McCLELLAN AFB

PRELIMINARY WASTE MINIMIZATION ASSESSMENT FOR THE 243 G BAY ELECTROPLATING FACILITY

Prepared by

CH2M HILL March 1990

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EXECUTIVE SUMMARY

CH2M HILL has completed a preliminary assessment for the electroplating facility (Bay 243G) to determine potential waste minimization measures that may be taken to reduce the amount of RCRA (Resource Conservation and Recovery Act) related hazardous waste that is generated from that facility. The RCRA related wastes are generated from the disposal of off-specification process solutions from electroplating and surface finishing operations.

During a review of the waste generation records at the facility, it was determined that the 10 largest waste-producing streams accounted for approximately 67 percent of the total volume of RCRA wastes disposed of in 1989. CH2M HILL concentrated on these 10 streams to provide a range of waste minimization alternatives. Various technologies for continuous purification, batch purification, and batch treatment prior to disposal at Industrial Waste Treatment Plant (IWTP) No. 2 are proposed for each stream. Section 3 of this report details each waste stream studied, Section 4 discusses general and specific waste minimization strategies for each waste stream, and Section 5 summarizes those findings in a table, with the preferred order of implementation.

The most promising way to minimize wastes generated in the electroplating facility does not use high-technology equipment (although this report does detail equipment that may be used to minimize wastes), but rather creates an organizational structure that promotes and rewards efforts to minimize wastes. As a method to achieve a higher awareness of the importance of waste minimization, CH2M HILL recommends the formation of a Process Solution Maintenance Team whose responsibilities will center around the preventative maintenance of process solutions.

The Process Solution Maintenance Team should have an organized chain-of-command and specific functions for each member. It may be necessary to create additional job openings in order for team members to devote the majority of their time to this function. The specific duties of the Process Solution Maintenance Team would be as follows:

- Perform filtration (particulate and activated carbon) of process solutions on a regular schedule, rather than only when a bath is out of specifications
- Make necessary chemical additions to baths under the guidance of QA/QC officials and as directed in the Technical Orders

- Perform bath "dummying" where required to remove tramp metals contamination
- Clean or replace cartridge filtration units as necessary
- Remove dropped parts from solution baths
- Maintain and repair process equipment and new technology insertions for waste minimization
- Batch treat off-specification process solutions (using technologies discussed in this report) to render them suitable for disposal through IWTP No. 2

An individual should be responsible for reviewing the decision to dispose of any process solution. This person would have the end authority for the disposal of a solution as a RCRA waste.

Specific technologies applicable to each of the 10 waste streams are detailed in Section 4. In addition, some general facility-related actions should be considered. These include:

- The high-capacity air handling system in G bay is contributing to blowing particulates entering process baths and contributing to degradation of the process solutions. Potential solutions would be to install and maintain filters on the incoming air, install removable fiberglass lids on the process tanks, or install cartridge filters on process tanks.
- Wide temperature fluctuations in G bay are causing some "ambient temperature" baths to become non-functional. The temperature fluctuations could be solved by upgrading HVAC controls in the facility or installing temperature controls on the individual process solution tanks. Tank covers may also be advisable.
- The almost universal method for agitation of process tanks in G bay is performed by air sparging. An air compressor is currently used to supply pressurized air for this application. The compressor is known to have an oil entrainment problem, and oil droplets are probably combining with the compressed air and being transferred to the process solutions. Electroplating and surface finishing solutions have a low tolerance for oil and grease. Also, sparging air through some alkaline solutions (especially those containing cyanide) creates a carbonate sludge on the bottom of solution tanks, leading ultimately

to the disposal of the solution as a RCRA waste. CH2M HILL recommends abandoning air sparging and, for most solutions, using in-tank pumps with integral cartridge filter units to agitate and filter the solutions simultaneously.

• IWTP No. 2 contains process equipment for chromic acid recovery. The system currently cannot be used because of damage to a sulfur removal tank. Repair of this tank would allow the recovery of chromic acid back to the plating shop.

Pure metals can be recovered from many process solutions by electrowinning. This process can be applied only to concentrated solutions on a batch basis; however, it is much more effective when combined with rinsewaters from the same plating line and with other process solutions that may contain a high concentration of the same metal. This is the case with nickel plating in the G bay electroplating facility. Four concentrated nickel streams and one nickel rinse stream could be combined with ion exchange and electrowinning to recover pure nickel metal and avoid generation of much of the RCRA waste leaving the facility. Currently, Metcalf & Eddy (M&E) is proposing a design for modifications to IWTP No. 2 to incorporate a rinse recycle system using ion exchange. If electrowinning technology is installed on the nickel line, it would be beneficial to work concurrently with M&E so that both systems are compatible.

CH2M HILL has attached vendor information on some commercially available technologies to assist in waste minimization at the electroplating facility. This information should not be considered as an endorsement for the particular vendor, but rather an initial recommendation of the technology for further evaluation. Other vendors may be available to supply similar equipment.

It should be noted that several recommendations in this report involve the mixing of one or more waste streams. Because of possible danger of fire, explosion, or toxic gas generation, CH2M HILL recommends careful consideration of mixing of components because waste streams may vary in concentrations and composition from one generation cycle to the next.

SECTION 1 INTRODUCTION

Section 1 INTRODUCTION

CH2M HILL is under contract with McClellan Air Force Base to perform various environmental consulting engineering services under Contract No. F04699-90-D-0035. The first task order delivered under this contract (Task Order No. 5000) was to perform a preliminary analysis of the electroplating facility in Building 243G. The analysis was performed to identify potential waste minimization technologies that may be applicable to reduce the amount of hazardous wastes generated at the shop and disposed of by outside contractors. This project does not address rinsewater treatment options, which are currently under study by another firm. This report presents the methodology used in the investigation and a strategy for waste minimization in the shop on both an immediate and long-term basis.

Most research done on waste minimization from electroplating facilities has been directed toward rinsewater minimization, treatment, and recycling. Some of these technologies are applicable to concentrated solution baths, but documented results of technology application in this area are sparse. As a result, some of the recommendations of this report must be qualified as potentially applicable, pending further evaluation and perhaps demonstration at the facility.

1.1 McCLELLAN AFB WASTE MINIMIZATION PROGRAM

McClellan AFB has a historical interest in waste minimization and is one of the leading Air Force Logistics Centers in this area. Recently, this interest has developed into a mandate to reduce basewide disposal of hazardous wastes by 50 percent, especially in the Maintenance Directorate (MA). This effort has its support from the highest levels in the Maintenance Directorate. To support the effort, each division under the Maintenance Directorate is forming Waste Reduction Assessment Teams (WRATs) to develop strategies and implement methodologies for waste reduction. At the time of this assessment, a WRAT for the division responsible for the electroplating facility was not yet formed, but it is hoped that when the team is finalized, it will review this preliminary assessment and provide comments.

1.2 METHODOLOGY OF THE PRELIMINARY ASSESSMENT

To prepare a preliminary assessment of waste reduction options for the electroplating facility, the project team performed the following tasks:

- 1. A facility walk-through to gain overall knowledge of the various plating processes and equipment present at the electroplating facility.
- 2. A data collection effort to determine quantities of RCRA hazardous wastes generated in the shop, process tank compositions, waste stream compositions, and process tank maintenance procedures.
- 3. Interviews with various members of the electroplating facility and support laboratories, wastewater treatment plant operators, and quality control specialists to determine work flow requirements, quality control procedures, and how/why wastes are generated. MA personnel interviewed are listed in Table 1-1.

Table 1-1
MA PERSONNEL INTERVIEW ROSTER

Name	<u>Department</u>	Job Description
Ray Esposito Clarence Svoboda Seymour Daniel Oscar Estela Elwin Jang James Costa James W. Shart Brick Mesman S. Meyer Glenn Young	MANPET MAQC MAQC MAQV MANE DEMS DRMO DEMS MANE MANE	Plating Facility Supervisor QA/QC Supervisor Process Chemist Waste Generation Data Engineering Wastewater Treatment #2 DRMO Supervisor CE Laboratory Engineering Environmental
Glenn Young	MAQV	Environmental

4. Contacts with outside chemical suppliers to determine maintenance and treatment potential for several proprietary chemical bath compounds.

Data collected from the above tasks were used to rank the waste streams by volume of generation. These waste streams were then assessed for the possible range of minimization options. The minimization options were then evaluated based on their effectiveness, technical feasibility, order-of-magnitude costs, maintenance requirements, and regulatory implications. Technologies and their applicability to the 10 largest waste streams are summarized in this report.

1.3 GOALS OF THE STUDY

As mentioned previously, the ultimate goal of this study is to provide a preliminary assessment of the electroplating facility and to answer three basic questions:

- What are the major RCRA waste streams produced at the electroplating facility?
- What causes these waste streams to be produced (e.g. off-specification plating baths, carbonate buildup in plating tanks, acid depletion for pickling baths)?
- What methods are available for minimization or treatment of these waste streams so that offsite disposal is not necessary (e.g., using new technologies, existing plant treatment systems, proprietary chemical additives, upgraded maintenance procedures, or a combination of the above)?

It should be noted that electroplating will inevitably produce some RCRA related wastes, and the 10 largest-volume waste-producing streams may not be eliminated entirely. However, the quantity of these RCRA related waste streams should be reduced considerably by implementation of some or all of the recommendations of this report.

SECTION 2 ELECTROPLATING FACILITY OVERVIEW

Section 2 ELECTROPLATING FACILITY OVERVIEW

The electroplating facility in 243G bay encompasses approximately 34,000 square feet. The main mission of the facility is to refurbish high-performance aircraft components as part of the maintenance services on the base. Operations other than electroplating, including vapor degreasing and paint stripping, are also performed in this bay. The electroplating lines include, but are not limited to, the following processes:

- Precious metal plating (gold, silver, platinum)
- Conventional and electroless nickel plating
- Cadmium plating
- Hard chrome plating
- Chemical milling of aluminum
- Acid stripping and pickling
- Alkaline cleaning and stripping
- Copper plating

The facility uses approximately 190 process tanks for production. The electroplating facility, sized with the ability to perform at "maximum effort" during a war situation, is somewhat out of proportion to the actual workload. The facility operates on a job-shop basis, and some plating lines may remain unused for extended periods until parts requiring that process are received.

The electroplating bay is composed of two levels. The upper level contains the process vessels and is the main working area. The lower level provides access to process piping and contains waste sumps to which various rinse tanks and process vessels drain.

The waste sumps receive rinsewaters and some process fluids from the electroplating operations. The tanks are segregated as to general waste classifications, and the contents are emptied periodically to the Industrial Waste Treatment Plant (IWTP) No. 2, where they are treated according to classification. Pretreated water from IWTP No. 2 flows to the main treatment plant (IWTP No. 1) where metals precipitation, flocculation, and sludge dewatering take place. The waste tanks are segregated and treated as follows:

• Miscellaneous (T-3) - Receives rinsewaters from the anti-rust operations, alkaline cleaning, iriditing, nickel plating, acid copper, ammonium nitrate, conversion coating, various dyes, chrome deoxidizing, hard anodize, and nickel strip. Wastes from this tank are

directed to IWTP No. 2 where the wastes can be either directly discharged to IWTP No. 1, or treated to reduce chrome prior to discharge depending on chemical analysis.

- Cyanide (T-4) Receives rinsewaters from processes containing cyanides. Effluent from this waste tank is treated by cyanide destruction using alkaline chlorination and is then discharged to IWTP No. 1.
- Miscellaneous Base (T-5) Receives rinsewaters from alkaline cleaning operations. Wastes from this tank are pumped to a central neutralization tank where they are combined with the wastes from the miscellaneous acid tank (T-6) for mutual neutralization. Depending on analysis, the effluent from T-5 may go directly to chrome reduction.
- Miscellaneous Acid (T-6) Receives rinsewaters from miscellaneous acid processes. Effluent from this tank is treated similarly to the procedure explained T-5.
- Chrome Rinse (T-7) Receives rinsewaters from all chrome plating operations. Wastes from this tank are directed to either chrome reduction and discharged to IWTP No. 1, or a chromic acid recovery system which contains ion exchange, concentrative evaporation, sulfur removal, and returned to the plating processes as regenerated chromic acid. Forward flow from the ion exchange units is returned to the rinse tanks.
- Chromic Acid (T-8) Receives spent chromic acid discharges from chromic acid anodize, chromic acid deoxidizer, and the hard chrome plating line. Wastes from this tank can be either sent to chrome reduction and discharged to IWTP No. 1, or directed to the chrome recovery system described earlier.
- Cadmium (T-20) Receives rinsewaters from the cadmium plating line. These rinsewaters are sent to a cadmium evaporative recovery unit that has recently been installed in the G bay facility. The overhead from the evaporator is reused as rinsewater, and the concentrated cadmium solution is returned to the plating process baths.

In addition to the above description of waste streams received by each tank, certain tanks receive concentrated streams (not rinsewaters) via floor drains from individual process tanks of the same classification.

It should be noted that the chromic acid recovery system is currently not operational because of a structural problem with the sulfide reduction tank. All spent chromic acid and chrome rinsewaters are being treated by chrome reduction and discharged to IWTP No. 1.

SECTION 3 WASTE GENERATION DATA AND DISCUSSION

Section 3 WASTE GENERATION DATA AND DISCUSSION

Waste generation data for the electroplating facility were obtained from the Hazardous Waste (HW) turn-in data base and the DRMO records. It should be noted that the HW turn-in data base is a fairly new system, and it is possible that earlier data (1986 through 1988) may not be as accurate or complete as the 1989 data. The HW turn-in system has sort features that identify waste streams by quantity, generating division, or waste stream classification.

The information collected from the HW turn-in data base was correlated with analytical information from the waste stream files available at MAQVE to provide data on a particular process tank, the waste composition, and the approximate annual volume generated.

The Technical Orders (TO) for the electroplating facility provided information on the process tank compositions normally encountered during a plating run when the process is within the TO specifications. The TO also gave information on the analytical methods used for quality control and maintenance of process solutions. The above parameters were combined to produce the information provided in Table 3-1. Data in this table are given in order of decreasing waste volumes.

As shown in Table 3-1, the 10 largest waste streams constitute 67 percent of the total waste volumes produced in 1989. Although other waste streams are considered in this report, these "top 10" waste streams were the primary focus of this study, having the greatest waste minimization potential. These 10 waste streams are as follows:

1. Chem-mill etching solutions from Tank 137. This solution is used in the chemical milling of aluminum. The solution, a proprietary mixture manufactured by TURCO, Inc., consists of sodium hydroxide, a sulfide solution to control the etch rate, and an aluminum catalyst. The bath operates between 190° and 200° F.

Approximately 7,450 gallons of wastes from this tank were generated in 1989, which accounts for approximately 26 percent of the total waste volume from the electroplating facility. The waste consists of the spent etching bath and sludges that accumulate on the bottom of the tank. The waste stream contains significant amounts of aluminum (up to 13 percent by weight) and sulfides (0.4 percent by weight) in an alkaline solution. Minor concentrations of tramp metals such as copper, zinc, and tin result from the etching of aluminum alloys.

Table 3-1

INTITUTE !

2. Electroless nickel plating solutions from Tanks 195 and 193. This plating solution is a proprietary mixture termed NIKLAD manufactured by Allied-Kelite, Inc. The plating bath is acidic and consists of nickel sulfate, sodium hypophosphite, acetic acid, sodium hydroxide, and organic chelating agents to prevent nickel precipitation in the bath. Plating in the bath is activated at an elevated temperature. At ambient temperature, the bath will not plate nickel, but at the operating temperature of 185° to 192° F the bath will plate.

Wastes from the electroless nickel plating tanks amounted to approximately 2,200 gallons in 1989, or approximately 7.6 percent of the total wastes produced from the electroplating facility. The waste streams consisted of a clear green solution with a pH of approximately 4.6. Nickel is the most significant residual component in the waste stream (approximately 1 percent by weight). Various chelating agents, as well as tramp metals such as lead, chromium, zinc, copper, and cadmium, are also present in the waste stream.

3. Electroless nickel stripping solutions from Tank 202. This process solution is alkaline and is a mixture of proprietary ingredients manufactured by ABCO[™]. The solution is a mixture of sodium carbonate and unspecified organic components and is used to strip nickel from parts prior to plating. The plating shop may also use an alternate stripping solution manufactured by SHIPLEY, Inc. under the name of CUPOSIT[™]. This is a mixture of nitrobenzene sulfonate, sodium hydroxide, and other organic compounds. Both solutions are designed to operate between 120° and 130° F.

The waste stream from the electroless nickel stripping procedures amounted to approximately 3,520 gallons in 1989. This constitutes approximately 12.2 percent of the total wastes generated in the electroplating facility for 1989. The waste solution consists of a dark alkaline (pH 10.4) liquid containing approximately 5 percent brown sludgy material. Major hazardous constituents of this waste stream are nickel (8.9-9.7 percent by weight), with lesser amounts of tramp metals such as zinc, cadmium, lead, and copper.

4. Chrome stripping solution from Tank 158. This process tank contains an alkaline sodium hydroxide solution (pH 13.5) and is used to strip chrome off of parts prior to plating. The strip tank operates at ambient temperature.

The waste stream from this stripping solution amounted to approximately 1,595 gallons in 1989, or approximately 5.5 percent of the total volume of wastes generated from the electroplating facility. The waste stream is an

alkaline sodium hydroxide solution containing significant amounts of hexavalent chromium (2.2 percent by weight) and trace quantities of tramp metals such as lead, copper zinc, and molybdenum.

5. Cadmium plating solutions from Tank 84. This cyanide-based plating solution is used for plating cadmium onto parts. The process solution operates at ambient temperature and contains cadmium oxide, sodium cyanide, and sodium hydroxide.

The waste stream from this process amounted to approximately 4,100 gallons in 1989, or approximately 14 percent of the total hazardous waste generation for the electroplating facility. The waste stream is alkaline (pH 13.8) and contains significant amounts of cyanide (3.8 percent by weight) and cadmium (5.8 percent by weight). Lesser amounts of tramp metals such as copper, zinc, silver, nickel, and lead are also present in the waste.

6. Silver stripping solution from Tank 3. This solution bath is used to strip silver from parts prior to plating. The solution is an alkaline, cyanide-based stripper containing sodium cyanide and sodium hydroxide. The solution bath operates at ambient temperature.

The waste stream produced from the silver stripping operation was approximately 840 gallons in 1989, or approximately 2.9 percent of the 1989 total waste volume from the electroplating facility. The waste stream is an alkaline solution, pH 13.9, with significant amounts of cyanide (6.1 percent by weight). Silver is present in the waste at approximately 434 milligrams/liter (ppm), and copper is present at approximately 788 ppm. Lesser amounts of tramp metals such as nickel, cadmium, zinc, and lead are also present.

7. Acid pickling solution from Tank 90. This solution is used to acid etch parts prior to plating. The bath contains a concentrated hydrochloric acid solution and operates at ambient temperature.

The waste stream from the acid pickling operation amounted to approximately 770 gallons in 1989, or approximately 2.7 percent of the total waste volume for that year. The waste is acidic and contains hydrochloric acid, approximately 822 ppm cadmium, 807 ppm zinc, and trace amounts of tramp metals such as chromium, copper, lead, and nickel.

8. Nitric acid wastes from the cleaning of the electroless nickel plating tank. This waste stream is produced when the electroless nickel tanks (Tanks 193 and 195) are cleaned. The nickel plating tanks tend to build up nickel deposits on the tank interior, thus causing poor bath performance. When this occurs, the plating bath is discarded and a concentrated nitric acid

solution is pumped from a tank on the lower level of the plating room into the plating tanks. The nickel deposits on the plating tank are dissolved, and the nitric acid solution is then disposed of.

The waste solution from the nickel tank cleaning process amounted to 770 gallons in 1989, or approximately 2.7 percent of the total waste volume for that year. The waste is acidic and contains nitric acid, nickel (1.4 percent by weight), and trace amounts of chromium and cadmium.

9. Alkaline derust solution from Tank 73. This process tank is used to remove rust deposits on parts prior to further surface finishing. The process solution operates between 125° and 200° F. The tank contains an alkaline derust agent composed essentially of sodium hydroxide.

The waste stream from this process contains both a free liquid and a brown sludge and is about 12 percent solids by weight. The total volume of waste generated from this process is approximately 715 gallons for 1989, or approximately 2.5 percent of the total waste volume for that year. The waste material is alkaline (pH 14.0) and contains sodium hydroxide, 867 ppm lead, 340 ppm zinc, 407 ppm chromium, and lesser amounts of copper, cadmium, nickel, and manganese.

10. Nickel stripping solution from Tank 5. This is an acid solution for stripping nickel from parts prior to plating. The solution bath operates at ambient temperature and contains a strong sulfuric acid solution.

The waste stream for this process amounted to approximately 660 gallons in 1989, or approximately 2.3 percent of the total waste volume produced in the electroplating facility for that year. The waste is acidic (pH <0.01) and contains sulfuric acid, 0.6 percent nickel by weight, 0.3 percent copper by weight, 22.1 percent sulfur by weight, 444 ppm zinc, and lesser amounts of chromium, silver, and lead.

It should be noted that the waste stream for the cyanide nickel strip from Tank 171 shown in Table 3-1 is a major contributor to the waste streams from the plating shop (4,100 gallons in 1989, or 14.2 percent of the 1989 total); however, this waste stream has not been mentioned for consideration in waste minimization technologies. This is because the electroless nickel stripping solution mentioned in the above waste stream explanations (ABCO^m or CUPOSIT^m solutions) has replaced the cyanide nickel strip process, and the cyanide-bearing waste stream has not been produced after 1989.

As mentioned previously, these 10 waste streams account for approximately 67 percent of the total waste volume produced by the electroplating facility in 1989. Section 4 of this report reviews the basic process technologies that were considered to reduce or treat the waste from these 10 sources. In addition to the 10

waste streams mentioned above, two additional generalized categories of wastes were considered for treatment/reduction potential: alkaline cleaners and acid stripping solutions. These categories were added because common technologies to minimize or treat these waste streams may be available and, unlike plating baths, these process solutions will continue to be a source of waste at the electroplating facility because they tend to become depleted or build up with metal impurities after heavy use.

SECTION 4

WASTE MINIMIZATION TECHNOLOGY OPTIONS FOR MAJOR WASTE STREAMS

Section 4 WASTE MINIMIZATION TECHNOLOGY OPTIONS FOR MAJOR WASTE STREAMS

Prior to detailing specific technology options for RCRA waste streams at the electroplating facility, it is important to classify the basic hierarchy of waste reduction technologies. This generalized hierarchy has been adopted by DOD to comply with future regulations regarding waste minimization technologies. Although this proposed hierarchy is not part of any regulatory compliance procedure as of the date of this report, the general order of implementation has a sound technical basis. Each component of the waste minimization hierarchy is summarized below.

PRIORITY 1: WASTE REDUCTION AT THE SOURCE OF WASTE PRODUCTION

For process solution baths at the electroplating facility, this component involves one of the following:

- Improving process solution maintenance procedures to extend the life of process baths
- Improving housekeeping practice
- Changing processes to substitute non-toxic or less toxic materials

Of the above methodologies, only the first two were considered in the scope of this report because of specific military specifications and processes required in the production of high-performance aircraft parts.

PRIORITY 2: WASTE RECOVERY AND REUSE

For process solutions in the electroplating facility, this methodology includes:

- Removing valuable metals or pure metal salts from spent process solution baths and returning these metals to the electroplating process
- Purifying off-specification process solution baths and returning them to the process from which they originated

PRIORITY 3: RECYCLING

This methodology includes either onsite or offsite recycling of the component baths, but not necessarily for the same use as the processes that generated the waste. Because of the complex nature of onsite recycling for other uses (i.e., potential use for the product, QA/QC requirements for military specifications, labor requirements, and plant facilities requirements) and because of the inherent increased liability incurred in offsite recycling facilities, this recycling option was not considered for the electroplating facility RCRA wastes except for the outside sale of scrap metal or pure metal salts.

PRIORITY 4: WASTE TREATMENT

This methodology includes methods to destroy, detoxify, or neutralize waste streams to make them less hazardous or potentially nonhazardous prior to their disposal.

PRIORITY 5: DISPOSAL

If a waste must be generated and it cannot be effectively handled by one of the above methods, methods must be invoked to reduce the amount of waste disposed of. "Secure" disposal sites with controlled monitoring and leachate collection must be used.

To simplify the above methodologies in order of priority, the goal is to find methods to not generate a waste by keeping the process solutions up to the QA/QC requirements. If a waste is generated, some method of purifying the waste must be found to reuse it in the process. If the first two methods fail, some method must be found to treat the waste to reduce its toxicity, volume, or both.

This section of the report explains the technologies deemed most appropriate for achieving the goals outlined above. The appropriate technologies will then be applied to the 10 waste streams outlined in Section 3.

4.1 SOURCE REDUCTION TECHNOLOGIES

The two methods of source reduction technologies mentioned earlier are to upgrade bath maintenance procedures and improve housekeeping in the electroplating facility. The housekeeping aspect is really not at issue, as the electroplating facility appears to be one of the cleaner electroplating operations reviewed by CH2M HILL. However, upgrading solution bath maintenance procedures (e.g., removing dropped parts) and improving environmental conditions in the facility appear to offer the potential for substantial reduction in the volume of process solutions that need to be disposed of by extending the life of process baths.

The general environmental and process factors in the electroplating facility that likely contribute to process solution degradation are related to blowing particulate matter, temperature extremes in the facility, and the use of air sparging for tank agitation. Each of these factors is briefly discussed in relation to their potential to increase the frequency of bath disposal in the electroplating facility.

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4.1.1 BLOWING PARTICULATES

The electroplating facility is equipped with a forced-air, positive pressure ventilation system that allows for good air movement in the shop. Although this is definitely preferred from an industrial hygiene perspective, the high air turnover in the building tends to introduce dust and dirt into the facility and deposit this material into process solutions. The dust/dirt eventually contributes to inferior bath performance and buildup of sludges, and leads to the disposal of the solution. During the facility walk-through by CH2M HILL, a film of dust and small debris could be seen on process solutions that were not being used.

Potential solutions to the particulate problems include the installation and maintenance of a dust filtration system on the air handling units, removable fiberglass tank covers, and continuous filtration of the process solutions using dedicated cartridge filter units for each tank.

Ray Esposito, Shop Foreman, has used small in-tank cartridge filters on selected tanks. His analysis is that the filtration systems keep the solutions much cleaner. Mr. Esposito wants to purchase more of these filtration units for other process tanks. The issue of bath filtration will be discussed further under General Bath Maintenance and Air Sparging.

As an aside to the small particulates problem, some process solutions contained small crickets floating in them. Shop foremen stated that this was a common problem. Although this is not related to the air handling system, the crickets could be considered a form of particulate contamination and could contribute to poor bath performance. Exterminating companies should be used to try to control the cricket problem in the vicinity of the electroplating facility. Tank covers for process solutions could alleviate (but not eliminate) this problem.

4.1.2 TEMPERATURE CONTROLS

Because the electroplating facility is in a large bay with high ceilings and because of the previously mentioned air handling system, temperatures fluctuate in the building. In the winter months it becomes very cold in the facility (especially over the weekends and holidays), and in the summer months it becomes very hot and humid in the building (contributed to by the heated process tanks). These extreme temperatures may not affect baths that are heated because temperature controls keep the solutions well above the facility air temperature in both winter and summer. However, solutions designed to operate at ambient temperature show definite detrimental effects from the temperature extremes in the building.

