March 2002
DTSC R-02-01
EPA 600/R-02/038

Environmental Technology Verification Report

Hydromatix 786E Ion Exchange Rinsewater Recycling System

Report Prepared by

California Environmental Protection Agency
Department of Toxic Substances Control

Under a cooperative agreement with
United States Environmental Protection Agency

Environmental Technology Verification Report

Hydromatix, Incorporated

Hydromatix 786E Ion Exchange Rinsewater Recycling System

By

California Environmental Protection Agency
Department of Toxic Substances Control
Office of Pollution Prevention and Technology Development
Sacramento, California

Notice

The verification report and verification statement found in this document were developed jointly by the United States Environmental Protection Agency (U.S. EPA) and the California Environmental Protection Agency (Cal/EPA), Department of Toxic Substances Control (DTSC). The verification study was funded in part by U.S. EPA under Cooperative Agreement number CR 824433-01-0 for the Pollution Prevention, Recycling, and Waste Treatment Systems (PPWTS) Pilot under the U.S. EPA Environmental Technology Verification (ETV) Program. The verification report and verification statement have been subjected to U.S. EPA's and Cal/EPA's peer and administrative review, and have been approved for publication.

The verification statement is limited to the use of the Hydromatix 786E Ion Exchange Rinsewater Recycling System for reducing the amount of regenerant waste to 17.1 ± 0.2 gal/ft^3 of resin. U.S. EPA and Cal/EPA make no express or implied warranties as to the performance of the Hydromatix 786E Ion Exchange Rinsewater Recycling System. Nor does U.S. EPA or Cal/EPA warrant that the Hydromatix 786E Ion Exchange Rinsewater Recycling System is free from any defects in workmanship or materials caused by negligence, misuse, accident or other causes. Mention of corporation names, trade names, or commercial products does not constitute endorsement or recommendation for use of specific products.

Foreword

The ETV Program was established by U.S. EPA to evaluate the performance characteristics of innovative environmental technologies across all media, and to report this objective information to the permitters, buyers, and users of environmental technology. U.S. EPA's Office of Research and Development (ORD), through the National Risk Management Research Laboratory (NRMRL), then established ETV pilot programs to enhance the transfer of technologies. Cal/EPA's DTSC partnered with U.S. EPA's ETV Program in late 1995 to establish the Pollution Prevention, Recycling, and Waste Treatment Systems Pilot. The PPWTS Pilot incorporated elements of the State of California certification program, and initially focused on the EPA Common Sense Initiative industry sectors including printing, electronics, petroleum refining, metal finishing, auto manufacturing, and iron and steel manufacturing.

The verification report found in this document reviews the performance of the Hydromatix 786E Ion Exchange Rinsewater Recycling System. The 786E system is used in various Metal Products and Machinery (MP&M) industries to treat rinse wastewaters, and features special provisions to minimize the regenerant waste volume produced. The 786E system treats rinse wastewaters by removing the cations and anions resulting from electroplating, cleaning, and anodizing operations, and minimizes the regenerant wastes produced by reusing portions of the regenerant waste solutions. Regeneration of ion exchange resins consists of a series of acid and base rinses which result in restored resin functionality. The regeneration technology utilizes a process logic controller (PLC), sensors, and associated plumbing for regeneration of the resins and for collection and reuse of portions of the regenerant waste solution. This verification report quantified the rinse wastewater treatment by measuring the volumes and concentrations entering and leaving the 786E system, and characterized the regeneration procedures by measuring the regenerant waste volume produced and by determining the regeneration efficiency.

Acknowledgments

Mr. Dick Jones, DTSC's Project Manager, and Mr. Edward Benelli, DTSC's Project Advisor, wish to acknowledge the support of those who planned and implemented the verification activities, and who prepared and reviewed this report. Ms. Norma Lewis of U.S. EPA's NRMRL in Cincinnati, Ohio was the Project Manager, while Ms. Lauren Drees acted as Quality Assurance Manager.

Aero-Electric Connector, Incorporated (AEC) of Torrance, California generously allowed their facilities and equipment to be used for the verification activities. The verification of the Hydromatix system could not have been conducted without an industry partner willing to host the testing activities, which included equipment installation, system monitoring, and sample collection. Both AEC representatives and those of Hydromatix contributed to this verification. AEC representatives included Messrs. Volker von Detten, Edgar Taracena, and Luis Castro. Hydromatix representatives included Messrs. Greg White, Amin Haq, and Jeremy Neel. Mr. Chubb Michaud of Systematix Chemical Engineers also contributed to the success of this project by serving as the technical representative for Purolite resins.

