

WATTS NICKEL AND RINSE WATER RECOVERY  
VIA AN ADVANCED REVERSE OSMOSIS SYSTEM

by

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## NOTICE

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## FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of materials that, if improperly dealt with, can threaten both public health and the environment. The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

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

Passage of the Pollution Prevention Act of 1990 marked a strong change in the U.S. policies concerning the generation of hazardous and nonhazardous wastes. This bill implements the national objective of pollution prevention by establishing a source reduction program at the EPA and by assisting States in providing information and technical assistance regarding source reduction. In support of the emphasis on pollution prevention, the "Waste Reduction Innovative Technology Evaluation (WRITE) Program" has been designed to identify, evaluate, and/or demonstrate new ideas and technologies that lead to waste reduction. The WRITE Program emphasizes source reduction and on-site recycling. These methods reduce or eliminate transportation, handling, treatment, and disposal of hazardous materials in the environment. The technology evaluation project discussed in this report emphasizes the study and development of methods to reduce waste.

E. Timothy Oppelt, Director  
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## ABSTRACT

An Advanced Reverse Osmosis System (AROS) manufactured by Water Technologies, Inc. was installed in the Hewlett-Packard (HP) Printed Circuit Division plant in Sunnyvale, California during an 8 month test program from December 1989 through July 1990. This report uses information from Hewlett-Packard to assess the effectiveness of the AROS unit in the recovery of Watts Nickel plating bath solution and rinse water. In addition, the report estimates the incremental cost savings resulting from reduced deionized water use, reduced wastewater volume being pretreated, lower effluent and sludge disposal quantities, and recovery of plating solution.

A major achievement was that rinse water quality was maintained at a low level of nickel contamination. The recycling of the rinse water resulted in a dramatic reduction in the use of new deionized water makeup for this plating process. The AROS unit also successfully produced concentrated Watts Nickel solution of adequate quality for reuse in the plating bath solution.

The HP cost evaluation showed an estimated net annual savings of approximately \$17,000/year through use of the AROS unit. This compares to a capital expenditure of approximately \$75,000 (\$62,600 for the unit, plus installation and training costs). For Hewlett-Packard, the payback period was approximately 4½ years and a return on investment of about 23 percent.

The AROS unit at HP was operated at less than 50 percent of its hydraulic capacity. The economic benefits would have been more favorable if the Watts Nickel plating process had operated for more hours and treated more printed circuit boards. For example, the plating solution dragout at HP was estimated to average only about 0.2 to 0.3 gph, whereas the AROS unit is designed to recover 2 to 3 gph of Watts Nickel solution: ten times as much as was actually recovered.

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## SECTION 1

### INTRODUCTION

#### PROJECT BACKGROUND

This study was performed under the California/U.S. Environmental Protection Agency (EPA) Waste Reduction Innovative Technology Evaluation (WRITE) Program, and was a cooperative effort between EPA's Risk Reduction Engineering Laboratory (RREL), the Office of Pollution Prevention of the California Environmental Protection Agency, the Hewlett-Packard Co. (HP) Printed Circuit Division, Sunnyvale, California operation, and Water Technologies, Inc., Edina, Minnesota, which supplied the Advanced Reverse Osmosis System (AROS) used in the test program. Under the WRITE Program, the cooperative efforts of the EPA and State or local environmental programs are used to identify, develop, demonstrate, and evaluate innovative pollution prevention techniques. Specifically, the WRITE Program provides engineering and economic evaluations plus information dissemination for methodologies that have the potential of reducing the quantity and/or toxicity of waste produced at the source of generation, or to achieve practicable on-site reuse through recycling.

#### PROJECT DESCRIPTION

An AROS unit manufactured by Water Technologies, Inc. was installed in the HP plant in Sunnyvale, California to treat and recover Watts Nickel sulfate plating bath solution and rinse water. This report uses information from HP, plus contractor testing, to assess the effectiveness of the AROS unit in the treatment and recovery of metal plating bath solution and rinse water. In addition, the report estimates the incremental cost savings resulting from reduced deionized water use, reduced wastewater volume being pretreated, lower effluent and sludge disposal quantities, and recovery of plating solution.

Prior to installation of the AROS unit, the overflow rinse water from the Watts Nickel plating process (approximately 1.3 million gal/yr) was added to the overall plating wastewater stream generated by HP operations (approximately 31 million gal/yr). At HP the overall plating wastewater stream is pretreated prior to discharge into the City of Sunnyvale sewer system. The pretreatment process includes chemical precipitation of metals using sodium hydroxide and ferrous sulphate; pH adjustment using sulfuric acid; and activated carbon adsorption. The chemical sludge generated by metals precipitation is dewatered and transported to a remote RCRA approved disposal site.

The makeup water to the rinse tank of the Watts nickel plating process is deionized by passage through an ion exchange resin. The ion exchange resin is regenerated using caustic and acid rinses. In addition, a percentage of the old resin is periodically replaced with new resin. The AROS unit recovers for reuse a high percentage of the deionized water used in the Watts Nickel plating process (approximately 1.3 million gal/yr) out of HP's total deionized water production of approximately 9 million gal/yr.

The AROS unit was installed in November 1989. After installation and debugging, the system was operated and tested from November 21, 1989 to December 18, 1989. The system was temporarily taken off line in late December, 1989 to allow HP to test and evaluate the plating bath solution quality

and to create a baseline of comparison for plating bath contents and performance. Results were considered acceptable and the AROS unit was restarted in January 1990, and the test continued through July 31, 1990. Counting one month in 1989 and seven months in 1990 the test totaled approximately 8 months.

This report summarizes the performance data provided by HP and also provides the results of a one day snapshot of the AROS unit operation as measured by chemical analyses of various process input and output streams. Section 1 provides background information about the project and Section 2 contains a technical description of the AROS unit. Existing data and the sampling program for the additional data are discussed in Section 3. Details about the design of the sampling program are provided in Section 4. The AROS unit performance was considered excellent by HP though some problems were experienced, as discussed in Section 5. Section 6 presents the economic analysis of the AROS system. Section 7 contains bibliographic information. Appendix A summarizes the field activities, Appendix B presents analytical sampling results, Appendix C summarizes the continuous data, and Appendix D is the Quality Assurance Project Plan.

## SECTION 2

### DESCRIPTION OF THE AROS UNIT INSTALLATION

The HP facility in Sunnyvale, California manufactures printed circuit boards for use in HP personal computers. As one step in the manufacturing process, Watts Nickel plating is used to plate a thin layer of conductive material on a non-conductive surface, like epoxy/plastic or ceramic. Watts Nickel is also widely used in other industries for decorative plating operations. During the test program the HP product line was operated from one to three shifts per day.

Figure 1 is a schematic flow diagram of how the AROS unit was used in the nickel plating operation at HP. In the upper half of the figure the flow of the production parts (printed circuit boards) is shown from left to right as follows:

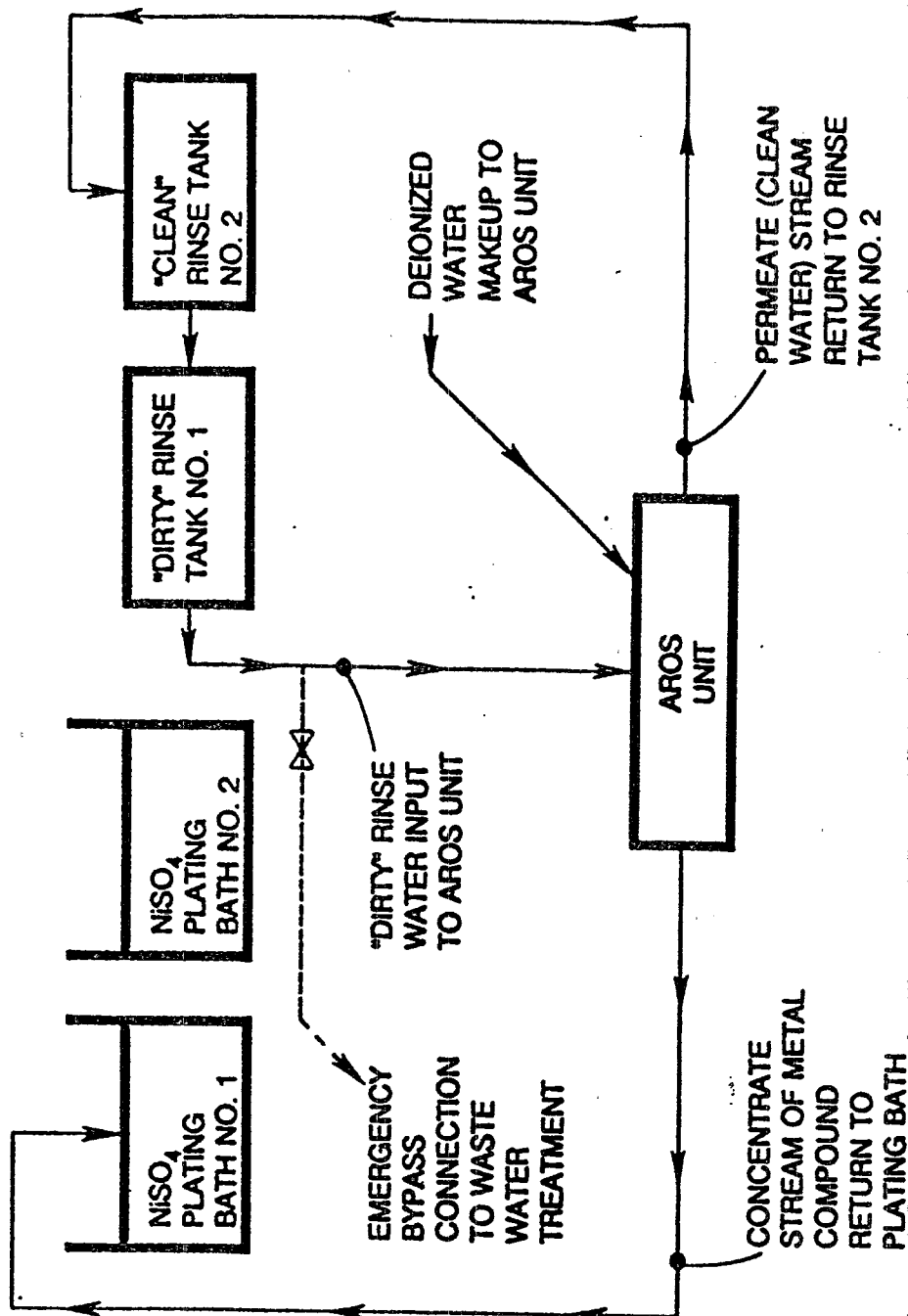
- First, the printed circuit (PC) boards are attached to moving racks. The moving racks carrying the parts move through the Watts Nickel sulfate solution plating bath of about 1,400 gallons capacity where the nickel plating is electrolytically applied to the PC boards. When the PC boards are removed from the bath, plating solution adheres to them. The PC boards are briefly held over the plating bath to allow plating solution to drip back into the plating bath before moving on. The plating solution that adheres to the PC boards is called "dragout."
- Second, the PC boards move through the "dirty" rinse tank which is the first of two rinse tanks in series. As shown in Figure 1, the clean rinse water enters the second rinse tank on the right and flows in the opposite direction (right to left) from the movement of the PC boards. In this way, the PC boards encounter the cleanest rinse water last just before exiting the second "clean" rinse tank. This method of having the parts and the rinse water move in opposite directions is called countercurrent rinsing. Each of the rinse tanks has a capacity of 450 gallons.

The AROS unit accepts as an influent the waste stream of overflow rinse water containing dissolved metal compounds from the "dirty" rinse tank. As illustrated in Figure 1, the AROS unit then treats this rinse water to separate out the metal compounds. This separation creates two product streams. First, a stream of deionized water called the permeate, and second, a liquid stream of concentrated metal compounds called the concentrate. Both of these product streams are reused in the production process. The permeate stream is returned to the "clean" water rinse tank. The concentrate stream of metal compounds is returned to the plating bath. This recycling eliminates the need for normal wastewater discharge, although, as seen in Figure 1, there is a standby emergency bypass connection to the existing wastewater pretreatment facility if needed. In addition to providing near zero discharge capability, the AROS unit also greatly reduces the volume of new deionized makeup water needed for the rinse tank. It also reduces the quantity of new nickel sulfate solution that must be added to the plating bath to maintain the required nickel concentration.

Because of the intermittent flow of various streams into and out of the AROS unit it is difficult to provide a snapshot of the flow volume in various streams. Reported flow volumes for the test period,

FLOW OF PARTS IN  
THE PLATING  
OPERATION

FLOW OF RINSE  
WATER IN THE  
RINSE TANKS



which comprised nearly 5000 hours, are approximately as follows:

- Rinse water cleaned and recycled = 190,000 gal. (38 gph)
- Concentrated Watts Nickel solution recycled = 1,100 gal. (0.2 gph)
- New makeup deionized water used = 31,000 gal. (6.2 gph)

During the test period the PC manufacturing line was operating at substantially below maximum capacity. The reader should note that the manufacturer reports that the AROS unit has a sustained capacity of 180 to 240 gph to clean and recycle rinse water, and 3 to 4 gph to recycle concentrated Watts Nickel solution. Obviously, the unit at HP was operating significantly below capacity and this adversely affected the economic benefits as discussed later in Section 6.

During the test period, the PC manufacturing line was operating substantially below maximum capacity. At maximum capacity the unit would have been operating about 8,500 hours annually and the estimated quantity of new deionized water saved would have been about 1.275 million gallons (150 gph) according to HP.

The AROS unit evaluated by HP is manufactured by Water Technologies, Inc. (WTI), Edina, Minnesota. WTI calls the unit their ZDR system; an abbreviation for zero discharge recovery. The heart of the AROS unit is a specialized reverse osmosis unit. Reverse osmosis is a physical process in which water containing dissolved materials can be separated from those dissolved materials. Pressure is applied to the solution on one side of a membrane barrier. Water passes through the membrane, but other materials including dissolved metal ions remain behind, thus becoming more concentrated. The membranes used are made of polyamide thin film plastics that can perform well under a wide range of pH (1 to 13.5) and high pressures (400 to 1100 psi) as needed to reconcentrate a wide range of dilute rinse waters to produce recycled plating bath solutions.

