes.

The reverse osmosis system concentrated the organic constituents present in the rinse water to levels of 2,800 to 3,500 mg/L. These concentrations suggest that the organic bath constituents are concentrated by the reverse osmosis equipment at rates that parallel the nickel concentration rates. No information was obtained in these tests regarding the quality and nature of the organic bath constituents. However, the low operating temperatures utilized by the reverse osmosis equipment (74 to 80 degrees F) should have prevented degradation of the organic molecules.

The quality of the cleaned rinse water produced by the reverse osmosis equipment was directly related to the quality of the feed solution pumped into the unit. Permeate produced by the reverse osmosis system averaged 89 to 134 mg/L nickel. These concentrations are acceptable for reuse as rinse water. However, this solution would not be acceptable for discharge to POTWs. It should be noted that the permeate quality could be significantly improved if the reverse osmosis system was utilized only on rinse water feed solution with lower (4,000 to 5,000 mg/L or less) nickel concentrations. TOC concentrations averaged 19.46 to 21.98 mg/L in the permeate solution suggesting that some of the organic compounds are able to permeate through the membrane. It is not known if some of the compounds have a greater tendency to permeate the membrane than others and this possibility should be investigated in future studies. Advantages of the reverse osmosis system include its relatively high production rates with respect to low concentration (less than 4,000 mg/L) feed solutions. Additionally, it requires lower capital investment (about \$50,000) than a comparably sized low temperature evaporation system. Energy costs to operate a reverse osmosis system would require only about \$2.50 per 1,000 gallons processed.

Disadvantages associated with a reverse osmosis system include its inability to concentrate the feed solution to levels beyond the 12,560 to 18,200 mg/L levels revealed in this study. This factor alone would prevent utilization of a stand alone reverse osmosis system at the Graham Plating facility due to the impracticalities associated with utilization of the concentrate produced by the system. Another disadvantage associated with the reverse osmosis system is the lower quality permeate produced by the system. This solution would most likely have to be reused within the plant and could not be discharged to the POTW.

Both the low temperature evaporation and reverse osmosis systems appear to offer advantages under specific operating conditions. The low temperature evaporation system appears to be best adapted to processing solutions with relatively high nickel concentrations. It can process these solutions such that concentrate solution comprised of eight percent or more nickel is produced along with a very high quality distillate solution. The reverse osmosis system is best

adapted to conditions where the feed solution is of relatively low nickel concentration. It can process the low concentration feed solution with relatively high efficiency to a level of about 4,000 to 5,000 mg/L. At this point the solution could be transferred to a low temperature evaporator or other acceptable process for further concentration. Utilizing the equipment within its optimum operating ranges would augment the ability of the systems to process the rinse water with maximum efficiency while supplying the electroplating operation with high quality concentrate, distillate, and permeate solutions for reuse. This relationship is consistent with the combined technology economic assessment provided above in which the two systems would be utilized in tandem. The implied rate of return of 27.6 percent associated with this assessment is the most favorable of the economic scenarios examined due to the projected utilization of the equipment under its most favorable conditions.

Electrical conductivity measurements taken during operation of both the low temperature evaporation and reverse osmosis systems could be of great value during actual plant operating conditions. The electrical conductivity data obtained in this project was well correlated with nickel concentration, TOC concentration, and membrane flux characteristics. Accurate assumptions regarding concentrate, permeate, and distillate quality could be based on electrical conductivity measurements taken throughout the work day. Further, the equipment could be automated to accumulate and discharge the various solutions based on in process electrical conductivity measurements that could activate pumps, valves, and/or switches when preset levels are attained.

## RECOMMENDATIONS

This study analyzes the performance of the two technologies with respect to processing rinse water from a nickel electroplating process. Additional tests utilizing rinse water from other electroplating lines using other metals should be performed to determine these technologies' usefulness with respect to processing the entire spectrum of rinse water streams that would be produced at a full-scale electroplating operation. Detailed analysis of all organic and inorganic rinse water components (organic brighteners, sulfate, chloride, etc.) would be useful in future studies to determine the effects of low temperature evaporation and reverse osmosis processing on the relative quality and quantity of these constituents.