DTSC's Project Manager and Project Advisor also acknowledge the efforts by DTSC's Technical Review Panel and Project Team members. The Technical Review Panel included Messrs. Tony Luan and John Wesnousky. Project Team members included Dr. Bruce LaBelle and Mr. Clay Booher of the Office of Pollution Prevention and Technology Development (OPPTD), and Drs. Russ Chin and Ruth Chang of DTSC's Hazardous Materials Laboratory (HML).

ENVIRONMENTAL TECHNOLOGY VERIFICATION STATEMENT

TECHNOLOGY TYPE:	**ION EXCHANGE RINSEWATER RECYCLING**
APPLICATION:	**TREATMENT OF METAL FINISHING RINSEWATERS FOR THE REMOVAL OF CATIONS AND ANIONS**
TECHNOLOGY NAME:	**HYDROMATIX 786E ION EXCHANGE SYSTEM**
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The Environmental Technology Verification (ETV) Program was created by the United States Environmental Protection Agency (U.S. EPA) to facilitate the deployment of innovative or improved environmental technologies through performance verification and information dissemination. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of innovative, improved, and more cost-effective technologies. The ETV Program is intended to assist and inform those individuals in need of credible data for the design, distribution, permitting, and purchase of environmental technologies.

The ETV Program works to document the performance of commercial ready environmental technologies through a partnership with recognized testing organizations. Together, with the full participation of the technology developer, the ETV Program partnerships develop plans, conduct tests, collect and analyze data, and report findings through performance verifications. Verifications are conducted according to an established workplan with protocols for quality assurance. Where existing data are used, the data must have been collected by independent sources using similar quality assurance protocols.

EPA's ETV Program, through the National Risk Management Research Laboratory (NRMRL), has partnered with the California Department of Toxic Substances Control (DTSC) under an ETV Pilot to verify pollution prevention, recycling, and waste treatment technologies. This verification statement provides a summary of performance results for the Hydromatix 786E Ion Exchange System.

TECHNOLOGY DESCRIPTION

Hydromatix Corporation (Santa Fe Springs, California) developed its 786E system to remove cations and anions from rinse wastewaters generated during metal finishing operations such as electroplating, cleaning, and anodizing. Regeneration of ion exchange resins consists of a series of acid and base rinses which result in restored resin functionality, while minimizing the volume of regenerant waste produced.

Hydromatix developed an ion exchange regeneration process for their Model 786E system which uses a programmable logic controller (PLC) system to coordinate acid and base rinse water reuse. This reduces the volume of regenerant chemicals wasted, and consequently the volume of regenerant wastewater produced. The Hydromatix system features packed bed, counter-current ion exchange columns with conductivity meters, PLC, and automatic valves to control the regeneration process. The cationic and anionic ion exchange columns are packed with Purolite PFC-100 H and Purolite PFA-300 OH resins (Purolite USA, Bala Cynwyd, Pennsylvania), respectively. By reusing portions of the regenerant rinses as make-up solutions for the next cycle, and by returning other rinses to the feed tank rather than to waste, the system is able to achieve a substantial reduction in the amount of chemicals used as well as in the amount of wastewater produced during each regeneration cycle.

Precipitation and clarification methods are traditionally used for conventional ion exchange regenerant waste treatment because they are able to process large volumes. These methods generally produce wastewaters which meet local Publicly Owned Treatment Works (POTW) or National Pollutant Discharge Elimination System (NPDES) requirements. The large volume of regenerant wastewater requiring precipitation and clarification treatment often precludes the use of evaporation as a disposal method, which could result in zero wastewater discharge from the facility.