In addition to the reverse osmosis membrane, the AROS unit contains pumps, valves, interim solution holding tanks, sensors and piping needed to manage the flows into and out of the membrane unit. The operation is automatically controlled by a computer program that monitors flow quality (using conductivity), flow volumes, and other operating parameters. Figure 2 is a schematic diagram of the major internal components of the AROS unit.

The space requirement for the AROS unit is relatively small. The unit is enclosed in a lidded box about 3 ft. high by 4 ft. wide by 8 ft long. The plumbing, electrical, and communications connections appear to be relatively simple and do not require major modifications to existing utilities.

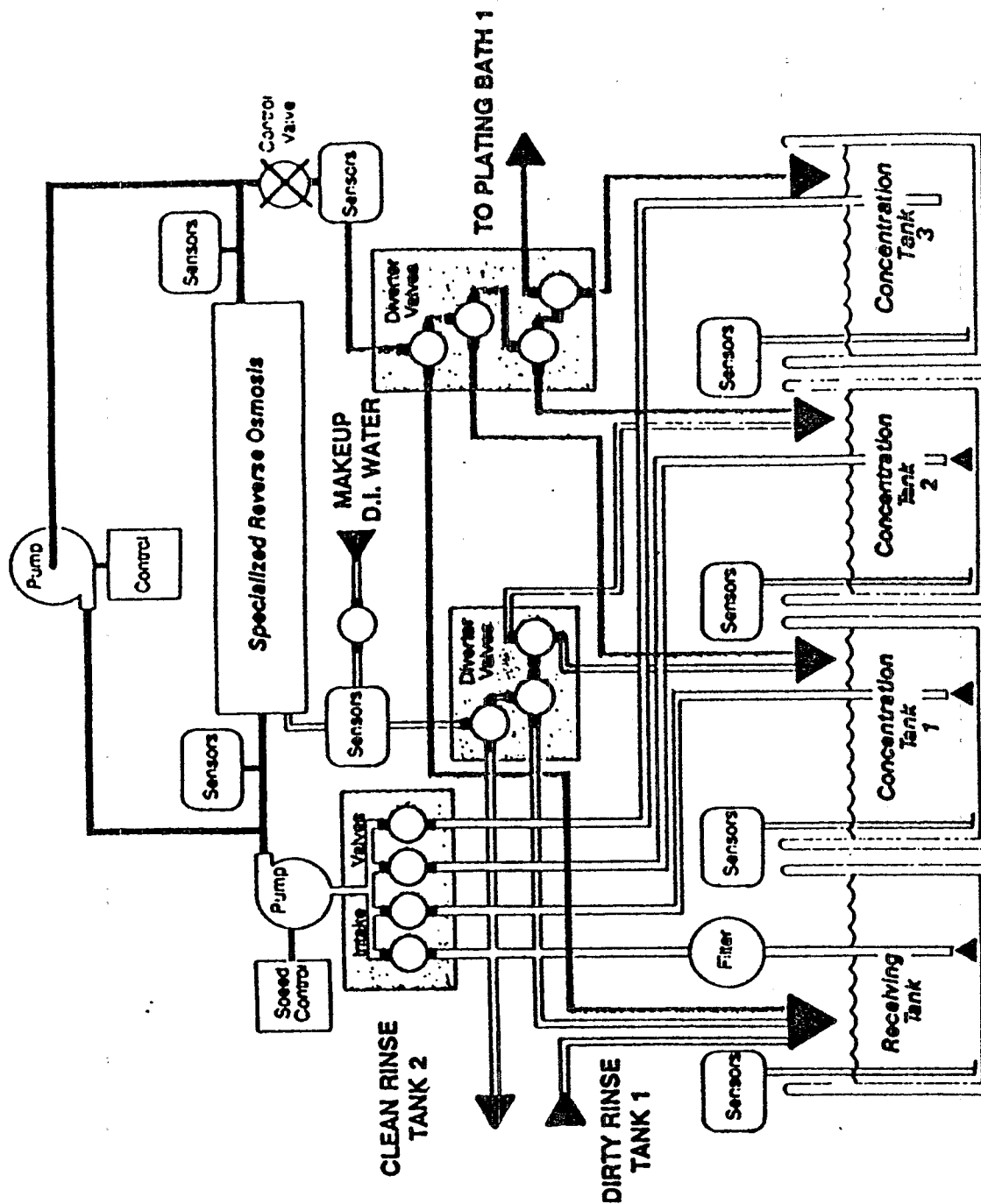


Figure 2. Schematic Diagram of Internal AROS Unit Components. (Source: Water Technologies)

## SECTION 3

### IDENTIFICATION OF DATA NEEDS

#### EXISTING DATA

Existing data was provided by HP about their sampling and monitoring program for the Watts Nickel plating process. The program included continuous monitoring (every 15 seconds) of parameters of flow volume, conductivity and pH at various monitoring points in the system. Streams monitored include the deionized rinse water make-up line, the emergency bypass line, the concentrate return line and the permeate return line.

In addition to the continuous monitoring program, plating bath No. 1 was sampled and analyzed weekly. Analyses were conducted for nickel, pH, Nikal PC-3 (Saccharin), boric acid, chloride and ductility. Boric acid is measured because its concentration is important to buffer the plating bath. Nikal PC-3 is an organic additive that must be maintained at a desirable level; neither too high or too low a concentration.

HP uses ductility testing as a key indication of the plating process performance. If the plated layer is too brittle, then future component failure can occur. HP was particularly concerned that the recovered concentrated Watts Nickel solution from the AROS unit might contain impurities that would adversely effect ductility, but the recovered solution proved satisfactory in this regard. The test is performed on a coupon removed from samples of printed circuit board products at final inspection and on a separate coupon plated from the recovered AROS unit concentrate. The procedure is as follows:

1. A sample is selected, plated on the coupon and identified with the appropriate lot numbers, part numbers, and date codes.
2. The thin metal sample sheet is removed from the coupon and the sample is subject to the following sequence:
  - a. A ball is pushed through the sample sheet at a controlled pressure and rate.
  - b. Pressure at which the metal sheet breaks is measured.
3. The results are compared with base line acceptable standards.

The samples plated from the recovered concentrate were all within the acceptable bounds of the ductility test, according to HP. However, to be conservative HP only recycled about half the concentrate recovered from the AROS unit. During the first months of the test period, HP discarded the recovered concentrate until satisfied that recycling would not harm product quality.

#### SAMPLING PROGRAM TO OBTAIN ADDITIONAL DATA

The purpose of additional sampling and monitoring was to spot-check the HP laboratory's analysis results and continuous monitoring readings with independent laboratory results.

Four streams were identified as target streams for sampling (Figure 1):

- Dirty rinse water stream influent to the AROS unit
- Deionized water (water make-up to the AROS unit)
- Permeate stream (recycled clean rinse water return)
- Concentrate Watts Nickel stream from the AROS unit (returned to the plating bath)

Laboratory analyses done for the samples are shown in Table 1 and include nickel, sulfate, chloride, pH, conductivity, TDS and TOC. Details about sampling activities are provided in Appendix A.

TABLE 1. ANALYSES DONE DURING ADDITIONAL SAMPLING AND MONITORING

PARAMETER	METHOD	DETECTION LIMIT <sup>a</sup>
Nickel	ICPMS <sup>b</sup>	0.01
Sulfate	EPA 300.6/IC <sup>c</sup>	0.1
Chloride	EPA 300.6/IC <sup>c</sup>	0.1
pH	EPA 9040/150.1	--
Conductivity	EPA 120.1	0.5
TDS	EPA 160.1	6
TOC	EPA 9060	0.5
Color	SM 204A	10

<sup>a</sup> All units are ppm except pH, conductivity (umhos/cm) and color (APHA platinum cobalt units)

<sup>b</sup> Inductively Coupled Plasma Mass Spectrometry - ICPMS (similar to EPA 200.8 - a recently accepted method)

<sup>c</sup> EPA 300.6/Ion Chromatography method - a similar equivalent method to EPA 300.0



## SECTION 4

### ANALYSIS OF SAMPLING RESULTS

Details of the one day sampling analysis results are shown in Appendix B and summarized in Table 2. The AROS unit achieved good separation of contaminants from the influent dirty rinse water. The composite permeate showed better than a 93% removal of nickel, sulfate, TDS and conductivity. Removal of chloride (76% removal) and TOC (77% removal) were less. The AROS unit normally achieves removals in the 95 to 97 percent range, as measured by on-line conductivity meters. The sampling done at 3 hour intervals happened to be grab samples (four were taken) that represented unusually high levels of conductivity (139 umhos/cm) and TDS (165 ppm). Normally, the conductivity of the permeate rinse water makeup is substantially less than 100 umhos/cm.

For comparison purposes, a continuous monitoring data summary was obtained from WTI for the period the sampling was conducted. Table 3 summarizes and Appendix C details the reading of conductivity, flow, etc. for four passes through the AROS unit membrane. Each pass indicated a different valve switch arrangement within the AROS unit that activated flow to or from internal tanks.

Conductivity values were averaged for each of the four passes and the removal rates were estimated based on discrete conductivity readings at various times listed in Table 3. A direct comparison of conductivity readings cannot be made between the data obtained from WTI and the results in Table 2 because of the internal storage of influents and effluents within the AROS unit. A snapshot does not necessarily reflect the performance over a longer time. In addition, the influent conductivity shown in Table 3 is the influent conductivity on the high pressure side of the membrane, not the influent conductivity entering the AROS unit. Similarly, the concentrate conductivity shown in Table 3 is not the concentrate level in the final product concentrate leaving the AROS unit. The concentrate conductivities shown in Table 3 are intermediate values achieved internally within the AROS unit.

The permeate conductivities shown in Table 3, however, are representative of the actual clean rinse water product, and average less than 80 umhos/cm based on 4 passes (compared to 139 umhos/cm in Table 2).

The continuous monitoring removal rates shown in Table 3 were higher than those obtained from SAIC's one-time sampling event. The continuous monitoring removal rates based on conductivity varied from 98.3 to 99.4 percent; whereas the snapshot (one time) sampling result indicated a removal rate of 93 percent, based on conductivity. One sample was composited over a period of 16 hours for the influent, and permeate samples. The concentrate was a composite of two shifts. Initially, Hewlett-Packard's laboratory was going to analyze the duplicate sample for accuracy and precision calculations. Split samples were collected for the Hewlett-Packard laboratory; however, the analysis was never done. As explained in Appendix A, no field blanks, equipment blanks and trip blanks were collected. The quality control related to each sample depended on the quality control procedures followed by the laboratory. Recovery rates for all parameters were within the acceptable range (refer to Appendix B). As previously discussed, the continuous monitoring results reflect internal sensors inside the AROS unit, and do not represent actual removals by the AROS unit, which usually range from about 95 to 97 percent based on conductivity.

TABLE 2. SAMPLING RESULTS OF AROS UNIT PERFORMANCE AT HEWLETT-PACKARD DURING ONE DAY<sup>a</sup>

Chemical	Unit	Influent <sup>b</sup>	Concentrate <sup>c</sup>	Permeate <sup>d</sup>	Deionized Water <sup>e</sup>	Detection Level	Percent Removal <sup>f,g</sup>
Nickel	ppm	650	52700	20.5	0.19	0.01	96.8
Chloride	ppm	120	7800	29	ND	0.1	75.8
Sulfate	ppm	1100	79000	18	0.11	0.1	98.4
pH	—	6	4.1	5.65	6.1	—	—
TDS	ppm	2525	171500	165	ND	6	93.5
Conductivity	umhos/cm	1985	53750	139.5	3.75	0.5	93.0
TOC	ppm	30.8	1625	7.01	0.74	0.5	77.2

Notes: <sup>a</sup> In case of duplicate analysis results, arithmetic averages are used.

<sup>b</sup> Composite sample taken over an 8 hour period with approximately 165 ml of sample taken every 30 minutes.

<sup>c</sup> Composite sample of two batches of concentrate taken at 11 a.m. and 4:30 p.m. respectively. Note that the unit only discharges concentrate periodically.

<sup>d</sup> Composite sample of four grab samples taken at approximately 3 hour intervals between 9:20 a.m. and 6 p.m.

<sup>e</sup> One-time grab sample.

<sup>f</sup> Percent removal was calculated based on the following formula:  $[(\text{Influent-permeate})/(\text{Influent} \times 100)]$ .

<sup>g</sup> Note that there is a significant time lag within the AROS treatment unit and significant rapid variations in the quality of the influent and permeate. Therefore, the percent removals shown should be considered a snapshot only and not necessarily representative of average percent removals.

TABLE 3. CONTINUOUS MONITORING RESULTS ANALYSIS (October 17, 1990)

WTI Readings											
Pass #1 Time	13:56	13:13	12:28	12:6	11:41	11:4	Average	St. Dev.	Removal (%)		
Conductivity Readings (umhos/cm)											
Influent Cond.	5984	5608	5368	6184	5256	4064	5411	684			
Permeate Cond.	12	12	11	106	157	77	63	56	98.8		
Pass #2 Time	14:28	14:3	13:22	12:39	11:56	11:17	Average	St. Dev.	Removal (%)		
Conductivity Readings (umhos/cm)											
Influent Cond.	2836	2804	2464	2460	2516	2804	2647	169			
Permeate Cond.	17	46	47	54	47	56	45	13	98.3		
Pass #3 Time	14:10	13:29	12:47	12:11	12:2	11:26	Average	St. Dev.	Removal (%)		
Conductivity Readings (umhos/cm)											
Influent Cond.	12368	10864	9648	11216	8208	11600	10651	1365			
Permeate Cond.	50	74	79	54	94	109	77	21	99.3		
Pass #4 Time	9:36	2:22	22:31	16:47	12:58	9:27	Average	St. Dev.	Removal (%)		
Conductivity Readings (umhos/cm)											
Influent Cond.	20384	20384	19968	19968	20800	21248	20459	453			
Permeate Cond.	149	131	123	127	128	128	131	8	99.4		

Note: Average levels are mathematic averages of six readings for each pass.  
Removal rates (%) were calculated base on the following formula: [(Influent-Permeate)/Influent \* 100].