Specifications for ambient temperature plating and process baths usually require that the baths operate somewhere around 70° to 72° F. If the ambient air temperature (and thus eventually the process bath temperature) varies significantly from this range, some components can precipitate from the solution, or the bath can be otherwise irretrievably damaged. Plant personnel mentioned that this was a particular problem with cyanide plating baths and other baths containing a high amount of dissolved solids. During cold weather, materials will precipitate out of the baths causing bath disposal. During warm weather, the ambient temperature baths exhibit reduced performance if their temperature exceeds the recommended range and are again disposed of.

It should be noted that one method of bath purification used by other electroplating facilities is purposely reducing the temperature of baths to "freeze out" contaminating carbonates. However, this is normally done under controlled conditions using portable chillers. The situation in the electroplating facility is one of uncontrolled precipitation which leads to bath disposal.

The recommended solutions to the temperature control problems would be to either more closely control the ambient air temperature in the facility or install automated heating/cooling devices in the affected process tanks. The first option may involve significant expenditures in HVAC controls because of the high air flow rate and the large size of the facility. Heating the process solution tanks could be more easily achieved using thermostatically controlled immersion heaters. Many types of immersion heaters for process tanks are available in a variety of construction materials (including Teflon). Appendix A provides vendor information on the types of immersion heaters available. Other vendors are available to provide similar equipment. Radiant heaters above the process tanks could also be used if overhead clearance is sufficient.

Cooling the tanks during warm weather could present more of a capital expenditure problem as cooling coils and water/refrigerant would be needed; however, portable chilling units could be provided to periodically induce precipitation of carbonate material.

The final recommendation on how to more closely control the temperature of ambient temperature process solutions will require further study of the above-mentioned alternatives in terms of their cost-effectiveness and feasibility; however, plant personnel believe that the lack of adequate temperature controls for the ambient temperature baths may be a significant reason for process solution turnover.

4.1.3 AIR SPARGING FOR SOLUTION AGITATION

Agitation is required for proper performance of many electroplating and metal finishing operations. Almost without exception, the method of agitation employed at the electroplating facility is air sparging. Compressed air supplied from a main air compressor is sparged into the bottom of solution tanks through a perforated diffuser pipe. The boiling motion of the air bubbles causes agitation in the tank.

In many cases, air sparging is an acceptable practice. However, it may be the cause of process solution turnover because of the potential for carbonate buildup in alkaline process solutions and because the air compressor has had severe oil entrainment problems. Most electroplating operations use a low pressure blower rather than a compressor to avoid oil entrainment.

In an alkaline solution, air sparging introduces carbon dioxide into the solution which consumes caustic in the solutions and causes metals to precipitate as carbonates. Air sparging also contributes to the oxidation of cyanides into carbonates, thus depleting the cyanide content of the bath, increasing carbonate content, and causing solution turbidity and sludge buildup. Carbonate buildup is normally checked under the analytical QA/QC program for alkaline plating tanks. Under current operating practices, if the carbonate levels are exceeded, the bath is "red-lined" and disposed of.

Based on interviews with shop personnel, carbonate sludge buildup in solution tanks is a major problem. This problem is magnified in ambient temperature baths during cold weather because of the temperature-related problems mentioned earlier. When sludge buildup becomes excessive, or when carbonate levels in the tanks cause the bath to be "red-lined," both the sludge material and solution are disposed of as a RCRA waste.

The oil entrainment problem with the air compressor represents another serious deficiency in the air sparging system. Electroplating and other process solutions are sensitive to oil and grease levels. These contaminants have a detrimental effect on plating quality and cannot be tolerated except in very minute quantities.

Apparently, the air compressor used to supply air to the sparging system has had severe mechanical difficulties, causing the oil and grease content of the compressed air to be very high. This oil and grease would be transferred directly to solution tanks during the sparging operation. There is no individual oil filtration units on the process tanks to remove this entrained oil.

The preferred method for solving the aforementioned problems would be to abandon the practice of air sparging solution tanks for agitation and use either mechanical mixing or in-tank recirculating pumps. In-tank recirculating pumps may be preferred because solution agitation can be accomplished simultaneously with the filtration methods mentioned under the preceding sections. An in-tank recirculating pump/filter system has been used on an experimental basis by the plating shop and found to be effective in providing both the necessary agitation and filtration. A manufacturer's brochure for the Flo King pumps purchased by the electroplating personnel is provided in Appendix B. Other manufacturers may supply similar items. This method is an elegant solution to both the particulate and agitation problems. It should be noted that some of the larger process tanks may require more than one pump/filter system to supply adequate agitation.

After prolonged periods of filtration, suspended particles on the filter cartridges may need to be periodically disposed of (the particular brand of pump/filter unit currently being used contains a disposable filter cartridge). This would potentially create a new waste stream for disposal at the electroplating facility. At the worst case, on a weight basis, disposal of the filter cartridges would be less costly than disposing of spent process solution baths, and methods could be used to compact or crush the cartridges in drums for volume reduction. It may be possible to clean and reuse the disposable cartridges by allowing them to air-dry and then removing particulates by shaking or beating them over a drum. The drum of particulates could later be disposed of.

An alternative to disposable cartridge filters would be to use "cleanable" filter units. These units are layered, rolled, filter material which plant maintenance personnel may unroll and clean regularly. The filters may be able to be cleaned in rinse tanks at the facility. The various manufacturers of these filters were not contacted as part of this report, and the compatibility of these cleanable filters with the existing Flo King equipment is not known at this time. The addition of hinged attachments to allow the pump/filter units to be raised out of the tanks more easily would also be a recommended modification.

Other methods to alleviate the oil and grease problem would be to install in-line air filters on the air lines to each tank or to the entire facility to trap oil and grease in the compressed air, or to use continuous carbon filtration of all air-sparged tanks to remove oils from the process solutions. Because of the continued use of air sparging, neither of these secondary options would alleviate the

carbonate sludge buildup or the cyanide oxidation problems, and installing dedicated carbon filtration units on all air-agitated solution tanks may not be cost-effective.

4.1.4 PROCESS BATH QA/QC AND MAINTENANCE PROCEDURES

The previous source reduction methodologies of this section dealt with specific facility changes that could extend the life (thereby decrease the waste) of electroplating process solutions. A general procedure would be to implement a rigorous Process Solution Maintenance Program to ensure that process solutions are maintained and replenished on a regular schedule, and to be responsible for the upkeep and maintenance of new equipment that is installed to minimize or treat wastes from discarded process baths.

These two areas of responsibility are critical to the success of a waste minimization program at the electroplating facility because the best way to minimize wastes is to keep the baths in specification (i.e., maintain and replenish solutions before they are "red-lined"). Also, if new equipment is installed to assist in this goal but no maintenance is performed, the technologies will soon become useless or cumbersome, and operators will abandon their use.

Based on CH2M HILL interviews with various personnel involved with the electroplating facility, preventative maintenance for process solutions is hampered by the lack of adequate staff to perform preventative maintenance, and the somewhat confused organizational responsibility of preventative maintenance and disposal orders for off-specification solutions.

Solution baths are sometimes discarded based on their inability to perform even when the chemical analysis shows that all measured parameters are within specification. As one interviewee said "The bath is OK but the platers say it is not working right, so the foreman gives orders to dump the bath and make up a new one." In this scenario, the bath may actually only require purification or replenishment to become useable, or the bath might not have become off-specification if continuous maintenance was performed on the solution from the outset. Currently, the foremen responsible for issuing the orders to "dump the bath" have no real incentive to do otherwise. From a production standpoint, re-making the bath is the quick and easy alternative to spending the time and effort needed to bring the bath back into specification.

Another contributing factor to the need for a comprehensive preventative maintenance program for process baths is the job-shop nature of the facility. A particular plating line may be active for a period of time due to a heavy workload for the particular plating process. After the workload is completed, the line (or certain tanks within the line) may remain idle for several weeks until it is reactivated to handle a new workload. This sporadic use of the plating lines may lead to an "out of sight, out of mind" mentality when the line is not in use. The

process solutions may not be as rigorously maintained when they are dormant. Many times a plater will re-activate a line that has been idle for some time only to find that some of the process solutions are no longer within specification.

CH2M HILL recommends that a Process Solution Maintenance Team be formed whose sole function is to maintain process solutions and equipment in the electroplating facility. The general functions of the maintenance team would be as follows:

- Filtration (particulate and activated carbon) of process solution baths on a regular schedule, regardless of whether the bath is within specifications.
- Make necessary chemical additions to baths under the guidance of senior QA/QC officials and the Technical Orders
- Perform bath "dummying" where required to remove tramp metals contamination
- Clean or replace cartridge filtration units as necessary
- Remove dropped parts from solution baths
- Maintain and repair process equipment and new technology insertions for waste minimization
- Batch treat waste process baths (using technologies to be discussed later) to render them suitable for disposal through the existing IWTP No. 2 or IWTP No. 1.

Some of these duties are currently performed by the chemical handlers in the facility, and these current handlers could become members of the proposed team. However, other team members will need to be added to meet the anticipated increased workload.

Another suggestion that may be implemented by MA would be to assign responsibility to an individual for reviewing the decision to dispose of any process solutions. When a request is made to dump a solution bath, this individual would review the analytical data and confer with QA/QC officials and the plating line foreman. If it is determined that the solution bath can be purified using existing technologies, the supervisory official would determine which method is most suitable and assign members of the Process Solution Maintenance Team to perform the treatment.

If bath purification or replenishment is unsuccessful, or if it is determined that it is not possible, the supervisory official would then determine a method to treat the waste so that it may be disposed of at IWTP No. 2 or No. 1. If this is not possible, then volume reduction measures should be investigated to reduce the volume of disposed fluid to offsite contractors.

A Process Solution Maintenance Program Manual should be prepared as soon as purification technologies and methods are chosen so that all operators are aware of the capabilities of new equipment and techniques. This manual would be a guide to the Process Solution Maintenance Team and the supervisory official to determine what technologies are applicable to a certain process solution or waste stream.

4.2 WASTE RECOVERY AND REUSE OPTIONS

In spite of preventative maintenance procedures, some process solution baths eventually become non-functional and need to be batch treated outside the process vessel. As mentioned in the introduction to this section, the baths can either be batch-purified and returned to service, or have the metals removed for recovery and the reduced-metal solutions sent to IWTP No. 2 for treatment. General methodologies for each of these options will be explained in this section.

4.2.1 BATCH PURIFICATION

Methods used for batch purification include chemical additions to remove trace contaminant metals, decanting of liquids and disposal of sludge from tanks and returning the supernatant liquid to process vessels, cleanup with ion exchange columns, and cleanup with electrodialytic membrane units. Batch treatment by particulate filtration and carbon adsorption is also feasible, but it has been assumed that this was performed under improved bath maintenance procedures described earlier. The equipment necessary to implement each of the above technologies is specific to a given process solution and a given contaminant. Each method is briefly described below.

4.2.1.1 Batch Purification by Chemical Addition

In general, this method involves the addition of a chemical agent to precipitate tramp metal species, such as iron, or other inorganic species, such as carbonates. A chemical (many times a proprietary agent manufactured by electroplating chemical suppliers) is added to the process solution, and precipitated contaminants are collected by decanting the supernatant liquids and filtration of the precipitated material. The precipitates would normally be disposed of as RCRA wastes, but would have a much reduced volume compared with the original solution bath.

4.2.1.2 Batch Purification by Decanting and Sludge Removal

Many of the process solutions that are discarded in the electroplating facility have a buildup of sludges on the bottom of the tank. The solution itself may be retrievable; however, the current practice is to discard the process solution with the sludge. Usually the solids content of the entire mixture is 5 to 10 percent sludge so it is possible that 90 to 95 percent of the volume of baths currently disposed of for this reason may be recoverable.

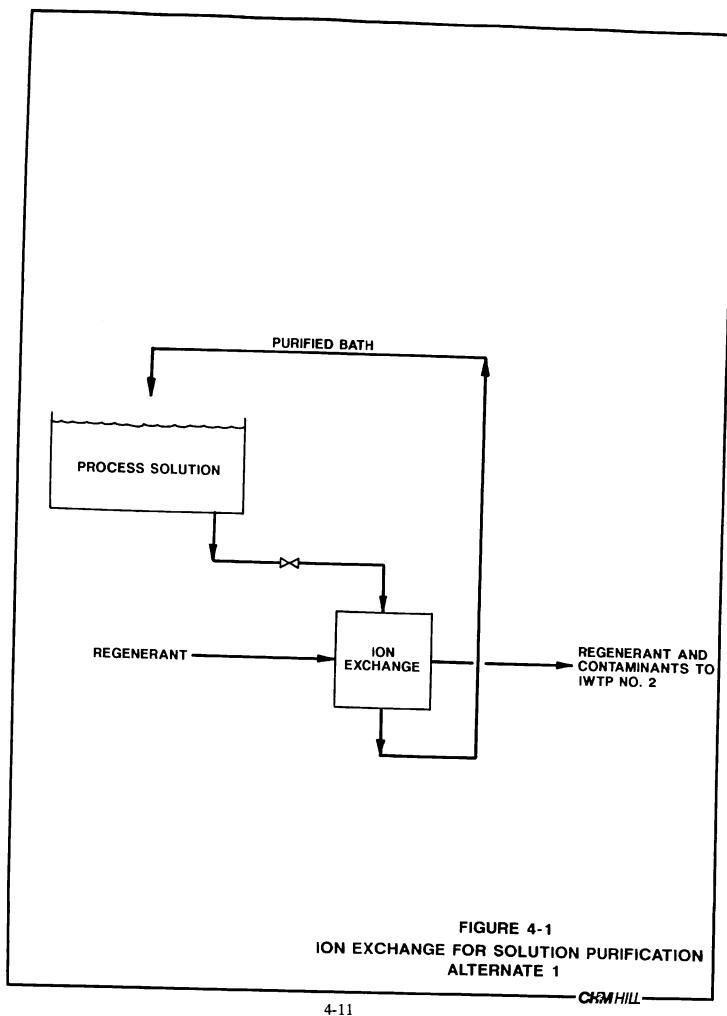
A simple process would be to acquire a portable tank for decanting process solutions when sludge buildup becomes a problem. The tanks could be cleaned of the sludge while the process solutions are batch treated for other contaminants in the portable tank. After the tank is clean, the purified solution may be returned to the process vessel and makeup solution added as required. The sludge would normally be disposed of as a RCRA waste.

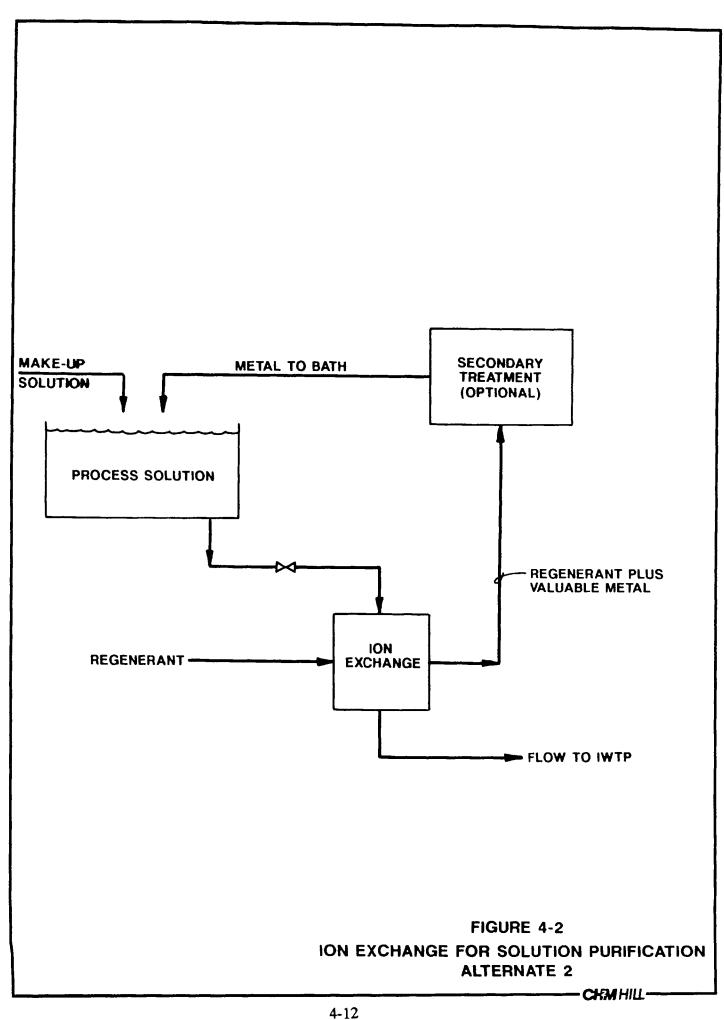
4.2.1.3 Purification by Ion Exchange

lon exchange systems are basically chemical filtration systems that remove either positively charged ions (cation exchange) or negatively charged ions (anion exchange) from a solution by using resin beads that exchange the appropriate ion from the resin with one from the solution. The technology is usually only used for dilute streams containing metals; however, studies have been performed for more concentrated waste streams. Ion exchange systems will concentrate dilute streams and hold the contaminants on the resin beads. The resin is periodically regenerated with a strong acid or base, producing a concentrated waste stream that can be reused directly, or directed to other treatment methods.

As applied to solution purification (contaminant metals removal), the system could be operated in two ways as shown in Figures 4-1 and 4-2. Figure 4-1 shows contaminant metals being removed from a plating bath by adsorbing onto the ion exchange resin, with the bath returning to the process vessel and the regenerant being directed to IWTP No. 2. Figure 4-2 shows the valuable plating metal being adsorbed onto the resin and the effluent containing the contaminant metals being directed to IWTP No. 2. The plating solution would then be recovered in the regenerant.

The benefits and drawbacks of each method depend on the type of solution to be ion exchanged. A study by Battelle, under contract to the U.S. Air Force Engineering and Services Laboratory, was conducted to determine the feasibility of using selective ion exchange to treat spent plating and process baths by removal of contaminant metals (the Alternative 1 method in this report). The results of the study showed that resin adsorption capabilities were highly variant on the type of bath being treated and the types of contaminant metals present. Backwash, regeneration, and rinse volumes for resin de-sorption were also significant in comparison with the amount of waste being treated.





4.2.1.4 Batch Purification by Electrodialysis

Electrodialysis systems remove metals by using induced current and semipermeable membranes to effect separation of ions in a solution. As with ion exchange systems, most industrial applications in the electroplating industry have been for dilute rinsewater streams, not direct treatment of process solutions.

4.2.1.5 Other Electrodialytic Processes

Figure 4-3 shows a process flow chart for an electrodyalitic process known as the IONSEPTM system. This system operates on the principle of removing metal cations from solution and precipitating them as a hydroxide sludge. The regenerated anion solution (in this case chromic acid) is returned to the bath. The IONSEPTM system will be discussed in more detail later in the report.

4.2.2 METALS REMOVAL TECHNOLOGIES

According to base personnel, process solutions are disposed of as RCRA wastes because the metals concentrations are usually too high for the IWTP No. 2 to accept. IWTP No. 2 is only designed to treat dilute wastewater streams from rinsing operations in the electroplating facility and contains no metals treatment or recovery facilities except for chromic acid recovery and chromate reduction. All metals are removed after the effluent from IWTP No. 2 has reached IWTP No. 1, where pH adjustment, lime addition, and flocculation occur to co-precipitate a variety of metals.

If metals could be removed directly from the waste streams, then a dual benefit would be obtained. Valuable solid metal, or pure metal salts, could be returned to the processes or sold as scrap, and the resultant low-metal process solutions could be more easily accepted for cyanide destruction and pH neutralization at IWTP No. 2 and for further metals treatment at IWTP No. 1. The two most widely used methods for metals recovery are electrowinning and chemical precipitation.

The problem with many of the process wastes involved in this study is that they are mixed metal streams and vary widely in pH. For example, the wastes from the electroless nickel stripping solution (Tank 202) contain a significant amount of nickel, but also contain a lower concentration of copper, lead, zinc, and cadmium, which have separate discharge limits from IWTP No. 1 in the NPDES permit. For most processes such as ion exchange, metals recovery and reuse, and chemical precipitation, only one metal or class of metals could be treated with a given process, thus potentially requiring several parallel treatment systems before the waste is acceptable to the IWTPs.

In the above example, it could safely be assumed that IWTP No. 2 could not accept this waste stream because of the nickel content, but no specific standards

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could be found during CH2M HILL's interview that would confirm exactly what metals in what concentrations would be considered excessive by IWTP No. 1 or IWTP No. 2. The decision process for accepting a concentrated waste stream at the IWTPs needs clarification and documentation before more definitive technology insertion recommendations can be made.

For this report, it has been assumed that the most prevalent metal is the cause of non-acceptance of the waste stream at the IWTPs, and that trace metal concentrations in any given process bath are of lower concern in relation to NPDES discharge limits.

4.2.2.1 Metals Recovery by Chemical Precipitation

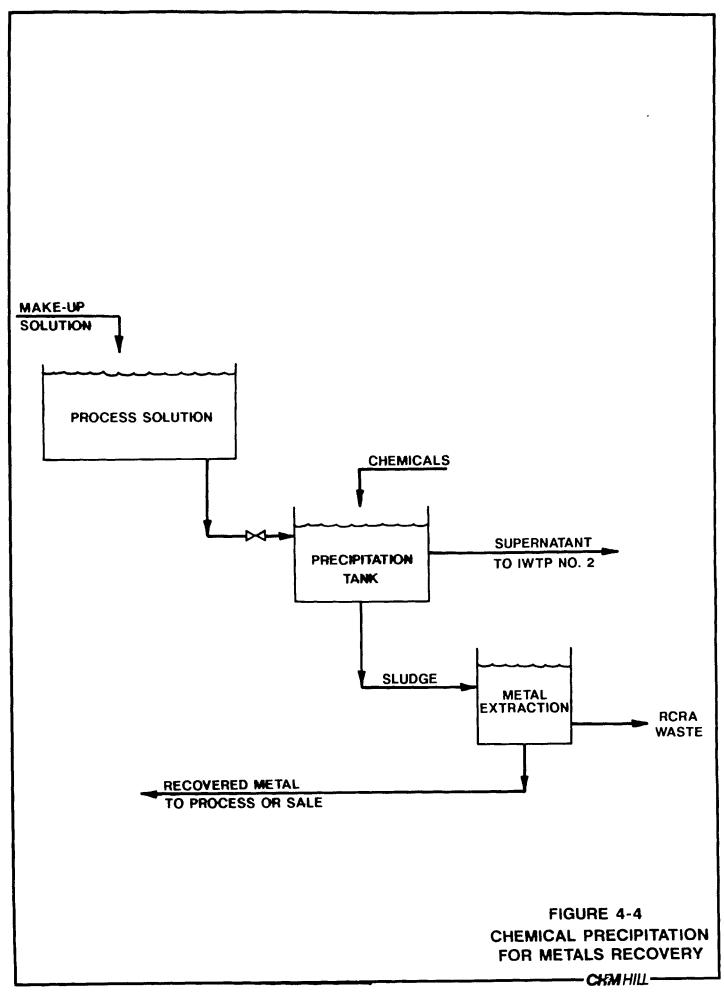
The generalized method of chemical precipitation was mentioned earlier in terms of a bath purification process for contaminant metals. The same general principle can be applied to the precipitation of a valuable metal for reuse within the shop. The main problem with this method is that the metal rarely precipitates in a form that is directly usable. For example, precipitation reactions that produce a metal hydroxide sludge are not desirable because the sludge is difficult to manage and not directly usable within the shop. It is not feasible to precipitate only one metal from a mixed metal solution. Mixed metal sludges are not desirable if metals are to be reclaimed. Thus, for metals recovery applications, the solid product produced by chemical precipitation usually has to be followed by some sort of conversion step to a usable product, or the solution to be treated must contain a high excess of a single metal.

The precipitation reactions can usually be carried out in a batch reactor with the addition of chemicals specific to the waste stream being treated. The supernatant liquid, now relatively free of the metal of concern, is directed to IWTP No. 1 for further treatment. The solid product continues on to a conversion step, and the end product is a usable metal-bearing product. A general process flow chart is shown in Figure 4-4.

Due to the complexity of the secondary treatment of the solid to form a usable product, the mixed-metal nature of the waste process solutions, and the potential QA/QC problems inherent in returning the product to the electroplating systems, CH2M HILL does not recommend that MA pursue metals recovery by chemical precipitation at this time, except for a proprietary process to precipitate nickel from electroless nickel baths (described later).

4.2.2.2 Metals Recovery by Electrowinning

Electrowinning is used to remove metals from wastewater by plating the target metal onto cathodes immersed in the electrowinning tank. The cathode can be physically stripped of the metal and reused, or disposable cathodes are also available. The electrowinning system can be used either to remove contaminant



metals from a process solution (a bath purification technique not mentioned earlier), or to remove the metal being plated in the bath (e.g., remove nickel from a nickel plating bath). After electrowinning, the resultant low-metal bath could be discharged to IWTP No. 2.

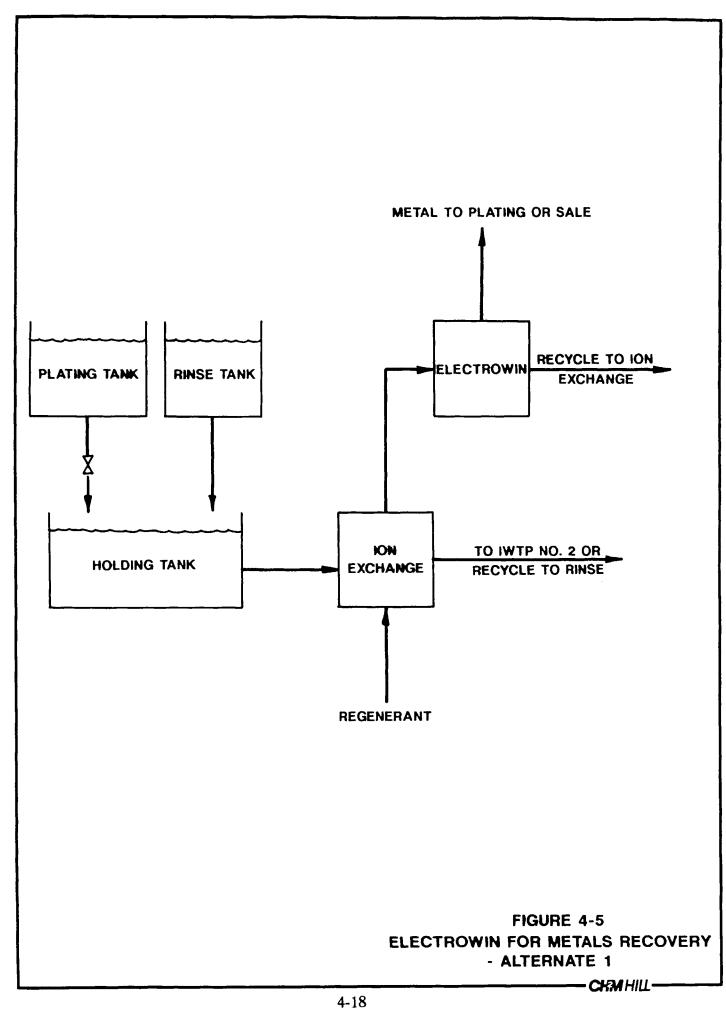
Electrowinning technology lends itself to solution baths that are concentrated because the rate of plate-out on the cathodes is directly proportional to the metal concentration in the solution. For this reason, treating plating baths for trace metal contaminants by using electrowinning only is not feasible. There are two types of electrowinning technology: High Surface Area Electrowinning, and Parallel Plate Electrowinning. The main difference between the two technologies is the ability of High Surface Area Electrowinning to remove residual metals concentrations down to more stringent effluent levels at a cost of additional equipment.