When Graham Plating implements the low temperature evaporation system in their new facility, on site testing should be performed to allow comparison of this full-scale system with the pilot-scale tests performed in this study. Detailed study of the performance of the concentrated rinse water that is returned to the plating bath should be performed. Efforts should be made to determine the effects of the recycled plating chemicals on plating quality and bath longevity. The information obtained from this study is based on very short term observations. Therefore, information should also be obtained regarding the long term performance of the full-scale system at the Graham Plating facility.

Evaluations of other technology options such as ion exchange and electrowinning should be performed in studies similar to those described in this project to provide a comparison with respect to all alternatives currently available. This project has provided a good foundation for

evaluating options for processing electroplating rinse waters. Execution of the additional studies recommended above would provide an accurate assessment of all of the available alternatives and their respective potential regarding the processing of electroplating rinse water.



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## APPENDIX

### QUALITY ASSURANCE

The quality assurance project plan submitted for this project (Miller, 1991) was written to validate the evaluation of a full-scale low temperature evaporation system at the new Graham Plating facility. However, Graham Plating has not been able to relocate their electroplating operations to their new facility. Therefore, evaluation of the full-scale low temperature evaporation process was not possible. An alternative study plan was developed in cooperation with Graham Plating and the U.S. Environmental Protection Agency project officer to evaluate both low temperature evaporation and reverse osmosis technologies on a pilot scale. The focus of this project was modified to evaluate these technologies with respect to their capabilities for processing electroplating rinse water produced from a nickel electroplating operation. Specifically, the tests were structured to examine the fate of nickel, total organic carbon, and electrical conductivity as the solutions were processed and concentrated through the low temperature evaporation and reverse osmosis systems.

Although significant modifications were made with respect to the project location and scope, great efforts were made to ensure that the quality assurance objectives established for this project were not compromised. The specifications outlined in the quality assurance project plan were followed with respect to sampling procedures, analytical procedures, instrument calibration, internal quality control checks, performance audits, and data reduction, calculation, validation and reporting. Compliance with these quality assurance objectives has resulted in high quality data validating the success of the two test methods. This simulation will have broad applicability to numerous electroplating firms.

### NICKEL ANALYSIS CALIBRATION

A five point calibration was used for all analyses performed by this technique, in accordance with the manufacturer's operating/procedures manual. The instrument software automatically processes the standards information and prepares a standard curve consistent with the data. Standard curves were manually plotted and checked on a recurring basis to ensure the accuracy of the computer algorithm, and to verify that the software was operating properly. An operating range was selected to ensure that the standard curve still had sufficient slope on the high end to facilitate unambiguous differentiation between sample concentrations of interest. Calibration curves were compared between analytical runs to verify proper standards preparation and consistent instrument performance.

## TOTAL ORGANIC CARBON ANALYSIS CALIBRATION

The Dohrmann instrument was calibrated with a single standard of potassium hydrogen phthalate in accordance with the manufacturer's operating/procedures manual. The calibration standard, usually 1000 mg-C/L, was chosen to be higher in concentration than the highest expected sample. Calibration was verified at approximately every 10th sample by rerunning the standard.

## MEASURES OF ACCURACY AND PRECISION

Results of precision and accuracy determinations for nickel and TOC are included in the accompanying table.

## NICKEL ANALYSIS

The wastewaters from the nickel plating process contained initially high levels (4,000 mg/L) of nickel and these levels were increased significantly in the concentration processes. By contrast, nickel levels in the treated water were much lower, ranging from 0.1 to about 100 mg/L. As a consequence, samples were run under several different wavelength conditions on the AAS, trying to match the analytical range to the sample concentrations to minimize the dilution factors. This resulted in a number of different check standards, one set for each concentration range. Check standards were monitored at approximately every 10 sample analyses, and were compared for consistency within the run and between runs. Drift in excess of 10% in check standard concentrations during the course of a run, which was occasionally observed, was used to reject data sets or parts thereof. A chart of check standard responses through a period in July, 1992, when the instrument was calibrated between 10 and 50 mg/L, is attached. As a matter of course, samples that were above or below the range of standards were rerun at a different dilution or wavelength.