## SECTION 5

### OVERALL SYSTEM PERFORMANCE

Overall the HP staff regard the AROS unit as having shown good performance during the test period. A major achievement was that rinse water quality was maintained at a low level of nickel contamination. This is critical to the quality of the Watts Nickel plating process, which in turn is crucial to the acceptability of the final PC board products. It was reported that no PC boards were rejected because of Watts Nickel plating deficiencies.

Conductivity is used as an indication of nickel contamination. In the rinse water, approximately 11 umhos of conductivity represent 1 ppm of nickel. Prior to using the AROS system the deionized rinse water supplied from the ion exchange units maintained a rinse water quality of 4 to 30 umhos conductivity. The AROS system generally supplied rinse water quality ranging from 25 to 40 umhos conductivity. The highest reading recorded during the test period was 211 umhos. The recycling of the rinse water resulted in a 98 percent reduction in the use of new deionized water makeup for this plating process.

The AROS unit also successfully produced concentrated Watts Nickel solution of adequate quality to return to the plating bath solution. About half the concentrate produced by the AROS unit was recycled. As discussed in Section 3, under existing data, HP did not start recycling concentrate until totally satisfied that the quality was satisfactory for reuse. After extensive testing for the first half of the trial period, HP did start recycling concentrate to supplement normal additions of fresh Watts Nickel solution. Fresh Watts Nickel solution is expensive at about \$5.00/gallon, so recovery and recycling of about 500 gallons represented a direct savings of \$2,500. Obviously, the savings would have been greater if the concentrate had been recycled during the entire trial period. It was also calculated that approximately 3 tons of category F006 sludge was not generated by the industrial waste water treatment system that otherwise would have been without recycling. The sludge produced is shipped to Arizona for treatment and recycling.

The AROS unit demonstrated excellent reliability during most of the test period. For example, during the period February 28 through June 29, 1990 the system was on-line 3,594 hours and experienced down-time of only 20 hours. However, mechanical failures experienced in July and August, 1990 caused down-time of over 200 hours during this period. The mechanical problems included failure of the pressure pump and two high pressure concentrate control valves, plus some minor leakage at fittings. A failure of the membrane occurred in September 1990, apparently caused by failure of a temperature sensor that resulted in membrane overheating. As a result, AROS systems are now equipped with cooling jackets around the pressure vessel to prevent membrane overheating. In addition, the manufacturer has upgraded the sensors used and the control software.

Over a period of several weeks the recycled Watts Nickel plating solution experienced an unacceptable build-up in the concentration of organic additive. A small in-line carbon filter was added to the system to remove these organics prior to recycling the concentrate solution back to the plating bath. The in-line carbon filter is installed directly into the pipeline connecting the concentrate discharge of the AROS unit to the Watts nickel solution plating Bath No. 1. The carbon cartridge required changing at approximately 10 day intervals.

## SECTION 6

### ECONOMIC ANALYSIS OF THE AROS SYSTEM

#### COST EFFECTIVENESS OF THE AROS SYSTEM IN THE HEWLETT-PACKARD PLANT SETTING

At HP the savings from use of the AROS unit were directly related to the incremental reduction in spending for the following cost items:

- Sewer discharge fees and fresh water cost, estimated by HP at \$0.004/gal. or \$4 per 1000 gal.
- Deionized (DI) water production cost, estimated by HP at \$0.0064/gal., or \$6.40 per 1000 gal.
- Plating wastewater treatment costs, estimated by HP at \$0.0062/gal., or \$6.20 per 1000 gal.

These plating wastewater treatment costs include:

- Labor
  - Power
  - Chemicals
  - Expendable parts and supplies replacement
  - Monitoring, e.g. analysis of influent and effluent
  - Sludge treatment, handling, manifesting, transport and disposal
- Purchase of new plating chemicals estimated by HP at \$5.00/gal. to make up for plating solution drag-out losses

The above listed cost items are the major incremental cost savings resulting to HP from use of the AROS system. As shown in Table 4, HP estimates the annual savings listed above to total \$26,250/year. Tables 5 and 6 provide additional cost details.

This incremental cost savings is balanced against the estimated annual expenditure for owning and operating the AROS system, as follows:

• Electrical Power	\$1629
• R.O. Membrane Replacement	\$2200
• Labor and Expendable Parts	\$5000
• Carbon Filters	\$ 90
• Telephone Modem Contact With AROS Mfg.	\$ 500
	<hr/>
	\$9419

TABLE 4. ESTIMATED ANNUAL INCREMENTAL SAVINGS FROM USE OF THE AROS UNIT  
AS REPORTED BY HEWLETT-PACKARD CORPORATION, 1990 COSTS

Item No.	Description	Estimated Savings (\$/gal)	Quantity (gal)	Total Annual Savings (\$)
1	Sewer Discharge Fees and Water Costs	0.004	1,275,000	5,100
2	Deionized (DI) Water Production Cost <sup>1</sup>	0.0064	1,275,000	8,160
3	Plating Wastewater Treatment Costs <sup>2</sup>	0.0062	1,275,000	7,905
4	Purchase of New Plating Chemicals at an 85 Percent Reduction	5.00	1260 x 0.85	5,355
Total Estimated Annual Savings				\$26,520

<sup>1</sup> DI water production cost is for chemicals, electricity and resin replacement only. No labor, depreciation or other costs are included because it is assumed that they would remain the same whether the AROS unit was used or not. See Table 5 for details of DI water production cost.

<sup>2</sup> Plating wastewater treatment cost includes sludge disposal, chemicals and electricity. As in note (1) above no labor, depreciation or other costs, are included because it is assumed that they would remain the same whether the AROS unit was used or not. See Table 6 for details of wastewater treatment cost.

TABLE 5. DETAILS OF DEIONIZED WATER PRODUCTION COST USED IN TABLE 4<sup>1</sup>

Item No.	Description	Annual Cost (\$)	Estimated Unit Cost (\$)
1	Electricity	\$13,440	6.73¢/KWH
2	Resin Replacement	\$10,000	---
3	Caustic (NaOH)	\$27,412	\$2.43/gal.
4	Hydrochloric Acid	\$ 5,151	---
5	Sulfuric Acid	\$ 2,475	\$0.57/gal.
6	Labor, Amortization and Other Costs	No Difference	---
TOTAL			\$58,478

<sup>1</sup> Approximate Annual Production of D.I. Water is 9.1 Million Gal.

$$\text{Cost per gallon} = \frac{\$58,478}{9.1 \text{ million gal/yr.}} = \$0.0064/\text{gal.}$$

TABLE 6. DETAILS OF WASTEWATER TREATMENT COST USED IN TABLE 4<sup>1</sup>

Item No.	Description	Annual Cost (\$)	Estimated Unit Cost (\$)
1	Electricity	\$ 46,455	6.73¢/KWH
2	Sludge Disposal	\$ 52,800	\$275/Ton
3	Caustic (NaOH)	\$ 49,941	\$2.43/Gal.
4	Sulfuric Acid	\$ 9,000	\$0.57/Gal.
5	Ferrous Sulphate	\$ 19,600	\$0.10/Lb.
6	Activated Carbon	\$ 16,900	---
7	Labor, Amortization and Other Costs	No Difference	---
TOTAL		\$194,696	

<sup>1</sup> Approximate Annual Volume of Water Treated is 31.25 Million Gal.

$$\text{Cost per gallon} = \frac{\$194,696}{31.25 \text{ million gal/yr}} = \$0.0062/\text{gal}$$

Subtracting \$9,419/Yr. from \$26,250/yr., HP estimates that the net annual savings from use of the AROS unit would be approximately \$17,100/yr. Investment is approximately \$75,000, which represents approximately \$63,000 for the AROS unit plus another \$12,000 for making the installation permanent and training of operating personnel. Dividing \$75,000 by \$17,100 results in a payback period of 4.4 years and a return on investment of 23 percent. As discussed below the economics would have been more favorable had the AROS unit been utilized to a higher percentage of its capacity.

#### COST EFFECTIVENESS OF THE AROS SYSTEM AT OTHER SITES

The AROS unit at HP was operated at less than 50 percent of its volumetric flow capacity and only about 10 percent of its design capacity to recover Watts Nickel solution. The economic benefits would have been more favorable if the Watts Nickel plating process had operated for more hours and produced more printed circuit boards. For example, the plating solution dragout at HP was estimated to average only about 0.2 to 0.3 gph, whereas the AROS unit is designed to recover 2 to 3 gph of Watts Nickel solution: ten times as much as was actually recovered. Similarly, the AROS unit volumetric design capacity for influent rinse water is over twice the volume of rinse water processed at HP.

A second economic factor is that at HP the AROS unit treated only a small fraction, e.g. about 3 percent, of the total site wastewater flow. Therefore, in its cost analysis HP made no allowance for reduced labor cost at its main wastewater pre-treatment plant. It was logical for HP to do this, since a 3 percent reduction in wastewater flow volume would not make a measurable difference in operating and maintenance labor. However, at another facility where the AROS unit treated a larger percentage of the total potential wastewater flow a labor reduction credit might have been included in the cost analysis.



Finally, the HP facility has a fully amortized wastewater treatment facility in place. Elsewhere at a new facility under design, it could be feasible to reduce the capacity and capital cost of the wastewater treatment facility because the inclusion of an AROS unit resulted in reduced design flow volume. In addition, a new facility would be able to reduce the capacity of its deionized water production system resulting in additional capital cost savings. Economic cost-benefit analysis will be different for each potential application of an AROS type unit depending upon the site specific situation. This demonstration indicates that there are many situations where the unit should be considered for its economic benefits as well as environmental waste minimization advantages.

## SECTION 7

### BIBLIOGRAPHY

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2. PEI Assoc. Inc. Characterization and Treatment of Wastes from Metal Finishing Operations. Order No. PB91-125 732/AS, March 1991.
3. Planning Research Corporation. Waste Audit Study, Printed Circuit Board Manufacturers, Department of Health Services, June 1987.
4. Water Technologies Inc., Various Items of Promotional Literature.
5. U.S. Environmental Protection Agency. Reducing Water Pollution Control Costs in the Electroplating Industry, EPA/625/5-85/016, Office of Research Program Management, Office of Research and Development. September 1985.

## APPENDIX A

### SUMMARY OF FIELD ACTIVITIES

#### SAMPLING RATIONALE

Streams in and out of the AROS unit were sampled on October 17, 1990 to obtain a one day snapshot of the system's operation. The samples were split for independent analysis by the HP laboratory and an outside laboratory. The sample results from the two laboratories were to be compared. However, the split samples provided to HP were lost and a direct comparison between the two laboratories cannot be provided.

#### SAMPLING PROCEDURES

The HP staff conducted the sampling. The sample containers were prepared by Western Analytical Services Laboratory, labels were filled out and chain of custody maintained, and samples placed in the bottles as explained in the QAPP (Appendix D). In addition to SAIC personnel, a representative from Water Technologies, Incorporated which manufactures the AROS unit, and Robert Ludwig with the California Department of Toxic Substances Control were also present to observe sampling.

Four liquid streams were sampled as shown in Figure A-1:

- 1) Influent to the AROS treatment unit, which is the rinse water from "Dirty" rinse tank No. 1
- 2) Deionized water used as makeup water to the AROS unit
- 3) Permeate (deionized "clean" water) produced by the AROS unit that is returned to "clean" rinse Tank No. 2
- 4) Concentrate, consisting of concentrated Watts Nickel plating solution produced by the AROS unit and returned to the plating bath.

Streams 1, 3, and 4 were collected as composites as described in the following subsections. Stream 2 was a one-time grab sample. Upon collection, all samples were stored on ice, with the exception of the concentrate (stream 4), which would have crystallized if put on ice. At the end of the day, samples were poured into the prepared bottles for shipment to the laboratory. A split sample of each (except deionized water) was provided to HP in bottles prepared by them. Samples from streams 1, 2, and 3 were shipped to the laboratory in a cooler with blue ice. The concentrate sample was shipped separately as a hazardous material and was not maintained on ice. The laboratory confirmed receipt of all samples in good condition the following morning at 10:00 a.m. Proper chain of custody procedures were used throughout.