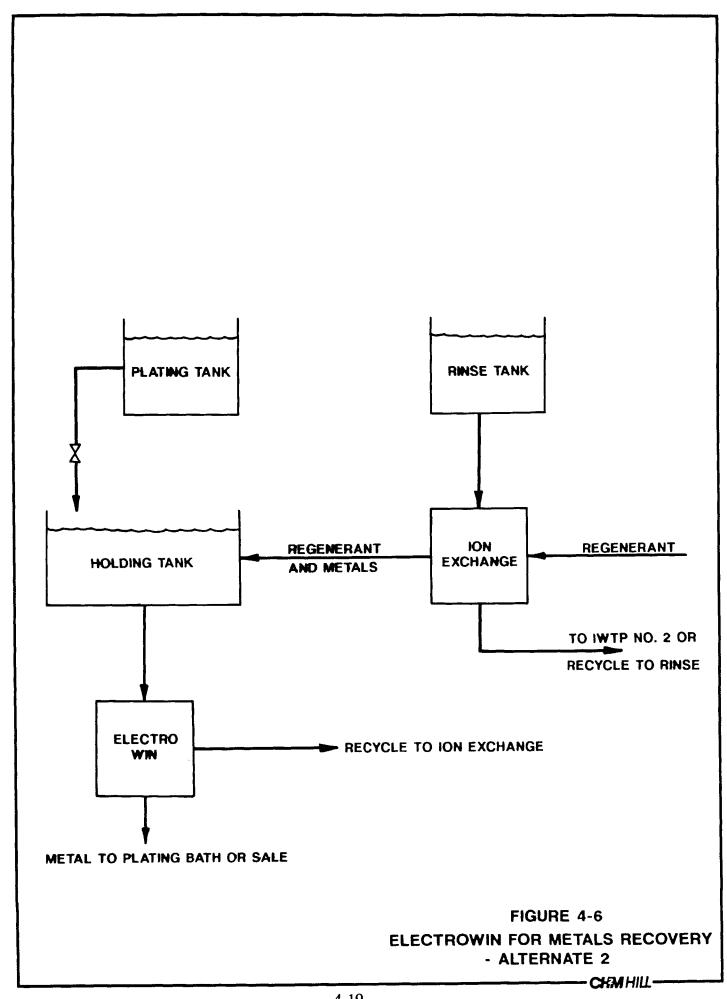
In the case of alkaline cleaning or acid stripping solutions, which by design only have trace metal concentrations, electrowinning may not be the preferred alternative, or the solutions may need to be concentrated before electrowinning is viable. Also, electrowinning technology is ion-specific, and thus only one metal at a time can be plated out in an electrowinning system to produce a salable scrap metal product.

Although solution baths with high metals concentrations can be electrowinned directly, an interesting and useful process can be used by combining rinsewaters with plating bath discharges, and using ion exchange technology in combination with electrowinning. Figures 4-5 and 4-6 show two such combinations.

In Figure 4-5, a process solution containing high metals concentrations is bled into a receiving tank containing rinsewaters from the same plating line. The resulting mixture, a fairly dilute solution, is passed through an ion exchange column where the metal to be recovered is adsorbed onto the resin. The forward flow effluent from the ion exchange unit is directed to IWTP No. 2 or recycled to the rinse tank. The ion exchange resin is periodically regenerated, and the concentrated stream is sent to an electrowinning system for metals recovery. The effluent from the electrowinning system is recycled back to the dilute collection tank.

Figure 4-6 shows a similar process except that only the rinsewaters pass through the ion exchange unit, with the regenerant being directed to a separate tank and mixed with periodic bath dumps from the same plating process. This concentrated solution is then electrowinned to recover the target metal, with the effluent from the electrowinning system being recycled back to the dilute holding tank. The advantage of this system is that surface active agents or additives in the plating baths would not affect resin performance. The disadvantage is that better concentration prior to electrowinning and more homogenous quality feed





to the electrowinning system are achieved when both the plating baths and the rinsewater are sent through the ion exchange system, at the expense of shortened ion exchange run time.

The above examples show that there are some advantages to combining spent concentrated solution baths with rinsewater streams to produce a dilute solution for ion exchange and subsequent metals recovery. This process has been used successfully by electroplating facilities and seems to work fairly well. The future expansion of IWTP No. 2 to become a closed-loop system, currently being studied by Metcalf & Eddy, should further study the integration of batch treatment and ion exchange for concentrated process solutions mixed with rinsewaters.

An alternative method for concentration of bath/rinsewater combinations prior to electrowinning would be to perform evaporative recovery rather than ion exchange. The overhead product from the evaporator may be useful as a recycle stream for rinsewater makeup. The size of a system required to perform evaporative recovery on bath/rinsewater combinations can be excessive in terms of capital and operating costs.

4.3 WASTE TREATMENT/VOLUME REDUCTION TECHNOLOGIES

This section discusses situations where process solutions are irretrievably depleted, and recovery and reuse options are non-existent or not cost-effective. The goal of waste treatment would be to convert the hazardous streams into nonhazardous waste streams suitable for treatment at IWTP No. 2, or to convert a large volume of waste into a small hazardous component and a larger nonhazardous component suitable for treatment in IWTP No. 2. The technology options chosen for consideration were chemical precipitation, evaporation, and bleeding concentrated solutions into IWTP No. 2.

4.3.1 WASTE TREATMENT BY CHEMICAL PRECIPITATION

This technology option is again similar to the other chemical precipitation options mentioned previously, except that the precipitated sludge would be hauled away as a RCRA waste. To reduce the volume of material hauled as sludge, sludge dewatering and drying could be instituted. After metal precipitation, the supernatant liquid could be discharged to IWTP No. 2.

Waste acid solutions could be neutralized with waste alkaline solutions to minimize chemical requirements. Separate holding tanks could be purchased to accumulate these solutions prior to mixing.

As with the previous chemical precipitation options, the precipitation method and the volume of sludge produced will vary with the solution bath constituents. Because of the varied precipitant materials and equipment necessary to make a workable system, and because of the labor intensive nature of the technology, this option should be considered as a "last resort" for treatment alternatives.

4.3.2 WASTE VOLUME REDUCTION BY EVAPORATION

Reducing the volume of waste process solutions via closed-loop evaporation is a simple and effective technology which is already in use at the electroplating facility. A cadmium recovery unit is in place to concentrate rinsewater solutions containing cadmium. In this existing system, the recovered cadmium is returned to process plating baths.

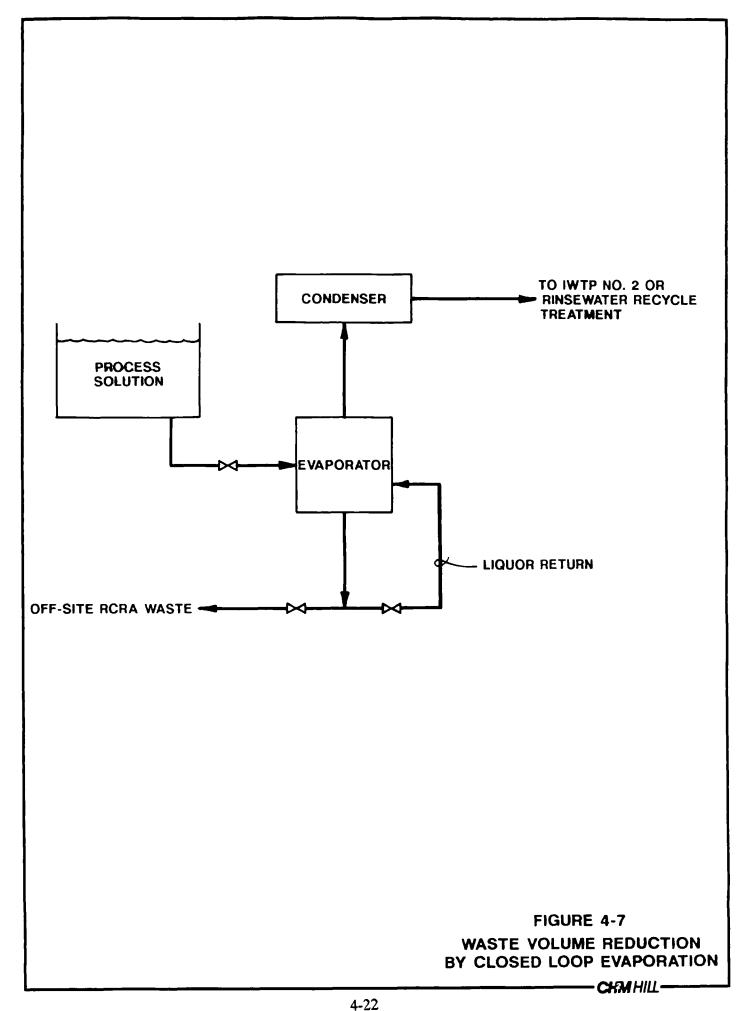
If off-specification process solutions are concentrated by evaporation, it would not be advisable to return the concentrated solutions to plating baths because contaminant metals and other undesirable impurities would also be concentrated and returned with the valuable metal. Therefore, the concentrated waste solution would be disposed of as a RCRA waste. The process solution could be continually recycled in the evaporation unit until maximum concentration was achieved. Depending on the types of additives in the process solutions, the overhead distillate may be usable as recycle for rinsewater, or disposed of at IWTP No. 1. A typical process diagram is shown in Figure 4-7.

Advantages of this method are that all process solutions are amenable to volume reduction by evaporative concentration, and a single evaporation unit can handle a variety of waste streams if construction materials are chosen properly.

4.3.3 TREATMENT BY BLEEDING INTO IWTP NO. 2

As mentioned previously, IWTP No. 2 has no capacity to treat metals except for chromium. Metals currently discharged to IWTP No. 2 flow to IWTP No. 1 where they are treated by classical precipitation methods. Concentrated solutions containing metals are not allowed to be discharged to IWTP No. 2 because of the potential for a "slug" of concentrated metals solutions to be discharged to IWTP No. 1, thus causing problems with the metals precipitation unit.

IWTP No. 1 has the capacity to co-precipitate metals of concern in the process solution baths. An alternative would be to slowly bleed concentrated process solutions into IWTP No. 2 for pH adjustment and/or cyanide destruction. The equalization of relatively small volumes of process solutions with large flows of rinsewaters could effectively even out the metals concentrations to IWTP No. 1 and eliminate potential upsets.



The advantage of this alternative is that it could be implemented immediately with little or no capital expenditures. According to Mr. Ron Eiler of the Sacramento Regional Sewer District, this concept is acceptable to the district as long as the discharge limits from IWTP No. 1 are not violated.

The disadvantage of bleeding concentrated solutions into IWTP No. 2 is that it would be diluting a concentrated stream in IWTP No. 2 only to again concentrate the metals in a sludge at IWTP No. 1 (not a preferred engineering solution). The volume of sludge generated at IWTP No. 1 would increase due to the additional metals loading from the concentrated solutions. The exact quantity increase in sludge production cannot be quantified at this time and will depend on the frequency and concentration of bath bleeds; however, the potential increase in sludge at IWTP No. 1 (also a RCRA waste) could offset the potential benefit of avoiding disposal of the concentrated solution bath as a RCRA waste.

Another potential disadvantage of bleeding concentrated solutions into the IWTP is the possible regulatory perception by EPA and the Department of Health Services that the base is trying to circumvent hazardous waste disposal regulations by the process of dilution. This is not the case because IWTP No. 1 has a metals precipitation unit to remove the metals of concern; however, the potential for misconceptions still exists. Modifications to permits and close regulatory liaison should be considered if this option is contemplated further. This option should be considered only as an interim method for RCRA waste reduction until more effective waste minimization options can be implemented.

Another similar option that has been discussed with base personnel would be to transport concentrated solution baths directly to IWTP No. 1 via drums or tanker trucks, thus bypassing IWTP No. 2 and the industrial waste line. Methods to bypass the industrial waste line are considered advantageous because of a current consent agreement that requires the cessation of use of this line by 1992. In studying this option, it would be difficult to treat the concentrated solutions directly at IWTP No. 1 without first going through treatment processes that would normally be performed at IWTP No. 1 (cyanide destruction, pH adjustment). Unless these unit processes are performed in a batch manner prior to transport to IWTP No. 1, the option of direct discharge to IWTP No. 1 is not recommended.

4.4 APPLICATION OF WASTE MINIMIZATION TECHNOLOGIES TO MAJOR WASTE STREAMS

This portion of the report applies the appropriate waste reduction technologies explained above to each of the 10 major waste streams. Note that the formation of a Process Solution Maintenance Team is a "technology" mentioned under

Source Reduction. This recommendation applies to all process solutions, although not mentioned specifically under the following discussions of individual waste streams.

4.4.1 CHEM MILL ETCHING SOLUTION

4.4.1.1 Source Reduction Technologies

The electroplating facility should consider discontinuing air sparging in this tank and providing recirculation pumps for agitation. Continuous filtration of this bath is not recommended because of the high solids content of the bath and the potential for clogging of the filters. Carbonate sludge buildup, in combination with hydrated aluminum scale deposits, is thought to cause poor etching performance, and the above-mentioned measures should reduce sludge buildup in the tanks. Martin Marietta, Inc. has switched from using a preblended etching solution to mixing its solutions in-house. Depending on the usage rate of the etching solution at G bay, this may be economical.

A specialized in-tank bath maintenance system developed by Eco-Tech of Canada is specifically designed to continuously purify components in an aluminum chemical milling bath. The system is designed to continuously remove aluminum and sulfides from the plating bath in the form of crystals (which may be re-sold) and simultaneously regenerate sodium hydroxide. Literature on the Eco-Tech system is provided in Appendix C. The main problem with this system is that it has to operate continuously. If the milling bath is dormant for a period of time, the system will stop functioning, and it can take a significant amount of time for the system to be re-started and reach equilibrium. Solutions require long residence times which makes equipment relatively large, floor space requirements high, and capital/maintenance costs relatively large for this system. The Eco-Tech system is not recommended for the chemical milling bath at the electroplating facility.

Another in-tank purification system involves the addition of sodium gluconate (a chelating agent) to the etching solution to keep hydrated aluminum scale from forming on tank walls. The sodium gluconate keeps the aluminum soluble and in solution. These so called "extended life" etch baths require fairly high work throughput rates to induce drag-out of the soluble aluminum. Fresh etching solution must also be periodically added to replace the drag-out losses. Information on this purification method is also given in Appendix C. Another drawback of this purification technology is that if the bath is ever deemed to be irretrievably spent, the chelating agents in the bath may upset waste treatment processes at IWTP No. 1 if that route of disposal is available. It is recommended that this bath maintenance technology be deferred until the results of the replacement of air sparging with pump mixing can be determined.

4.4.1.2 Waste Recovery and Reuse Options

Batch purification by chemical addition is possible with the Chem-mill solution tank. The excess aluminum can be precipitated by dilution and cooling of the solution. The aluminum hydroxide precipitate can then be decanted from the supernatant liquid. If the pH of aluminum hydroxide sludge and any precipitated carbonates is less than 12.5, the solids may not be considered a hazardous waste because aluminum is not toxic. The sludge may be disposed of as a non-regulated waste material. The presence of sulfides in the sludge may cause it to be regulated as a "reactive waste."

The supernatant liquid could be returned to the milling bath and replenished with fresh chemicals.

4.4.1.3 Waste Treatment/Volume Reduction Technologies

Chemical precipitation of metals by either the method mentioned in the previous section or sulfide oxidation and neutralization methods could be performed. After chemical precipitation, the supernatant liquid could be discharged directly to IWTP No. 2. However, an even more expedient alternative would be to bleed waste solutions directly into IWTP No. 2 with no pretreatment. Aluminum is not a toxic heavy metal, and the current waste discharge permit from IWTP No. 1 does not specify limits for aluminum. By bleeding the alkaline solution to the sulfide oxidation tank at IWTP No. 2 (which needs to be fixed), followed by pH adjustment, the effluent quality should be acceptable to IWTP No. 1. No RCRA waste would be produced by this method, and aluminum should co-precipitate with other metals at IWTP No. 1. It should be remembered that if any chelating agents are added to the Chem-mill solution, as mentioned under Source Reduction Methods, then sludge flocculation processes at IWTP No. 1 may be adversely affected. The chelates can be "broken" by the addition of proprietary chemical additives.

Concentration methods using evaporation are doubtful. The volume of Chem-mill etchant solution baths is rather large, and it may take significant amounts of time, or large evaporator capacity to reduce the waste volume by this method. Also, the solution contains a fairly high amount of dissolved solids which may hamper evaporation equipment.

4.4.2 ELECTROLESS NICKEL PLATING SOLUTIONS

4.4.2.1 Source Reduction Technologies

A continuous bath maintenance procedure should allow extended service of the nickel plating solutions. This method is a proprietary formulation manufactured

by Witco Corporation to be used with the NIKLAD™ electroless nickel process. Most references recommend continuous replenishment and bath maintenance on electroless nickel baths because of their inherent instability.

The maintenance procedure, given in Appendix D, includes continuous filtration with a 5-micron filter unit. This is particularly important because a spontaneous decomposition reaction may be triggered by particulate matter from the atmosphere or from parts being plated. Because of this, CH2M HILL recommends removable tank covers in addition to continuous filtration as an added protection from particulate contamination. Because of the organic stabilizers in the bath, activated carbon filtration is not recommended. The filtration schedule should also help with a reported mold buildup problem in the plating tank due to the organic stabilizers.

The data sheet by Witco also gives specific testing and replenishment methods for keeping the solution within specified limits.

The bath maintenance procedure looks somewhat labor intensive; however, an automated chemical addition system developed especially for electroless nickel baths has been developed by Palmer Industries. Data on this system are also given in Appendix D. Because of the sensitivity of electroless nickel plating solutions to parameters such as pH, temperature, nickel concentrations, particulate matter, and agitation, maintaining this solution will be a challenge.

A current problem with electroless nickel baths is the tendency of the bath to plate nickel onto the sides of the tank walls. This may contribute to the bath becoming unusable. A impressed-current system developed by Palmer Industries (ENSTAT^m) can be used to alleviate this problem. A data sheet on this impressed-current system is given in Appendix D. This system may also eliminate the nitric acid tank-stripping waste mentioned in Section 4.4.8; however, it would be necessary to replace the existing plastic-lined tank with a stainless steel one. This type of impressed-current system has been used by McClellan AFB in the past.

4.4.2.2 Waste Recovery and Reuse Options

Because of the inherent instability of electroless nickel plating solutions and the large amount of specialized reaction agents present in the bath, methods for waste recovery and reuse using batch purification by chemical addition, ion exchange or electrodialysis methods with subsequent return of the plating solution to the plating tank could not be found.

This is because as the nickel solution is used, a buildup of a variety of reaction by-products such as orthophosphite, hypophosphite, and sulfate is formed. The bath maintenance procedures mentioned earlier help to reduce the effect of these reaction by-products; however, removing them from solution using batch purification processes would probably cause spontaneous decomposition of the bath.

Witco has developed a metals recovery system using proprietary chemical additives which break down the chelating agents and allow the nickel to plate out to very low levels. This metal plate-out system is given on data sheets in Appendix D. This method is very similar to electrowinning, but no electric current is used. Specialized chemicals are added, along with a high surface area substrate such as steel wool, and the nickel plates onto the steel. The resulting nickel plated wool may be sold as scrap or disposed of as a nonhazardous waste, or nickel may be stripped from the wool in a separate tank and recovered in pure metal form by ion exchange. This system claims to remove nickel concentrations to the 5 to 10 ppm range. Supernatant liquid from the above process could be bled to IWTP No. 2. No additional chelate destruction should be necessary on the supernatant liquid, but this should be verified prior to implementation of this technology. This metals recovery/treatment system looks very promising from both a cost and operational standpoint.

Classical induced current electrowinning is an option for recovery of nickel from plating baths; however, the chelating agents would need to be destroyed before electrowinning to allow the nickel to plate out on the cathode. Lancy International, Inc. supplies an electrowinning system especially designed for chelated nickel plating solutions. A vendor brochure for this system is given in Appendix D. Because of the potential advantages of the chemical plate-out method, induced current electrowinning is not recommended for the electroless nickel bath at this time unless this stream is combined with other nickel-bearing wastes.

4.4.2.3 Waste Treatment/Volume Reduction Techniques

Witco has developed a spent NIKLAD^m electroless nickel batch treatment to remove nickel from the waste stream, with disposal of the precipitate as a RCRA waste, directing the supernatant liquid to IWTP No. 2. Witco claims that this process can reduce nickel content in the supernatant liquid to less than 1 ppm. A process data sheet from Witco is supplied in Appendix D.

The waste treatment system is a two-step process involving precipitation of nickel, followed by flocculation to produce a sludge. Different chemical additions are required for each step, but only one tank (the original plating tank) is required. Sludge produced from this process would be a RCRA waste, and sludge dewatering and drying may be possible to reduce the amount of waste sent to RCRA disposal sites.

Evaporation and concentration of spent nickel plating baths is a feasible alternative; however, this technology is not as promising as the Witco process mentioned above.

Bleeding the spent nickel baths into IWTP No. 2 could be performed as long as the chelating agents were destroyed beforehand. The nickel bath could go through pH adjustment, and the nickel metal would eventually be treated and coprecipitated with other metals at IWTP No. 1. Bleed-in rates for the nickel solution were not determined in this report and would depend on the IWTP No. 2 plant flow rates at the time of mixing; however, a 500-gallon batch of spent nickel bath should be able to be disposed of within a period of days.

4.4.3 ELECTROLESS NICKEL STRIPPING SOLUTIONS

NOTE:

Technologies mentioned under this section are for the ABCO™ nickel strip solution. CUPOSIT™ solution is used as an alternate but contains a different formulation. Some or all of the technologies mentioned for the ABCO™ product cannot be applied to the CUPOSIT™ material because of the differences in their formulation.

4.4.3.1 Source Reduction Technologies

The ABCO™ stripping product is an alkaline formulation containing sodium hydroxide and a mixture of proprietary organic chemicals. Continuous filtration and periodic alkaline addition are the only bath maintenance procedures recommended by the manufacturer. This solution also contains organic stabilizers, and activated carbon filtration is not recommended. It is recommended that air sparging be discontinued for agitation of this solution, and in-tank filtration/circulation pumps be purchased to reduce sludge buildup in the tanks. A data sheet on the ABCO™ product is given in Appendix E.

4.4.3.2 Waste Recovery and Reuse Options

Batch purification to remove contaminant metals or metals recovery options for this waste stream were difficult to determine because of its severely alkaline nature. Both ion exchange and electrowinning technologies are more applicable to a slightly acidic waste stream; however, pH adjustment would not allow the bath to be returned to the process. Ion exchange also works well only on dilute solutions. The nickel content of this waste is very high (8-10 percent).

One alternative for metals recovery would be to dilute the alkaline stripping solution with nickel rinsewaters and run the solution through a cation exchange resin. The effluent, now stripped of metal cations, could be directed to IWTP No. 2 for further treatment.

During acid regeneration of the resin with acid, the concentrated regenerant containing the cations (nickel included) could be sent to an electrowinning system. This stream may be too acidic to electrowinning directly, so pH adjustment to approximately 6 may be necessary. The solution could be electrowinned of the nickel, and the effluent either directly discharged to IWTP No. 2 or recycled back

to the IX unit for another pass. This methodology is similar to the process flow diagram given in Figure 4-5, except the electrowinning influent would need pH adjustment prior to initiating electrowinning activities.

Another alternative would be to direct nickel rinsewaters only to an ion exchange system, with the forward flow directed to IWTP No. 1. The acid regeneration containing the metals would combine with the alkaline nickel strip waste in a holding tank. The effluent from this tank should be much less basic than the original alkaline stripper and could be electrowinned directly. This is a similar process to that shown in Figure 4-6. Care would have to be taken not to precipitate the nickel as a hydroxide sludge during the neutralization process.

The advantage to the latter methodology is that a secondary pH adjustment step is eliminated. A potential disadvantage is the possible presence of chelating agents in the stripping bath that would interfere with electrowinning operations.

4.4.3.3 Waste Treatment/Volume Reduction Techniques

The data sheet given in Appendix E gives a batch treatment method for spent nickel stripping solutions by pH adjustment and treatment with sodium hypochlorite. This causes the nickel to precipitate as nickel oxide, a much more easily handled solid material than a nickel hydroxide sludge. The data sheet states that after treatment by this method, the resultant supernatant liquid is "completely biodegradable." The data sheet does not specify quantities of hypochlorite necessary to precipitate the nickel, and manufacturers' representatives were not helpful in this regard.

Another batch treatment method given by ABCO™ is precipitation of nickel by forming nickel hydroxide through dilution and pH adjustment. As mentioned previously, metal hydroxide sludges are difficult to work with in the electroplating facility, and this method of treatment is not recommended unless other alternatives fail.

Volume reduction by evaporation is feasible for this waste stream; however, the distillate may not be usable for recycle to the rinse water tanks because organic agents may be carried over in the evaporation process.

Bleeding the solution to IWTP No. 2 is worth considering for this waste stream. Before proceeding with this option, it should be determined if the "unspecified organic constituents" contain any chelating agents that would cause problems at IWTP No. 1.

4.4.4 CHROME STRIPPING SOLUTION

4.4.4.1 Source Reduction Technologies

Replacement of air sparging with continuous filtration with in-tank pump/filter units should be implemented for this process solution. Continuous or periodic carbon filtration should also be considered. Because the solution operates at ambient temperature, heating/cooling controls should be installed to regulate the bath temperature. As mentioned in Section 4.1.2, an alternative to in-bath temperature controls would be to devise a method to more accurately regulate air temperature in the electroplating facility.

IONSEP™ manufactures a continuous unit to purify alkaline chrome stripping baths. The unit is based on the principle of electrodialyses described earlier and regenerates sodium hydroxide for return to the stripping bath. The chromium contained in the bath is separated by the electrodyalitic cell and is converted to chromic acid which can be used in the chromium plating tanks. Vendor literature on the IONSEP™ system is given in Appendix F.

This IONSEP™ system has been installed in industrial shops, but its long-term effectiveness is not known. Further vendor contacts should be made as to the long-term feasibility of IONSEP™ to this application. If the bath maintenance procedures outlined in Section 4.4.4.1 are not successful in reducing the volume of wastes produced, this IONSEP™ system should be investigated more fully.

4.4.4.2 Waste Recovery and Reuse Options

Because of the alkaline nature of the waste and the high concentration of hexavalent chromium, no waste recovery and reuse options can be recommended for this waste stream.

4.4.4.3 Waste Treatment/Volume Reduction Techniques

The hexavalent chromium can be reduced to the trivalent (insoluble) state by pH adjustment and reduction with a strong reducing agent such as sulfur dioxide gas. This process is used at IWTP No. 2 for chrome reduction, and it would not seem cost-effective to construct a small batch unit to treat only this bath.

The most expedient solution to the disposal of this solution as a RCRA waste would be to bleed the solution into the chrome reduction system at IWTP No. 2. This could be accomplished by bleeding the alkaline solution into the Miscellaneous Base Sump (T-5) or the Miscellaneous Sump (T-3) and diverting the flow from these sumps to the chrome reduction system. This system would convert the chromium to the trivalent state and discharge it to IWTP No. 1 for precipitation.

The spent bath could also be combined with the chrome recovery system by bleeding the bath into the Chrome Rinse Sump (T-7) or the Chromic Acid Sump (T-8); however, pH adjustment of the alkaline solution would probably be necessary prior to discharge to either of these sumps. The benefit of this option is that the chrome in the stripping solution would be recovered rather than discharged to IWTP No. 1.