On average, one duplicate and one spike analysis was run with each 10 samples analyzed. Duplicates were invariably within 5% relative difference throughout the project. Differences as large as 10% would not have compromised the experimental objectives of the project because of the distinct differences in treated water versus concentrates. So duplicate results strongly supported the appropriateness of the nickel data for evaluating project results.

Spike results also were satisfactory for meeting project needs. Most spike recoveries fell between 90-100%, with a few values in the 80% range. In a few cases, spike values fell significantly below 80%, suggesting errors in preparation or spike levels inappropriate to sample concentrations. These spikes were prepared over and rerun to verify accurate performance of the instrument.

Calibration standards were also routinely inserted into sample sets to serve as checks on instrument performance during the course of a run. One standard was inserted for each 10 samples analyzed; the concentration of the standard was varied so that in our standard 30 sample runs, three different standards were routinely checked. Instrument responses to these standards

were used in conjunction with check standard data to indicate drift or other problems with instrument performance.

Numerous samples were rerun during the course of the several months of analytical support provided this project. Reruns were performed to ensure comparability of results over time and to check on results that were out of range or suspect due to problems with check standards or spikes. These reruns regularly confirmed the validity of the original determinations, often to within 1% relative difference.

Specific conductivity was measured at the point of sample collection and was used as a control parameter in the experimental procedure. These data were used to guide analytical decisions on concentration range, dilution, and spike levels. They were also used as a final validation of the nickel data, since the relationship between specific conductance and nickel concentration was shown to be consistent within a sample set. In a few instances, deviations from this relationship were used to identify samples for reanalysis. Such reanalysis often revealed problems in the initial concentration measurement and justified adjusting reported concentration values.

#### TOTAL ORGANIC CARBON

Duplicate samples and reruns (on subsequent dates) were used to verify accuracy of TOC results. Only selected samples were analyzed for TOC, to give indications of trends in organic carbon concentration through an experimental run. The range of concentrations was large, 1 to 26,000 mg/L, and to some degree, paralleled the nickel results. The duplicates and reruns strongly supported the reproducibility of the instrumental measurement, and calibration checks suggested the results were accurate. Comparisons to changes in nickel and conductivity data also served as an independent check on TOC values.

A duplicate and spike was prepared for the set of five samples run for chloride and sulfate determination. Percent differences between duplicates was 1% or less and spike recoveries ranged from 99 to 104% for these analyses.

#### REFERENCE STANDARDS

The laboratory did not participate in any external performance evaluation (PE) for nickel analysis by AAS during the course of this project. The laboratory is involved in a quarterly PE exercise with the USGS, but the low metals concentrations in these PE samples argues for their analysis by ICP/MS. We have consistently performed well in these PE exercises, with nickel analysis always yielding good to very good results. The significance of these PE exercises to the current project is that they provide an independent check on the nickel standard quality.

A Spex certified standard was used for nickel analysis in this project (a copy of the Certificate of Analysis for the nickel standard is attached). Check standards were prepared, whenever feasible, from a separate stock, and consistently resulted in measured concentrations at or near the anticipated concentration.

No reference standards were used for TOC or inorganic ion determinations. Standards for these determinations were prepared from the highest quality reagent chemical available on an analytical balance with a calibration history traceable to NIST. Previous performance in the USGS PE program has shown that we have performed well on sulfate and chloride analysis by ion chromatography.

## DETECTION LIMITS

Instrument detection limits were never an issue in this study. Concentrations were quite high in the untreated samples and concentrates so that 1 mg/L as a bottom limit offered treatment efficiencies of greater than 99.9%. Some measurements were made in the 0.1 to 1 mg/L range, well within the capabilities of the instrument. Most were made in the 10 to 50 mg/L range. Performance at the lower end, the operationally defined project detection limit, was verified by evaluating the quality of the standard curve and the results of sample spikes. This lower limit of 0.1 mg/L proved more than adequate in addressing all of the samples.

Some treated samples had TOC levels below 10 mg/L. These levels are near the effective lower limit of TOC determination for our instrumental system. However, the exact value of the TOC at this level was less critical to the project than just knowing concentrations were in this range. Consequently, no formal determination of lower detection limit was performed. Duplicate determinations at 16 mg/L showed consistent results. Evaluation of 5 and 10 mg/L standards in this low range yielded quantitation at 93% of nominal. The treated water also provided a very clean matrix for these measurements, contributing to our confidence in the results.