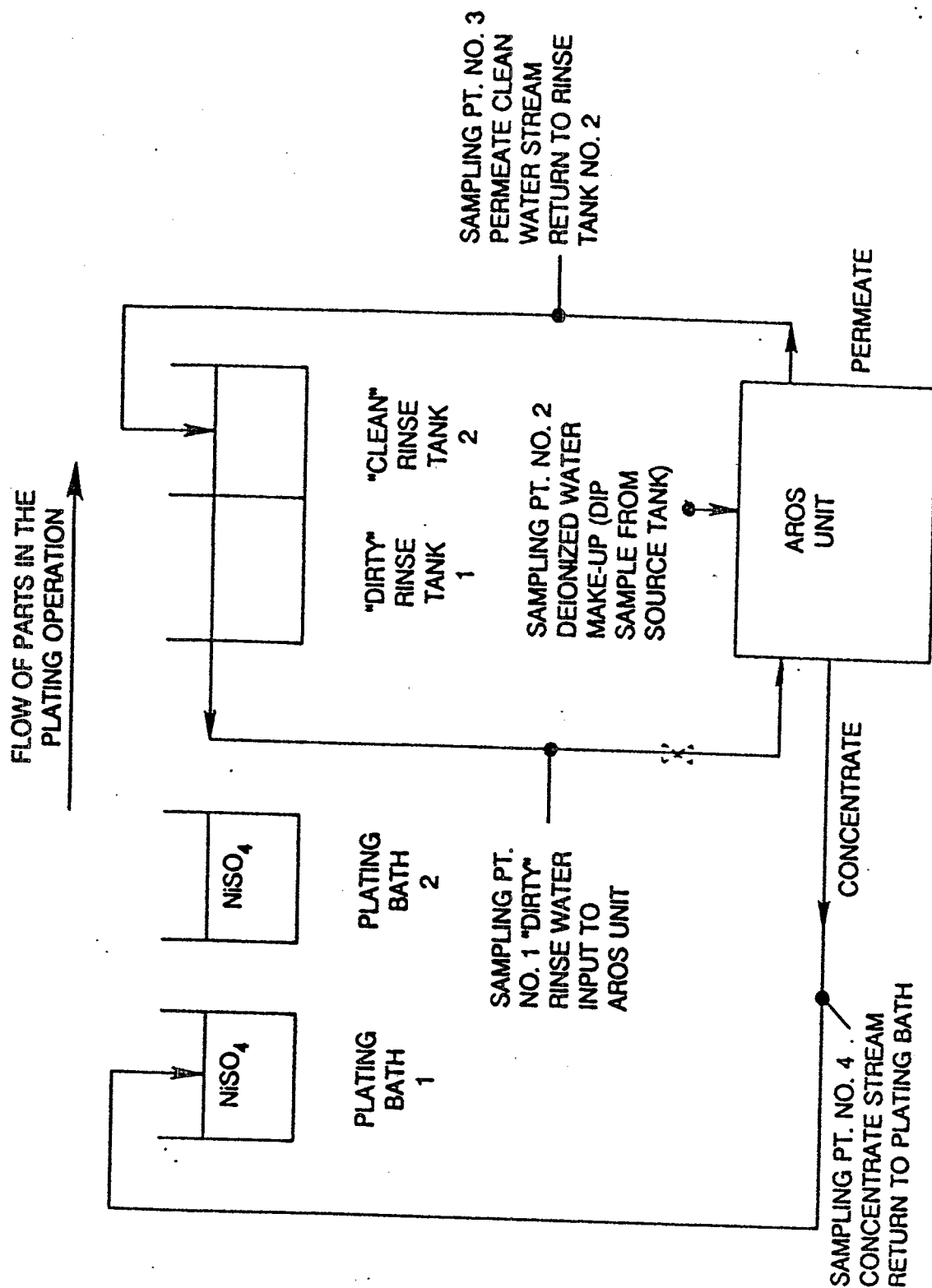


Figure A-1. Sampling Points Shown on Schematic Diagram of the Advanced Reverse Osmosis System (AROS) for the Nickel Plating Operation

### Sampling of the Influent Stream

An ISCO sampler was installed to automatically take samples of the influent to the AROS unit. Due to the time lapse during treatment of the wastewater entering the unit, the influent to the unit cannot be calibrated directly with the effluents (permeate and concentrate) from the unit. However, collection of samples automatically throughout the day gave a good indication of the influent composition. Beginning at 9:15 a.m., the ISCO was programmed to obtain approximately 165 mL of influent every 30 minutes. The composite sample was collected from the ISCO sampler at 5:15 p.m. To chill the sample, ice was packed around the Nalgene collection bottle located within the sampler.

### Sampling of the Deionized Water

A one-time grab sample of deionized water was taken from a tap of the deionized water production system at approximately 1:35 p.m. Hewlett Packard did not want a split sample of the deionized water, as they are already knowledgeable about its composition.

### Sampling of the Permeate

Grab samples of permeate were obtained from a tap off the AROS unit, at approximately three hour time intervals throughout the day, for a total of four grab samples. The first sample was collected at 9:20 a.m. As each sample was collected, it was placed into an acid-rinsed plastic one gallon bottle. This composite bottle was kept on ice in a cooler. After the final sample was collected the composite container was mixed by shaking. The composite sample was then poured into properly labeled bottles prepared with preservative for the various analyses.

### Sampling of the Concentrate

Concentrate is discharged from the AROS unit periodically, not continually. Two grab samples of concentrate were collected from different batches. The first batch was discharged from the AROS unit at about 11:00 a.m. It was collected in a five-gallon bucket. The contents of the bucket were swirled to mix, and a sample was poured into a one-liter glass bottle. This sample was not chilled due to the likelihood of crystallization of the highly concentrated plating solution. The remainder of the batch was poured into a 55-gallon drum that HP uses to collect the concentrate. At approximately 4:30 p.m., another batch of concentrate was discharged from the unit. A sample from this batch was collected in the same manner as the first. The contents of both liter bottles were then poured into a compositing container (one-gallon plastic), and swirled to mix. The bottles prepared by the laboratory were filled with concentrate and shipped to the laboratory for analysis as described below.

### **PACKING, PRESERVATION, AND TRANSPORT OF SAMPLES**

All bottles were taped with duct tape to prevent loosening of the caps in transit. The samples of influent, deionized water, and permeate were placed into a small cooler, and packs of blue ice were put in. Remaining gaps were filled with styrofoam "peanuts," and a small amount of ice was added to the top. The laboratory confirmed that the samples were still cold when they arrived the next morning.

The concentrate was packed separately and shipped as a hazardous material. Hewlett-Packard provided the information, box, and proper label for this shipment. Taped bottles were placed deep in an absorbent material in the box. The remaining airspace was stuffed with crumpled newspapers. The box was sealed and marked with the proper shipping name of the substance, "Corrosive Liquid, N.O. S.," and the UN number, UN1760. The Federal express office accepted the samples after a "shipper's certification for restricted articles" was completed. The agent determined that since the total quantity consisted of more than one quart, the shipment would have to go on a cargo rather than a passenger plane. The laboratory confirmed receipt of this box by 10 a.m. the following morning.

APPENDIX B  
WEST COAST ANALYTICAL  
SAMPLING RESULTS

June 25, 1987

To Our Customers:

Ref: Sample Storage Policy

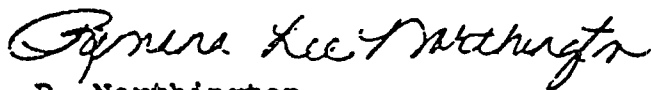
With each report as it is completed we include a Sample Storage Card. This card is to be returned each time, so that proper handling of your samples can be maintained.

Our policy as stated on the card will be adhered to in the future unless we receive back the sample storage card indicating samples to be returned at customers expense. We do not store samples after 30 days of job being completed.

Thank you in advance for your cooperation.

Sincerely,

WEST COAST ANALYTICAL SERVICE, INC.



R. Northington  
Controller

RN/ds



October 31, 1990

SAIC  
1720 E. Wilshire Ave.  
Santa Ana, CA 92705

Attn: Ilknur Erbus-White

JOB NO. 16864

A

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LABORATORY REPORT

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Samples Received: Nine (9) water samples and three (3) product  
Date Received: 10-18-90  
Purchase Order No: R5503467


The samples were analyzed as follows:

<u>Samples Analyzed</u>	<u>Analysis</u>	<u>Results</u>
Four (4) samples	Nickel by ICPMS	Table 1
One (1) sample	QC Summary for ICPMS	Table 2
Four (4) samples	Chloride and Sulfate by EPA 300.6/IC	Table 3
One (1) sample	QC Summary for EPA 300.6/IC	Table 4
Four (4) samples & two (2) duplicates	pH by EPA 9040/150.1	Table 5
Four (4) samples & two (2) duplicates	Total Dissolved Solids by EPA 160.1	Table 6
Four (4) samples & two (2) duplicates	Conductivity by EPA 120.1	Table 7
Four (4) samples & two (2) duplicates	Color by SM 204A	Table 8

Page 1 of 7

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Michael Shelton  
Technical Director

  
D. J. Northington, Ph.D.  
President

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WEST COAST ANALYTICAL SERVICE, INC.

SAIC  
Ms. Ilknur Erbas-White

Job # 16864  
October 31, 1990

---

LABORATORY REPORT

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<u>Samples Analyzed</u>	<u>Analysis</u>	<u>Results</u>
Four (4) samples	Total Organic Carbon (TOC) by EPA 9060	Table 9
One (1) sample	QC Summary for EPA 9060	Table 9

WEST COAST ANALYTICAL SERVICE, INC.

SAIC  
Ms. Ilknur Erbas-White

Job # 16864  
October 31, 1990

---

LABORATORY REPORT

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TABLE 1

Parts Per Million (mg/Kg)

<u>Sample No.</u>	<u>Nickel</u>
Concentrate	52700
DI Water	0.19
Influent	650
Permeate	20.5
Detection Limit	0.01

Dates Analyzed: 10-23-90 & 10-25-90

TABLE 2

QC Summary for Nickel by ICPMS

Sample ID: Permeate

<u>Sample</u>	<u>MS</u>	<u>% Recovery</u>	<u>MSD</u>	<u>% Recovery</u>	<u>RPD</u>
20.5	107	87	107	87	0

Spike Level: 100 ppm

Date Analyzed: 10-25-90

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Job # 16864  
October 31, 1990

LABORATORY REPORT

TABLE 3

Parts Per Million (mg/L)

by EPA 300.6/IC

<u>Sample ID</u>	<u>Chloride</u>	<u>Sulfate</u>
Concentrate	7800**	79000**
DI Water	ND	0.11
Influent	120	1100*
Permeate	29	18
Detection Limit	0.1	0.1

ND-Not Detected

\*\* values in mg/Kg

Dates Analyzed: 10-19-90  
10-22-90\*

TABLE 4

OC Summary for EPA 300.6/IC

Sample ID: Permeate

<u>Component</u>	<u>Orig.</u>	<u>Dup</u>	<u>RPD</u>	<u>MS</u>	<u>MSD</u>	<u>1 Rec.</u>	<u>RPD</u>
Chloride	29	29	0	17	17	81	0
Sulfate	18	18	0	31	31	65	0

Detection Limit: 0.1 ppm

Spike Level = 20 ppm

WEST COAST ANALYTICAL SERVICE, INC.

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Ms. Ilknur Erbas-White

Job # 16864  
October 31, 1990

LABORATORY REPORT

TABLE 5

by EPA 9040/150.1

<u>Sample ID</u>	<u>pH (Units)</u>
Concentrate	4.1
Concentrate (DUP)	4.1
DI Water	6.1
DI Water (DUP)	6.1
Influent	6.0
Influent (DUP)	6.0
Permeate	5.7
Permeate (DUP)	5.6

Date Analyzed: 10-25-90

TABLE 6

Parts Per Million (mg/L)

by EPA 160.1

<u>Sample ID</u>	<u>Total Dissolved Solids</u>
Concentrate	172000
Concentrate DUP	171000
DI Water	ND
DI Water DUP	ND
Influent	2490
Influent DUP	2560
Permeate	180
Permeate DUP	150
Detection Limit	6

ND-Not Detected

Date Analyzed: 10-19-90

WEST COAST ANALYTICAL SERVICE, INC.

SAIC  
Ms. Ilknur Erbas-White

Job # 16864  
October 31, 1990

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LABORATORY REPORT

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TABLE 7

Micromhos Per Centimeter  
(umhos/cm)

Sample ID                      Conductivity by EPA 120.1

Concentrate	53700
Concentrate DUP	53800
DI Water	3.7
DI Water DUP	3.8
Influent	1980
Influent DUP	1990
Permeate	137
Permeate DUP	142
Detection Limit	0.5

Date Analyzed: 10-19-90

TABLE 8

APHA Platinum Cobalt Units

Sample ID                      Color by SM 204A

Concentrate	9700
Concentrate DUP	9500
DI Water	ND
DI Water DUP	ND
Influent	140
Influent DUP	140
Permeate	ND
Permeate DUP	ND
Detection Limit	10

ND-Not Detected

Date Analyzed: 10-19-90

WEST COAST ANALYTICAL SERVICE, INC.

SAIC  
Ms. Ilknur Erbas-White

Job # 16864  
October 31, 1990

---

LABORATORY REPORT

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TABLE 9

Parts Per Million (mg/L)

by EPA 9060

<u>Sample ID</u>	<u>Total Organic Carbon (TOC)</u>
Concentrate	1610
Concentrate DUP	1640
Influent	30.8
Permeate	6.91
Permeate DUP	7.10
DI Water	0.74
External Reference Standard	20 (100% Recovery)
Detection Limit	0.5

Date Analyzed: 10-26-90

Client Name: SAIC Phone No. 714-542-9411  
1730 E. Wilshire Ave. Fax No. 714-542-5389  
Santa Ana 92705 Proj. No. \_\_\_\_\_  
 Technical Contact: Ilknur Erbus-white Proj. Name \_\_\_\_\_

**CHAIN OF CUSTODY**

**WEST COAST ANALYTICAL SERVICE, INC.**  
 9840 Alburta Ave Santa Fe Springs, CA 90670  
 Phone 213/948-2225 FAX 213/948-5850

WCAS Job No. W16864

Date Sampled \_\_\_\_\_ Condition of Samples \_\_\_\_\_

Sample #/Description/Remarks	Sample Matrix	Analyses Requested											
✓ Di (3 containers)	Water												
✓ Permeate	"												100
✓ Influent	"												
✓ Concentrate	product												100

Total # of Containers 12

Relinquished by: (Company & Signature)

Received for Lab by:

Date/Time

Mary M. McLeary

10-18/10 50

A.M.