Volume reduction by evaporative concentration is viable for waste minimization for this waste stream. Overhead streams may be suitable for rinsewater recycle after demineralization, and the concentrate could be disposed of as a RCRA waste.

4.4.5 CADMIUM PLATING SOLUTIONS

4.4.5.1 Source Reduction Technologies

Continuous particulate filtration with periodic carbon filtration is recommended for this plating bath. The removal of air sparging and thermostatically controlled temperature regulation are also recommended.

McClellan has recently purchased IVD aluminum equipment to substitute for a portion of the cadmium plating in the facility. Although this will not eliminate the production of cadmium-related plating wastes, it may significantly reduce the volume generated.

4.4.5.2 Waste Recovery and Reuse Options

Lancy International, Inc. supplies an electrowinning system designed for cyanide-bearing metal plating baths. This system, given in Appendix G, can recover cadmium metal while simultaneously destroying cyanide complexes. The effluent from this electrowinning system could be discharged to IWTP No. 2, or possibly sent to the cadmium recovery system installed in the electroplating facility. It should be noted however that the cadmium recovery unit would concentrate contaminants in the bath that were the cause of its original red-line condition.

4.4.5.3 Waste Treatment/Volume Reduction Technologies

The existing cadmium recovery system could be used to reduce the volume of waste from this waste stream; however, it is not recommended that the concentrated cadmium solution be returned to process baths because this practice would return other contaminants also.

Batch evaporation of cadmium with disposal of the concentrate is viable, especially because a cadmium evaporation system is already in place.

Bleeding the waste to IWTP No. 2 is possible. However cadmium is a highly toxic metal with low discharge requirements. This option should be closely studied before implementation.

4.4.6 SILVER STRIPPING SOLUTION

4.4.6.1 Source Reduction Technologies

Continuous particle filtration and periodic carbon filtration are recommended for this process bath. Because the bath is alkaline and operates at ambient temperature, temperature control measures outlined earlier are recommended for the bath.

4.4.6.2 Waste Recovery and Reuse Options

The Lancy Electrowinning system for cyanide baths mentioned for the cadmium plating waste stream could be applicable; however, the stream contains mixed metals and the potential success of electrowinning technologies to this waste is not likely.

4.4.6.3 Waste Disposal/Volume Reduction Technologies

Bleeding this waste stream to IWTP No. 2 via the cyanide sump would seem to hold the best potential for this low-volume waste stream. Evaporation could also be considered as an option.

4.4.7 ACID PICKLING SOLUTION

4.4.7.1 Source Reduction Technologies

Continuous particulate filtration and periodic carbon filtration are recommended for this process solution.

An acid purification unit manufactured by Eco-Tech, Inc. uses a reciprocating ion exchange unit to continuously purify metal cations from acid pickling baths. This system can be used continuously to treat the baths or can be used on a batch basis.

The reciprocating ion exchange unit purifies the acid and returns it to the pickling bath. Regenerants containing the metal cations could be directed to IWTP No. 2 for treatment, or sent to an electrowinning system. Appendix H gives vendor information and typical process flowsheets for this technology. The APU/DCU system looks very promising not only for this particular waste stream, but for other acidic stripping or pickling waste streams.

The IONSEP™ process could be applicable to this technology. This process would remove contaminant metal cations as a hydroxide sludge and regenerate the acid. This option would be preferred if effluent from an APU unit could not be electrowinned or further processed into a useful product.

4.4.7.2 Waste Recovery/Reuse Options

Batch treatment using an APU/DCU system could be used after the bath has become excessively contaminated. One or two APU/DCU systems in a portable configuration could be used to batch treat this and other acidic stripping and pickling baths.

Direct electrowinning or other metals recovery technologies for the spent bath are not recommended for this waste stream because no one metal is predominant in the spent solution. Also, the levels of contaminant metals present (approximately 800 ppm maximum) are not concentrated enough to allow electrowinning technologies to be effective.

It may be possible to use the concentrated regenerant produced from the APU/DCU unit to recover metals by electrowinning; however, the mixed metal content of this solution would make electrowinning of questionable feasibility.

The solution could be combined with other cadmium baths mentioned earlier, except those containing cyanide that have not undergone cyanide destruction, and sent to an ion exchange/electrowinning system. The effectiveness or advisability of this alternative depends on the disposition of other cadmium-containing waste streams.

4.4.7.3 Waste Treatment/Volume Reduction Technologies

Batch chemical precipitation was not deemed appropriate because of the large volume of neutralization chemicals necessary for this highly acidic waste stream and because of the relatively low metals concentration in the waste.

Bleeding the solution into the acid sump and allowing discharge to IWTP No. 2 is a viable option for this waste because the volume of spent solution would only be approximately 300 gallons at a time and because the concentration of heavy metals is fairly low and would require less flow dilution to avoid upsets at IWTP No. 1.

It may also be feasible to mix this acidic waste with other alkaline wastes in a batch mode for neutralization and precipitation of metals. However, as mentioned in Section 4.3.1, the hydroxide sludges generated would be difficult to handle, making this method of treatment somewhat labor-intensive.

4.4.8 NITRIC ACID WASTES FROM ELECTROLESS NICKEL PLATING TANK CLEANING

4.4.8.1 Source Reduction Technologies

As mentioned in Section 4.4.2, the use of nitric acid to strip plated nickel off of the plating tank may be discontinued by use of the Enstat technology developed by Palmer Industries, Inc. Another method of source reduction that could be easily implemented would be to recycle the nitric acid bath back to the storage tank and use it several times before discarding.

An APU/DCU unit could be installed to remove nickel from the nitric acid solution on a continuous or batch basis. The waste regenerant from this process could be combined with waste concentrates from the electroless nickel plating tank and the electroless nickel stripper solutions to provide an excellent feedstock to an electrowinning system.

An IONSEP™ system could also be used to remove metals as hydroxide sludges while purifying the acid.

4.4.8.2 Waste Reuse/Recovery Options

Batch treatment with an APU/DCU unit is an option for regenerating a spent bath from this waste stream. Regenerant could be sent to an electrowinning system, bled to IWTP No. 2, or disposed of as a RCRA waste.

Electrowinning the bath directly could be performed; however, it may not be costeffective to do so. It would be more effective to concentrate the nickel in the solution and combine it with other concentrated nickel solutions prior to electrowinning.

4.4.8.3 Waste Treatment/Volume Reduction Techniques

Batch precipitation is not recommended for this waste stream because of the high acidity and low volume of the waste stream. Mixing the waste with other alkaline wastes for neutralization and precipitation is possible, but not preferred because of the reasons mentioned in Section 4.3.1.

Evaporative recovery is not recommended because of the high acidity of the waste, requiring special materials of construction for the evaporator system.

Treatment by bleeding into IWTP No. 2 via the Miscellaneous Acid Sump (T-6) could be easily accomplished.

4.4.9 ALKALINE DERUST SOLUTION

4.4.9.1 Source Reduction Technologies

Particulate filtration and periodic carbon filtration are recommended for this bath because of the reported buildup of sludgy solids in the alkaline derust tank. The bath should also be decanted if sludge buildup becomes apparent.

An ultrafiltration system developed by Lancy Technologies that is especially developed for maintaining high-temperature alkaline baths may be useful in maintaining the bath within specifications. Vendor literature on this system is given in Appendix H. Although the ultrafiltration system will not remove metal cations, it can help to keep entrained oil, grease, and particulates out of the bath.

4.4.9.2 Waste Reuse/Recovery Options

This waste stream is alkaline and contains relatively low concentrations of mixed metals. As such, it is not amenable to batch precipitation, ion exchange, or electrowinning.

4.4.9.3 Waste Treatment/Volume Reduction Techniques

Evaporation of the waste stream with off-site disposal of the concentrated solution is a viable option for this waste stream; however, the "dirty" nature of the waste may cause problems with evaporation equipment.

The best treatment option for the waste would be to bleed the solution to IWTP No. 2 via the Miscellaneous Base Sump (T-5).

4.4.10 NICKEL STRIP SOLUTION

4.4.10.1 Source Reduction Technologies

Continuous particulate filtration and periodic carbon filtration of this solution are recommended to maintain bath life. An APU/DCU unit could be used to remove contamination metal cations (mainly nickel and copper), with the regenerated acid returning to the process and the cations discharged to an electrowinning system or to IWTP No. 2.

An IONSEP^m system could be used to remove metal cations as a hydroxide sludge, and regenerate the stripping bath.

4.4.10.2 Waste Recovery and Reuse Options

An APU/DCU unit could be used on a batch treatment basis to rejuvenate the contaminated nickel stripping solution. Regenerant could be combined with other concentrated nickel-bearing waste streams for electrowinning.

Direct electrowinning or direct ion exchange is not appropriate because of the fairly low metals concentrations in the waste stream.

The waste stripping acid could be diluted with nickel-bearing rinsewaters and reconcentrated in an ion exchange column. The regenerant from this column could then be electrowinned with metals from the process bath and the nickel-bearing rinsewaters recovered.

4.4.10.3 Waste Treatment/Volume Reduction Technologies

This waste solution could be bled to IWTP No. 2 via Sump T-6; however, nickel is a regulated metal in the IWTP No. 1 discharge, and the bleed-in rates should be carefully controlled. Nickel precipitation by metal hydroxide treatment is also difficult.

Evaporative concentration of this solution is possible; however, the waste is extremely acidic and special equipment would be necessary to account for the highly corrosive nature of the waste.

4.4.11 MISCELLANEOUS ACID STRIPPERS

4.4.11.1 Source Reduction Technologies

Bath maintenance procedures should be instituted as described in previous sections.

A portable APU system could be purchased to purify acidic waste streams, with the regenerant sent to IWTP No. 2, to an electrowinning system, or to disposal as a RCRA waste. The regenerant streams from acid strippers will, in general, produce mixed-metal streams not suitable for electrowinning applications.

An IONSEP™ system may be the best choice for a portable unit to purify acid baths because the metals are removed as a hydroxide sludge, not as a high-volume regenerant. IONSEP™ units are produced as a portable system for such applications.

4.4.11.2 Waste Recovery/Reuse Options

The APU and IONSEP™ systems mentioned above, operated on a batch basis, would qualify under such options.

Because the metals contained in acid stripping solutions are mixed, potential recovery of salable metal products is not considered feasible at this time.

4.4.11.3 Waste Treatment/Volume Reduction Technologies

Bleeding waste acid stripping solutions into IWTP No. 2 via Sump T-6 is a possible option. The mixed metal concentrations are usually relatively low in these wastes, and IWTP No. 1 could possibly handle the increased metal loading from periodic acid stripper bath discharges.

Evaporative concentration is not recommended for these types of wastes because of their severely corrosive nature.

Batch precipitation of metals is not recommended for these waste streams because of the high amount of caustic necessary to neutralize the waste, and the equipment necessary for sludge precipitation and dewatering.

4.4.12 ALKALINE CLEANERS

4.4.12.1 Source Reduction Technologies

Alkaline cleaning solutions are difficult to deal with because the metals in these solutions are not in the form in which recovery or continuous removal is viable.

Removal of air sparging equipment and replacing with pump mixing and continuous filtration are procedures that should be implemented for all alkaline baths.

The Lancy Ultrafiltration system given in Appendix I appears to be an acceptable method of removing impurities (other than metal contaminants) from alkaline cleaning baths, thus extending their life. The ultrafiltration system could be purchased so that more than one alkaline bath could be treated in a batch mode.

Palm Instruments also manufactures an automatic alkaline cleaning bath control system that automates the replenishment of chemicals to the solutions. The controller can replenish more than one bath. Information on this controller is given in Appendix J.

4.4.12.2 Waste Recovery and Reuse Options

The batch treatment using ultrafiltration is one method to purify alkaline cleaning baths and return them to the process. As previously mentioned however, this method will not remove contaminant metals.

Metals recovery is not generally applicable for alkaline cleaning baths unless they could be combined with other waste streams and the combined stream sent to an electrowinning system.

4.4.12.3 Waste Treatment/Volume Reduction Technologies

Concentrative evaporation is technically feasible, but not economically attractive for waste volume reduction of alkaline cleaning solutions. The large energy input to significantly reduce the waste volumes from these solutions would make this option low in priority.

Bleeding waste alkaline solutions into Sump T-5 and allowing them to be treated through IWTP No. 2 would be an attractive alternative. The metals concentrations in these solutions are relatively low, and treatment in IWTP No. 1 should not be affected if the dilution flows are properly calculated. This option could be an interim waste minimization step until more rigorous methods are instituted in the electroplating facility.

SECTION 5

SUMMARY OF WASTE MINIMIZATION STRATEGIES AND PREFERRED ORDER OF IMPLEMENTATION

Section 5

SUMMARY OF WASTE MINIMIZATION STRATEGIES AND PREFERRED ORDER OF IMPLEMENTATION

Table 5-1 summarizes all technologies mentioned for each major waste stream considered in this report. The order of implementation of a technology for a given waste stream is a combination of several factors:

- The hierarchy of the technology, e.g., source reduction would rank higher than waste treatment options
- The capital cost outlay for a technology, e.g., bath maintenance techniques would rank higher than buying equipment to reduce waste volumes
- The ability of the technology to minimize or treat the waste stream, e.g., IONSEP™ technology may be more effective for a given waste stream than electrowinning, although both cost approximately the same and are in the same hierarchy.
- The time it would take to implement the technology, e.g., bath maintenance techniques should be tried first; if this fails to minimize waste volume, then specific technology insertions may be necessary as a secondary measure

Table 5-1 shows that, in general, bath maintenance should be instituted for all waste streams immediately. As stated previously, this is the best methodology to minimize waste production in the shortest time and with the minimum capital outlay. Some waste streams may require additional measures as the results of the bath maintenance program on waste production become apparent.

The drawback to considering waste minimization technologies on a waste-by-waste basis is that several waste streams can be combined for metals recovery and reuse options. A good example is the nickel waste streams produced at the electroplating facility. Nickel strippers, electroless nickel plating baths, and other nickel-bearing waste streams could be combined such that technologies not applicable to the individual wastes are now applicable to the combination of the wastes.

Figure 5-1 shows a possible nickel recovery system for the combined nickel rinsewater and process solutions. As mentioned previously, Metcalf & Eddy is currently investigating ion exchange recovery of metals at IWTP No. 2. If this process is included for nickel-bearing waste streams, then process solutions could be introduced into the system, and metals recovery via electrowinning technology could become a viable option.

TABLE 5-1 WASTE MINIMIZATION TECHNOLOGY SUMMARY TABLE

TO VOY OF C	ज्ञ⊪	-
	٠,	GAL. FILTER
4		5500 NA
SOLUTION NO LONGER USED		SOFF
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		500
		1440
		550
	_	009
NA		
	_	337
1	_	840
1	_	450
_		Varies
-	_	Varies

KEY TO TABLE

^{1 =} RECOMMENDED FOR IMMEDIATE IMPLEMENTATION

^{2 -} RECOMMENDED FOR IMPLEMENTATION ONLY IF ALT. 1 DOES NOT SIGNIFICANTLY REDUCE WASTE GENERATION

^{3 =} TECHNOLOGY MAY BE APPLICABLE, BUT PILOT WORK SHOULD PRECEED IMPLEMENTATION NA = TECHNOLOGY IS NOT APPLICABLE FOR THIS WASTE STREAM, OR HAS A HIGH CHANCE OF FAILURE

TABLE 5-1 WASTE MINIMIZATION TECHNOLOGY SUMMARY TABLE (Continued)

					WASTE RI	WASTE RECOVERY AND REUSE	REUSE
GENERAL PROCESS	WASTE STREAM/PROCESS	TANK NO.	TANK	BATCH	DECANT	ION EXCHANGE	ELECTRO- DIALYSIS
Aluminum Etching	Chem Mill Etchant	137	5500	2	-	NA V	NA
Chromic Plating	Nickel Strip (CN)/NOT PRoc	171	1	SOLUTION	SOLUTION NO LONGER USED	R USED	
Electroless Nickel	Electroless Nickel Strip	202	1000	N A	A V	က	က
Electroless Nickel	Electroless Nickel Plate	193	200	က	AN A	N N	NA A
Chrome Plating	Alkaline Chrome Strip	158	1440	က	က	က	A A
Precious Metals	Silver Stripping	က	550	¥	¥ Z	က	က
Cadmium Plate	Cadmium/NACN	2	009	V V	ဗ	င	င
Electroless Nickel	Nitric Acid Clean Ni Tk	Lower Level		3	NA	3	3
Cadmium Plate	Acid Pickling	06	337	က	N A	ဧ	ဇ
Cadmium Plate	Alkaline Deruster	23	840	3	-	2	5
Precious Metals	Nickel Strip/H2SO4	2	450	¥ Z	N A	က	ဇ
Alkaline Cleaners in General	Various	Varies	Varies	က	-	NA NA	NA
Acid Strippers in General	Various	Varies	Varies	3	1	8	ε

KEY TO TABLE

1 = RECOMMENDED FOR IMMEDIATE IMPLEMENTATION

2 - RECOMMENDED FOR IMPLEMENTATION ONLY IF ALT. 1 DOES NOT SIGNIFICANTLY REDUCE WASTE GENERATTION

3 - TECHNOLOGY MAY BE APPLICABLE, BUT PILOT WORK SHOULD PRECEED IMPLEMENTATION

NA - TECHNOLOGY IS NOT APPLICABLE FOR THIS WASTE STREAM, OR HAS A HIGH CHANCE OF FAILURE

TABLE 5-1 WASTE MINIMIZATION TECHNOLOGY SUMMARY TABLE (Continued)

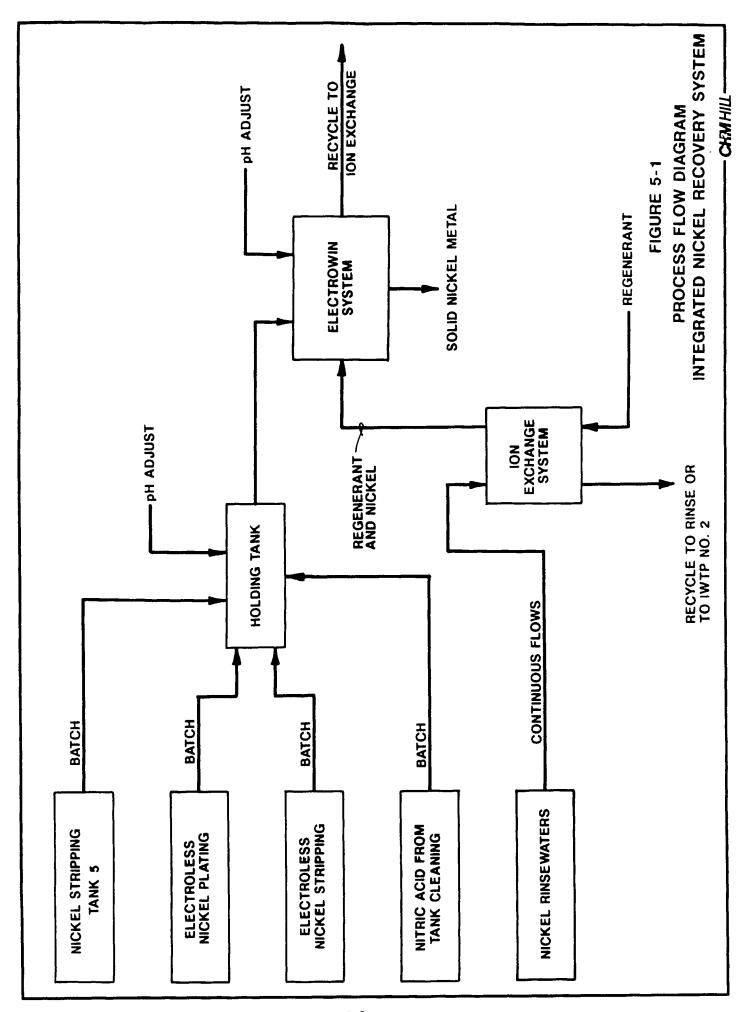
				METALS RECOVERY	OVERY	WASTE	WASTE TREATMENT/VOLUME	NOLUME
GENERAL PROCESS	WASTE:STREAM/PROCESS	TANK NO.	TANK GAL.	PRECIPI- ELE TATION	ELECTRO- WIN	PRECIPI- TATION	EVAPORA- TION	BLEED TO IWTP #2
Aluminium Etching	Chem Mill Etchant	137	5500	NA	Ą Z	2	NA	-
Chromic Plating	Nickel Strip (CN)/NOT PRoc	171				SOLUTION	SOLUTION NO LONGER USED	SED
Electroless Nickel	Electroless Nickel Strip	202	1000	3	က	-	ေ	2
Electroless Nickel	Electroless Nickel Plate	193	200	-	2	-	ဗ	2
Chrome Plating	Alkaline Chrome Strip	158	1440	NA	Y Z	6	e e	2
Precious Metals	Silver Stripping	6	550	¥ Z	2	က	es .	2
Cadmium Plate	Cadmium/NACN	\$	009	NA V	2	ဇ	ဇ	က
Electroless Nickel	Nitric Acid Clean Ni Tank	Lower		က	2	က	ဇ	2
Cadmium Plate	Acid Pickling	8	337	က	2	က	က	2
Cadmium Plate	Alkaline Deruster	73	840	Y Y	Y Y	က	ဇ	2
Precious Metals	Nickel Strip/H2SO4	S	450	က	2	က	6	2
Alkaline Cleaners in General	Various	A N	Varies	ဇ	VA	က	6	2
Acid Strippers in General	Various	A N	Varies	6	2	က	NA NA	2

KEY TO TABLE

1 = RECOMMENDED FOR IMMEDIATE IMPLEMENTATION

^{2 =} RECOMMENDED FOR IMPLEMENTATION ONLY IF ALT. 1 DOES NOT SIGNIFICANTLY REDUCE WASTE GENERATION

^{3 =} TECHNOLOGY MAY BE APPLICABLE, BUT PILOT WORK SHOULD PRECEED IMPLEMENTATION NA = TECHNOLOGY IS NOT APPLICABLE FOR THIS WASTE STREAM, OR HAS A HIGH CHANCE OF FAILURE



This is just one example of combining similar streams containing a valuable commodity and recovering that commodity. There may be other examples of combining waste streams at the electroplating facility that could result in similar metals recovery processes.

SECTION 6 BIBLIOGRAPHY

BIBLIOGRAPHY

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APPENDIXES

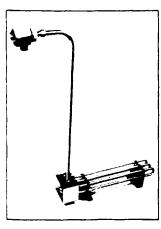
APPENDIX A

GLO QUARTZ IMMERSON HEATERS VENDOR INFORMATION



GLO-QUARTZ ... SPECIALIST IN ELECTRIC HEATING.



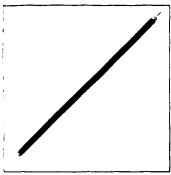


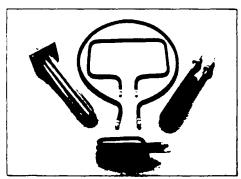




IMMERSION HEATERS

From heating a concentrated acid to 500°F to keeping oil flowing at -30°F. Glo-Quartz provides the right heater to meet your specific requirements. Both standard and custom designed units are available in a variety of sheath materials including copper, stainless steel, titanium, incoloy, quartz, teflon and various nickel alloys. Heater mounting options include square, round or rectangular flange mountings; 1" thru 3" NPT screw plug sizes and builchead fittings.



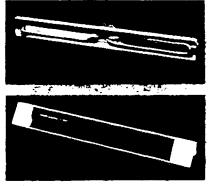


TUBULAR ELEMENTS

The most versatile of all heaters, these quality units are available in a variety of sheath materials and diameters. Tubular elements can be manufactured in lengths exceeding 20 feet and formed into a variety of standard or custom shapes. Bulkhead fittings and a variety of terminations are also available.







RADIANT HEATERS

Gio-Quartz offers a wide variety of radiant heaters with custom designed units available up to 12 feet in length. Using either a quartz tube, quartz lamp or metal sheath incolory element as the heat source. A wide variety of materials can be processed quickly and efficiently. We can also manufacture replacement heating elements for radiant panels or heating systems.









TEMPERATURE CONTROLS

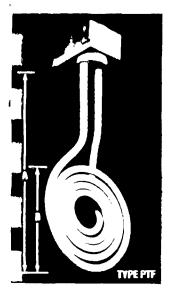
Every process heating application requires a suitable temperature control system. Glo-Quartz offers a wide partiety of temperature controls, from a low cost electronachanical thermostat to a solid state digital displacement. Let us help match the proper temperature controls to your heating system.

GLO-QUARTZ ... SPECIALIST IN TEFLON IMMERSION HEATING.

All Glo-Quartz Teflon* heaters consist of a heavy wall Teflon tube extruded over a grounded stainless steel heating element.

Shown below are some standard heater configurations. Special custom configurations. are available.

*Registered Dupont trademark

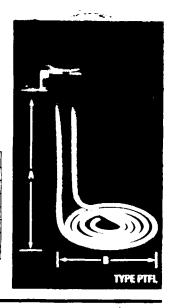


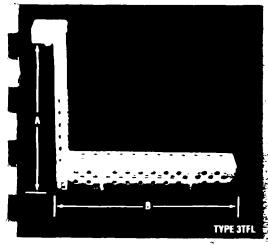
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bir 43.3	1	180		

SINGLE PHASE ONLY LONGER "A" DIMENSION AVAILABLE, POLYPROPYLENE OR TEFLON GLIARD AVAILABLE.

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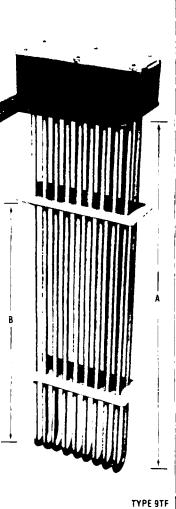
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APPENDIX B

FLO KING PUMP VENDOR INFORMATION



1320 Bennett Drive, Longwood, FL 32750 (407) 331-4634



Forty years or better, filtration system manufacturing has changed very little indeed. We are still experiencing seal leaks, "O" ring leaks from top cover of filter chamber, and at base of chamber due to faulty seal, or elsewhere, at joints and fittings.

Cavitation or starving of pump is also a concern, pressure drop across each filter cartridge in filter chamber, is also concern for sizing a filter system. You as the owner, plater, or engineer must ignore the cost of the filtration system, and advised it takes 1 H.P. motor and pump to filter the plating solution at 3000 GPH, in which the flow rate is sized for 1.0 Sp. Grv. WATER!!!

Why do you use this type of filter system? Because you are unaware that there is THE NEW CONCEPT of filtration, and you are content to filter a plating bath the same way, as was done forty years ago!!

THE NEW CONCEPT is FLO KING filter systems, which is 1/2 the cost of filtration systems available today!

FLO KING filter systems are unique, in that the negative side or suction end holds the filter cartridge. This puts a vacuum on the cartridge, eliminating channeling, pressure drop across filter cartridges, and utilizes each fiber of the media to its full potential. This results in cartridges holding 2 to 3 times more solids before plugging up, making in-tank filtration possible due to a much smaller pump and filter housing, eliminating all leaks and spills.

This unique method of filtration lead to the development of a NEW CONCEPT in filter media, the "MAGNUM" filter cartridge. The "MAGNUM" filter cartridge uses a special blanket made of polypropelene fiber, designed for conditions found in the plating industry. It has the ability to filter 100 micron down to 1 micron without plugging up. The "MAGNUM" can be cleaned and reused over 100 times without losing its filtration capabilities.