Instrument detection limits for inorganic ions were not a factor in this study as the few samples analyzed had concentrations of analytes at or above 10,000 mg/L.

## METHOD BLANKS

A distilled water blank was used in each calibration of the AAS. A reagent blank, containing a concentration of  $\text{HNO}_3$  (the diluent for all diluted samples, and the stabilizer for undiluted samples) similar to all of the samples, was analyzed, on average, with every 10 samples. Because of the fairly high levels of nickel in nearly all of the samples, the reagent blank results, usually between 0 and 0.05 mg/L in the higher concentration measurement ranges (10-50 mg/L), were never considered significant. Blanks in the lower concentration measurement range (0.1-1.0 mg/L) were indistinguishable from zero.

TOC and ion chromatographic analyses employed a distilled water blank, and consistently gave results indistinguishable from zero.

## DATA REDUCTION

Data reduction was limited to adjusting direct concentration measurements from the instruments for dilution factors, and to providing % difference and % recovery values for

duplicate and spikes, respectively. Calibration on the instrumental systems allows for direct readout of concentrations on all samples analyzed. In some cases, dilution factors can also be built into the instrument program. However, we rarely used this latter feature, preferring instead to maintain close contact with the actual instrument response for the sample. Dilution corrections were made manually (spreadsheet) after examination of the instrument results. All data transcriptions to the spreadsheet were checked and rechecked, sometime by two different individuals. The spreadsheet was also used for calculation of % difference and % recovery information.

# Graham Plating Nickel Analyses: 1st Pilot Lab Study

Summary of T-Sample Nickel and Selected TOC Data									
Client ID #	Lab ID #	Conductivity $\mu$ mhos/cm	NI Analysis Date	Meas'd Ni Conc, mg/L	Additional Information	Meas'd Ni Conc, mg/L	TOC mg/L	QC Comment	
T-1	92-1894	16,600	12-May	3,890	duplicate	4,140	990	dupe: 1040	
T-2	92-1895	36	9-Jun	2.5			2.6		
T-3	92-1896	20,000	12-May	11,900					
T-4	92-1897	24	9-Jun	0.8					
T-5	92-1898	23,100	12-May	5,710					
T-6	92-1899	24	9-Jun	0.3					
T-7	92-1900	25,900	12-May	6,540					
T-8	92-1901	22	9-Jun	0.4					
T-9	92-1902	30,400	12-May	7,360					
T-10	92-1903	25	9-Jun	0.7					
T-11	92-1904	33,100	12-May	8,440			2,170		
T-12	92-1905	20	9-Jun	0.2			1.6		
T-13	92-1906	36,800	12-May	9,680					
T-14	92-1907	24	9-Jun	0.5					
T-15	92-1908	41,300		no sample found					
T-16	92-1909	20	9-Jun	0.2					
T-17	92-1910	44,200	30-Jul	17,600					
T-18	92-1911	15	9-Jun	0.5	spike	97.6%			
T-19	92-1912	48,200	30-Jul	20,100					
T-20	92-1913	15	9-Jun	0.2	duplicate	0.2			
T-21	92-1914	53,400	30-Jul	22,600			3,500	dupe: 3500	
T-22	92-1915	841	22-Jul	161	duplicate	161	42		
T-23	92-1916	59,000	2-Jul	25,600	rerun: 7/30	25,900			
T-24	92-1917	193	9-Jun	33.2					
T-25	92-1918	65,800	2-Jul	29,000	duplicate	28,900			
T-26	92-1919	29	9-Jun	1.5					
T-27	92-1920	73,500	2-Jul	37,000					



T-28	92-1921	19	9-Jun	0.4		13,000
T-29	92-1922	95,300	2-Jul	65,400		6.3
T-30	92-1923	27	9-Jun	1.0		26,000
T-50	92-1926	-	26-Jun	179,000		550
T-51	92-2015	10,150	22-Jul	2,540	rerun: 7/23	6.3
T-52	92-2016	31	9-Jun	2.2		
						2,590