White copy: Job Env. Yellow copy: Return with Lab Results Pink copy: Client at time of Delivery

W16864



APPENDIX C

WATER TECHNOLOGIES INC.  
CONTINUOUS DATA SUMMARY

■ File Programs Archive Options System Calib Rinse Membrane Log  
Hewlett Packard ZDR-513 PASS #1 PERFORMANCE DATA 10-17-90  
Watts nickel (LAST 6 TIMES IN PASS) 14:29:14

	10-17	10-17	10-17	10-17	10-17	10-17
date	10-17	10-17	10-17	10-17	10-17	10-17
time of day	13:56	13:13	12:28	12: 6	11:41	11: 4
Pass time, min	18.72	18.18	9.28	3.45	7.22	6.32
Rinse time, min	16.62	16.02	6.92	1.45	1.50	2.80
Control time, min	18.58	18.02	9.27	2.58	7.20	6.30
Rinse volume, gal	21.9	21.4	6.4	0.3	0.0	0.5
Control volume, gal	17.4	17.4	14.0	6.0	14.7	12.8
Permeate temperature, deg F	74	75	73	80	79	71
Concentrate temperature	71	71	70	75	71	69
Pump flow rate, gpm	5.02	5.03	4.34	3.69	3.70	3.67
Permeate flow rate, gpm	1.40	1.44	1.31	0.58	0.29	0.38
Concentrate flow rate, gpm	0.53	0.49	0.47	1.87	1.80	1.80
System pressure, psi	217	217	195	118	74	79
Membrane pressure drop, psi	12	12	5	3	2	2
Feed tank conductivity, umho	5984	5608	5368	6184	5256	4064
Permeate conductivity, umho	12	12	11	106	137	77
Concentrate conductivity	4776	4344	3068	7928	1968	1186
Membrane separation ratio	388.5	365.5	282.5	74.9	12.5	15.3
Min. permeate conductivity	12	11	11	71	73	32

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■ File Programs Archive Options System Calib Rinse Membrane Log  
Hewlett Packard ZDR-513 PASS #2 PERFORMANCE DATA 10-17-90  
Watts nickel (LAST 6 TIMES IN PASS) 14:30:20

	10-17	10-17	10-17	10-17	10-17	10-17
date	10-17	10-17	10-17	10-17	10-17	10-17
time of day	14:28	14: 3	13:22	12:39	11:56	11:17
Pass time, min	3.97	7.00	9.23	11.27	14.83	12.67
Rinse time, min	3.90	7.00	9.23	11.10	11.87	11.20
Control time, min	1.27	3.70	4.50	5.40	9.17	6.43
Rinse volume, gal	8.3	8.7	11.9	13.0	11.6	11.5
Control volume, gal	2.1	3.5	4.2	4.9	5.7	4.8
Permeate temperature, deg F	79	80	80	79	80	78
Concentrate temperature	76	75	75	75	76	74
Pump flow rate, gpm	4.94	5.03	5.00	4.36	3.69	3.67
Permeate flow rate, gpm	3.15	0.75	1.00	1.20	0.98	1.00
Concentrate flow rate, gpm	1.78	1.40	1.11	0.69	0.40	0.46
System pressure, psi	564	137	175	197	186	197
Membrane pressure drop, psi	7	11	2	0	3	0
Feed tank conductivity, umho	2835	2804	2464	2460	2516	2804
Permeate conductivity, umho	17	46	47	54	47	54
Concentrate conductivity	9516	8288	7840	7664	8544	7928
Membrane separation ratio	591.0	180.0	167.8	142.5	182.8	142.5
Max. concentrate conductivity	9516	9120	9024	9120	8912	8944

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File Programs Archive Options System Calib Rinse Membrane Log  
Hewlett Packard ZDR-513 PASS #3 PERFORMANCE DATA 10-17-90  
Watts nickel (LAST 6 TIMES IN PASS) 14:31:28

	10-17	10-17	10-17	10-17	10-17	10-17
date	10-17	10-17	10-17	10-17	10-17	10-17
time of day	14:10	13:29	12:47	12:11	12: 2	11:26
Pass time, min	6.90	6.93	7.40	4.97	6.05	9.07
Rinse time, min	6.20	6.43	5.97	4.10	3.80	5.27
Control time, min	0.00	0.00	0.00	0.30	0.00	0.00
Rinse volume, gal	9.0	8.6	7.5	10.9	3.7	5.0
Control volume, gal	0.0	0.0	0.0	0.2	0.0	0.0
Permeate temperature, deg F	83	83	83	83	82	81
Concentrate temperature	81	80	80	81	79	78
Pump flow rate, gpm	4.95	5.02	5.01	3.54	3.69	3.69
Permeate flow rate, gpm	2.89	1.49	1.40	3.02	0.91	0.87
Concentrate flow rate, gpm	0.58	0.58	0.60	0.64	0.58	0.62
System pressure, psi	729	385	388	850	244	281
Membrane pressure drop, psi	12	5	3	7	4	4
Feed tank conductivity, umho	12368	10864	9648	11216	8208	11600
Permeate conductivity, umho	50	74	79	54	94	109
Concentrate conductivity	20384	19040	18848	22688	15520	18080
Membrane separation ratio	410.5	257.0	239.5	423.5	165.8	166.5
Max. concentrate conductivity	20384	19040	18848	22688	15536	18080

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File Programs Archive Options System Calib Rinse Membrane Log  
Hewlett Packard ZDR-513 PASS #4 PERFORMANCE DATA 10-17-90  
Watts nickel (LAST 6 TIMES IN PASS) 14:32:33

	10-17	10-17	10-16	10-16	10-16	10-16
date	10-17	10-17	10-16	10-16	10-16	10-16
time of day	9:36	2:22	22:31	16:47	12:58	9:27
Pass time, min	4.62	4.75	5.05	4.95	4.85	4.61
Rinse time, min	1.02	3.50	2.93	3.40	3.37	4.11
Control time, min	2.88	2.92	2.25	2.52	2.65	2.77
Rinse volume, gal	2.5	6.3	4.8	6.1	6.2	7.0
Control volume, gal	1.7	1.7	1.3	1.3	1.4	1.1
Permeate temperature, deg F	84	86	87	83	82	8
Concentrate temperature	82	84	85	81	80	7
Pump flow rate, gpm	1.70	1.51	1.50	1.47	1.44	1.5
Permeate flow rate, gpm	0.96	1.02	1.04	1.00	0.98	1.0
Concentrate flow rate, gpm	0.82	0.58	0.56	0.51	0.53	0.5
System pressure, psi	951	1003	999	998	999	95
Membrane pressure drop, psi	10	8	9	8	7	
Feed tank conductivity, umho	20384	20384	19963	19968	20800	212
Permeate conductivity, umho	149	131	123	127	128	12
Concentrate conductivity	32448	30432	29792	30400	30688	3068
Membrane separation ratio	218.5	231.8	242.3	239.8	239.8	240.
Max. concentrate conductivity	32448	30432	30112	30400	30688	3068

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**APPENDIX D**  
**QUALITY ASSURANCE PROJECT PLAN**  
**FOR**  
**THE EVALUATION OF AN ADVANCED REVERSE**  
**OSMOSIS SYSTEM AT THE SUNNYVALE, CALIFORNIA**  
**HEWLETT-PACKARD FACILITY**

July 20, 1990

Submitted to:

U.S. Environmental Protection Agency  
Risk Reduction Engineering Laboratory  
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QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM  
for  
RREL Contracts/IAGs/Cooperative Agreements/In-house Projects

Contractor: Science Applications International Corporation

QA Project Plan Title: The Evaluation of An Advanced Reverse Osmosis System at the Sunnyvale, California Hewlett-Packard Facility

Revision Date: 7/20/90

**COMMITMENT TO IMPLEMENT THE ABOVE QA PROJECT PLAN:**

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Curtis J. Schmidt  
Signature

8/5/90  
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**APPROVAL TO PROCEED IN ACCORDANCE TO THE ABOVE QA PROJECT PLAN:**

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Date  
9-6-90  
Date

RREL (QAP/P AF)  
(Sept 1988)



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## 1.0 INTRODUCTION

The objective of the Waste Reduction Innovative Technology Evaluation (WRITE) Program is to identify, develop, demonstrate, and evaluate innovative pollution prevention techniques. The WRITE Program is part of the EPA Risk Reduction Engineering Laboratory's (RREL) pollution prevention research program and is a cooperative effort between the U.S. EPA and state and local environmental programs to identify, develop, demonstrate and evaluate innovative pollution prevention techniques. Specifically, the Waste Reduction Program provides engineering and economic evaluations plus information dissemination for methodologies that have the potential of reducing the quantity and/or toxicity of waste generated at the source, or to achieve practicable on-site reuse through recycling.

An Advanced Reverse Osmosis System (AROS) manufactured by Water Technologies, Inc. (WTI), Minnesota was installed in the Hewlett-Packard (HP) plant in Sunnyvale, California to treat and recover nickel sulfate plating rinse water. The technology provides zero discharge capability. A test program is ongoing to evaluate the effectiveness of the AROS in the treatment and recovery of metal plating rinse water and compare its costs with that of an existing chemical precipitation system.

The AROS unit was installed in November 1989. After initial installation and debugging, the system was tested from November 21, 1989 to December 18, 1989. The system was temporarily taken off-line at the end of 1989, to allow HP to test and evaluate the plating bath quality and to create a baseline of comparison for bath contents and performance. Results were considered acceptable and the AROS unit was restarted in January 1990, and has been operated on-line since then.

The operation of the AROS unit is monitored with computers using specialized software. Samples are collected and analyzed by HP to assess the chemical content of various streams into and out of the AROS unit. This plan describes proposed activities

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required to assess the AROS technology and evaluate data. Results of the evaluation will be presented in a final report and papers will be submitted to related technical journals and conferences.

The existing wastewater treatment system for plating wastes is designated by HP as the "Water Purification System" (WPS). A schematic of the processes is shown in Figure 1-1.

The design flow of the WPS is about 120 gallons per minute (gpm). Rinse waters from various plating operations, including nickel, copper and tin are collected. Sulfuric acid is added to lower the pH and ferrous sulfate is added to reduce the bivalent copper to monovalent copper and to form ferrous complexes with the free EDTA that is used as a complexing agent to solubilize copper. The pH is then raised to 11 in a separate tank with the addition of sodium hydroxide causing metals to precipitate out as hydroxide salts. The chemical addition is done in a completely mixed reactor. Settling occurs in three subsequent tanks. Chemical sludges are pumped to a recessed plate filter press system for dewatering. Dewatered sludges are disposed to an off-site RCRA-approved facility at a reported cost of about \$275/ton for transport and disposal. Dewatered sludge generation is about 11.5 tons per month.

The effluent from the recirculation/settling tanks is pumped to 48 ultrafilters for final polishing. Solids collected by the filters are recirculated back to the recirculation/settling tanks. The filtrate is discharged to the city sewer.

A minor side stream from the ultrafiltration system is an hydrochloric acid solution used to periodically (every 3 months) clean the filter elements. The acid solution can be used several times for cleaning. Approximately 50 gallons of acid are used every 6 months. The acid collected from the cleaning process is pumped into an onsite low flow high concentration neutralization system for batch treatment and disposal. HP has good records for the performance and costs of the existing water treatment system.

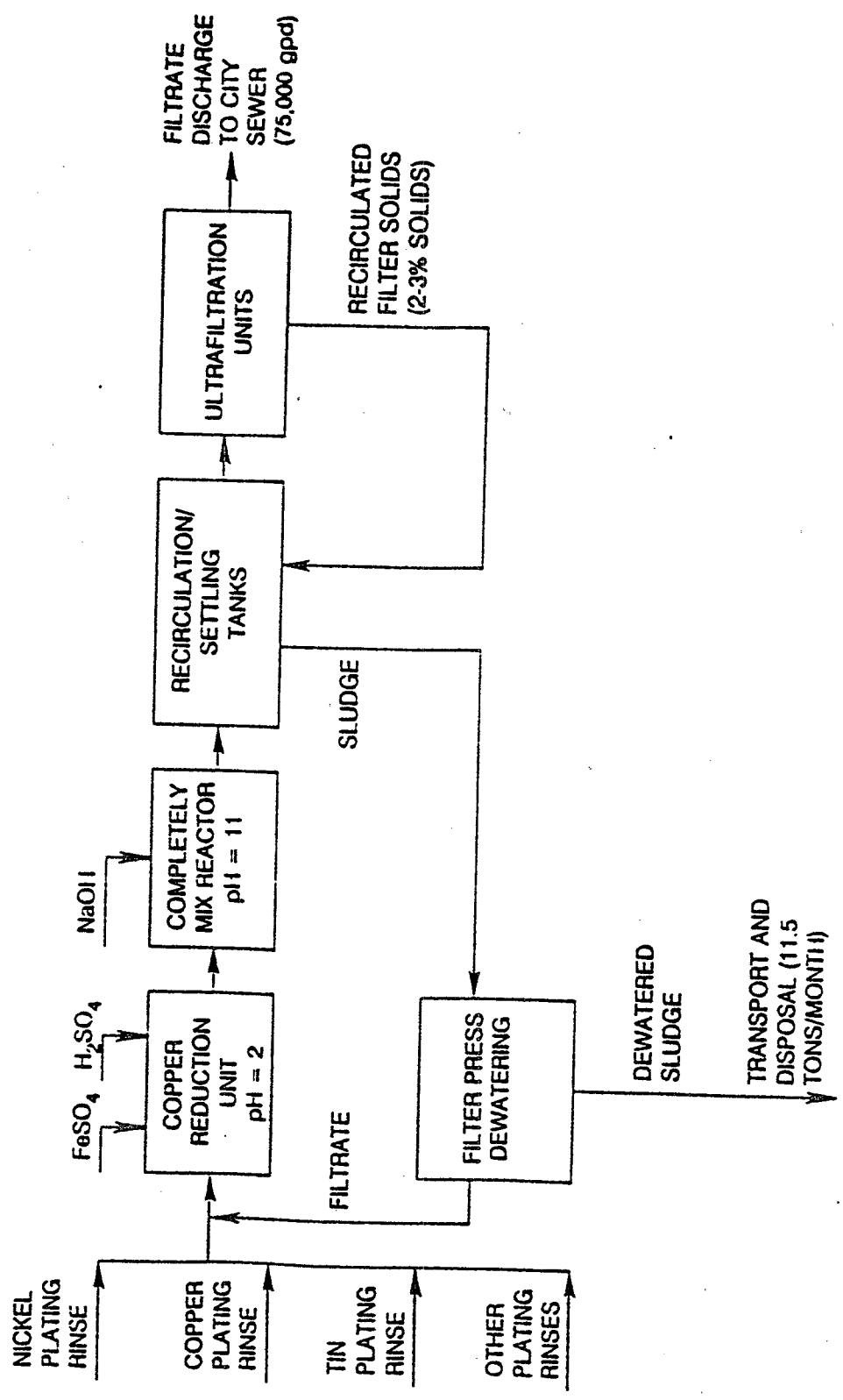


Figure 1-1. Schematic Diagram of the Existing Water Purification System (WPS)

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## 2.0 PROJECT DESCRIPTION

### 2.1 Background

The demonstration AROS unit is currently installed to treat and recycle nickel sulfate and nickel chloride rinse solutions. A schematic flow diagram of the plating process units and the AROS unit is shown in Figure 2-1. The nickel plating line consists of two plating baths followed by a "dirty" rinse tank and a "clean" rinse tank. Rinse water flows countercurrent to the flow of the items being plated. The overflow from the "dirty" rinse tank is the influent to the AROS unit at a flow of about 4 to 5 gpm. The treated effluent (permeate) produced by the AROS unit becomes the clean water supply to the "clean" rinse tank. A supply of fresh deionized water provides additional makeup water to replace evaporative losses.