A 10 micron 10 inch depth tube filter cartridge has 3.5 sq. ft. of solids holding capacity. The "MAGNUM" has 35 sq. ft., ten times the holding capacity of the standard of the industry. The FLO KING model BX3000 filtration system has a 1/3 H.P. motor, holds two 20 inch "MAGNUM" cartridges and has a solids holding capacity of 140 sq. ft.

THE NEW CONCEPT is of course, the FLO KING filter system. Suction through the filter media and into the pump, which in turn discharges back into the vessel or plating tank giving the solution a counter-flow agitation, which in turn speeds up plating deposition on part being plated.

FLO KING media is excellent for hard chrome plating at $165^{\circ} \pm 5^{\circ}$, where polypropelene wound cartridges do not hold up. They are excellent for removing sludge from low temp. black oxide baths. With the high flow rate they are perfect for electroless nickel baths at 195° . The electronic industry is regarding FLO KING filter systems a must, for particulate removal in acid copper, copper pyro-phosphate, SolderOn, tin/lead fluoborate, nickel, gold, rhodium etc..

THE NEW CONCEPT for organic removal, is the FLO KING refillable carbon pack, and resin pack for gold recovery from rinse water following gold plate etc.

For more information about your operation please feel free to contact us and let us help you with your purification and filtering requirements.

THE MOST VERSATILE FILTER SYSTEM AVAILABLE

THE NEW CONCEPT

FLO KING COUNTER-FLOW FILTER SYSTEM



USING 4 1 IN-TANK FILTER SYSTEM

solves many oil, contaminant, agitation, and carbon treating problems for the following operations.....

PLATING SOLUTIONS

Keeps solution filtered while skimming action keeps solution surface free of oil, dust, etc.

ELECTROLESS METAL APPLICATIONS

High agitation with filtration. Increase life of heaters and solution while producing quality plating.

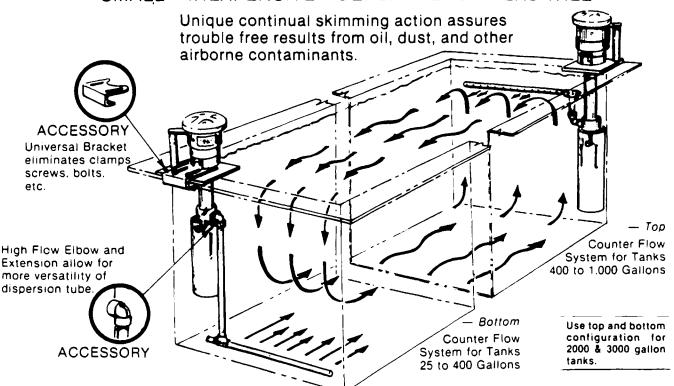
CLEANING SOLUTIONS

Doubles and Triples life of solution. Cuts cleaning cycle as much as ten times. Eliminates agitation bar.

ACTIVATING TANKS ... ETCHING SOLUTIONS ... RINSE TANKS PHOTO RESIST ... PHOSPHATE ... ELECTRO CLEANERS

PUMPS / FILTERS / AGITATES / TREATS

SMALL • INEXPENSIVE • DEPENDABLE • VERSATILE



Innovative Ideas for Industry Since 1964

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APPENDIX C

CHEM-MILL ETCHING VENDOR INFORMATION

ECO-TECH CRYSTALIZATION	C-1
ECO-TECH EXTENDED LIFE BATHS	C-1

Industrial Finishing

A MICHOCK PUBLICATION

Recovery of Aluminum Finishing Wastes

Recovery of Aluminum Finishing Wastes

The recovery of aluminum finishing wastes is particularly challenging because the value of many aluminum finishing chemicals, such as caustic and acid, is often quits low. The aqueous chemistry of aluminum also presents some unique problems.

Yet, despite these apparent draw-backs, recovery of aluminum finishing wastes in anodizing lines and other finishing processes is being done. Advantages of recovery include reduced process chemical use, less chemical destruct waste treatment requirements and a cutback in waste treatment sludge formation.

On-stream recovery of eluminum finlishing waste is being done with the following processes:

- austic etchina
- D phosphoric acid bright dipping
- ☐ sulfuric acid anodizing
- phosphoric acid anodizing
- ☐ Integral color anodizing
- ☐ electrolytic coloring
- **Chromating**
- nitric and hydrochloric acid electro-
- chemical etching
- water recycling

Caustic etching

Before anodizing, aluminum is usually etched in solutions of caustic soda to varying degrees, depending on the finish deared. In Japan, where a relatively shiny finish is common, the metal loss is in the order of 30 g/m² (0.098 oz/ft¹). In Europe and North America, where more of a matte finish is desired, the metal loss is typically 80 g/m² (0.26 oz/ft²).

Etching produces sodium aluminate and hydrogen gas:

2AI + 2NaOH + 2H₁O __ 2NaAIO₁ + 3H₁ !

A "conventional" caustic etch (with no additives) has a typical working composition as in Table 1. These etch solutions are moderately viscous and experience typical dragout rates of 0.2 L/m² (0.005 gal/t²) of work processed, which is about twice that of water or diffute sulfunc acid.

If the aluminum concentration is

By Craig Brown
Vice-President and General Manager
Eco-Tec Limited, Toronto, Ontario

allowed to increase enough, the sodium aluminate will hydrolyze, liberating free caustic sods and hydrated slumina or aluminum hydroxide precipitate:

NAAIO, - 2H10 - NAOH + AI(OH), *

Theoretically, the liberated free caustic can be used for more etching. Unfortunately, under normal stch bath conditions, the alumina precipitates as a rock-like hydrate on the tank walls and heating coils.

To avoid problems associated with precipitation, chelating agents, such as sodium gluconats, are often added to tie up the aluminum and prevent precipitation. The aluminum builds up to the point where the rate of dragout equals the rate of etching. The bath no longer requires regular dumping, and its life

Table 1. Caustic Steh Fermulations

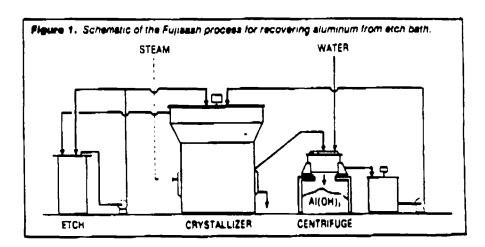
	Conventional Etch	Extended Life Etch
NaOH (free)	70 g/L	60 g/L
Al	50 g/L	130 g/L
Dragout	0.2 Č/m²	0.6 L/m²

can be significantly extended. A typical "extended life" or "never dump" bath analysis is shown in Table 1. The viscosity of these solutions is much higher than the "conventional" etch solution, and, as a result, dragouts are much higher—typically 0.6 L/m² (0.02 gal/ft²). Caustic soda must be continually added to these "extended life" etches to allow etching to continue and to replace dragout losses. Consumption of caustic soda is therefore not effectively reduced by this method.

It is possible to induce the precipitation of Al(OH), from conventional etch solutions in a separate crystallizer, preventing precipitation in the etch tank. Crystallization depends on the variation in solubility of metal salts in a chemical solution as a function of temperature and solution composition.

Caustic soda is continually liberated for use in etching aluminum, and additions of fresh caustic are therefore only required to replace dragout losses. As the bath is much more dilute than the "extended life" etch, dragout losses are lower. As a result, caustic soda purchases can be reduced by as much as 80%.

The low aluminum concentration in the etch when the regeneration process is employed allows excellent etching performance. More importantly, bath composition is constant, providing uni-



The aluminum sulfate waste or by-product produced by the system can be neutralized in the plant waste treatment system or concentrated by evaporation and sold as liquid alum. Table 3 shows projected costs when this approach is applied to recovery of dragout from a typical phosphoric/nitric bright dip operation.

A DCU/APU employed in conjunction with a vacuum evaporator in a Japanese anodizing plant has been recovering phosphoric/sulfuric acid bright dip solution at over 98% efficiency since its March 1980 startup. No phosphoric acid purchases were needed during the initial six months of operation.

The large recovery potential from processes of this type makes it extremely attractive. When total operating costs of 15.8e/kg (7.1e/lb) are subtracted from a value of 50e/kg (27e/lb) for 80% phosphoric acid, an installation consuming 2.2 million kg (2430 tons) per year can save up to \$170,000 per year based upon a recovery efficiency of approximately 90%.

Sulfuric sold enodizing

Most anodizing is done in sulfuric acid electrolytes; some of the aluminum oxide layer dissolves during anodizing. It has been estimated that under normal anodizing conditions 15 g/m² (0.05 oz/ft²) of aluminum are dissolved per hour of anodizing time. Eventually the aluminum reaches a level where it impairs the process and quality of the film. At this point all or part of the bath must be discarded and replaced with fresh solution.

In March 1977 the first APU was started up to purify a sulfuric acid anodizing bath. Since then a large number of similar units have been installed around the world. Users of these units have reported a number of benefits, including improved anodizing quality, better throwing power, reduced cooling requirements. Iower voltage and reduced labor requirements.

A typical APU can remove approximately 4 kg/hr (6.8 lb/hr) of aluminum from a sulfunc acid anodizing bath while maintaining the bath aluminum content at 10 g/L (1.3 oz/gal). Table 4 lists typical field data.

Phosphoric acid anodizing

Significant interest has been shown in phosphone acid anodizing. Being considerably weaker than sulfune acid, phosphoric acid anodizing baths can be purified by either an APU or DCU. The APU can remove aluminum phosphate impurities as waste. Taking into consideration the relatively high value of phos-

Table 3. Economics of Phosphoric/Nitric Bright Dip Recovery (per pound of 80% H.PG, recovered)

	Amount (Ib)	Cost (S)
66°Be sulfuric acid	0 245	0.0086
Lime	0.265	0 0053
Steam	8.450	0.0421
Sludge disposal	2 310	0.0058
Labor		0.0094
Total operating costs		0.0712

Table 4. Typical APU Results for H₂SO, Anodizing Recovery

	Flow rate (L/hr)	H,SO. (g/L)	Al (g. L)
Feed (bath)	298	183.8	12.2
Product	296	175.0	4.2
Waste	175	13.0	12.0
Water	173	0	0

phoric acid and the role of the phosphate ion as a pollutant, the best approach appears to be a DCU/APU system. A DCU converts the aluminum phosphate impurities to free phosphoric acid: an APU conserves sulfuric acid regenerant.

Some dilution of the phosphoric acid may occur during treatment. An atmospheric evaporator that operates directly on the bath may be necessary to remove this excess water.

Integral color anodizing

A vanety of weak organic and inorganic acids are used in integral color anodizing, including sulfosalicylic, sulfophthallic and boric. These baths are more sensitive to dissolved aluminum impurity buildup than conventional sulfuric acid baths. The aluminum content has to be maintained at a much lower level and within closer limits. The high replacement value of these baths necessitates some means of impurity removal.

Rates of aluminum buildup in these baths have been estimated at 27 g/m² (0.088 oz/gal) of work per hour of anodizing time at 2.58 A/dm² (23.9 A/ft²).

The low free acidity of these electrolytes allows the use of decationizers for aluminum removal, and conventional cation exchange has already been used extensively for this application. The large amount of downtime for frequent regenerations and the high acid regeneration dosages normally required allow considerable room for improvement. An estimated 378 L (100 gal) of 10% suffund acid are required to regenerate 1 kg (2.2 lb) of aluminum from cation resins used in this application. This represents 7.36 times the theoretical dosage. Use of a DCU/APU can

reduce this to about 1.5 times theoretical, a significant improvement. Several systems of this type have been recently installed with good success. A DCU to remove approximately 2.5 kg/hr (5.5 lb/hr) of aluminum from a Duranodic integral color anodizing bath is available. An APU is used to recover excess regenerant from this unit. The fully automated DCU typically operates on a 20-minute cycle time: 10 minutes in service and 10 minutes for regeneration, so the bath can be consistently maintained at a low level of aluminum contamination.

Electrolytic coloring

Because of its low electrical requirements, electrolytic (two-step) coloring has found recent acceptance in the North American architectural anodizing industry. The electrolytes employed are based on nickel, copper, cobalt or tin salts, frequently in conjunction with a weak organic acid. After coloring, the aluminum must be rinsed well prior to sealing. Loss of valuable metallic salts due to dragout is appreciable.

Recovery of metal salts from these nnsewaters is advantageous for three reasons:

- ☐ Salts of cobalt, tin, nickel and (to a less extent) copper represent significant value.
- ☐ While aluminum hydroxide sludge is not considered a hazardous waste, this classification could change if significant quantities of these other, more toxic metals are present in the sludge.
- ☐ Complexing agents added to the electrolytes frequently render conventional chemical precipitation techniques ineffective.

Numerous reciprocating flow ion exchange systems recovering copper

form etch rates and consistent product quality.

Caustic etch regeneration systems of this type have been used extensively in Japan for a number of years with excellent success. Fujisash industries of Kawasaki City has commercialized the process and developed a standard stackage. Over 20 of these systems have been installed to date. Alcoa has more recently introduced a similar system.

Figure 1 shows a schematic of the Fujisash process. Etch solution is continually pumped from the etch tank to the crystallizer. Hydrated alumina precipitates from the solution and settles to the bottom of the crystallizer. Regenerated etch solution, with lower aluminum and higher free caustic levels, flows back to the etch bath directly from the top of the crystallizer. Crystals of hydrated alumina are periodically withdrawn from the crystallizer bottom and dewatered with a centrifuge.

The hydrated alumina crystals produced by the system contain only 7 to 15% water and bear no resemblance to the watery aluminum hydroxide "sludge" produced by conventional waste treatment systems. Table 2 lists a typical assay of this material, which is basically equivalent to commercial "Bayer hydrate" and can be sold to a variety of end users. The amount of sludge generated in the final waste treatment system can be reduced by typically 75% if this system is employed.

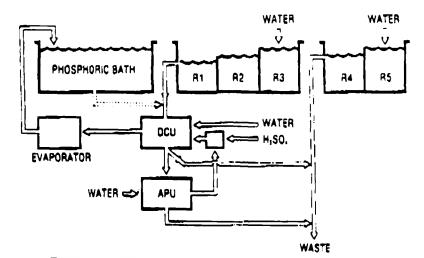
Energy consumption for the system is minimal: electricity for agitators, pump motors and the centrifuge and small amounts of steam to compensate for heat radiation losses from the equipment.

Caustic etch regeneration systems of this type offer a number of important advantages:

- up to 80% reduction in caustic soda purchases
- ☐ 100% elimination of purchases of gluconates and other proprietaries
- dramatic size reduction in sludge dewatering equipment and resulting sludge disposal problems
- ☐ consistent bath composition and operation with good product quality ☐ production of a salable hydrated alu-
- mina by-product

 Phosphoric acid bright dipping
 Concentrated phosphoric acid baths

Figure 2. Phosphoric acid recovery system utilizes a decationizer unit (DCU) and an acid purification unit (APU).



	Percent
Ai,0,	64.88
SiO,	0.07
Fe,O,	0.04
CaO	0.06
Mg0	0.15
Na _t O	0.27
K ₂ O	0.02
Other	0.122 to 0.534
Loss on ignition pH (10.7)	34.38

(usually with additions of nitric, sulfuric, acetic and/or chromic acid) are used to polish aluminum. After brightening, the adhering bright solution, contaminated with aluminum, must be immediately rinsed from the metal with water. Typically 15 g/m² (0.05 oz/ft²) of aluminum are etched. Due to the high acid concentration (70 to 80% H₂PO₄) and high viscosity, dragout losses are appreciable, around 0.3 to 0.4 L/m² (0.0073 to 0.0098 gal/ft²), compared to about 0.1 L/m² (0.0024 gal/ft²) for a "water-like" process solution.

If the aluminum contamination is removed, the rinsewater can be reconcentrated to bath strength by vacuum evaporation and recycled to the bath. Most previous purification systems based upon cation exchange or acid retardation have failed, mainly because the cost of treating the final waste stream and disposal of the resultant

sludge was excessive. The decationizer unit/acid purification unit (DCU/ APU) approach has greatly reduced these problems.

The DCU/APU are ion-exchange systems that utilize reciprocating flow ion exchange, which allows eluting a purified acid product from resin with only water. In the APU "up stroke," acid feed solution containing a metallic sait impurity is metered into the bottom of the resin bed. The free acid is sorbed by the resin, and the metallic sait by-product solution flows from the top of the bed. In the "down stroke," water desorbs the acid from the resin, producing a purified acid product.

Figure 2 shows a typical phosphoric acid recovery system. Concentrated rinsewater flows to the DCU where cationic impurities exchange onto the resin; the purified process solution, with most of the aluminum removed, flows to an evaporator for concentration to bath strength and recycling. The DCU is periodically regenerated with acid and washed with water. The dilute washings flow to waste, and the concontrated portion of the spent recenerant flows to the APU. The acid in the spent regenerant solution is retained by the APU, and the impurities are rejected in the APU by-product stream. Water slutes the acid from the APU, which, after refortification with a small amount of fresh acid, can be reused by the DCU. The APU can remove 90% of the metallic impurities and recover about 90% of the free acid fed to it.

and nickel sulfate from electroplating rineswater have demonstrated that cation-exchange systems can effectively recover metallic salts from rineswater. Several similar systems have recently been installed in two-step electrolytic coloring operations with excellent success.

Figure 3 shows a typical recovery system layout for the Alcan Anoiok process. Rinsewater flows from the first rinse tank through a decationizer (DCU), which removes the metals and discharges the purified rinsewater to waste. Careful sulfuric acid regeneration of the DCU yields a concentrated metalic sulfate product solution at a pH of 1 to 1.5. If necessary, the recovered product can be treated with a modified APU or descidification unit (DAU) to raise the pH of the solution to bath operating levels. The final product is collected and analyzed prior to recycling to the color bath.

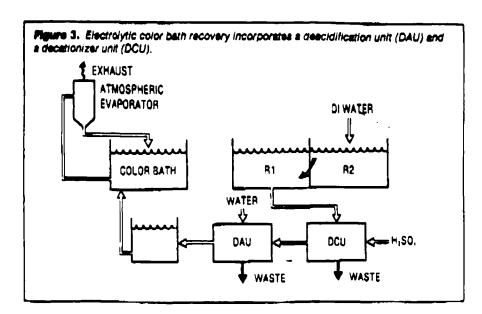
Most color baths operate near room temperature, so evaporation losses are negligible. Atmospheric evaporators are sometimes necessary to remove sufficient water from the bath to allow recycling the recovered product. This is not feasible or necessary for most tin-based electrolytes.

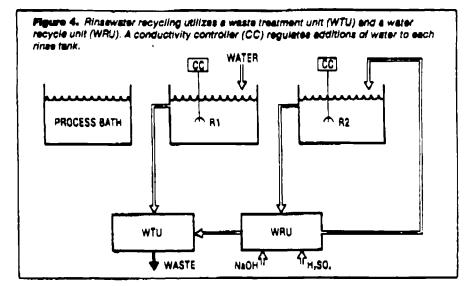
It is important to recover the metallic salts in a purified form to avoid contamination of the color bath. When recovering this utilize, for example, it became necessary to utilize a cation resin with a high selectivity for tin to avoid recovery of aluminum contamination. Precipitation of stannic oxide in these rinsewaters also presents some challenging filtration problems. A DCU and filter are being employed to recover tin sulfate from Reynolds Trucolor.

Chromate processes

Chromate, either as chromic acid or the potassium (or sodium) sait, is used in several aluminum finishing processes, including chromic acid anodizing, conversion coating, desmutting, dichromate sealing and hard chrome plating. To avoid pollution hazards, a recent trend has drifted away from chrome-based processes, even though these baths are often better understood, easier to control, superior in performance and less expensive to operate than the chrome-free baths. The availability of viable chrome recovery systems suggests a reevaluation of any moves in this direction. Recovery of chromate from plant wastewater reduces costs and simplifies waste treatment. These considerations may tip the scale back in favor of chromate.

Pollution results from chromate proc-





esses in two ways: dragout to rinsewater and process bath dumps due to contamination with aluminum and/or trivalent chromium.

The use of ion exchange for recovery of purified chromic acid from plating rinsewaters and purification of hard chrome plating baths is well established. This technology has recently been applied with excellent results to various chrome processes in finishing aluminum.

Specialty etching

Dilute nitric acid and hydrochloric acid solutions, often with additions of various organic acids and/or small amounts of other inorganic acids, are used for electrochemically etching sluminum coils. These baths become contaminated with dissolved aluminum and must be dumped frequently.

Because these baths are often fairly dilute, they lend themselves to purification by cation exchange better than acid retardation. It is usually necessary to utilize an APU in conjunction with the

decationizer to improve chemical regeneration efficiency sufficiently to make recovery truly viable.

Several DCU/APU systems are being used by manufacturers of lithographic plates for removal of aluminum from hydrochloric acid graining solutions. Etchants of similar compositions, used for aluminum capacitor foils, can also be punified by this method.

Water recycling

Large quantities of water are consumed in rinsing work after each process bath. Most of the water can be easily deionized by cation/anion exchange and reused. Indeed, as deionized water is required for several rinses and makeup of many baths, the same unit can serve both purposes.

Recovery of rinsewaters simplifies waste treatment because it is much easier and cheaper to treat a concentrated, low-volume stream than a dilute, high-volume stream. Reaction tanks, reagent pumps and clarifiers are all reduced in size. Moreover, the char-

acter of sludge precipitated from concentrated wastewater is generally superior to that produced from dilute solutions.

The nature of the rinsing process is such that typically only 10% of the water is used to rinse 90% of the chemicals from the work. Conversely, 90% of the water is consumed in actually rusing only 10% of the chemicals. Operating costs in ion exchange are mainly related to chemical loadings and only to hydraulic flows to a very minor degree. These two principles can be used to great advantage in designing a viable rinsewater recovery system.

Figure 4 illustrates this point; although only one step is shown, several processes would normally be tied together in a similar manner. Conductivity controllers (CC) mounted in rinse tanks R1 and R2 control additions of water to the tanks. The first rinse, R1, is maintained at about one-tenth of bath strength through additions of a very small amount of water, equivalent to about 10 times the rate of dragout. Overflow from R1 flows directly to the waste treatment unit (WTU), Desired final rinsewater purity in R2 is main-

tained through additions of large amounts of recovered water. The overflow from R2 flows to a deionizer or water recycle unit (WRU), consisting of cation and anion exchangers, where it is purified and recycled.

Automatic regeneration of the WRU may occur once or twice each hour for five minutes if reciprocating flow techniques are used, eliminating the need for a duplicate standby unit. Regeneration yields a low-volume, concentrated liquid that flows to the WTU.

General considerations

Particular attention should be paid to the acid/alkali balance in the plant effluent when evaluating the recovery of acid or caustic. It makes little sense to install an APU on the anodizing bath if it becomes necessary to purchase virgin acid to neutralize spent caustic etch solution. Similarly, a "conventional" decationizer may be adequate for an integral color anodizing bath where spent solution is available to neutralize the large amount of excess acid in the spent regenerant. If one wishes to install a caustic etch regeneration system, it normally makes sense to con-

currently install the more efficient DCU/ APU system in lieu of the conventional decationizer.

Increasing attention is being paid to neutral earts in industrial effluents, such as sodium sulfate. Conventional waste treatment utilizes chemical neutralization, aggravating this pollution problem. Recovery techniques effectively reduce neutral sait discharges.

Technology for recovery of chemicals and water in finishing aluminium is available and finding wide acceptance. Recovery of even inexpensive process solutions, such as sulfuric acid anodizing baths, can be shown to be worthwhile when side benefits such as pollution control and process stabilization and improvement are considered along with direct economic savings. The benefits of recovery of more expensive and/or toxic chemicals are even more pronounced.

Any existing or proposed aluminum finishing facility should have chemical and water recovery to abate pollution, reduce costs and improve process performance.

APPENDIX D

ELECTOLESS NICKEL PLATING VENDOR INFORMATION

BATH MAINTENANCE PROCEDURES (WITCO)	D-1
PALMER INDUSTRIES CHEMICAL FEED SYSTEM	D-5
PALMER INDUSTRIES ENSTAT SYSTEM	D-7
CHEMICAL PLATE-OUT METOD (WITCO)	D-11
LANCY ELECTROWIN SYTEM FOR ELECTROLESS NICKEL	D-15
WITCO CHEMICAL PRECIPITATION METHOD	D-17

Niklad™ 794

Electroless nickel-phosphorus process

Allied-Kelite Division, Witco Chemical Corporation 2701 Lake Street, Melrose Park, 1L 60160, (312) 344-4300

Supersedes 021583

052083

Introduction.

The Niklad 794 electroless nickel process is designed to deposit a uniform, bright nickel-phosphorus alloy onto properly prepared metal surfaces such as stainless steel, alloy steels, cast iron, copper and its alloys, titanium, beryllium and nickel alloys, magnesium, and onto various non-conductive materials. It is particularly suited for electroless nickel plating of aluminum alloys due to its high tolerance to metallic impurities. (Your Allied-Kelite representative can recommend pretreatment cycles for these substrates.)

Niklad 794 is supplied as three liquid concentrates — Niklad 794A, Niklad 794B, and Niklad 794HZ. These concentrates provide all the necessary purified chemical formulations for make-up and replenishment of the Niklad 794 plating solution. Replenishment additions can be made to the solution at operating temperatures.

Users of the Niklad 794 process have the option of adjusting bath pH with either ammonia-free Niklad 794pH or ammonium hydroxide. Niklad 794pH is a liquid additive that does not produce objectionable ammonia fumes and has the added benefit of improving bath life.

Advantages.

- Exceptionally bright deposits
- Tolerant to metallic impurities
- Economical to operate
- Consistent high rate of deposition with maximum stability
- Replenishment made at operating temperature
- All-liquid system
- · Allows for barrel plating of parts
- Meets specifications MIL-C-26074B, AMS 2404A and AMS 2405

Equipment.

The following equipment is suggested for optimum Niklad 794 operation.

Tank and heating.

Tanks fabricated from high-density polypropylene are suggested. An excellent plating arrangement is a twin-tank setup, with one tank held in reserve. If the nickel begins to deposit on the production tank, the solution can immediately be filtered into the standby tank, chemically adjusted and operated. Therefore, the twin-tank arrangement can eliminate costly downtime. The plated tank is then stripped and cleaned to serve as the standby tank.

Derated electric immersion heaters fabricated from 304 or 316 stainless steel, titanium or anodized titanium; or steam TEFLON* Supercoil are satisfactory for heating the solution.

DuPont trademark

Agitation.

Clean, mild air agitation or mechanical work rod agitation is satisfactory. The rate of agitation can affect the deposition rate.

Filtration.

Continuous filtration, though not necessary, is suggested for optimum results. A 5-micron bagtype filter provides adequate filtration for most installations. Use periodic filtration if continuous filtration is not feasible.

Solution makeup and operating conditions.

Solution makeup. <u>Material</u>	100 Gallons	100 Litres
Niklad 794A (Ni source/replenisher)	8 gallons	8 litres
Niklad 794B (complexor/buffer)	15 gallons	15 litres
Water (distilled or deionized)	Balance	Balance

Witco

CAUTION: Before making up the working solution, refer to Safety And Handling section on last page to be certain you are aware of the protective gear you must wear, and the first-aid procedures to follow in the event of accidental exposure to the liquid concentrates or solutions.

1. Fill plating tank 1/2 full with deionized or

distilled water.

Add 8% Niklad 794A by volume (of final volume), mix well.

Add 15% Niklad 794B by volume (of final volume), mix well.

 Adjust to operating volume with deionized or distilled water and mix thoroughly.