EVALUATION DESCRIPTION

The central claim made by Hydromatix is that their technology reduces the volume of regenerant waste produced. The ratio of gallons of waste produced per cubic foot of resin regenerated, the specific volume, is smaller than in conventional ion exchange systems. This smaller specific volume allows more waste management options and assists metal plating facilities in achieving zero wastewater discharge. Thus, the primary objectives of the evaluation were to determine (1) the specific volume of regenerant waste produced, and (2) the cation and anion exchange capacities restored during regeneration. Secondary objectives include providing information for potential end-users and metal reclaimers, and observing the system during normal operating conditions in order to evaluate worker health and safety. Only the Hydromatix system was evaluated to achieve the primary and secondary

objectives of this study; no other competing ion exchange technologies were investigated. The manufacturer and users provided basic cost data. Users also provided information on system performance, reliability, and waste generation. The evaluation verified, through independent testing, the following performance parameters:

1. Regenerant waste volume produced
2. Cation and anion exchange capacities restored
3. Rinse wastewater volume treated
4. Masses of acid and base volume consumed
5. Masses of metal species in the regenerant waste

Five test runs lasting approximately one week each were conducted over a three month period at Aero-Electric Connectors, Incorporated (AEC) in Torrance, California. Details of the evaluation, including data summaries and discussion of results may be found in the report entitled U.S. EPA ETV Report, Hydromatix 786E Ion Exchange Rinsewater Recycling System.

VERIFICATION OF PERFORMANCE

Performance results of Hydromatix Corporation's 786E Ion Exchange Rinsewater Recycling System, are summarized as follows (all data calculated at the 90 percent confidence level):

- Regenerant waste specific volume: 17.1 ± 0.2 gallons of waste per cubic foot of resin (gal/ft^3). The cationic regenerant waste produced during four test runs averaged 302 gallons for 18 ft^3 of resin, yielding a specific volume of 16.8 ± 0.2 (gal/ft^3). The anionic regenerant waste produced during five test runs averaged 313 gallons for 18 ft^3 of resin, yielding a specific volume of 17.4 ± 0.1 gal/ft^3.

- Cation and anion exchange capacities restored: Cation and anion capacities restored were 94.5 ± 6.8 and 88.7 ± 1.7 percent over five test runs, respectively. Compared to new resin material, the remaining cationic resin capacity averaged 96.0 ± 2.1 percent, and the remaining anionic resin capacity averaged 79.9 ± 1.8 percent. For the cation resin, the resin utilization was found to be 46.6 ± 4.6 percent using three test runs, and the regenerant efficiency was 29.9 ± 28.8 percent using two test runs. For the anion resin, the resin utilization was found to be 57.2 ± 36.5 percent over two test runs, while the regenerant efficiency was 32.0 ± 3.7 percent using two test runs.

- Rinse wastewater volume treated: 75,565 ± 9,663 gallons average, measured over five test runs, containing typical cations and anions found in plating shop wastestreams.

- Masses of acid and base consumed: 144.3 pounds of HCl measured over two test runs, and 119.7 pounds of NaOH per regeneration cycle measured over five test runs. The regenerant solution volumes were 271 ± 11.6 gallons of acid, and 274.4 ± 6.5 gallons of base, each measured over five test runs. The volumes of concentrated acid and base in the regenerant solution volumes were 38.9 gallons of 37 percent HCl, and 18.7 gallons of 50 percent NaOH.

- The masses of metal species in the regenerant waste: The average masses and ranges of representative metal species were found to be: 113.8 ± 89.7 g with a range of 24.9 to 272.5 g for copper, 175.3 ± 70.5 g and 47.5 to 227.9 g for nickel, and 580.8 ± 411.5 g and 65.6 to 1,078.7 g for zinc. Metal species were determined using four test runs.

Original signed by E. Timothy Oppelt, 4/2/02

E. Timothy Oppelt, Director Date
National Risk Management Research Laboratory
Office of Research and Development
United States Environmental
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Original signed by Kim Wilhelm, 3/15/02

Kim Wilhelm, Acting Chief Date
Office of Pollution Prevention
and Technology Development
Department of Toxic Substances Control
California Environmental Protection Agency

AVAILABILITY OF VERIFICATION STATEMENT AND REPORT

Copies of the public Verification Statement are available from the following:

(NOTE: Appendices are not included in the Verification Report.
Appendices are available from DTSC upon request.)

United States Environmental Protection Agency/NSCEP
P.O. Box 42419
Cincinnati, Ohio 45242-2419
Web site: http://www.epa.gov/etv/library.htm *(electronic copy)*

Department of Toxic Substances Control
Office of Pollution Prevention and Technology Development
P.O. Box 806
Sacramento, California 95812-0806
Web site: http://www.dtsc.ca.gov/ScienceTechnology/etvpilot.html
or http://www.epa.gov/etv *(click on partners)*

NOTICE: U.S. EPA and California Environmental Protection Agency (Cal/EPA) make no expressed or implied warranties as to the performance of the technology described in this verification. Verifications are based on an evaluation of technology performance under specific, predetermined criteria using appropriate quality assurance procedures. The end-user is solely responsible for complying with any and all applicable federal, state, and local requirements

Photo 1. Aero-Electric Connector, Inc. facilities in Torrance, California, showing installation of Hydromatix 786E Ion Exchange treatment system and associated equipment.