Client ID #	Lab ID #	Conductivity $\mu$ mhos/cm	Ni Analysis Date	Meas'd Ni Conc, mg/L	Additional Information	Meas'd Ni Conc, mg/L	TOC mg/L	QC Comment
T-53	92-2017	11,800	12-May	2,960				
T-54	92-2018	33	9-Jun	0.8				
T-55	92-2019	14,000	12-May	3,700	duplicate	3,810		
T-56	92-2020	27	9-Jun	0.3				
T-57	92-2021	16,300	12-May	4,430				
T-58	92-2022	21	9-Jun	<0.1				
T-59	92-2023	17,900	30-Jul	5,820	spike	94.8%		
T-60	92-2024	20	9-Jun	<0.1	duplicate	<0.1		
T-61	92-2025	19,100	30-Jul	6,250			1,200	
T-62	92-2026	22	9-Jun	<0.1			<1	
T-63	92-2027	20,600	30-Jul	7,000				
T-64	92-2028	23	9-Jun	0.2				
T-65	92-2029	22,200	30-Jul	7,700				
T-66	92-2030	31	9-Jun	<0.1				
T-67	92-2031	24,200	22-Jul	7,900	rerun: 7/30	8,150		
T-68	92-2032	23	9-Jun	0.3				
T-69	92-2033	26,000	22-Jul	8,750				
T-70	92-2034	36	9-Jun	0.2				
T-71	92-2035	25,800	22-Jul	8,400			1,700	dupe: 1700
T-72	92-2036	33	9-Jun	<0.1			3.8	
T-73	92-2037	26,000	22-Jul	8,550				
T-74	92-2038	22	9-Jun	<0.1				
T-75	92-2039	30,000	22-Jul	9,850				
T-76	92-2040	25	9-Jun	1	spike	99.4%		
T-77	92-2041	33,200	12-May	8,960				
T-78	92-2042	18	9-Jun	<0.1				
T-79	92-2043	33,400	22-Jul	11,600	duplicate	11,800		
T-80	92-2044	18	9-Jun	0.2	duplicate	0.2		
T-81	92-2045	36,500	22-Jul	13,000			2,500	
T-82	92-2046	19	9-Jun	0.1			<1	
T-83	92-2047	22	9-Jun	0.3				
T-84	92-2048	40,100	15-May	9,540	Value is low			
T-85	92-2049	45,800	26-Jun	17,200	rerun: 7/22	17,800		
T-86	92-2050	424	9-Jun	79.5				
T-87	92-2051	60,000	26-Jun	26,800	22-Jul	26,700		
T-88	92-2052	76	9-Jun	10.6				

Client ID #	Lab ID #	Conductivity $\mu$ mhos/cm	Ni Analysis Date	Meas'd Ni Conc, mg/L	Additional Information	Meas'd Ni Conc, mg/L	TOC mg/L	QC Comment
T-89	92-2053	-	26-Jun	45,000	spike	96.7%		
T-90	92-2054	-	26-Jun	108,000	duplicate	111,000		
T-91	no lab #	-	24-Jul	43,700	duplicate	44,000	140	
T-92	92-2055	-	26-Jun	128,000			2,600	
T-95	92-2056	-	26-Jun	8,940				
T-96	92-2057	-	9-Jun	1.9				
T-97	92-2058	-	9-Jun	1.9				
T-98		-	no sample found					
T-99	no lab #	-	22-Jul	<1				
T-00	92-2059	-	15-May	1,070				
T-200	92-2060	-	9-Jun	<0.1				
T-201	92-2061	-	9-Jun	3.4	duplicate	3.5		
T-203	92-2062	-	22-Jul	186,000	30-Jul	184,000		
T-204	92-2063	-	15-May	89,400 ppm	duplicate	84,700ppm		
T-205	92-2064	-	30-Jul	121,000	duplicate	121,000		
T-206	92-1872	-	25-Mar	1,850				
T-207	92-2391	-	29-Jun	5,380	30-Jul	5,340		
T-208	92-2392	-	22-Jul	9.1	duplicate	9.0		
T-209	92-1871	-	25-Mar	2,950				
T-210	92-1868	-	22-Jul	2,650				
T-211	92-1870	-	22-Jul	2,650				
T-212	92-1873	-	25-Mar	1,890				
T-213	92-1869	-	25-Mar	5,060	2x high?			
T-214	92-2393	-	29-Jun	5,380	30-Jul	5,340		
T-215	92-2394	-	29-Jun	46				
T-216	92-2395	-	29-Jun	1,590	duplicate	1,580		
T-217	92-2396	-	29-Jun	1,580	duplicate	1,590		
T-218	92-2397	-	29-Jun	2,770	duplicate	2,740		
T-219	92-2398	-	29-Jun	2,800	duplicate	2,770		
T-220	92-2399	-	29-Jun	2,730	22-Jul	2,680		
T-221	92-2400	-	29-Jun	2,780	duplicate	2,750		
T-222	92-1866	-	25-Mar	4,820				
T-223	92-1867	-	25-Mar	4,940				
T-999	92-2065	-	19-Jun	42,000	26-Jun	43,800		