5. If necessary, adjust pH to operating range using Niklad 794pH (or 1:1 ammonium hydroxide) to raise pH, or 25% sulfuric acid to lower pH. CAUTION: Niklad 794pH (or 1:1 ammonium hydroxide) can cause a violent reaction and spurting if improperly added to the bath. With vigorous agitation, add small, frequent amounts of Niklad 794pH (or 1:1 ammonium hydroxide) over the entire surface area of the bath rather than large, infrequent amounts to a concentrated area.

6. Heat to operating temperature. Solution is now ready for operation.

Operating conditions.

	Range	Typical
Solution temp.	160-212F (71-100C)	195 F (91 C)
Solution pH*	4.5-5.2	4.8
Bath loading	0.25 - 1.0 (t²/gal (0.6 - 2.5 dm²/L)	0.6 ft ² /gal (1.5 dm ² /L)

If pH is permitted to drop out of operating range, the buffering capability of the solution is reduced. This may require large additions of Niklad 794pH (or ammonium hydroxide) to restore pH value to normal limits.

Bath loading.

Optimum bath loading (ratio of plating surface area to solution volume) is determined by bath operating temperature, deposition rate desired and frequency of maintenance additions. As shown in Figure 1, a bath loading of 0.25 ft²/gal (0.6 dm²/L) at an operating temperature of 195F (91C) provides a good deposition rate with a practical replenishment schedule. Of course, greater loading conditions and increased overall deposition rates are obtained by decreasing the time interval between bath replenishments.

Deposition rate.

Deposition rate largely depends on operating temperature of the electroless nickel bath. The plating solution can be varied over a wide temperature range and maintained for long periods at maximum temperatures without causing any spontaneous decomposition. Plating rates at various temperatures over the life of the Niklad 794 solution are approximately as indicated in Figure 1.

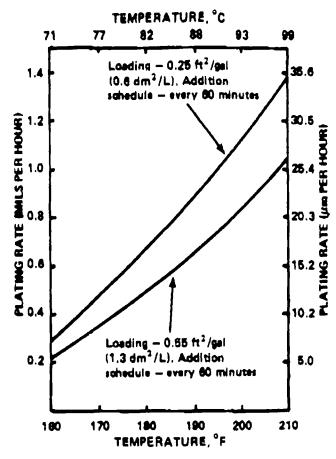


Figure 1. Niklad 794 plating rate vs. operating temperature at various surface area loading conditions.

Solution maintenance.

CAUTION: Before replenishing the working solution, refer to Safety And Handling section on last page to be certain you are aware of the protective gear you must wear, and the first-aid procedures to follow in the event of accidental exposure to the liquid concentrates or solutions.

Additives.

Additives used for solution maintenance are:

Niklad 794A (nickel source/replenisher)

Niklad 794HZ (reducer/stabilizer)

Niklad 7948* (complexor/buffer)

Niklad 794pH (An optional additive used instead of ammonium hydroxide to adjust pH)

Niklad 794B should be added at a rate of light 100 gal, regeneration (0.251./100 L/regeneration). Where high dragout losses are encountered, such as barrel operations, larger adds of Niklad 794B may be required.

Niklad 794A/Niklad 794HZ replenishment.

Replenishment additions of Niklad 794A and Niklad 794HZ are based on the percent nickel metal activity of the plating solution. An Allied-Kelite Niklad Test Kit can be used for a quick, simple determination of the percent nickel metal activity, or a more precise burette titration analysis can be employed (both methods are described in the Percent Nickel Metal Activity Determination section.) After determining the percent nickel metal activity of the Niklad 794 plating solution, use Table I to obtain required amounts of Niklad 794A and Niklad 794HZ.

Table 1. Conversion of percent nickel metal activity to required replenishment additions of Niklad 794A and Niklad 794HZ.

%Nickel-Metal Activity	Nikiad 794A Required floz/gal mL/L		Niklad 79411Z Required	
By Analysis			fi oz/gal	mL/L
100	None	None	None	None
90	1	8	1	8
80	2	16	2	16
70	3	24	3	24
60	4	32	4	32
50	5	40	5	40

To calculate required additions for nickel metal activity not given in the table, use the following formulas:

- 1. ft oz/gat Niklad 794A
 required = (100 % nickel metal activity) × 0.1
 mL/L Niklad 794A
 required = (100 % nickel metal activity) × 0.8
- 2. fl oz/gal Niklad 794HZ
 required = (100 % nickel metal activity) × 0.1
 mL/L Niklad 794HZ
 required = (100 % nickel metal activity) × 0.8
 Replenishment procedure is as follows:
- 1. Slowly add required amount of Niklad 794A to plating solution, with agitation.
- Slowly add required amount of Niklad 794HZ to plating solution, with agitation.
- 3. Adjust solution pH to operating range using Niklad 794pH (or 1:1 ammonium hydroxide) to raise pH. or 25% sulfuric acid to lower pH. CAUTION: Niklad 794pH (or 1:1 ammonium hydroxide) can cause a violent reaction and spurting if improperly added to the bath. With vigorous agitation, add small, frequent amounts of Niklad 794pH (or 1:1 ammonium hydroxide) over the entire surface area of the bath rather than large, infrequent amounts to a concentrated area.

NOTE: Materials can be added to the plating solution at operating temperature if caution is used. Vigorous agitation and mixing are necessary. Do not add materials directly over parts being plated.

General guide to bath operation.

- 1. If nickel metal activity percentage drops below 50%, do not make additions until bath has cooled to room temperature.
- 2. A complete solution replenishment of the plating solution's nickel content occurs when 10 fl oz/gal (80 mL/L) Niklad 794A have been added.
- 3. A Niklad 794 deposit of 1 mil square foot requires 7.8 floz of Niklad 794A and 7.8 floz of Niklad 794HZ. (A Niklad 794 deposit of 10 µm/dm² requires 10 mL of Niklad 794A and 10 mL of Niklad 794HZ.)
- 4. For repetitive work, additions can be made on a predetermined or continuous schedule based on occasional checks of bath concentrations.
- 5. Proper chemical cleaning of parts to be plated with electroless nickel is most important to insure maximum performance. Pretreatment procedures for both rack and barrel plating of various base materials are available from your Allied-Kelite representative.
- 6. Though the Niklad 794 system is tolerant to impurities, the system can be affected by the drag-in of specific impurities such as palladium, cadmium, certain wetting agents, and sulfur containing compounds. Good rinsing is imperative prior to the Niklad 794 bath. Filter out solid particles as they appear.
- 7. pH papers in the appropriate range are suitable for pH control.
- 8. Deposition rate decreases as plating bath pH decreases. Maintain pH in proper operating range using Niklad 794pH (or 1:1 ammonium hydroxide) to raise pH, or 25% sulfuric acid to lower pH.
- 9. Bulk parts are best plated in slowly rotating barrels with good solution recirculating characteristics.

Percent nickel metal activity determination.

Niklad Test Kit analysis.

Kit Content: 1 -4-oz glass sample bottle

- 1 16-07 bottle of titrating solution (0.0575M EDTA)
- 1 —2-oz dropper bottle of ammonium hydroxide
- 1 1-oz bottle of indicator powder (murexide mix)
- I Dropper marked at 5 mL
- 1 Dropper calibrated from 0-5 mL

Procedure:

- 1. Fill 4-oz glass sample bottle about ½ full with water.
- Using marked dropper, add 5 mL cooled sample of Niklad 794 plating bath to the 4-oz bottle and mix.
- 3. Add ammonium hydroxide until sample solution turns deep blue in color.
- 4. Add a small amount of indicator powder to sample solution and mix. The sample solution should be straw colored.
- 5. Fill calibrated dropper to the zero mark with titrating solution and add this to the sample solution. Shake, if the solution does not turn violet, continue with steps 6 and 7. If solution color is violet, STOP. This means the plating bath is at or below 50% activity. If this occurs, cool bath to room temperature and add 5 floz/gai (40 mL/L) each of Niklad 794A and Niklad 794HZ. Reheat bath to operating temperature and redetermine nickel metal activity percentage starting with step 1.
- 6. Refill calibrated dropper to the zero mark again with titrating solution. With mixing, add a few drops at a time to the sample solution until its color changes to violet.
- 7. Record total number of millilitres required to obtain color change.

Calculation:

% nickel metal activity

= total mL titration × 11.2

Burette titration analysis.

Reagents: Concentrated ammonium hydroxide (NH₄OH)

Murexide indicator mix (1 gram of murexide mixed with 100 grams C.P. sodium chloride)

EDTA (disodium dihydrate salt), 0.0575M, 21.4101 g/L

Procedure:

- 1. Pipette 10 mL cooled sample of Niklad 794 plating solution into 250 mL Erlenmeyer flask.
- 2. Add about 100 mL of distilled water.
- Add 15 mL of concentrated ammonium hydroxide
- 4. Add about 0.2 gram murexide indicator mix.
- 5. Titrate immediately with standard EDTA solution to endpoint color change of light brown to violet.

Calculation:

% nickel metal activity = mL EDTA \times 5.62

Safety and handling.

Store Niklad 794A, Niklad 794B, Niklad 794HZ, and Niklad 794pH in tightly closed containers. Avoid contact with eyes, skin and clothing. Wear chemical-resistant gloves and chemical safety goggles when handling liquid concentrates and solutions. Wash thoroughly after handling.

In case of skin or eye contact, immediately flush with large amounts of water for at least 15 minutes and call a physician. If swallowed, call a physician immediately.

In case of spill or leak, absorb with an inert ingredient such as sand, earth or vermiculite. Dispose of residue consistent with federal, state and local regulations.

Niklad 794A, Niklad 794B, Niklad 794HZ and Niklad 794pH are for industrial use only. Read pertinent Material Safety Data Sheet and product label before using, Also, carefully read and follow product data information and label instructions associated with all chemicals and solutions mentioned in this technical bulletin.

Standard packages.

Niklad 794A	_	5 gallons: 55 gallons
Niklad 794B	_	5 gallons; 55 gallons
Niklad 794HZ	_	5 gallons: 55 gallons
Niklad 794pH	_	5 gallons; 55 gallons
NOTE: Additives of	of the N	iklad 794 system are not
damaged by freez	ing. If	frozen, thaw to room
temperature and n	nix well	before using.

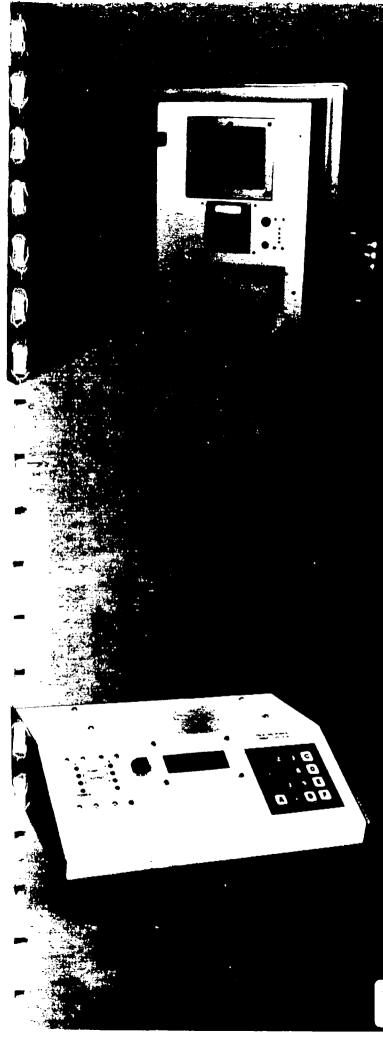
Warranty.

We warrant our goods to conform to our standard specifications. This Warranty is in lieu of any and all other warranties or guarantees and our obligation hereunder for breach of such warranty is limited to either refund of the purchase price or replacement of said goods as we may elect. We make no other warranties, express or implied, including those of MERCHANTABILITY and FITNESS FOR A PARTICULAR PURPOSE.

We believe that the statements, technical information and recommendations contained herein are reliable, but are given without warranty or guarantee of any kind except as specified above. Beyond that specified above, we assume no responsibility for any loss, damage or expense, direct or consequential, arising out of the use of, or inability to use, our goods. Their quality and suitability for any particular purpose or use should be confirmed by the user's own tests.

NOTE: All gallon measurements are U.S. Gallons





AUTOMATIC ANALYZER MODEL 850CL

- Closed loop controller for electroless Ni and Cu
- Completely self contained analysis and replenishment system
- Measures pH. concentration, bath and sample temperature
- Nema 4× enclosures for harsh environments
- Printer records results in real time
- Intelligent operating system

PROGRAMMABLE CHEMICAL ADDITION SYSTEM MODEL 225-ASA

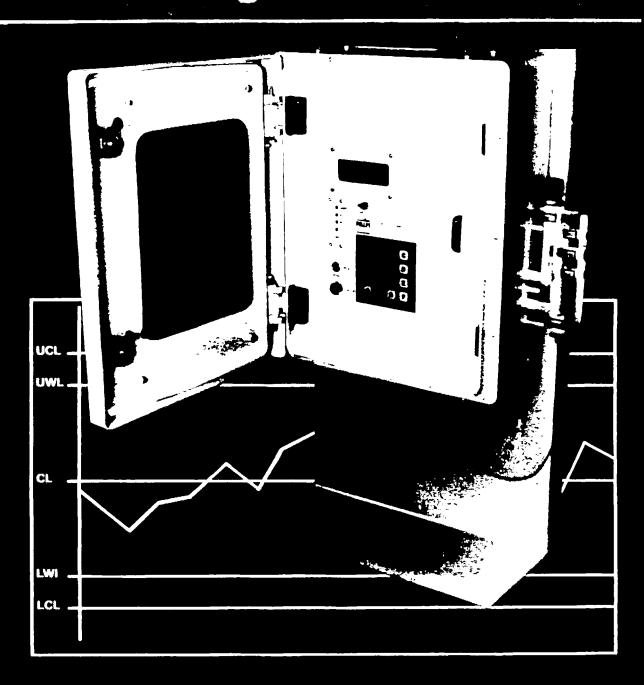
CAPABILITIES:

- Nema 4× enclosures for solid state relays
- Dead man timer stops pump in event of system failure
- Real time clock
- Up to three components per bath
- Up to seven bath addition and chemical tracking
- Baths need not be the same composition
- Simple data entry
- Liquid crystal data display

OPTIONS:

- Bath temperature measurement
- Printer for hard copy of transactions

PALM INSTRUMENTS introduces a new generation of Plating Bath Controllers



Closed Loop Controller and SPC System Model 1100 SL

Analyzer and replenishment system for electroless Ni and Cu
Measures pH, concentration, and temperature
Interface up to twenty-four controllers for data collection
PC type processor collects data and generates SPC charts
Controllers are microprocessor operated and may be used as stand alone units
Communicates with central processor via RS-232 data transfer
Controllers are equipped with LCD readouts and keypads for ease of use
Advanced record keeping ability and statistical analysis of data is featured

General Information:

- Palm Instruments manufactures a broad line of pH. specific ion. ORP and conductivity monitors/controllers
- All equipment utilizes state-of-the-art dedicated microprocessor operating systems
- Palm offers custom system development for customer's specific application. Contact us for details.

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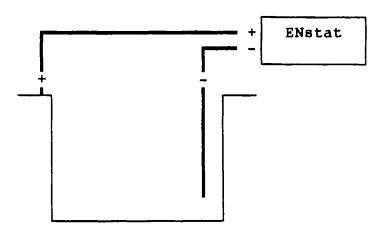
ENSTAT

PASSIVATION SYSTEM FOR

STAINLESS STEEL PLATING TANKS

ENstat is an electrochemical system for continuously passivating the stainless steel components used in electroless plating baths to prevent plate-out, while also avoiding the need to strip and condition the tank with nitric acid on a daily basis. The use of this system also minimizes disposal of spent nitric acid solutions and the cost of their waste treatment.

The ENstat is based upon the electrochemical technique of anodic protection, which has been used for many years prevent corrosion equipment in the chemical industries, and process more recently to avoid deposition on equipment electroless in plating solutions. anodic terms, protection is a method of producing a passive film



upon the surface of stainless steel by means of a small impressed current at a predetermined and carefully controlled potential. This film (which is very similar in composition to that produced by nitric acid passivation) prevents deposition of electroless nickel or electroless copper, while also avoiding corrosion of stainless steel components.

The difference between this technique and the "reverse current passivation" method used by some platers in the past, is that with "deplating" the potential of the stainless steel is not controlled. Instead a relatively large current (typically 10 to 40 ma/ft²) is discharged from the metal to keep it from plating. This discharge, however, also causes the stainless steel to corrode and become etched, especially at the welds. With ENstat a very small current (typically 1 to 2 ma/ft²) is used to maintain the potential of the metal within a desired range and little, if any, corrosion occurs.

Not only will the ENstat system protect the plating tank, but the instrument will also passivate and prevent plate-out on any stainless steel component electrically connected to it, including pumps, heat exchangers, filters, and their associated piping. Because protection is not dependent upon current flow, the passivating potential does not change significantly with distance from the cathode. In one installation, passivation was found to extend more than 60 feet inside a 1 inch diameter pipe and its potential varied by only 0.3 mv.

Hundreds of anodic protection systems have been installed on electroless plating tanks in Europe, some for more than twenty years. These installations have been very successful and have typically allowed the plating solutions to remain in the same tank for as long as two months without transfer and without nitric acid passivation.

INSTRUMENT

The ENstat is a precision potentiostat, specifically designed to protect stainless steel equipment in electroless plating solutions. It utilizes a state-of-the-art microelectronic design to assure stable and trouble free operation. A backup battery is also provided to continue the tank's protection during a power failure.

The unit is capable of providing a sustained output of 10 amperes, and thus is adequate for even the largest electroless plating tanks. A overload protection circuit is also provided, which will shut down the unit and sound an alarm in the event of a short circuit. The ENstat also includes digital meters to display both the actual and set potential and the current being discharged from the tank, as well as LED indicators for power, battery supply, short circuit, overload and open circuit conditions.

The unit is supplied in a dust and water tight, NEMA 12 aluminum enclosure suitable for plating environments, and is complete with two 25 feet long, neoprene insulated cables for the anode and cathode connections.

SPECIFICATIONS

Input
Reference
Output
Battery
Size

110 VAC, 60 Hz at 3 amperes
0 - 1200 millivolts
0 - 10 amperes at 0 - 1.2 volts
1.3 amp-hour lead acid to supply
up to 0.5 amperes for 1 hour
5½" H x 17½" W x 10½" D



Recondition Flat Rate

PALM INSTRUMENTS, INC.

DESCRIPTION Ni Watchdoq model PC-670ENC Basic unit Including: Data printer and pH measurement	<u> </u>	RICE
including: Lata printer and pH measurement		
	3	3860.00
Options:		
Bath temperature measurement option each (1 to 3)	\$	250.00
Automatic solution replenishment option	\$	675.00
Double bellows metering pump each	3	520.00
Printer paper 200 ft roll	\$	10.50
Disposable cuvettes case = 140 ea	\$	24.00
Model 225-ASA Programmable solution addition unit with printe Remote pump control each bath (min three) Double beliows metering pump each Solution temperature option (ea bath)		225.00 520.00
Model 850-CL Closed Loop Ni Controller including: CRT screen, full keyboard, and data printer Ni metal and pH measurement		
Sample and bath temperature measurement		
Sample pump and Ni replenishment pump (two component) Automatic solution replenishment	و ۾	10600.00
varomatic polation replentanment	3.	10000.00
pH adjustment pump	9	410.00

Model B-2020 bench top analyzer	
Ni concentration and pH with ATC	\$ 2250.00

Model 1100-SL Closed Loop Ni controller including:		
Ni metal pH measurement		
Sample and bath temperature measurement		
Automatic solution replenishment		
Sample pump and Ni replenishment pump (two component)		
	\$ 8200.	00
Options:		
pH adjustment pump	\$ 410.	00
Remote printer	\$ 350.	00
SPC System for the above controllers New Units	\$ 2000.	00
For up-to 8 units Upgrade	\$ 2400.	00

AFTER SHIPHENT OPTIONS CONTACT FACTORY FOR PRICING

\$ 500.00



PALM INSTRUMENTS, INC.

NEW PRODUCT ANOUNCEMENT

DESCRIPTION PRICE

Model MP2000CC SOLUTION CONDUCTIVITY CONTROLLER including:

Measures solution concentration
Measures solution temperature
High-low audible alarms
Closed loop - microprocessor controlled system
Automatic solution replenishment
In tank electrodeless sensor - no contacts to foul
NEMA enclosure for operating system
Replenisher addition volume tracking for cost analysis
Four solution analysis capability from one controller

System with one sensor and pump outlet \$ 2850.00

Options:

Replenishment pump - single component \$	410.00
Replenishment pump - double component \$	520.00
Additional solution sensor	950.00
Remote data output port (For SPC system) \$	450.00
Remote data printer \$	350.00

SPC System for the above controller
For up-to 8 units

\$ 2000.00

Price effective AUGUST 30, 1989

Niklad™ WT 110

Plate-out system for electroless nickel plating solutions

Allied-Kelite Division, Witco Corporation 2701 Lake Street, Melrose Park, IL 60160, (312) 450-7435

040187

Introduction.

The Niklad WT 110 process is designed to remove nickel from spent electroless nickel plating solutions without generating sludge that can require further costly processing. The plate-out process works on most electroless nickel (EN) bath formulations provided that the nickel is not overly complexed with certain very stable complexors or overstabilized to the point where no plating is obtained. The single Niklad WT 110 additive significantly weakens the complexing ability of various chemicals, and allows the nickel to be plated to very low concentrations in certain electroless nickel baths. Niklad 794, Niklad 795, and Niklad 796 baths respond well with this plate-out process.*

In a typical spent Niklad 794 bath, a total addition of approximately 3% by volume of Niklad WT 110 over 6 hours can reduce the nickel metal content to 5-10 ppm. Results will vary depending upon type of bath, contaminants, and treatment cycle parameters.

This process is easy to use, utilizes standard EN tanks, and can reduce or eliminate further waste treatment requirements, providing a simple and cost-effective method of treatment.

 Other Niklad baths can be treated, but may require a modified process cycle. Refer to your Allied-Kelite representative for further information.

Advantages.

- No high-nickel-content sludge generated
- No special equipment required
- Nickel is plated out as nickelphosphorus alloy
- Simple process supplied as a single liquid additive

No sludge disposal

uses standard EN tanks; no complicated setups, treatment, or filters needed

Possible scrap value

Doesn't require highly skilled operators

Equipment.

In general, equipment that is used for Niklad plating baths is suitable for the WT 110 process.

Heating.

The bath must be heated to about 190F (88C) during treatment. Stainless steel, Teflon*, or Tefloricoated stainless steel heaters may be used. Derated electrical heaters as used in EN plating tanks are suggested if electrical heaters are used.

*Du Pont trademark

Tanks.

Tanks fabricated from polypropylene, polyethylene, or stainless steel are suggested. While the plating tank can be used as the plate-out tank, using a separate plate-out tank will avoid interruptions in production due to lengthy stripping times.

Agitation.

Agitation is required to mix the process chemicals into the solution. Mechanical or clean air agitation is satisfactory.

Treatment cycle.

CAUTION: Before handling or using the Niklad WT 110 process, refer to the Safety and handling section on page 3 to be certain you are aware of the protective gear you must wear, and the first-aid procedures to follow in the event of accidental exposure to liquid concentrate or solutions.

I. Refer to all applicable federal, state, and local regulations, and determine the limit of nickel that is allowed in the waste effluent. This limit should include the dilution from non-nickel-bearing waste water from rinsing and other water sources in the plant that will be in the waste stream. Analyze a cooled sample of the bath to be treated for nickel metal concentration (refer to Analytical control section).



- 2. If a separate plate-out tank is used as recommended, pump spent plating solution into plate-out tank and heat to 185-190F (85-88C). Maintain this temperature throughout the process.
- Slowly, with agitation, adjust the solution pH to 5.0 - 5.2 using dilute caustic soda (25% or less) solution.
- 4. Begin plating onto a large workload (1.5 ft²/gal [3.7 dm² Litre] or more) of clean scrap material with agitation. Large work loads speed the process. Steel or aluminum is satisfactory. Good agitation is essential.
- 5. Initially, add 1% (one gallon per 100 gallons [1 Litre per 100 Litres] of solution being treated) Niklad WT 110 to the plating bath with agitation. As the bath plates out, the pH tends to drop and Niklad WT 110 is consumed, slowing the plating rate (measured by the rate of decrease in nickel content by analysis). Maintain the pH at 5.0-5.2, and make additions of 1% Niklad WT 110 to the plating bath hourly or as required. With experience, the rate of plate-out can often be estimated by the amount of gassing produced by the plate-out reaction. As the bath depletes, smaller increments (0.5%) of Niklad WT 110 can be used.
- 6. Analyze and record the nickel content hourly during the plating process. When the nickel concentration reaches 0.08 oz/gal (0.6 g/L) or less, raise the pH to 5.5-6.0 with dilute (25% or less) caustic soda solution, and continue the plate-out procedure.
- 7. Continue the plate-out procedure, maintaining the proper pH, temperature, and Niklad WT 110 additions until the desired nickel content is obtained. If within all applicable regulations, discharge the effluent to the sewer.

Operating notes.

- 1. When adjusting pH with 25% caustic soda solutions, add very slowly with good agitation to minimize the formation of excess nickel hydroxide precipitates. On concentrated EN solutions, more dilute caustic solutions or ammonia additions may be helpful until some of the nickel has plated out. Excess precipitate can initiate plating or seeding on itself.
- 2. When treating spent solutions such as Niklad 794 or Niklad 796, the procedure typically requires 4-7 hours, and consumes a volume of Niklad WT 110 that is approximately equal to 3% of the bath, to reach a nickel concentration of 5-10 ppm.
- 3. The plate-out rate increases directly with higher surface area of workload, higher pH, higher temperature, higher agitation, and frequency of additions (assuming adequate additions of Niklad WT 110).
- 4. More stably-complexed EN solutions and those formulated with slower plating rates require longer treatment times.
- 5. As the plate-out reaction proceeds, the pH tends to lower and Niklad WT 110 is consumed. These factors are not critical, but will slow the reaction if not properly maintained.
- 6. If a production EN plating bath has stopped plating due to 'poisonous' contaminants or overstabilization and is to be treated and dumped, additional pre-treatments are necessary to get the plate-out reaction started. Often the contaminants responsible for the overstabilization can be sufficiently reduced by electrolyzing at low current densities using DC current with an insoluble anode. Using carbon anodes at 2-5 amp/ft² (21.6-54 amp/meter²) with good agitation for an hour may be sufficient. Lowering the temperature to about 140F (60C)during the electrolytic dummying will be less corrosive on the carbon anode. Remove the anode when not in use.

Analytical control.

Nickel metal determination using EDTA.

Reagents: Conc. ammonium hydroxide (NH₄OH)

Murexide indicator mix

EDTA (disodium dihydrate salt) 0.0575 Molar, 21.41 g/L)

Procedure:

- 1. Pipette 20 mL cooled sample of nickel bath into 250 mL Erlenmeyer flask.
- 2. Add about 100 mL of distilled water.
- 3. Add 10 mL of concentrated ammonium hydroxide.
- 4. Add about 0.2 gram of murexide indicator mix, or one tablet.
- 5. Titrate immediately with standard 0.0575M EDTA solution to an endpoint color change of light brown to violet. This can change abruptly.

Calculation:

oz nickel metal/gal = mL EDTA × 0.0225 g nickel metal/L = mL EDTA × 0.1688

NOTE: This EDTA method is accurate down to about 500 ppm nickel content. Use appropriate analytical methods, such as colorimeter, atomic absorption, or polarograph analysis for accurate results at lower concentrations. Consult your Allied-Kelite representative for specific instrument and method recommendations.