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Appendices A-H **included as separate volumes**

List of Abbreviations and Acronyms

AEC	Aero-Electric Connectors, Inc.
ATL	Advanced Technology Laboratories
Cal/EPA	California State Environmental Protection Agency
CCR	California Code of Regulations
CFR	Code of Federal Regulations
DI	Deionized
DTSC	California Department of Toxic Substances Control
EC	Electrical Conductivity
ETV	Environmental Technology Verification
GAC	Granular Activated Carbon
GF	George Fisher
gpm	Gallons per Minute
HCl	Hydrochloric Acid
H&SC	Health and Safety Code
HML	Hazardous Materials Laboratory
HAZWOPER	Hazardous Waste Operations and Emergency Response
lbs	Pounds
meq/ml	Milliequivalents per liter
mg/L	Milligrams per Liter
ml	Milliliter
MP&M	Metal Products and Machinery
MSDS	Material Safety Data Sheet
N/A	Not Available
NaOH	Sodium Hydroxide
ND	Non-Detectable
NPDES	National Pollutant Discharge Elimination System
NRMRL	National Risk Management Research Laboratory
OPPTD	Office of Pollution Prevention and Technology Development
ORD	EPA's Office of Research and Development
OSHA	Occupational Safety and Health Administration
pH	Negative Log of the Hydrogen Ion Concentration
PLC	Programmable Logic Controller
P or G	Plastic or Glass
PPWTS	Pollution Prevention, Recycling, and Waste Treatment Systems
POTW	Publicly Owned Treatment Works
ppm	Parts per Million
PVC	Polyvinyl Chloride
QA/QC	Quality Assurance/ Quality Control
U.S. EPA	United States Environmental Protection Agency
uv	Ultraviolet
µS/cm	MicroSiemens per Centimeter
WIRS	Waste Identification and Recycling Section
w/v	Weight per Volume

1.0 Introduction

The Environmental Technology Verification (ETV) Program was created by the United States Environmental Protection Agency (U.S. EPA) to facilitate deployment of innovative technologies through performance verification and report publication. The goal of the ETV Program is to enhance environmental protection by substantially accelerating the acceptance and use of innovative, improved, and cost-effective technologies. The ETV Program is intended to assist and inform those individuals in need of credible data for the design, distribution, permitting, and purchase of commercially-ready environmental technologies.

U.S. EPA's ETV Program, through the National Risk Management Research Laboratory (NRMRL), has partnered with the California Department of Toxic Substances Control (DTSC) under an ETV Pilot to verify pollution prevention, recycling, and waste treatment technologies. The Pilot focuses on the hazardous waste management technologies used in the EPA Common Sense Initiative industry sectors: printing, electronics, petroleum refining, metal finishing, auto manufacturing, and iron and steel manufacturing.

The ETV Pollution Prevention, Recycling, and Waste Treatment Systems Pilot gives developers the opportunity to have their technology evaluated under realistic laboratory or field conditions. The ETV Pilot selects market-ready environmental technologies from both the private and public sectors. The evaluation provides information necessary for the ETV Program verification. By completing the verification and distributing the results, U.S. EPA establishes a baseline for acceptance and use of these technologies.

This ETV report documents the evaluation of the Hydromatix 786E Ion Exchange Rinsewater Recycling System developed by the Hydromatix Corporation (Santa Fe Springs, California). This system removes cations and anions from rinse wastewaters generated during metal finishing operations such as electroplating, cleaning, and anodizing. The Hydromatix 786E system employs recycling to reduce the amount of waste produced during regeneration of the ion exchange resins. DTSC evaluated the Hydromatix 786E system in Spring 2001 at Aero-Electric Connectors, Incorporated (AEC) in Torrance, California.