# Graham Plating Nickel Analyses: 2nd Pilot Lab Study

## Summary of T-Sample Nickel and Selected TOC Data

Client ID #	Lab ID #	Conductivity $\mu$ mhos/cm	Ni Analysis Date	Meas'd Ni Conc, mg/L	Additional Information	Meas'd Ni Conc, mg/L	TOC mg/L	QC Comment
RO-1	92-2275	10,100	19-Jun	2,590				
RO-2	92-2276	9,300	19-Jun	2,350			9.1	
RO-3	92-2277	559	11-Jun	52.6			590	
RO-4	92-2278	10,300	19-Jun	2,610				
RO-5	92-2279	9,500	19-Jun	2,340				
RO-6	92-2280	9,200	19-Jun	2,420				
RO-7	92-2281	500	11-Jun	44.4				
RO-8	92-2282	10,000	19-Jun	2,580				
RO-9	92-2283	498	11-Jun	44.3				
RO-10	92-2284	10,400	19-Jun	2,750				
RO-11	92-2285	9,900	19-Jun	2,530				
RO-12	92-2286	10,400	19-Jun	2,630		2,710		
RO-13	92-2287	10,890	19-Jun	2,689	duplicate	57.2%		
RO-14	92-2288	512	11-Jun	44.0	spike			
RO-15	92-2289	11,390	19-Jun	2,970				
RO-16	92-2290	540	11-Jun	46.6			690	
RO-17	92-2291	11,860	19-Jun	3,150			9.2	
RO-18	92-2292	567	11-Jun	48.0				
RO-19	92-2293	12,620	19-Jun	3,320				
RO-20	92-2294	610	11-Jun	50.5				
RO-21	92-2295	13,290	19-Jun	3,600				
RO-22	92-2296	650	11-Jun	53.4				
RO-23	92-2297	14,090	21-Jul	3,860				
RO-24	92-2298	698	11-Jun	56.9	spike	97.0%		
RO-25	92-2299	14,900	21-Jul	4,130	duplicate	4,240		
RO-26	92-2300	714	11-Jun	57.9	duplicate	58.7		
RO-27	92-2301	16,040	19-Jun	4,420	duplicate	4,410	970	
RO-28	92-2302	793	11-Jun	63.6			13	
RO-29	92-2303	17,140	19-Jun	4,473	spike	85.3%		
RO-30	92-2304	845	11-Jun	66.4				
RO-31	92-2305	18,370	19-Jun	5,110				
RO-32	92-2306	932	11-Jun	72.2				