Warranty.

We warrant our goods to conform to our standard specifications. This Warranty is in lieu of any and all other warranties or guarantees and our obligation hereunder for breach of such warranty is limited to either refund of the purchase price or replacement of said goods as we may elect. We make no other warranties, express or implied, including those of MERCHANTABILITY and FITNESS FOR A PARTICULAR PURPOSE.

We believe that the statements, technical information and recommendations contained herein are reliable, but are given without warranty or guarantee of any kind except as specified above. Beyond that specified above, we assume no responsibility for any loss, damage or expense, direct or consequential, arising out of the use of, or inability to use, our goods. Their quality and suitability for any particular purpose or use should be confirmed by the user's own tests.

Safety and handling.

CAUTION: Niklad WT 110 is a liquid reducer and contains a large concentration of sodium hypophosphite. Avoid contact with skin, eyes, and clothing. Use with adequate ventilation. Do not swallow. Can be strong irritant to eyes and skin. Can be harmful if swallowed. Store in tightly closed containers away from direct heat, alkalies, and oxidizers.

PROTECTIVE EQUIPMENT: When handling Niklad WT 110 liquid concentrate and solutions, wear rubber gloves, chemical safety goggles, and chemical-resistant clothing. Wash thoroughly after handling.

FIRST AID: In case of skin or eye contact, immediately flush with large amounts of water for at least 15 minutes. For eye contact, immediately call a physician. If swallowed, immediately call a physician. If inhaled, remove victim to fresh air and call a physician.

SPILL OR LEAK: Wear protective equipment during cleanup of a spill or leak. Absorb spill or leak with an inert material such as sand, earth or vermiculite. Dispose of residue consistent with federal, state and local regulations.

REQUIRED READING: Niklad WT 110 is for industrial use only. Read Material Safety Data Sheet and product label before using. Also, follow supplier's safety recommendations for all other chemicals mentioned in this technical bulletin.

Standard packages.

Niklad WT 110 - 5 gallons. 55 gallons

NOTE: Niklad WT 110 is not damaged by freezing. However, it may freeze or separate due to cold. If frozen, thaw to room temperature and mix well before using.

NOTE: All gallon measurements are U.S. Gallons.

ALLIED-KELITE

Your single source for tried and proven, brand-name metal treating and finishing products, industrial cleaning products, and support material and equipment.

Surface Preparation Products

- Isoprep® Cleaners...and etchants, derusters, deoxidizers, desmutters, descalers, paint strippers and rust-preventive oils
- Alprep^{tw} Cleaners...cleaners and activators expressly formulated to prepare aluminum surfaces for Niklad^{tw} electroless nickel plating
- Ferroprep[™] Cleaners...soak cleaners and electrocleaners expressly formulated to prepare ferrous alloy surfaces for Niklad electroless nickel plating
- Multiprep[™] Cleaners...picklers, descalers, desmutters and activators expressly formulated for a wide variety of basis metals that are to be plated with Niklad electroless nickel

Plating Products

- Niklad™ Electroless Nickel Systems...nickel-phosphorus, nickel-boron, and polyalloy processes and companion products for engineering, electronic and printed circuit board applications
- Barrett SN® Sulfamate Nickel System...high-purity, low-stress nickel plating for electroforming and plating applications
- Isobrite® Brighteners...additives and systems for bright nickel, chromium, zinc, cadmium and copper electroplating
- ARP® Specialty Chemicals...includes metal strippers, wetting agents, bath contamination control additives, mist suppressants, foam control additives, chromate coating tester, blackening salts, and immersion tin and zincate processes

Protective/Pre-Paint Coating Products

- Iridite® Chromate (Or Chromate-Free) Conversion Coating Systems
- Keykote® Zinc, Iron And Manganese Phosphatizing Systems
- Irilac® Clear Protective Treatments
- Iridize[™] Zinc Anodize Coatings

Industrial Cleaning Products

■ Chemicals for cleaning and maintaining machinery, paint lines, refinery and petrochemical equipment, vehicles and aircraft

Support Material And Equipment

■ Everything that's needed to support your operation including basic chemicals, anodes, polishing equipment, tanks, rectifiers, automatic feed systems, heaters, test apparatus and kits.



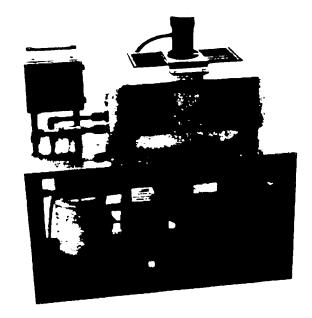


LANCY INTERNATIONAL, INC.

TECHNICAL BULLETIN

NUMBER 3203

LANCY ELECTROLESS BATH TREATMENT CELL



The Lancy E.B.T. Cell uses metallic electrodes which enhance the deposition of the recovered metal. The cell design permits recovery from low concentration rinsewaters and process baths without usual cathode polarization problems.

The Lancy Electroless Bath Treatment Cell (E B T) provides a recovery technique that will handle most spent electroless dumps and rinses without sludge generation.

An E.B.T. Cell will collect metals as a non hazardous slab. The metals are easily stripped from reusable cathodes following recovery from these rinses or baths:

- Spent electroless copper
- Spent electroless nickel
- · Spent electroless cobalt
- Drag out rinses from electroless process solutions

FEATURES

- Simple operation
- Removes metal as a non hazardous slab
- Versatile application copper - nickel - cobalt - palladium
- Inexpensive metallic electrodes
- Low energy requirements

BENEFITS

- Eliminates strong reducing agents
- No sludge by-product
- Addresses difficult waste treatment problems of chelated electroless dumps and rinses
- Low operating cost
- Operates at 250% or higher efficiency

SPECIFICATIONS

Typical Operating Parameters

Temperature: 140°F (60°C)

pH range: 8.3 - 8.8

Metal recovery rate: 0.1 lbs/hr/ft2

Materials of Construction

Tank

Polypropylene

Frame

FRP coated tubular steel

Electrodes

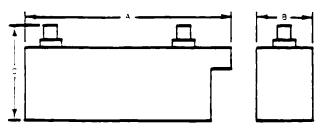
Stainless steel, lead alloy, aluminum. Karbate 22^{rm}.

mild steel

Agitator Shaft

Stainless steel Alloy 20

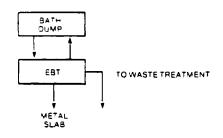
Tank Dimensions



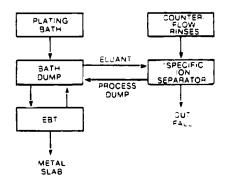
		Dimensions		Cathode	Cell	Weights	
Series	A. Length	B. Width	Overall C. Height	Area (sq. ft.)	Capacity (gallons)	Shipping (pounds)	Operating (pounds)
3221	38 '	18	22	1	13	436	340
3222	65 ·	23 ·	56 °	4	60	1,234	1,224
3223	80 '	31 '	74 "	10	220	2,499	3,678
3224	96 ·	31 '	75 ·	20	260	3.044	4.488
3225	115	31	75 *	30	330	3,542	5,440
3226	119	31	88 ⁻	50	440	4.587	7,344
3227	145	31	88 '	80	560	5,379	8,976
3228	171	31 ′	88 -	100	670	5.824	10,170

TYPICAL ELECTROLESS TREATMENT PROCESSES

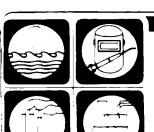
Dumped Bath Treatment



Combined Treatment For Baths and Rinses (Sludgeless)







LANCY INTERNATIONAL, INC.

525 West New Castle Street, P.O. Box 490, Zelienopie, PA 16063 • Phone (412) 452-9360

TELEX 86-6259

SPENT NIKLAD EN BATH TREATMENT

- 1). Transfer solution to a treatment tank
- 2). Heat solution to 150°F
- 3). With agitation adjust pH to 6.0-7.0 with 25% sodium hydroxide
 - 4). Check and maintain sodium hypophosphite level at 26.0-30.0 g/l (3.5-4.0 ozs/gal). Adjust hypo level by using Niklad 790.
 - 5). Slowly add with agitation Niklad WT Additive. Initial add of 0.2% by volume is required. Keep temperature at this step not higher than 150°F. Higher temperature will cause violent reaction.
 - 6). Spent EN solution will start to decompose at this stage.
 - 7). Continue treatment maintaining the proper pH, temperature, sodium hypophosphite and WT Additive additions until desired nickel content is obtained.
 - 8). Allow nickel powder sludge to settle to the bottom of the tank. The effluent can now be polished through ion exchange to reduce nickel level below 2ppm.

 With this treatment nickel level in the effluent is reduced to about 5.0 ppm

Witco

NR/df

Witco Corporation 1250 North Main Street Los Angeles, CA 90012 TEL 213-222-0201 In CA 800-223-4520

Anil Lashkari
Electronics
Service Supervisor
Allied-Kelite

CHEMICAL COSTS TO TREAT 1 GAL NIKLAD 1000

SODIUM HYDROXIDE	AMOUNT REQUIRED 10% by vol. of 25% NaoH solution	0.08¢
SODIUM HYPOPHOSPHITE (\$1.40/lb	25% by volume of 600 g/l hypo	\$1.75
WT ADDITIVE (\$110.61/gal)	0.3% by volume	0.33¢
	TOTAL	\$2.16

About 20% less for Niklad 794, 795, 719 type baths as nickel concentration is 6.0 g/l while it is 7.5 g/l in Niklad 1000.

NR/df

WITCO CHEMICAL CORPORATION Allied-Kelite Division 1250 North Main Street Los Angeles, CA 90012

Attn: ANil Lashkari

PURCHASE ORDER NO.

Pacific Spectrochemical Laboratory, Inc.

Chemical and Spectrographic Analysis

2558 Overland Avenue Los Angeles, California 90064

April 20, 1989

SEMIQUANTITATIVE ANALYSIS

Ni P 2.82 Na 2.00 0.68 A1 0.030 Si 0.017 Mg 0.0041 Cr 0.0071 Ca 0.0078 Cu 0.0015 Гe 0.013 Other elements

nil

per Syamala P WITCO lab 1-15-90 23 pm

Respectfully submitted,

PACIFIC SPECTROCHÉMICAL LABORATORY, INC.

CHAM HILL TELEPHONE CONVERSATION RECORD CALL TO Witco Xaboratory
Sus mala PHONE NO. 213/222 -0201 Syamala DATE 1-15-90
TIME 230 PROJECT NO. SACA8722-0022 SUBJECT NIKLAD WT Additive 1. Anil fashkari was not in today so the receptionist connected me to the lab. 2. Ayamala informed we that the WT Additive in the "Apent NIKLAD EN Bath Freatment" instructions is not WT-110. She is not oure if they have a data sheet but she took my name & said Anil would lither send me a data sheet or talk to me a what it is tomor-3. Fyangela Rail the WT Additive was not sodium hypophosphite. 4. The nickel powder concentrations fiven in the instructions we gercentages basel na her if the units were mg/mg-she said ges. D-21

Niklad™ Waste Treatment Process

For electroless nickel and cobalt plating solutions

Allied-Kelite Division, Witco Corporation 2701 Lake Street, Melrose Park, IL 60160, (312) 450-7435

Supersedes 050885

071387

Introduction.

The Niklad Waste Treatment Process is designed to remove nickel and cobalt metals from spent electroless plating solutions and rinses. The 2-component process (Niklad Precipitant and Niklad Flocculant) chemically reduces the solution nickel content to less than I ppm. without requiring the purchase of costly treatment equipment. The process produces an effluent suitable for discharge into sanitary sewers, and a metal sludge suitable for landfill disposal in approved sites or resale for electrolytic metal recovery, which can make your waste treatment process help pay for itself.

Advantages.

 Components are in liquid form —— Niklad Precipitant and Niklad Flocculant Easy to measure and use

 Chemically reduces metal content No costly treatment equipment required

 Produces effluent suitable for sewer discharge and metal sludge suitable for landfill or resale Treated wastes can easily be disposed of; sludge can be resold for metal content

Equipment.

In general, equipment that is used for Niklad plating baths is suitable for the Waste Treatment Process.

Tanks.

Tanks fabricated from polypropylene, polyethylene, or stainless steel are suggested.

Agitation.

Agitation is required to mix the process chemicals into the solution. Mechanical or clean air agitation is satisfactory.

Filtration.

Continuous filtration is not required. The settled metal compound precipitate can be run through a filter press to increase its solid content.

Treatment cycle.

CAUTION: Before handling or using the Niklad Waste Treatment Process, refer to the Safety and handling section on page 2 to be certain you are aware of the protective gear you must wear, and the first-aid procedures to follow in the event of accidental exposure to liquid concentrates or solutions.

- 1. Allow the spent Niklad plating bath to cool to room temperature before treatment.
- 2. Dilute the bath so that the nickel content is between 500 and 2500 ppm.
- 3. Determine the amount of Niklad Precipitant required using the following procedure:
 - A. Pour 1000 mL of the diluted bath into a 1500 mL beaker.
 - B. Adjust the pH of the sample to 8-9 (electrometric) by slowly adding 50% caustic soda solution to the beaker with agitation.
 - C. Pipette 1.0 mL of Niklad Precipitant into the beaker. If a precipitate forms, stir the solution and let it settle.
 - D. Repeat step C until no new precipitate is formed with the addition of Niklad Precipitant.



THE HADELESS NICKEL SPECIALTY PRODUCTS

- E. The last addition of Niklad Precipitant that produced a precipitate determines the amount necessary to treat your effluent. For every 1.0 mL added that produced a precipitate, 1 gallon of Niklad Precipitant must be added per 1000 gallons of effluent (1 Litre per 1000 Litres).
 - Example: If no precipitate formed from the fifth 1.0 mL addition, then four gallons of Niklad Precipitant are required per 1000 gallons of effluent for treatment.
- 4. Raise the pH of the bath to 7-8 by slowly adding 50% caustic soda solution to the beaker with agitation.
- 5. Add the required amount of Niklad Precipitant with continuous agitation.
- 6. Add 1 pint per 1000 gallons (0.125 Litres per 1000 Litres) of Niklad Flocculant with agitation to accelerate the rate of settling.
- 7. Mix well for 15 minutes, then allow to settle for one hour.
- 8. Check metal content of effluent. If within EPA and other applicable regulatory guidelines, filter through a filter press to separate and compact the metal sludge. The effluent can now be discharged into the sanitary sewer, and the sludge properly disposed of or sold to nickel reclaimers.

Warranty.

We warrant our goods to conform to our standard specifications. This Warranty is in lieu of any and all other warranties or guarantees and our obligation hereunder for breach of such warranty is limited to either refund of the purchase price or replacement of said goods as we may elect. We make no other warranties, express or implied, including those of MERCHANT-ABILITY and FITNESS FOR A PARTICULAR PURPOSE.

We believe that the statements, technical information and recommendations contained herein are reliable, but are given without warranty or guarantee of any kind except as specified above. Beyond that specified above, we assume no responsibility for any loss, damage or expense, direct or consequential, arising out of the use of, or inability to use, our goods. Their quality and suitability for any particular purpose or use should be confirmed by the user's own tests.

Safety and handling.

CAUTION: Niklad Precipitant and Niklad Flocculant are harmful if swallowed. Do not swallow. Avoid contact with skin, eyes, and clothing. Store in tightly closed containers at room temperature and away from strong acids.

PROTECTIVE EQUIPMENT: When handling Niklad Precipitant and Niklad Flocculant liquid concentrates and solutions, wear rubber gloves, safety goggles, face shield and chemical-resistant clothing. Wash thoroughly after handling. The area immediately around the plating tank should be ventilated.

FIRST AID: In case of skin or eye contact, immediately flush with large amounts of water for at least 15 minutes. For eye contact, immediately call a physician. If swallowed, immediately call a physician. If inhaled, remove victim to fresh air and call a physician.

SPILL OR LEAK: Wear protective equipment during cleanup of a spill or leak. Absorb spill or leak with an inert material such as sand, earth or vermiculite. Dispose of residue consistent with federal, state and local regulations.

REQUIRED READING: Niklad Precipitant and Niklad Flocculant are for industrial use only. Read Material Safety Data Sheets and product labels before using. Also, follow supplier's safety recommendations for all other chemicals mentioned in this technical bulletin.

Standard packages.

Niklad Precipitant — 5 gallons, 55 gallons Niklad Flocculant — 5 gallons, 55 gallons

NOTE: Keep Niklad Precipitant and Niklad Flocculant from freezing. If frozen, thaw to room temperature and mix well before using.

NOTE: All gallon measurements are U.S. Gallons.



APPENDIX E

ELECTROLESS NICKEL STRIPPING VENDOR INFORMATION

ABXO DATA SHEET	E-1
ABCO PRECIPITATION METHOD	E-3

U.S. DEPARTMENT OF LABOR Occupational Safety and Health Administration

Form Approved
OMB No. 44-91347

MATERIAL SAFETY DATA SHEET

Required under USDL Safety and Health Regulations for Ship Repairing

Shippuilding,	ind S	hippreaki	ng (29 CFR 1915, 1916, 1917)				
		SEC.	TION I			7	
ABCO PRODUCTS OF SACRAMENTO EMERGENCY TELEPHONE NO: (916) 447-9931							
ADDRESS, Number, Street, City, State, and ZIP Co P.O. Box 9613, Sacramento, CA	de) 958	23				7	
CHEMICAL NAME AND SYNONYMS			: Nickel Stripper 99			1	
THE MICAL FAMILY			Proprietary			7	
						- 1	
SECTION	11 -	,	RDOUS INGREDIENTS			4	
PAINTS, PRESERVATIVES, & SOLVENTS	*	(Unital	ALLOYS AND METALLIC COATINGS	*	(Umas)		
PIGMENTS			BASE METAL]	
CATALYST		ļ	ALLOYS				
VEHICLE			METALLIC COATINGS				
SOLVENTS			FILLER METAL PLUS COATING OR CORE FLUX] .	
ADDITIVES			OTHERS]	
OTHERS						1	
HAZARDOUS MIXTURES	0F (THER LI	عادان ، بحدث المحادث ا	*	(Units)		
Sodium Carbonate CAS	S No	. 139-	02-6	> 50	Not as	tablished	
Organic Compound Non-hazar	rdou	is acco	rding to the criteria of	₹ 50		11	
paragraph (d) of the OSHA }	Haza	ird Com	munication Standard. All per-				
health effects of the chemi	i ca 1	(s) is	opercies, safe handling, and disclosed of this MSDS			-	
SEC	TION	N 111 - P	HYSICAL DATA				
SOILING POINT ("F.)	no	ne	SECIFIC GRAVITY (H20=1)	8.3	3 1bs/d	a l	
VAPOR PRESSURE (mm Hg.)	N/	A	PERCENT, VOLATILE BY VOLLME (%)	N/A			
VAPOR DENSITY (AIR+1)	N/	A	EVAPORATION RATE		4		
SQLUBILITY IN WATER	com	plete					
APPEARANCE AND ODOR White to yel	low	granul	es				
SECTION IV -	FIRE	E AND E	XPLOSION HAZARD DATA				
FLASH POINT (Method used) N/A			FLAMMABLE LIMITS LM		Ųe1		
EXTINGUISHING MEDIA WATER Spray							
SPECIAL FIRE FIGHTING PROCEDURES	aid	eve ar	nd skin contact			1	
UNUSUAL FIRE AND EXPLOSION HAZARDS	AI.					1	

PAGE (1)

(Continued on reverse side)

None

Form OSHA-20 Rev. May 72

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	•	SECTIO	NV - HEA	LTH HAZARD DATA
THRESHOLD LI	MIT VALUE			
EFFECTS OF O	VEREXPOSURE	Eye & Ski	n Contact	: may cause minor irritation.
Inhalation	: Dust may			o the respiratory tract.
lb minutes irritation fresh air	. Get medi develops, Call physi	cal attent get medica cian. INS r having c	ion. SKI lattenti ESTION: novulsion	lush eyes with flowing water for at least N: Wash with mild soap and water. If on: INMALATION: Move exposed individual Never give fluids or induce vomiting if s. Get medical attention EACTIVITY DATA
				ZACTIVITI DATA
STABILITY	UNSTABLE			NS TO AVOID
STABILITY	UNSTABLE		CONDITION	NS TO AVOID
INCOMPATABIL	STABLE	X	CONDITION	
INCOMPATABLE OXIDIZING	STABLE	X evoid)	CONDITION	NS TO AVOID
INCOMPATABIL OXIDIZING & HAZARPOUS O	STABLE SITY (Materials to agents ECOMPOSITION 6	X evoid)	CONDITION	NS TO AVOID

SECTION VII - SPILL OR LEAK PROCEDURES

Biodegradable. Large quantities may pose an aquatic hazard. Carefully sweep up material and transfer to a suitable container (steel drum) for disposal. Avoid creating a dusty atmosphere. After sweeping, flush area where spill occurred with water. Dilute water residue mixture may be flushed to sewer with large amts. of water waste disposal of material as solid waste. Small quantities may be flushed to sewer with large amounts of water. Discharge to a public sewerage authority should coincide with all applicable local permits and notification requirements. Follow all ciemical pollution control regulations.

	SECTION VIII	- SPECIAL PROTECT	TON INFORMATION	
Use NIOSH ap	proved dust mask	if ventilation is	inadequate.	
VENTILATION	Is recommended		SPECIAL	
	MECHANICAL (General) Is required.		OTHER	
PROTECTIVE GLOV Ceneral purpo	res se nloves		otection cal workers glasses, c	lo not weer lens
STHER PROTECTIV			W: 1171 1851 2 3 W 2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	

SECTION IX - SPECIAL PRECAUTIONS							
Precautions to be Taken in Handlin Precautionary [abe] Information	ción: CAUTION! May cause irritation. Avoid contact						
	g. Wash thoroughly after handling.						
OTHER PRECAUTIONS formal warel	nouse storage.						
The state of the s							

PAGE (2)

Form OSPLA-20 Rev. May 72



PRODUCTS CO. of SACRAMENTO

ETERGLÁIS — PAINT STRIPPERS

RUST REMOVERS - SOLVENTS & ANTI-CORROSION OILS

PHOSPHATE & CONVERSION CONTINGS + Olber Products, Processe and Equipment for Industry

P. O. Box 9613 / Sacramento, California 95823 / Telephone (916) 447-9931

ABCO NICKEL STRIP 99

For Stripping Nickel From Steel

ADVANTAGES

- 1) No corrosive liquids involved.
- 2) Easy to dispose of when exhausted.
- 3) Contains no cyanide./
- 4) Fast stripping rate.

EQUIPMENT NEEDED

- 1) Tank can be mild steel, polypropylene, or lined with liner to withstand the required temperature.
- 2) Agitation is needed. (Clean mild air, or mechanical.)
- 3) Heaters large enough to maintain 120° to 150°F;

Bath Make-up

- 1) Fill the tank half full with water. 120° to 150°F. Add 2.5 lbs. of ABCO NICKEL STRIP 99 for each gal. of working solution.
- 2) Fill the tank to working capacity. Stir bath to dissolve powder. Heat to 120° to 150°F. Adjust pH to 8.5 to 10.5. If below 8.5, add Soda Ash to bring into range.

CAUTION

May cause skin or eye irritation. Avoid contact with skin, eyes and clothing. Avoid breathing dust. Use with adequate ventilation. In case of contact, flush skin or eyes with plenty of water, for eyes, get medical attention.

IMPORTANT NOTICE REGARDING THIS INFORMATION

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PHOSPHATE & CONVERSION COATINGS + Olber Products, Procuse and Equipment for laduity

P. O. Box 9813 / Sacramento, California 95823 / Telephone (918) 447-9931

ABCO NICKEL STRIP 99 For Stripping Nickel From Steel

Bost Cleaning

- 1) Parts should be clean before entering the stripping bath.
 The normal method of cleaning steel should be adequate.
- 2) Rinse parts well before stripping.
- 3) Immerse parts in stripping solution, being careful not to touch sides of the tank, or the heating coils. Parts should be spaced, to avoid laying flat together. For maximum performance, fill tank with as many parts as possible.

Stripping Rate

A new bath, when used at optimum conditions, should strip nickel at a rate of 1 to 2 mils per hour. Time for stripping Electroless Nickel is dependent on the phosphorous content.

ANALYTICAL PROCEDURES

- 1) Pipet a 1 ml. sample into a 250 ml. Erlenmeyer flask.
- 2) With a graduate, add 50 ml. of distilled water, 10 ml. of ammonia buffer (1 molar ammonium chloride and ammonium hydroxide) and one tablet of murexide indicator (0.4 mg. tableted with potassium chloride). Swirl to dissolve.
- 3) Fill a 50 ml. buret with 0.1m disodmum EDTA and titrate the solution until the color changes from yellowish-green to deep purple.
- 4) Read the buret. The ml. of EDTA used, multiplied by 0.78, gives the ounces of nickel metal per gallon of plating solution.

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PRODUCTS CO. of SACRAMENTO

ETERGENTS - PAINT STRIPPERS

RUST REHOVERS - SOLVENTS & ANTI-CORROSION OILS

PROSPRATE & CONVERSION CONTINGS + Older Products, Process and Equipment for Industry

P. O. Box 9813 / Sacramento, California 95823 / Telephone (916) 447-9931

ABCO NICKEL STRIP 99 For Stripping Nickel From Steel

Disposal

One of the better advantages of ABCO NICKEL STRIP 99 is the disposal. It contains no chemicals that would be considered hazardous or unacceptable in a public treatment center. It is completely bio-degradable after removal of nickel.

The following are two methods of nickel removal.

- 1) Nickel hydroxide precipitation through pH adjustment.
- 2) Nickel oxide precipitation through treatment with sodium hypochlorite.

Method #1 can be better accomplished if the spent bath is diluted as much as possible after adjusting the pH to 11.5-12.5.

Method #2 gives much less precipitate, however, the solution must stay in the alkaline pH range and should be filtered as soon as possible to prevent rehydration of the oxide to nickel hydroxide.

NOTE: "Material Safety Data Sheet" available on request.

IMPORTANT NOTICE REGARDING THIS INFORMATION

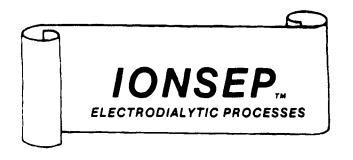
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E = 5

APPENDIX F CHROME STRIPPING SOLUTION VENDOR INFORMATION

IONSEP RECOVERY SYSTEM

F-1



FOR:

MORE EFFICIENT USE OF CHEMICALS

MORE RELIABLE PROCESSING

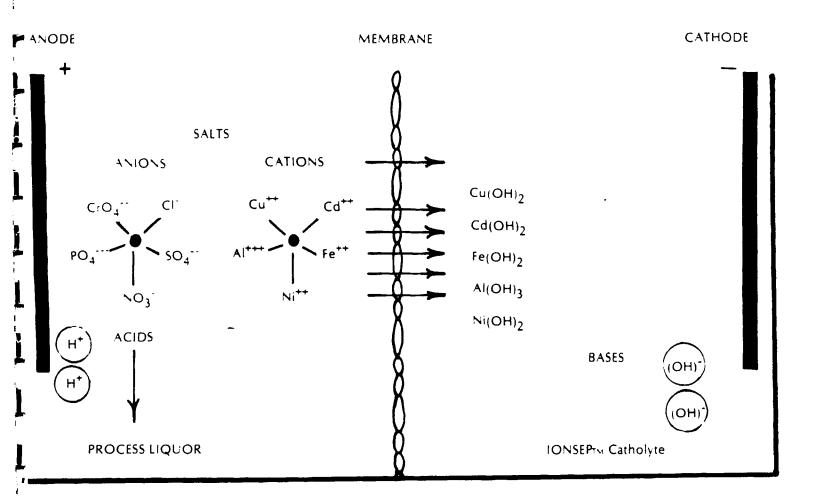
CLOSED LOOP PROCESSING

Each year thousands of tons of heavy metal waste are made in chemical processing that must be securely landfilled. This waste (inefficient use of process chemicals) increases cost of manufacture and results in a high and increasing cost of waste disposal. Ionsep's new technology provides for more efficient use of chemicals, more reliable processing and closed loop processing.