This ETV report describes the evaluation approach taken by DTSC and the quality control criteria required for the field sampling and testing activities. The report also includes sampling and testing results, and calculations for feed rinsewater, regenerant waste, resin capacities, and product deionized (DI) water quality. Original field notes, raw analytical data, calculation sheets, and reference manuals are included in Appendices A - H. Appendices are included as separate volumes, and are available from DTSC upon request. The report includes discussions of the chemicals used in regeneration, and the characteristics of the regenerant waste. Worker health and safety is reviewed, and results of end-user surveys are reported. An overview of waste management regulations is provided, followed by a summary of DTSC's verification activities and sampling results. Lastly, Hydromatix's vendor comments are presented.

1.1 Background

Metal Products and Machinery (MP&M) industries generate waste streams containing metals and their salts through plating operations such as electroplating, cleaning, etching, anodizing, and stripping. A thin film of chemical solution is retained on any workpiece as it is removed from a plating bath. This chemical film is then washed from the workpiece in a subsequent clean water rinse. Metals and salts found in the chemical film are thus transferred to the clean water, producing a rinse wastewater. The concentrations of metals and salts found in the rinse wastewater are a function of their concentrations in the plating baths, the production rate through the plating baths, and the amount of plating solution transferred to the rinsewaters with each workpiece.

The rinse wastewaters contain a diluted mixture of all the upstream plating bath chemistries, which are a mixture of raw plating chemicals and byproducts from chemical reactions in the plating baths. Waste streams originating from MP&M industries often are characterized as a hazardous waste requiring treatment prior to disposal.

One method of treating rinse wastewaters utilizes ion exchange resins to remove both metals and salts, yielding a DI product water which can be reused in rinsing operations. The resin materials are contained in separate cationic and anionic ion exchange columns. Rinse wastewaters are first passed through the cation exchange column, where metals are exchanged for hydrogen ions. The rinse wastewater is then passed through the anion exchange column where anions are exchanged for hydroxide ions, producing DI water at a neutral pH. Ultimately, the ion exchange capacity of the resin is exhausted, and the material must be regenerated by removing the accumulated metals and anions and replacing them with hydrogen and hydroxide ions. The regeneration process results in the production of a regenerant wastewater, which comprises a smaller, more concentrated volume than the original rinse wastewater treated.

The Hydromatix 786E Ion Exchange System uses two pairs of cation and anion resin columns to enable continuous operation; one resin pair operates while the other is being regenerated or is in standby. The cation resin column is regenerated by a strong acid, hydrochloric acid, and the anion column is regenerated by a strong alkali, sodium hydroxide. The Hydromatix system reduces the regenerant wastewater volume produced by recycling portions of the water rinses used in regeneration. Raw chemical usage is also minimized by reusing portions of the acid and base regenerant solutions. The 786E system uses a programmable logic controller (PLC) to manage the treatment and regeneration processes.

Ion exchange replaces precipitation and clarification for the treatment of electroplating rinsewaters. While the precipitation and clarification process is able to treat large volumes efficiently, it may not meet increasingly stringent discharge requirements such as the U.S. EPA's pending MP&M Rules. Both treatment methods produce a residual sludge which is typically a hazardous waste and must be further treated before disposal.

The central claim made by Hydromatix is that their technology reduces the volume of regenerant waste produced. The ratio of gallons of waste produced per cubic foot of resin regenerated, the specific volume, is smaller than in conventional ion exchange systems. This smaller specific volume allows more