Client ID #	Lab ID#	Conductivity $\mu$ mhos/cm	NI Analysis Date	Meas'd NI Conc, mg/L	Additional Information	Meas'd NI Conc, mg/L	TOC mg/L	QC Comment
RO-33	92-2307	19,860	19-Jun	5,880				
RO-34	92-2308	1,050	11-Jun	77.5				
RO-35	92-2309	21,200	19-Jun	6,360				
RO-36	92-2310	1,160	11-Jun	84.9			1,500	
RO-37	92-2311	23,200	19-Jun	7,050			20	
RO-38	92-2312	1,330	11-Jun	93.4	spike	92.2%		
RO-39	92-2313	25,000	19-Jun	7,296		96.9%		
RO-40	92-2314	1,520	11-Jun	103	spike			
RO-41	92-2315	27,000	21-Jul	8,730	rerun: 7/24	8,880		
RO-42	92-2316	1,770	11-Jun	118	duplicate	118		
RO-43	92-2317	29,000	21-Jul	9,310				
RO-44	92-2318	2,080	11-Jun	135				
RO-45	92-2319	31,000	19-Jun	9,700				
RO-46	92-2320	2,440	21-Jul	140	Value a bit high		2,400	
RO-47	92-2321	32,800	30-Jul	11,500			46	
RO-48	92-2322	2,880	21-Jul	164				
RO-49	92-2323	35,100	19-Jun	11,340				
RO-50	92-2324	3,470	21-Jul	196				
RO-51	92-2325	37,000	19-Jun	12,460				
RO-52	92-2326	3,720	21-Jul	210	duplicate	212		
RO-53	92-2327	37,300	19-Jun	12,560			2,800	
RO-54	92-2328	1,054	11-Jun	79.4			16	dupe: 16
RO-55	92-2329	-	21-Jul	1,320				
RO-56	92-2330	-	21-Jul	1,320				
RO-57	92-2331	6,270	21-Jul	1,425			340	
RO-58	92-2332	242	11-Jun	14.5			1.9	
RO-59	92-2333	6,780	21-Jul	1,550				
RO-60	92-2334	240	11-Jun	15.6	spike	98.0%		
RO-61	92-2335	7,120	21-Jul	1,680	spike	82.2%		
RO-62	92-2336	280	11-Jun	18.6				
RO-63	92-2337	7,910	21-Jul	1,880				
RO-64	92-2338	289	11-Jun	19.7				
RO-65	92-2339	8,550	21-Jul	2,110				
RO-66	92-2340	317	11-Jun	22.4				
RO-67	92-2341	9,460	21-Jul	2,380	duplicate	2,350	540	
RO-68	92-2342	376	11-Jun	25.3	duplicate	25.0	4	

Client ID #	Lab ID#	Conductivity $\mu$ mhos/cm	Ni Analysis Date	Meas'd Ni Conc, mg/L	Additional Information	Meas'd Ni Conc, mg/L	TOC mg/L	QC Comment
RO-69	92-2343	10,840	21-Jul	2,740				
RO-70	92-2344	434	11-Jun	31.0				
RO-71	92-2345	12,040	21-Jul	3,090				
RO-72	92-2346	498	11-Jun	35.0				
RO-73	92-2347	13,660	21-Jul	3,580				
RO-74	92-2348	584	11-Jun	41.1				
RO-75	92-2349	-	21-Jul	3,510				
RO-76	92-2350	-	11-Jun	41.1				
RO-77	92-2351	16,310	21-Jul	4,440	spike	82.8%	990	rerun: 980
RO-78	92-2352	753	11-Jun	52.9	spike	100.5%	11	
RO-79	92-2353	19,520	30-Jul	5,750		70.4		
RO-80	92-2354	1,023	11-Jun	70.7	duplicate			
RO-81	92-2355	22,400	30-Jul	6,800				
RO-82	92-2356	1,280	11-Jun	86.6				
RO-83	92-2357	26,700	21-Jul	7,900				
RO-84	92-2358	1,740	11-Jun	117	97.0%			
RO-85	92-2359	32,800	30-Jul	10,600				
RO-86	92-2360	2,700	24-Jul	182	duplicate	181		
RO-87	92-2361	38,700	22-Jul	13,200	rerun: 7/24	12,800	2,800	rerun: 2,800
RO-88	92-2362	4,070	24-Jul	283	duplicate	282	71	
RO-89	92-2363	44,100	30-Jul	16,000				
RO-90	92-2364	6,480	24-Jul	479	duplicate	475		
RO-91	92-2365	48,300	24-Jul	17,900	duplicate	17,800		
RO-92	92-2366	9,190	21-Jul	790	spike	92.1%		
RO-92	92-2366	9,190	rerun: 7/21	840	spike	90.3%		
RO-93	92-2367	47,900	21-Jul	18,200			3,500	
RO-94	92-2368	833	11-Jun	61.6	duplicate	61.9	12	