The AROS unit generates a concentrate at different intervals, depending upon the conditions of flow, conductivity and pH within the AROS unit. Sensors and controls required to manage the membranes set the valves on and off to allow or prevent flow to or from the concentrate stream pipes (Figure 2-2). The concentrate is returned to the plating bath, as shown in Figure 2-1.

The AROS unit is basically a reverse osmosis (RO) unit with a highly sophisticated design that allows recovery of rinse water and plating bath solutions. The AROS differs from other reverse osmosis units by its ability to use tolerant membranes that do not require pH adjustment to neutral. Membrane materials and system components have been specially adapted to plating environments and can concentrate dilute rinse solutions to near bath strength (initial conductivity of the solution) without the need for additional concentration technology. The unit also contains a continuous monitoring system that monitors the influent, permeate and concentrate temperatures, flow rates and conductivities every 15 seconds. At the HP facility, the unit has demonstrated the ability to produce concentrate at a nickel concentration that is about 40 to 50 percent of the original bath strength. Figure 2-2 is an schematic diagram of the AROS unit.

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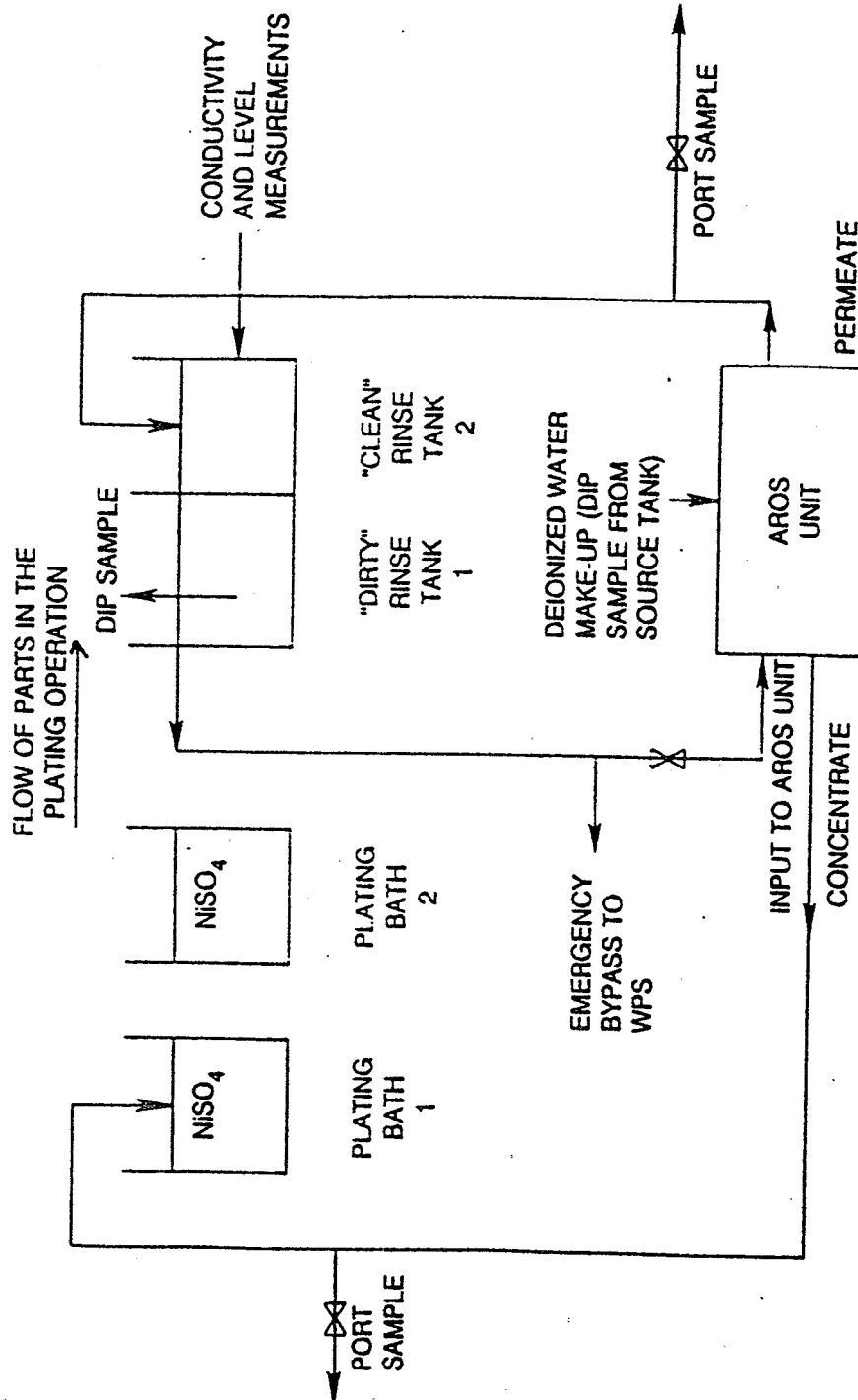


Figure 2-1. Schematic Diagram of the Advanced Reverse Osmosis System (AROS) for the Nickel Plating Operation And Proposed Sample Locations

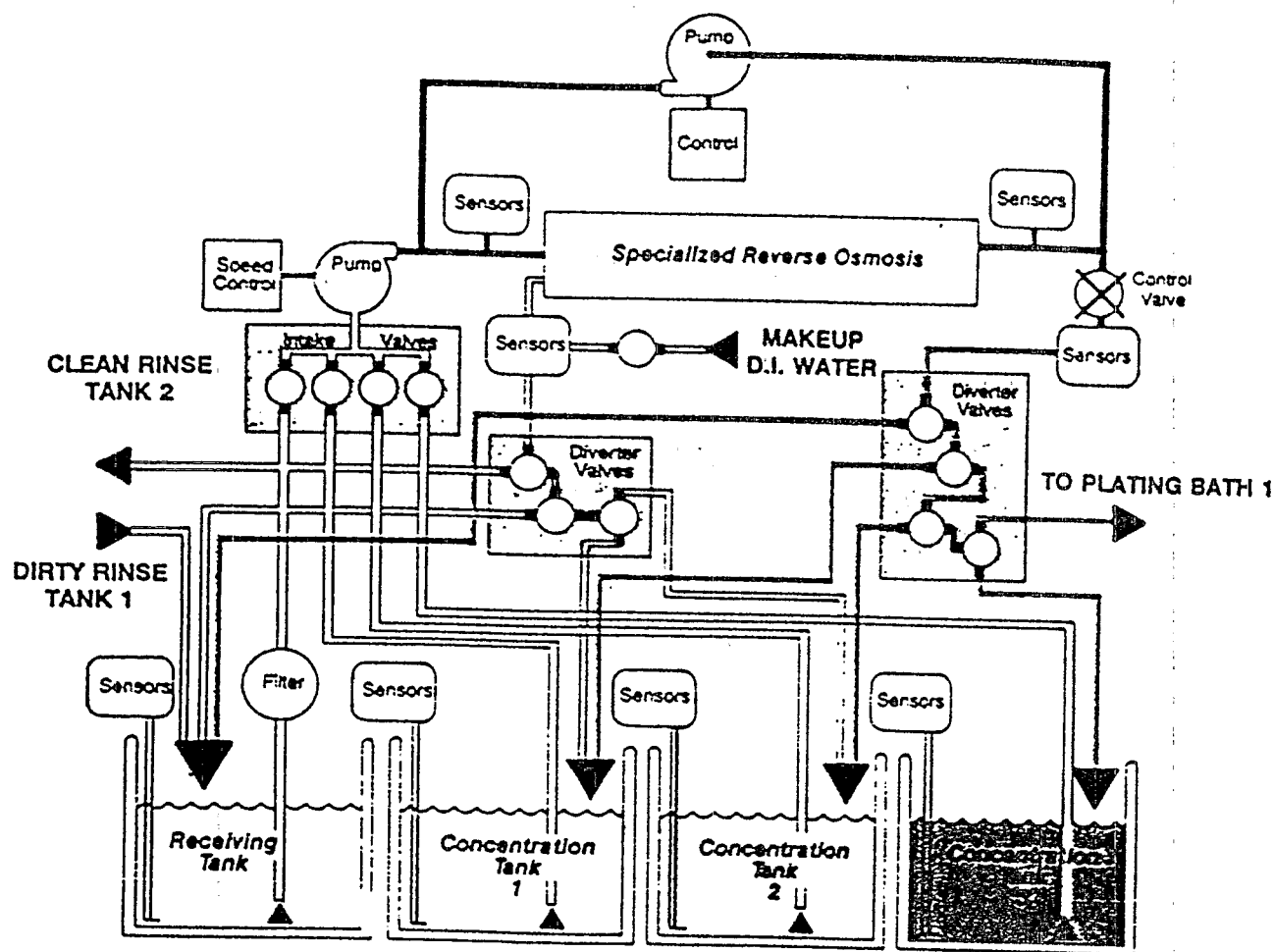


Figure 2-2. Inside of a Typical AROS Unit\*

\* Courtesy of Water Technologies, Inc.

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## 2.2 Existing Sampling and Monitoring Program

### 2.2.1 Existing WPA Sampling and Monitoring Program

The existing WPS operation performance is monitored by an on-going sampling program of the effluent. The effluent from the ultrafilters is collected using 50 ml samplers 6 times a day at three-hour intervals during the first two day-shifts. These samples are composited into one sample which is analyzed for parameters such as nickel, copper, iron and pH. Sludge disposed to an offsite facility is not analyzed by HP.

### 2.2.2 Existing AROS Sampling and Monitoring Program

The existing monitoring program for the AROS includes the parameters of flow, conductivity and pH at various points in the system. Streams monitored include the deionized water makeup line, the emergency overflow to the WPS line, the concentrate return line and the permeate return line shown in Figure 2-2. Readings are taken every 15 seconds and are displayed on screen instantaneously at an onsite monitor located at the facility. Preset values of conductivity and flow control the valves.

In addition to the continuous monitoring described above, the first plating bath is sampled and analyzed weekly, collecting 1 liter samples. The analyses conducted are nickel, pH, Nikal PC-3 (saccharin), boric acid, chloride, and ductility of a "plate" from the concentrate solution. Nickel concentration is measured to estimate how much nickel is recovered and returned to the plating bath. The quantity of recovered nickel reduces the amount of new nickel that must be added to the plating bath to maintain the proper concentration for optimum plating conditions. Boric acid (approximately 50 ppm) is measured because the amount of boric acid needs to be determined in the plating bath solution for buffer. Nikal PC-3 containing an aqueous solution of organic salts (the only salt identified on MSDS is sodium saccharin) is added for plating operations and needs to be maintained at a desirable level (1.4 ppm). Chloride (approximately 15 ppm) is present in the plating bath as nickel chloride and needs to be maintained at a certain desirable level. Ductility tests are run to check for impurities.

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Other analyses are conducted to estimate the purity of the return concentrate. During the reverse osmosis process, chemicals other than nickel are concentrated. These parameters include VersaCLEAN 400, containing sulfamic acid and resistant breakdown products. VersaCLEAN 400 is present in the plating bath water as a residue from previous cleaning operations, and resistant breakdown products result from the high operating temperatures of the nickel plating bath. Buildup of these compounds present in the concentrate stream may affect the ductility of the nickel layer plated on the circuit boards. The ductility is tested to determine the suitability of the return concentrate to maintain the quality in the nickel plating bath. Impurities have to be kept to a minimum to avoid brittleness.

There are two kinds of ductility tests. The first one is visual and the other uses a ball bearing method. For visual ductility tests, a 3-inches by 5-inches brass panel is first plated using the concentrate solution. The piece is then inspected visually for impurities (discoloration, spots, dark or light areas, etc.). For ball bearing tests, a piece of stainless steel panel, 2 inches by 2 inches, is placed in the concentrate solution and plated. The thin metal is then peeled from the plate and subjected to a laboratory ductility test developed by HP. The thin metal rectangle is placed in a ductility test apparatus and a ball bearing is pushed slowly into it. The distance the ball moves before breaking the sheet is measured and compared to the known distance that provides satisfactory ductility.

During the first month of operation of the AROS unit, concentrate was collected into 55-gallon drums before being returned to the nickel plating bath solution. Chemical and ductility tests were run to evaluate plating quality of the concentrate to establish the integrity of the concentrate. Chemical tests included nickel concentration and pH measurements. Ductility tests were both visual and ball bearing type.

The first 19 tests were run on the plating bath solution to establish the baseline organic impurities concentrations. Then, the AROS unit was hooked up and subsequent ductility test results were compared to the baseline results. These tests showed that the



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integrity of the plating bath solution would not be in jeopardy when the concentrate stream is in-line with the plating operation.