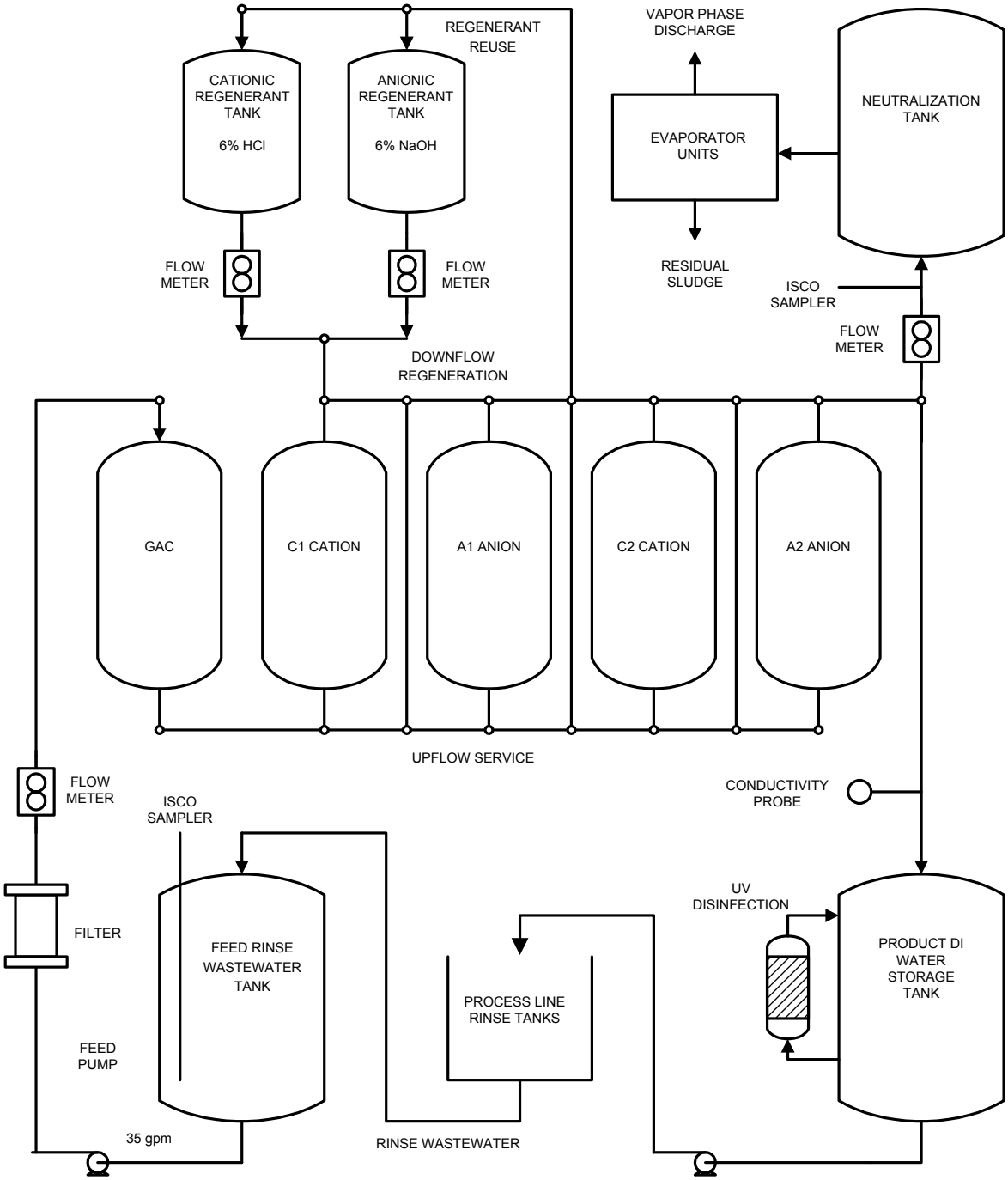
waste management options and assists metal plating facilities in reducing their wastewater discharge. Thus, the primary objectives of the evaluation were to determine (1) the specific volume of regenerant waste produced, and (2) the cation and anion exchange capacities restored during regeneration. Secondary objectives include providing information for potential end-users and metal reclaimers, and observing the system during normal operating conditions in order to evaluate worker health and safety. Only the Hydromatix system was evaluated to achieve the primary and secondary objectives of this study; no other competing ion exchange technologies were investigated, and only basic cost data was compiled. The evaluation verified, through independent testing, the following performance parameters:

1. Regenerant waste volume produced
2. Cation and anion exchange capacities restored
3. Rinse wastewater volume treated
4. Masses of acid and base volume consumed
5. Masses of metal species in the regenerant waste

Photo 3. Ion exchange columns - 6' tall.

2.0 Technology Description

Figure 1. Hydromatix 786E Ion Exchange System



2.1 Treatment

Rinse wastewaters containing metals and their salts are generated from plating operations at Aero-Electric. AEC keeps contaminants such as oils and grease, and oxidizers out of the rinse wastewater to protect and extend the life of the ion exchange unit, but as a precaution the rinse wastewater is routed through a carbon filtration unit to remove any organic compounds that may be present. The next step in the Hydromatix 786E operation is removal of cations from the feed rinse wastewater by passing the waste stream through a column of cation exchange resin.

The 786E system uses columns with an empty volume of 24.7 cubic feet. This volume holds both inert material and active ion exchange resin. In each column there are 2.6 cubic feet of sand at the bottom, 2.4 cubic feet of inert polymer at the top, and 0.6 cubic feet of support and distribution systems. With room for some expansion, this leaves 18 cubic feet of active ion exchange resin in both the cationic and anionic columns.