IONSEP PROVIDING NEW TECHNOLOGY

Acids are reacted with bases and metals everyday to make salts. If it was possible to convert salts in an aqueous solution back to separate acids and bases, acids and bases could be used again and again. Ionsep has developed and is now making available commercially a simple process for converting any salt in an aqueous solution into the separate acid and base of the salt using electricity in a membrane electrodialytic process. This simple capability of converting a salt to an acid and a base makes IONSEP Electrodialytic Processes proadly useful in chemical processing.

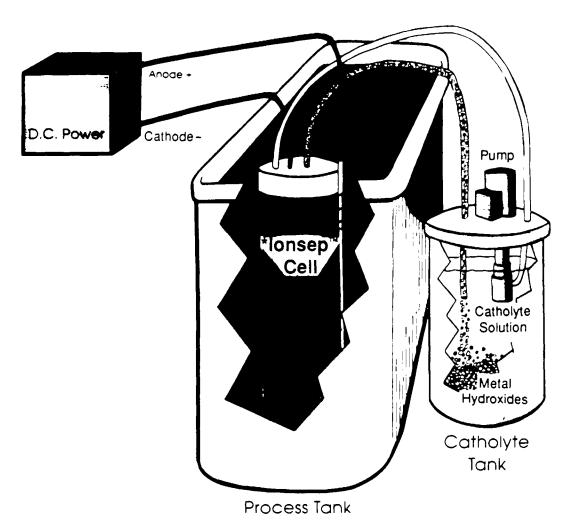
CONVERSION OF SALTS



THE IONSEP

A SIMPLE PROCESS

The IONSEP process comprises a membrane electrochemical cell, designed for immersion in a process liquor, a rectifier, a process liquor containing metal salts, an IONSEP catholyte solution and a pump to flow the catholyte solution through the cell. The membrane separates the process liquor from the catholyte solution and acts as an "Electrochemical Traffic Controller" that lets metal cations go from the process liquor through the membrane (electrofilters the metals) into the catholyte solution and keeps anions in the process liquor. The metal cations are continuously converted to hydroxides in the catholyte solution and the anions are continuously converted to acids in the process liquor. The hydroxides of multivalent metals (cadmium, zinc, iron, copper, aluminum, calcium, etc.) are substantially insoluble in the catholyte and can be removed for use. The IONSEP process is unique in that salts of multivalent metal cations can be converted. There is essentially no electrodeposition of metals. The IONSEP process can be operated at reproducible capacities for months. The capacity of the IONSEP process is varied by voltage.



ROCESS ...

A BROADLY USEFUL PROCESS

CHROMIC ACID PLATING LIQUORS

- All metal impurities removed and maintained at low level
- Permits recycle of rinse or rinse concentrate to plating
- Provides reliable plating with less power and higher rates
- Provides closed loop processing for chromic acid plating

CHROMATING LIQUORS

- Metals removed and acids reformed continuously
- Liquors maintained at composition for best chromating
- Disposal of chromating liquor essentially eliminated
- Provides for more reliable chromating, lower use of chemicals and potentially closed loop processing.

ACIDS—ETCHING, ANODIZING, PICKLING ETC.

- Acids reformed continuously and metals removed
- Composition of liquors maintained essentially constant
- Provides more reliable processing and more efficient use of chemicals
- Potential for closed loop processing

CAUSTIC ELECTROSTRIP—SODIUM CHROMATE SOLUTIONS

 Converts sodium chromate to sodium hydroxide for reuse and chromic acid for another use with removal of multivalent metal impurities.

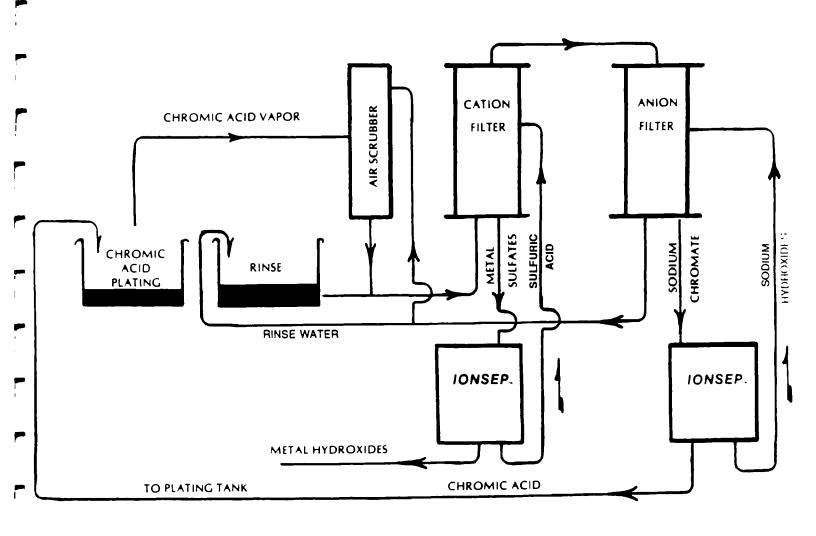
ORGANIC DYES FOR METALS

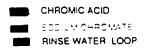
- Dyes restored-metal impurities removed from dyebath
- Provides more reliable dyeing and lower dye usage
- Potential for closed loop processing.

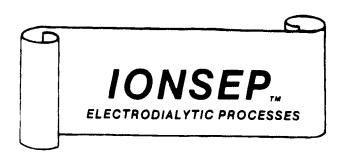
CLOSED LOOP PROCESSING

A process with no loss of chemicals to the environment (closed loop) may not be possible. But, it is desirable to lower cost of manufacture by operating more reliable and efficient processes while reducing waste. *IONSEP* electrodialytic processes provide two important steps towards closed loop processing: (1) purification and restoration of acidic and alkaline process liquors: and. (2) reforming and separating chemicals used to regenerate ion filters.

IONSEP'S CLOSED LOOP SYSTEM







Ionsep Corporation provides:

- *equipment
- *installation
- *maintenance
- *chemicals
- *technical service

For IONSEP Electrodialytic Processes.

License required to operate IONSEP Electrodialytic Processes.

U.S. Patent Nos. 4,325,792 and 4,439,293 and 4,636,288 4,652,351 and 4,684,453 and patents pending.

> Publication Date: May 16, 1988 Printer: Sprint Quality Printing, Inc.

IONSEP CORPORATION INC. P.O. Box 258 Rockland, DE 19732

302-764-7849 302-798-7402 302-475-2198

APPENDIX G

CADMIUM CYANIDE VENDOR INFORMATION

LANCY ELECTROWIN

G-1

Series 3220

This series of Electroless Bath Treatment Cells is used
for the batch treatment of spent electroless bath dumps and
rinsewaters. Lancy EBT cells recover electroless copper.
nickel, cobalt and palladium in non Eszardous slabs. After
recovery, the metals can be easily stripped from reusable
catnodes. This series is available in eight standard models.
ranging in size up to 670 gallons capacity

SERIES	S 3230		DI	MENSIO	NS
MGDEL NO	SATHOSE SLATE AREA	OEL. SARACITY Gallins		A 01-	- E G-*
3231	4	7	36	24	35
3232	88	14	36 "	24	35
3233	12	21	48 "	36	35
3234	16	28	48 "	36	35
3235	20	35	48	36	35
3236	24	42	52 "	36	 3 5

For small capacities, the ECONO-CELL version of Series 3200 can be used. Like the heavy duty Series 3200. ECONO-CELLS reduce sludge generation and eliminate the reintroduction of impurities into the process stream. The Series 3230 is available in six standard sizes ranging from 7

Series 3230

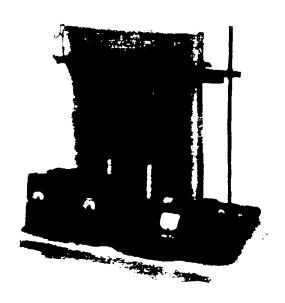
to 42 gallons capacity

Note: Dimensions on ail series may vary depending on application

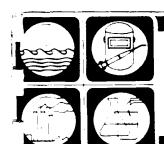
•	SERIES	S 3220		DI	MENSIO	NS
	MODEL NO	CATHODE PLATE AREA	CAPACTY CAPACTY Capacts	_ENG~-		HE GH*
	3221	4	7	36 "	24 '	35 ′
	3222	8	14	36 "	24 "	35 "
-	3223	12	21	48 "	36 "	35 "
	3224	16	28	48 "	36	35 "
-	3225	20	260	96 "	31	75 "
	3226	50	440	119"	31	88 "
_	3227	80	560	145 "	31 "	88 "
	3228	106	670	171	31 "	88

Series 3240

This series of Electrolytic Cells is designed specifically for the batch destruction of cyanide plating baths. Series 3240 cells reduces operating costs of standard chemical treatment systems by electrolytic destruction of the cyanides. Series 3240 Electrolytic Cells are custom designed by Lancy for each specific application



3231 ECONO CELL



LANCY INTERNATIONAL, INC.

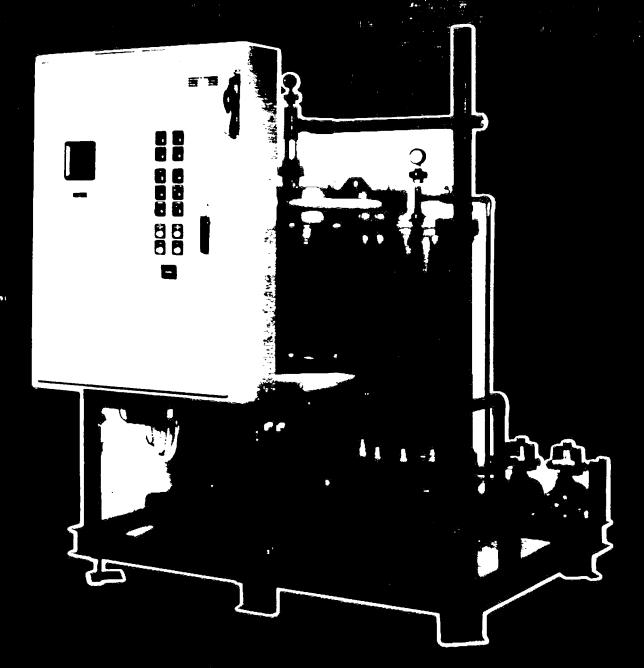
525 West New Castle Street, P.O. Box 490, Zelienople, PA 16063 • Phone (412) 452-9360

TELEX 86-6259

APPENDIX H ACID PICKLING SOLUTION VENDOR INFORMATION

APU SYSTEM H-1

ECO-TEC





APU: Practical separation of acids from metal salts

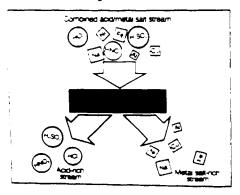
Liquid mixtures consisting of inorganic acids (sulfuric, hydrochloric, phosphoric, nitric) and dissolved metals (iron, nickel, copper, aluminum, etc.) are often discarded by industry. In the steel industry. for example, sulfuric or hydrochloric pickling acids are considered spent and are discarded once the dissolved iron concentration builds up. In the mining industry, on the other hand, hydrometallurgical leach circuits containing dissolved metal salts may require purging due to a build-up of acid. While the cost of replacing these acid or salt solutions is significant. the high cost of waste treatment is often a more important consideration.

Separation of the acid and dissolved metal has generally been considered impractical—until now.

The Eco-Tec Acid Purification Unit, or APU, is a unique system developed for the separation of dissolved metallic salts. The separated acid or metal may be recycled for reuse. The system uses only water in its process. No other chemicals and virtually no energy are required.

The outstanding capabilities and features of this field-proven system have justified acid-metal separation in a large number of applications that were not previously even considered:

- Steel and galvanized steel pickling, using sulfuric or hydrochloric acid
- Copper and brass pickling, etching and brightening, using sulfuric or nitric acid
- Aluminum anodizing, using sulfuric acid
- Hydrometallurgical ore leach circuits
- Stainless steel and titanium pickling, using nitric and hydrofluoric acids
- Electroplating rack or rework stripping, using nitric acid
- Aluminum bright dipping or electropolishing, using phosphoric and nitric acids or phosphoric and sulfuric acids
- Stainless steel electropolishing, using phosphoric and/or sulfuric acids
- Spent regenerants from acid cycle cation exchangers.



Benefits and features

Reduced acid consumption: Much of the acid (40 to 70 percent) used in metal-finishing operations such as pickling, anodizing or etching, is lost when a spent bath is discarded. An APU processes this acid for reuse. It reduces acid consumption essentially to the amount required to dissolve the metal and make up for dragout losses.

Reduced waste treatment: Since acid is purified and reused, alkali requirements for neutralization are reduced. In some cases, such as with lime neutralization of sulfuric acid, less sludge is generated, resulting in lower disposal charges.

Reduced labor costs: Continuous purification of acid process baths eliminates the need for decanting or discarding and replenishing. Because these operations cannot be carried out during regular working hours, eliminating them often results in a saving of premium labor charges.

Reductions in acid handling also reduce the possibility of personal-injury accidents.

Reduced production downtime: Acid process baths can be decanted and replenished only when production is stopped. In many plants, this results in costly downtime and lost production. Continuous purification using an APU can extend the life of an acid processing bath indefinitely.

Uniform production quality: The composition of an acid processing bath changes as it is operated. The dissolved metal concentration increases, while the free acid concentration decreases. Consequently, immersion times in the process bath must be varied. With too short a time in the bath, the surface finish may not be acceptable. With too long a time, acid is used up needlessly. Only an experienced, conscientious operator can ensure that optimum immersion times are maintained.

With an APU, however, a constant bath composition is maintained, resulting in a consistent surface finish at a constant immersion time. The acid processing operation then lends itself to automation.

Metal electrowinning facilitated: The presence of acid often interferes with the efficient operation of electrowinning processes. The APU can be used economically to continuously remove acid from electrowinning solutions.

Increased energy efficiency. Some acid processing baths must be operated at elevated temperatures in order to extend their life once they become contaminated. By maintaining a bath with a constant, low contaminant level, lower temperatures malbe used and energy savings gained.

Similarly, electrolytic acid processes such as aluminum anodizing, require higher voltages as contaminant levels rise in the process solution. By maintaining the bath at a low contaminant level, lower voltages may be used. And since the process is more energy efficient, less cooling is required to dissipate the waste heat generated at higher voltages.

Low operating costs: The process uses only water. The only energy consumed is that used to pump acid and water through the unit. The process is so simple that is requires a minimum of supervision and maintenance.

Low capital cost: Individual units start as low as \$12,000, depending on the application

Low installation costs. The APU comes pre-assembled and skid-mounted to keep shipping and installation costs to a minimum.

Compact size: APUs vary in size: depending on capacity. However, a small unit requires only about 6 ft² (0.6 m²) of plant space, while a large unit requires only about 25 ft² (2.3 m²). For greater capacities, multiple units are used

Full automation: The APU needs only to be switched on and off. No special backwashing, regeneration or other manual operations are normally required

High reliability: Improvements made during years of operating experience, and use of quality components, have resulted in equipment which is simple and reliable

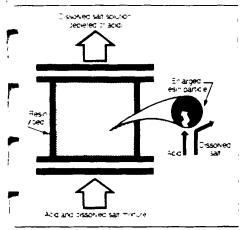
Corrosion-resistant components. Nonmetallic wetted surfaces throughout and a two-part epoxy-coated framelensulationg equipment life, even in the most corrosive environments.

The APU separates inorganic acids and dissolved metal salts.

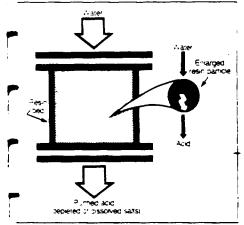


Poperating principle

The APU employs Eco-Tec's patented Recoflor technique for efficient operation. The heart of the APU is a ped of special corption resin. This resin has the unique ubility to sorb strong mineral acids yet exclude metal salts. This is not an ion exchange process since only water is used to desorb the acid from the resin.



During the sorption step, the acid and metal salt mixture ted up through the resin bed. Acid is sorbed into the isin while the remaining dissolved metal salts are liected as midly acidic solution leaving from the top of the bed. Depending on the metal salt, this solution may be waste-treated or diverted to an electrowinning cell for metal.



During the desorption step, water flows down through the resin bed. Acid is desorbed from the resin and esplaced from the pottom of the oed. City water is pically adequate for this step.

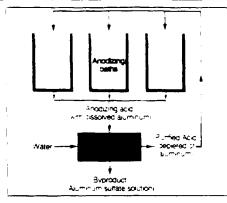
Due to the system's design, dilution of the acid is kept to an absolute minimum. In some cases, this purified acid is more concentrated than the contaminated acid ed. The resin is stable under normal operating conditions for many years without the need for regular inplacement or any special treatment.

Typical applications

Standard APU models of varying Lapacity are used in a variety of configurations for different applications. Following are some typical examples:

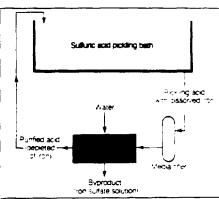
Aluminum anodizing

A single APU is typically used to maintain a number of aluminum anodizing baths at a constant dissolved aluminum concentration. Bath dumps are virtually eliminated, consistent anodic thicknesses are achieved at constant voltages, and acid consumption is reduced.



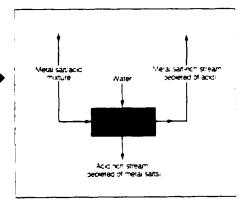
Sulfuric acid steel pickling

Pickling solution is first passed through a depth media filter to remove scale and other undissolved solids, then through the APU to remove dissolved iron. Acid and alkali (used for neutralization) consumption is reduced, waste sludge volume is reduced (if lime is used for neutralization), and composition of the pickling bath remains constant for a consistent surface finish.



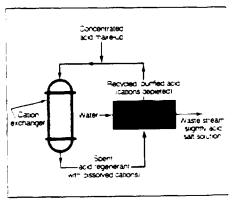
Acid removal from metal salt solutions

Electroplating or electrowinning metals out of metal salt solutions often generates acid that must be removed from the system. An APU is used to remove excess acid from the metal salt solution. No chemicals are required for this process so no additional materials are introduced into the metal salt solution.



Cation exchange regenerant recycle

An APU is used to recover excess acid from acid cycle cation exchange spent regenerant solutions. The recovered acid is fortified with concentrated acid and reused as fresh regenerant. When used with Eco-Tec cation exchangers, it can reduce acid consumption to a level approaching the theoretical requirement.



Eco-Tec: Acid Purification Unit (APU)

Equipment description

Standard models of varying capacity are available for a variety of applications. Where the requirements cannot be met by a single APU, multiple units may be used.

A standard APU consists of a cartridge filter, a vessel containing the sorption resin. plastics piping, valving and a control system, all mounted on a rugged, epoxycoated steel frame. Small APU models use hydro-pneumatic tanks to meter the flow of acid and water through the resin bed Larger units use a microprocessor-controlled flow sensor.

Options include additional pre-filters pumps, and high-temperature-resistant construction. Eco-Tec can also supply all necessary tanks, pumps, valves, controls etc. to complete the system.

Equipment selection

Selection of the appropriate model is done by Eco-Tec process engineers, based on information collected from the client using an applications survey form. For example, an APU for a pickling or anodizing process is selected on the basis of acid concentration, desired dissolved metal concentration, and the metal build-up rate in the process bath. The APU is selected to remove metal at a rate equivalent to the build-up rate. Thus, a constant dissolved metal concentration in the process bath can be maintained.

In the event that the acid and metal mixture is somewhat unusual. Eco-Tec can perform treatability studies on bench scale or pilot plant equipment at its own laboratory. Only about 25 gallons (100 litres) of the client's acid is required to generate sufficient pilot plant data to properly select the correct APU model

A small pilot APU is also available on a rental basis for the client to evaluate the process in his own plant

Technical service

Each APU assembled at Eco-Tec's manufacturing facility is tested prior to shipment. Installation of the pre-assembled units is simple and can be accomplished by the client's own personnel.

Eco-Tec field engineers provide on-site start-up assistance operator training and continue to be available for consultation at any time.

All statements information and recommendations contained herein are its our knowledge, true and accurate. However no guarantee or warrants is quien expressed or moled into rishall any statement in commation or recommendation constitute a recresseriation less set forth in an agreement signed our Economic timited.

Typical results

Sulfuric acid	190	182	13
	10	5 5	6
Sulfuric açid	127	116	10
Iron	36	10 5	21
Nitric acid	514	581	10
Nickel & copper	99	47 5	70 8
Sulfuric acid	128	113	18
Hydrogen peroxide	41	35	7
Copper	13 3	5 9	9.2
Hydrochloric acid	146	¹ 46	10
	34	25	15
Natric acid	150	139	4 5
Hydrofluoric acid	36	28 8	7 2
ron	29	8 7	20 3
Nickei	7 02	2 1	4 9
Chrome	7 33	2 2	5 1
Sulfuric acid	61 3	54 9	5 88
Sodium	7 8	0 8	5 56

A pre-assembled iskid-mounted APU (arregula) iffle blant space and is readily installed







APPENDIX I

ALKALINE DERUST

LANCY ULTRAFILTRATION

I-1



LANCY INTERNATIONAL, INC.

TECHNICAL BULLETIN

NUMBER 3401

LANCY ULTRAFILTER

The Lancy Ultrafilter provides metal working facilities, metal finishers, platers, food processors, and chemical processors with a rugged filtration unit for hot or corrosive liquids and contaminated waste waters.

The Lancy Ultrafiltration Unit

- Separates stabilized oil water emulsions.
- · Removes oils and greases from hot, dirty alkaline baths:
- Separates paints and coatings from rinse waters:
- · Separates "hard-to-handle" materials

FEATURES

The key feature of the Lancy Ultrafilter is the unique inorganic membrane and rigid inorganic membrane support. Compared to the organic membranes and supports of conventional UF units, the refractory nature of the Lancy membrane system makes it impervious to chemical attack in virtually every industrial wastewater application, even at process temperatures of 200° F and above

The norganic membrane will reject all particulate matter, oil, grease, polymers, and emulsion micelles above a molecular weight of approx 20.000, while water, solvent, ions and smaller molecules will pass through

BENEFITS

Versatility

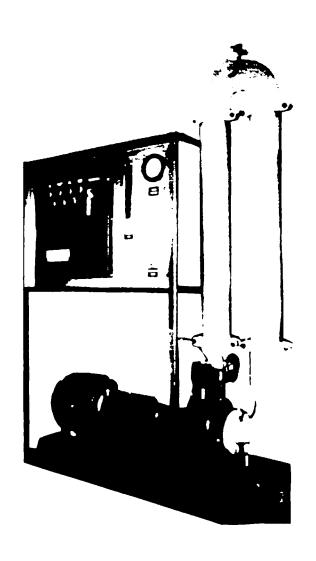
The mechanical and chemical stability of the Lancy Ultrafilter permits use over a broad range of pH, temperature, and corrosive, abrasive and solvating chemical environments at temperatures up to and above 200° F. Pre and post chemical treatment requirements are minimal. Systems are available for both batch and continuous operations

Low Maintenance

The inorganic tubes and membranes provide extended module life at high performance levels. This decreases down time and maintenance costs, resulting in increased productivity.

Compact

With the high permeation rates attainable, processing is maximized in a minimum amount of floor space. A typical 6,000 gallon per day oil water system occupies a $2^{7} \times 4^{1/2}$ skid.



Low Energy Requirements

The high ratio of surface to volume allows minimal feed volume per unit area. Pumping costs are thus minimized

Because the tubes and supports will not crush or compact at high temperatures, no precooling is required and the filtrate is returned close to operating temperature. Cooling and reheating costs are therefore less than those for organic membrane systems.

OPERATION

Figure 1.
Continuous Cleaning of Typical Hot Alkaline Cleaner

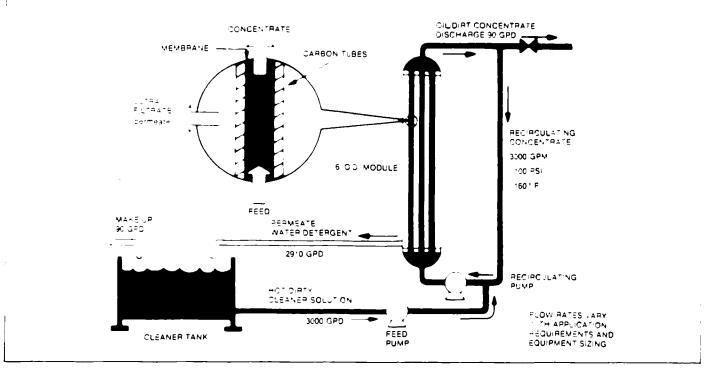


Figure 1 shows a typical arrangement for continuous removal of solids, free oils and emulsified oils to extend the life of an alkaline cleaning bath.

Hot process feed is continuously pumped into the filtration loop. A second pump circulates the feed through the tubes in the filtration loop. A second pump circulates the feed through the tubes in the filtration module. Water improve fluids and dissolved ions are forced at right angles to the bulk liquid flow through the membrane layer and walk of the portions tube to produce a clear filtrate. This filtrate (permeater is returned to the cleaning tank at a concentration and temperature close to that it the operating bath.

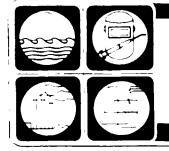
Within the futration loop, the reject concentrate, containing the dirt, metal particles, oils, etc., is recirculated together with fresh feed make up. A fraction of this circulating concentrate is continuously purged to maintain the fluid concentration in the loop at a specified value. This reject efficient may be discharged or used elsewhere.

For applications where the goal is to recover and concentrate the oils or solids rejected by the membranes, or to reduce disposal costs the same basic equipment would be piped in a batch arrangement so the filtrate (permeate) is discharged and the concentrate is recirculated to the feed tank. For a high-flow, continuous system, the concentrate can be directed to additional membrane modules connected in series

Operating Parameters

Temperature 200° F (93 ° C) or higher Feed Pressure 20 to 200 psi (138 to 1379 kPa) pH Range 1 to 14 Permeate Recovery: 90 to 98*

Not affected by most solvents



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APPENDIX J ALKALINE BATH CONTROLLER (PALM INDUSTRIES)



PALM INSTRUMENTS, INC.

PALM MODEL MP2000CC

SOLUTION CONDUCTIVITY CONTROLLER

Closed Loop Controller and SPC System for Alkaline Cleaners

- * Analyser and replenishment system for alkaline cleaners
- * Measures solution conductivity and concentration
- * Microprocessor operating system for maximum flexibility, advanced record keeping, and statistical data handling
- * In-tank, maintenance free sensor
- * Automatically adds liquid replenishers
- Records replenisher useage to allow tracking for cost analysis
- * Controllers are equipped with LCD readouts and keypads for Lasy use
- * NEMA enclosure for harsh environments
- * Four different baths can be controlled by one stand alone unit
- * Communicates with central processor via RS-232 data transfer
- * Data can be analysed by PC based SPC software
- * Up to eight controllers can be interfaced for data collection
- * Hard copies of the data can be printed