### 2.3 Purpose and Experimental Design

The purpose of additional sampling and monitoring is to compare the HP laboratory's analysis results with the ones of an independent laboratory (SAIC's) and to obtain a one-day snap shot of the AROS unit operation at the facility. It is planned to conduct additional analyses on a daily composite sample collected from each of the following streams (see Figure 2-1):

- Concentrate stream (returned to the plating bath)
- Permeate stream (recycled rinse water return)
- Influent stream to the AROS unit (the dirty water rinse overflow)
- Deionized water (water makeup to the AROS unit)

Due to the internal storage capacity of the AROS unit, the concentrate and deionized water (DI) makeup streams are not continuous (Figure 2-2). Only a discrete sample can be taken from these two streams, whereas, a one-day composite over a period of 16 hours will be collected from the influent and permeate streams. Dip samples will be taken from the "dirty" rinse tank 1 and the deionized water, storage tank for the influent stream to the AROS unit and deionized water samples. Concentrate and permeate samples will be obtained from ports. Temporary storage of the concentrate during the 16-hour sample may be necessary due to the non-continuous nature of the concentrate flow.

The collected samples will be split and analyzed by both the HP laboratory and SAIC's laboratory. HP analyses to be run on the samples are listed in Table 2-1. SAIC's laboratory analyses will include parameters listed in Table 2-2. All measurements made by SAIC, except TOC/Color, are critical measurements. The TOC and/or Color analyses are proposed as possible methods to monitor the organic impurities. One or both of these methods will be used. However, it is possible that these general methods may not be able to distinguish between the buildup of a particular compound or compounds that affect the

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quality of the metal plate and higher concentrations of organic compounds in general. In other words, the buildup of a particular compound or compounds (not identified) would be a problem; however, no noticeable change in the total organic content is observed, i.e., the concentration of the offending compound is only a very small fraction of the total organic or "colored" (UV and visible) compounds.

TABLE 2-1  
 ANALYSES WHICH WILL BE DONE BY HEWLETT-PACKARD

Parameter	Method	Detection Limit (ppm)	Stream
Nickel (1)	AA	0.5	Influent to AROS Permeate
pH (2)	pH meter	NA	Concentrate Influent to AROS Permeate
Conductivity (2)	Cond. meter	NA	Concentrate Influent to AROS Permeate

- (1) The collected composite sample will be split and analyzed by the Hewlett-Packard Laboratory and SAIC's Laboratory
- (2) One-day of monitoring data (every 15 seconds) will be provided by Hewlett-Packard

NA Not applicable

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TABLE 2-2

ANALYSES PROPOSED FOR ADDITIONAL SAMPLING AND MONITORING

<i>Parameter</i>	<i>Method (a)</i>	<i>Detection Limit (ppm)</i>	<i>Stream</i>
Nickel	AA (EPA 249.1) or (EPA 200.7)	0.04	Concentrate
		0.015	Influent to AROS
	AA (EPA 249.2) or (EPA 200.7)	0.001	Permeate
		0.015	DI water
Sulfate	Gravimetric (EPA 375.3) or Ion chromatography (EPA 300.0)	—	Concentrate
			Influent to AROS
			Permeate
			DI water
Chloride	Titrametric (EPA 325.3)	—	Concentrate
			Influent to AROS
			Permeate
			DI water
pH	pH meter (EPA 150.1)	—	Concentrate
			Influent to AROS
			Permeate
			DI water
Conductivity	EPA 120.1	—	Concentrate
			Influent to AROS
			Permeate
			DI water
TDS	EPA 160.1	—	Concentrate
			Influent to AROS
			Permeate
			DI water
TOC (b)	EPA 415.1	—	Concentrate
			Influent to AROS
			Permeate
			DI water
Color (b)	EPA 110.3	—	Concentrate
			Influent to AROS
			Permeate
			DI water

(a) Methods for Chemical Analysis of Water and Wastes, EPA/600/4-79/020, March 1979.

(b) Methods are optional; one of the two methods will be selected.

#### 2.4 Comparative Cost Estimates for WPS and AROS

An economic evaluation will be made of the AROS system on a side-by-side basis with the existing WPS system. The capital cost of the AROS is \$62,650. The savings from the AROS unit are directly related to the reduction in spending for:

- Water and sewer charges related to discharges to the city sewer
- DI production
- Batch waste treatment for small quantities of waste and sludge treatment
- Sludge transport and disposal
- Purchase of new plating chemicals to make up for drag-out losses
- Power costs for the WPS
- Ultrafiltration membrane replacement
- Labor costs for the WPS (it may be impossible to quantify the slight difference, if any, resulting from a 5% volume reduction)
- Liability costs (if applicable)
- Worker health and safety training costs (if applicable)

The AROS unit will be analyzed for costs of:

- Power
- Replacement of the AROS membranes
- Labor
- Part replacement

Savings related to the recirculation of streams in the AROS unit, paybacks, health and safety benefits, and trade-offs, will be examined. HP has generally kept good records of costs that can be analyzed to conduct this evaluation.

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## 2.5 Organization and Responsibilities

A project organization and authority chart is shown in Figure 2-3. The California DHS and HP are cooperating with the Risk Reduction Engineering Laboratory (RREL) on this evaluation. Mr. Curtis Schmidt is the SAIC Work Assignment Manager and is responsible for the technical and budgeting aspects of this work assignment. Mr. Thomas Wagner is QA Manager and prepared this QAPjP and is responsible for QA oversight on this work assignment. Mrs. Ilknur Erbas-White will handle the day-to-day activities of the project.

## 2.6 Schedule

The sampling is scheduled for mid to late August or early September, 1990.

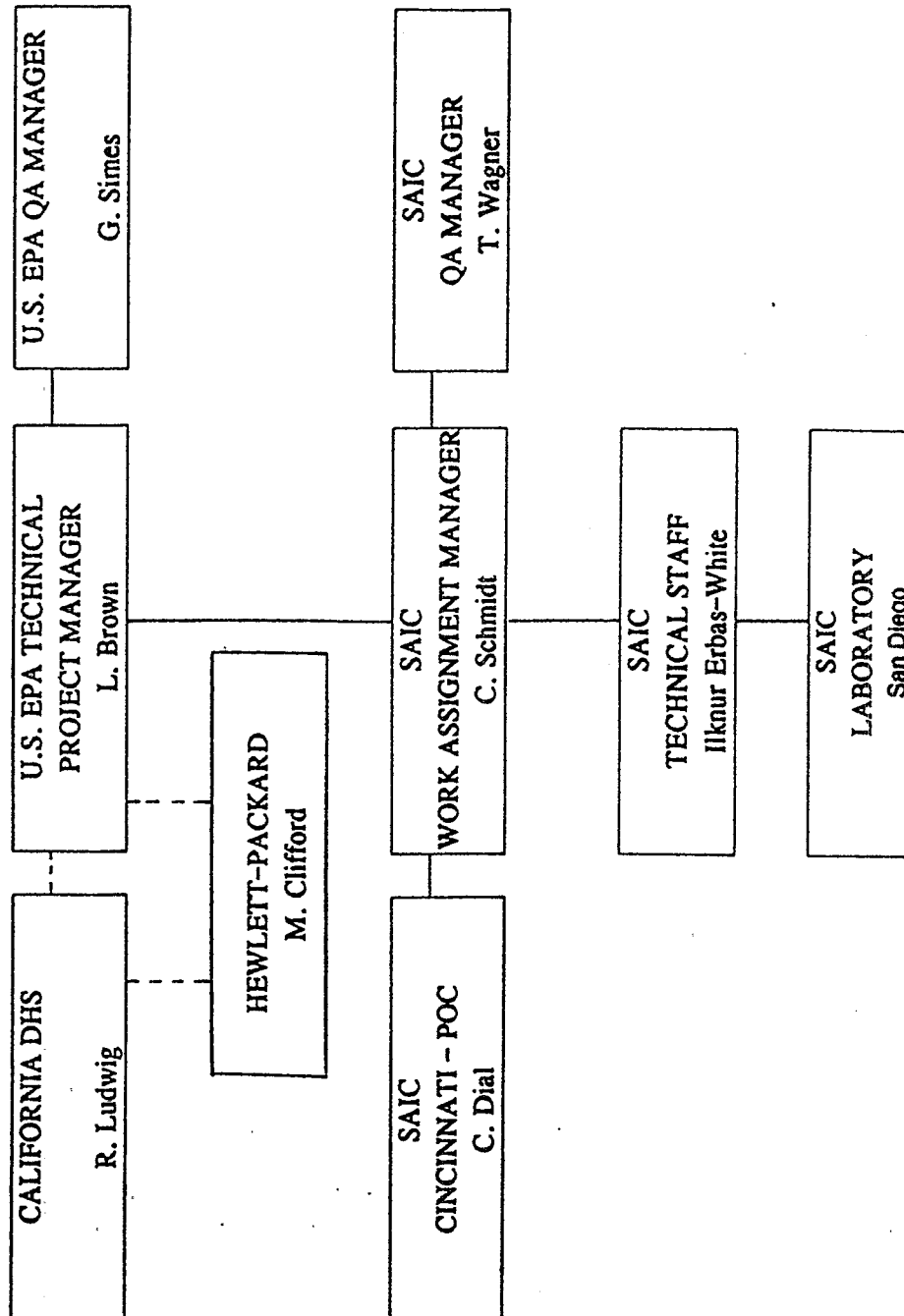


Figure 2-3. Project Organization

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### 3.0 QUALITY ASSURANCE OBJECTIVES

#### 3.1 Precision, Accuracy, Completeness, and Method Detection Limits

Objectives for accuracy, precision, method detection limits, and completeness for the critical measurements are listed in Table 3-1. Accuracy (as percent recovery) will be determined from matrix spike recovery for nickel, sulfate, and chloride, and from laboratory control samples for pH, conductance and TDS. Precision (as relative percent difference) will be determined from the results of matrix spike duplicates for nickel, sulfate, and chloride, and from laboratory duplicate analyses for pH, conductance and TDS. The completeness will be determined from the number of data meeting the criteria in Table 3-1 divided by the number of samples collected.

#### 3.2 Representativeness and Comparability

Representativeness and Comparability are qualitative parameters. The samples obtained will be as representative of a typical day's operation as the day's operation is typical. Regardless of how typical the operation is, the purpose will be accomplished because a independent comparison of the HP laboratory's analysis will be obtained. The data obtained in this program will be comparable because all the methods are taken from a standard EPA reference manual.

#### 3.3 Method Detection Limits

It is anticipated that the deionized water and the permeate sample might have values below the method detection limits. Since both streams approach a "distilled water" matrix, listed detection limits should apply. All other streams are also aqueous streams; therefore, the only adjustments that should be necessary are those caused by dilutions necessary to remain within the calibration range of the methods.

TABLE 3-1. Quality Assurance Objectives for Critical Measurements

Parameter	Method (a)	Accuracy (b) (as % recovery)	Precision (c) %	Method Detection Limit (mg/l)	Completeness %
Nickel	249.1	75 - 125	20	0.04	100
	249.2	75 - 125	20	0.001	100
Sulfate	375.3	75 - 125	20	10 (d)	100
pH	150.1	± 0.3 pH units	0.1 pH units	NA	100
Conductivity	120.1	80 - 120	10	NA	100
TDS	160.1	80 - 120	10	2	100
Chloride	325.3	75 - 125	20	0.1	100

(a) References are to "Methods for Chemical Analysis of Water and Wastes", EPA/600/4-79/020

(b) Determined from MS/MSD analyses for Nickel, Sulfate and Chloride; others determine from laboratory control samples. pH is determined as bias rather than percent recovery.

(c) Determined as relative percent difference for MS/MSD analyses for Nickel, Sulfate and chloride; others determined from duplicate laboratory analyses. pH which is calculated as the range of duplicate analyses.

(d) Based on 50 ml sample and 1 mg of Barium Sulfate weighed.

NA - Not applicable

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#### 4.0 SITE SELECTION AND SAMPLING PROCEDURES FOR CRITICAL MEASUREMENT

The sampling points were shown in Figure 2-2. Each influent stream (dirty water rinse overflow and DI makeup) and each effluent stream (concentrate returned to the plating bath and permeate added to the final rinse bath) of the AROS unit will be sampled.

The one-day composite sample schedule is given in Table 4-1. HP personnel will collect the samples and SAIC will observe the collection and handling of the samples.

Table 4-2 lists each parameter to be determined, the required preservation method, the maximum holding, the nominal analytical volume, the minimum volume required for analysis including QC, and the sample size to be obtained in the field. The sample container size is roughly twice the minimum required volume.

A separate 1 liter bottle will be used for each of the four streams to preclude any cross contamination. The aliquots for the composite samples will be stored in 5-gallon plastic containers with lids until all portions are obtained. These 5-gallon containers will be mixed by swirling and dispensed into triplicate bottles of 500, 180 and 2000 ml each and preserved according to Table 4-2. One set will be given to HP, one will be shipped to the laboratory for analysis, and one will be shipped to the laboratory and held in reserve.

The DI water permeate and concentrate samples (aliquots) will be obtained from taps in these lines. These taps will be opened momentarily and flushed into a waste container prior to obtaining each aliquot. The influent samples (aliquots) will be obtained by dipping a 1 liter container into the dirty rinse tank near the outfall to the AROS influent line.

Sample bottles will either be purchased from I-CHEM (precleaned) or cleaned by the procedure for metals in SW-846, 3rd. Ed., Chapter 3.

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TABLE 4-1. Proposed Sampling Schedule

<i>Location</i>	<i>Sampling Procedure</i>
Influent to the AROS unit.	Composited over a period of 16 hours during two shifts. Two 2-liter dip samples from the dirty rinse tank will be taken per shift. Final composite will be split; one sample will be sent to SAIC's laboratory, the other will be analyzed by the Hewlett-Packard laboratory.
Permeate	Composited over a period of 16 hours during two shifts. Two 2-liter samples will be taken per shift from a sample port. Final composite will be split; one sample will be sent to SAIC's laboratory, the other will be analyzed by the Hewlett-Packard laboratory.
Concentrate	One 8-liter sample will be collected during the two shifts. When the concentrate flow occurs as observed from the AROS unit computer monitor, a sample will be collected from the sample port, or temporary storage will be provided to get an 8-liter sample. The sample will be split; one sample will be sent to SAIC's laboratory, the other will be analyzed by the Hewlett-Packard laboratory.
DI water	One 8-liter sample will be collected from the DI water storage tank. The sample will be split; one sample will be sent to SAIC's laboratory, the other will be analyzed by the Hewlett-Packard laboratory.

TABLE 4-2. Sample Volumes, Preservation and Holding Times

Parameter	Preservation	Maximum Holding Time	Nominal Analytical Volume (ml)	Type of QC	Minimum Required Volume (ml)	Sample Container Size, ml(oz)
Nickel	HNO <sub>3</sub> , pH<2	6 months	100	MS/MSD	300	500 (16)
Chloride	None	28 days	50	MS/MSD	150	see below
Sulfate	Cool 4° C	28 days	50	MS/MSD	150	see below
pH	None	None*	25	Duplicate	50	see below
Conductivity	Cool 4° C	28 days	100	Duplicate	200	see below
TDS	Cool 4° C	7 days	100	Duplicate	200	2 liter <sup>b</sup> (1/2 gallon)
Color	Cool 4° C	48 hours	50	Duplicate	100	see TDS above
TOC	H <sub>2</sub> SO <sub>4</sub> or HCl, pH<2, Cool 4° C	28 days	25	MS/MSD	75	180 (6)

(a) The pH will be checked in the laboratory to determine the correspondence with the HP laboratory and the on-line measurements.

(b) This 2-liter sample container will be cool to 4° C and used for chloride, sulfate, pH, conductivity, TDS, and color, if necessary.

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The field personnel will document, on data sheets (Figure 4-3), the date and time each aliquot is obtained from each stream. The volumes obtained for each aliquot will be the same because the same 1 liter container (one bottle for each stream) will be filled each time an aliquot is obtained. (The 1 liter container will be filled more than once to obtain the required volume for each aliquot or sample.)

The amount of preservative added will also be recorded. Samples will be labeled (see Figure 4-4) and shipped by overnight delivery service to the laboratory in coolers containing ice. If "blue" ice is used in the coolers, samples will be initially cooled with regular ice prior to being packed in the coolers with blue ice.

The *Chain of Custody Record* shown in Figure 4-5 will be completed for each cooler shipped to a laboratory.

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QUALITY ASSURANCE AND QUALITY CONTROL FORMS  
FOR THE HEWLETT-PACKARD AROS UNIT SAMPLING EVENT

SAMPLE: COMPOSITE GRAB DIP PORT OTHER  
(Circle) DUPLICATE BLANK -----

SAMPLE NUMBER: \_\_\_\_\_

SAMPLE TYPE: LOW CONC. AVERAGE CONC. HIGH CONC.  
(Circle)

SAMPLE TAKEN AT: DATE \_\_\_\_/\_\_\_\_/90 TIME: \_\_\_\_:\_\_\_\_ AM/PM

FOR COMPOSITE SAMPLES ONLY:

Composite 1: SHIFT:\_\_\_\_ DATE:\_\_\_\_/\_\_\_\_/90 TIME: \_\_\_\_:\_\_\_\_ AM/PM VOL: \_\_\_\_1/ml  
Composite 2: SHIFT:\_\_\_\_ DATE:\_\_\_\_/\_\_\_\_/90 TIME: \_\_\_\_:\_\_\_\_ AM/PM VOL: \_\_\_\_1/ml  
Composite 3: SHIFT:\_\_\_\_ DATE:\_\_\_\_/\_\_\_\_/90 TIME: \_\_\_\_:\_\_\_\_ AM/PM VOL: \_\_\_\_1/ml  
Composite 4: SHIFT:\_\_\_\_ DATE:\_\_\_\_/\_\_\_\_/90 TIME: \_\_\_\_:\_\_\_\_ AM/PM VOL: \_\_\_\_1/ml  
Composite 5: SHIFT:\_\_\_\_ DATE:\_\_\_\_/\_\_\_\_/90 TIME: \_\_\_\_:\_\_\_\_ AM/PM VOL: \_\_\_\_1/ml  
Composite 6: SHIFT:\_\_\_\_ DATE:\_\_\_\_/\_\_\_\_/90 TIME: \_\_\_\_:\_\_\_\_ AM/PM VOL: \_\_\_\_1/ml  
Composite 7: SHIFT:\_\_\_\_ DATE:\_\_\_\_/\_\_\_\_/90 TIME: \_\_\_\_:\_\_\_\_ AM/PM VOL: \_\_\_\_1/ml  
Composite 8: SHIFT:\_\_\_\_ DATE:\_\_\_\_/\_\_\_\_/90 TIME: \_\_\_\_:\_\_\_\_ AM/PM VOL: \_\_\_\_1/ml

COMMENTS: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Figure 4-3. Sampling Data Sheet

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8400 Westpark Drive, McLean, Virginia 22102

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<i>Location:</i>	<i>Project No.:</i>
<hr/>	
<i>Sample Date/Time:</i>	
<hr/>	
<i>Sample No.:</i>	<i>Sample Location:</i>
<hr/>	
<i>Analysis:</i>	
<hr/>	
<i>Collection Method:</i>	<i>Purge Volume:</i>
<hr/>	
<i>Preservative:</i>	
<hr/>	
<i>Comments:</i>	
<hr/>	
<i>Collector's Initials:</i> _____	

Figure 4-4. Example Sample Label

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### Figure 4-5. Chain of Custody Record

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## 5.0 ANALYTICAL PROCEDURES AND CALIBRATION

Analytical procedures for all critical measurements are referenced in Table 3-1. The only other measurement is color or TOC that will be performed according to method 110.3 or 415.1 from the same reference. These are all EPA procedures and specify the required calibration to be performed. The samples for metal analysis will be digested according to the procedure in Section 4.1.3 of the same reference. For those procedures requiring a calibration curve, the calibration will be verified after this sample set is run. For example, there will be two samples for metals analysis by flameless AA plus three QC samples for a total of five samples. After initial calibration, these five samples will be analyzed followed by a calibration check that must agree within  $\pm 20$  percent of its original value.



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## 6.0 DATA REDUCTION, VALIDATION, AND REPORTING

Data will be reduced by the procedures specified in the methods and reported by the laboratory in the units also specified in the methods. The work assignment manager or his designee will review the results and compare the QC results with those listed in Table 3-1. Any discrepancies will be discussed with the QA Manager.

All data will be reviewed to ensure that the correct codes and units have been included. After reduction, data will be placed in tables or arrays and reviewed again for anomalous values. An inconsistencies discovered will be resolved immediately, if possible, by seeking clarification from the sample collection personnel responsible for data collection, and/or the analytical laboratory.

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## 7.0 INTERNAL QUALITY CONTROL CHECKS

Due to the nature of this project, the collection of field blanks, equipment blanks, and trip blanks are not deemed necessary. The internal QC checks appropriate for the measurement methods to be utilized for this project are summarized in Table 7-1. These items are taken from the methods and the QC program outlined in Section 3 of this QAPjP. Because the number of samples to be analyzed for this project is small, all samples and related QC will be analyzed in one batch.

For this project, a matrix spike/matrix spike duplicate (MS/MSD) or laboratory duplicate analysis will be performed on two samples, because the pure water streams (DI water and permeate) and the contaminated water streams (AROS influent and concentrate) are considered different matrices.

TABLE 7-1. Internal QC Checks

Parameter	Method	Initial Calibration	Calibration Checks	Method Blank	MS/MSD (a)	Duplicate Sample Analysis	QC Sample
Nickel	249.1 or 249.2	Blank plus 3 standard	± 20%	1	Yes 50 - 100 ppm Yes 10 times D.L.	NR	NR
Chloride	325.3	Titrant standardized against NaCl solution	NR	1	Yes 5 - 15 ppm	NR	NR
Sulfate	375.3	NR (b)	NR	1	Yes 50 - 150 ppm	NR	NR
pH	150.1	2 points (c)	± 0.1 ph unit	NR	NR	Yes	Yes (c)
Conductivity	120.1	4 points	± 20%	NR	NR	Yes	Yes
TDS	160.1	NR (b)	NR	1	NR	Yes	Yes
TOC	415.1	Varies with instrument	± 20%	1	Yes (d)	NR	NR
Color	110.3	NR	NR	1	NR	NR	NR

(a) Anticipated values (mg/l) are given for concentrate stream; permeate and DI water are expected to be low (D.L.). The influent to the AROS will be intermediate between these two extremes.

(b) Daily check of balance required.

(c) Anticipated pH is 4.

(d) No expected value is available for concentrate stream. Permeate and DI expected to be low (D.L.). Spike levels for AROS influent and concentrate will have to be determined.

NR - Not Required

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## 8.0 PERFORMANCE AND SYSTEM AUDITS

No audits are planned for this project.

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## 9.0 CALCULATION OF DATA QUALITY INDICATORS

### 9.1 Accuracy

Accuracy for nickel, sulfate, chloride, and TOC will be determined as the percent recovery of matrix spike samples (two per matrix). The percent recovery is calculated according to the following equation:

$$\% R = 100\% \times \left[ \frac{C_i - C_o}{C_i} \right]$$

where

%R = percent recovery  
 $C_i$  = measured concentration in spiked sample aliquot  
 $C_o$  = measured concentration in unspiked sample aliquot  
 $C_i$  = actual concentration for spike added

Accuracy for the other critical measurements, except pH, will be determined from laboratory control samples according to the equation:

$$\% R = 100\% \left[ \frac{C_m}{C_i} \right]$$

where

%R = percent recovery  
 $C_m$  = measured concentration of standard reference material  
 $C_i$  = actual concentration for standard reference material

For pH, accuracy will be determined as bias according to the equation:

$$B = pH_m - pH_t$$

where

B = bias  
 $pH_m$  = measured pH of standard reference material  
 $pH_t$  = actual pH of standard reference material

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## 9.2 Precision

Precision will be determined from the difference of percent recovery values of MS and MSDs for nickel, sulfate, chloride, and TOC, and duplicate laboratory analyses for other parameters. The following equation will be used for all parameters except pH:

$$RPD = \frac{[C_1 - C_2] \times 100\%}{[C_1 + C_2] / 2}$$

where

RPD = Relative percent difference  
 $C_1$  = The larger of two observed values  
 $C_2$  = The smaller of the two observed values

Precision for pH will be estimated by calculation of the range using the following equation:

$$D(pH) = pH_1 - pH_2$$

where

$D(pH)$  = precision limits for pH  
 $pH_1, pH_2$  = observed values for duplicate samples

## 9.3 Completeness

Completeness will be calculated as the percent of valid data points obtained from the total number of samples obtained.

$$\% \text{ Completeness} = \frac{VDP}{TDP} \times 100$$

where

VDP = number of valid data points  
 TDP = total number of samples obtained.

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## 10.0 CORRECTIVE ACTION

Corrective actions will be initiated whenever quality control limits (e.g., calibration acceptance criteria) or QA objectives (e.g., precision, as determined by analysis of duplicate matrix spike samples) for a particular type of critical measurement are not being met. Corrective actions may result from any of the following functions:

- Performance evaluation audits
- Technical systems audits
- Interlaboratory/interfield comparison studies

All corrective action initiations, resolutions, etc. will be implemented immediately and will be reported in Sections One and Two (Difficulties Encountered and Corrective Actions Taken, respectively) in the existing monthly progress reporting mechanisms established between SAIC and EPA-RREL, and in the QA section of the final report. The QA Manager will determine if a correction action has resolved the QC problem.

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## 11.0 QA/QC REPORTS TO MANAGEMENT

This section describes the periodic reporting mechanism, reporting frequencies, and the final project report which will be used to keep project management personnel informed of sampling and analytical progress, critical measurement systems performance, identified problem conditions, corrective actions, and up-to-date results of QA/QC assessments. As a minimum, the reports will include, when applicable:

- Changes to the QA Project Plan, if any.
- Limitations or constraints on the applicability of the data, if any.
- The status of QA/QC programs, accomplishments and corrective actions.
- Assessment of data quality in terms of precision, accuracy, completeness, method detection limit, representativeness, and comparability.
- The final report shall include a separate QA section that summarizes the data quality indicators that document the QA/QC activities that lend support to the credibility of the data and the validity of the conclusions.

For convenience, any QA/QC reporting will be incorporated into the already well-established monthly progress reporting system between SAIC and EPA-RREL for all TESC Work Assignments. Any information pertaining to the above-listed categories will be reported under Sections One thru Three (Difficulties Encountered, Corrective Actions Taken, and Current Activities, respectively) in the monthly reports.



July 21, 1993

Format review for R-2022

**WATTS NICKEL AND RINSE WATER RECOVERY VIA AN ADVANCED REVERSE OSMOSIS SYSTEM**

Project Officer: Lisa M. Brown

This draft report requires a few adjustments before it is ready for publication. This review is for format only, not for editorial or for content. Needed changes are listed below. Assistance in making these changes can be found in the Handbook for Preparing Office of Research and Development Reports.

1. The Abstract should have the work done under statement as the last paragraph. (samples enclosed). Delete advertising of the company name and/or address at the top or bottom of pages in Appendix B
2. The final camera ready copy must be typed within the image area shown on the enclosed typing guide sheets with the page numbers centered. All text, including figures and tables, must fit within the image area. Furnish originals or very good reproducible copies of the figures and tables.
3. Your Project Summary has been sent out for editing and will be returned to you as soon as possible.
4. After the adjustments are made, prepare your project report/project summary package for clearance using the forms indicated in the checklist for clearance packages. After clearance, we will need the adjusted camera-ready copy of the report plus two copies if the report is going to NTIS only or camera-ready plus one if the report will be printed. Adjust the project summary and supply us with **corrected hard copy and a 3 1/2 in. disk, with latest revisions in WordPerfect 5.1 format. Be sure to include all tables and graphics (with format identified) on the disk.**

If you have any questions, please call.

Robert M. Roetker (513) 569-7926