The 786E system uses Purolite (PuroliteUSA, Bala Cynwyd, Pennsylvania) PFC-100 H strong acid cationic exchange resin, which features a sulfonic acid functional group. The resin is composed of spherical, 560 micron diameter polystyrene beads with a total exchange capacity of 1.9 eq/L (wet form, volumetric) when new. Hydrogen ions in the cationic resin are displaced by metals in the incoming rinse wastewater. Next, the waste stream exits the cationic resin column and flows through the anion resin column. The system uses Purolite PFA-300 OH strong base type II anionic exchange resin, which features a quaternary ammonium functional group. The anion resin is similarly composed of 560 micron polystyrene beads, with a total exchange capacity of 1.4 eq/L (wet form, volumetric) when new. Hydroxide ions in the anionic resin are then displaced by anions in the waste stream. The resultant DI water flows to the product DI water tank, where it can be reused in plating shop operations. At the end of the process, ultraviolet light is applied to recirculating water to control the growth of bacteria in the lines or tanks.

DI water production continues until the resin limiting capacity is exceeded; a conductivity sensor detects the ionic contamination resulting from resin exhaustion. Approximately 75,000 gallons of rinse wastewater are treated per run, with flowrates ranging from 35-45 gallons per minute (gpm), by the 786E system at AEC. The volume of rinse wastewater treated per run is dependant on the concentration of metals and their salts in the waste stream, with higher concentrations resulting in earlier exhaustion of the limited capacity of the resin material.

2.2 Regeneration

The regeneration cycles for the 786E system cation column are completed first, followed by regeneration for the anion column. Hydromatix uses upflow service, and downflow regeneration, in a counter-current flow system. In counter-current flow systems, high quality product DI water is obtained because the treated water passes through the most highly regenerated portion of the resin bed immediately before it exits the column. The 786E System utilizes George Fisher (GF, Tustin, California) UniDirectional Motorized Ball Valves to perform the rinse wastewater treatment and resin column regeneration control functions. While the GF valve operation is controlled by the PLC, the actual position of the valve can be verified by either inspecting the top of the valve, or by viewing the control panel.

The regeneration cycle consists of the following sequences:

1) *Tank capacity checks:* Prior to beginning regeneration, the operator must ensure that sufficient regenerant waste tank capacity and product DI water volume are available. The regenerant waste tank will be receiving approximately 600 gallons of waste solutions, and the consumption of product DI water will be about 75 gallons. The solution level in cationic and anionic regenerant tanks is maintained at the high level by Flowline (Los Alamitos, California) Smart Trak float switches located inside the tanks. The chemical composition of the cationic and anionic regenerant solutions is maintained at six percent by Solu Comp (Irvine, California) SCL-470 conductivity cells and SCL-C-200 Analyzers. High and low level conditions, as well as the solution concentrations are displayed on the control panel. Subsequent steps in the regeneration cycle will require that the volume of 30 percent hydrochloric acid and 50 percent sodium hydroxide in the holding drums be sufficient for replenishment of the regenerant tanks.

2) *Displacement of DI water from the cation bed and into the feed tank:* Initiated by pushing the regeneration button, several valves open and close which isolate the treatment train to be regenerated from the standby train, allowing both regeneration and treatment to continue simultaneously. When the acid feed pump starts, acidic solution begins to displace the waste rinsewater solution remaining in the cation bed pore space. The displaced waste rinsewater is directed to the feed tank rather than to waste, which minimizes the ultimate waste volume to be treated later. Approximately one-third of the bed pore space volume is directed back to the feed rather than to waste. This step continues until a mid-level float switch in the cationic regenerant tank is triggered.

3) *Removal of metals from the cation bed by means of the fresh acid and depositing the spent acid into the neutralization tank:* Once the waste rinsewater is displaced from the resin bed the process of removing the retained metals can begin. Fresh acid is passed through the cation bed at a flow rate of 810 gpm. As the acid comes into contact with the resin beads, it exchanges metals from sites within the resin with hydrogen ions supplied by the acid. One or more hydrogen ions are supplied for every mono or polyvalent cation. As the acid solution works its way down through the column it gradually becomes concentrated with metals and is ultimately directed through the waste neutralization line into the regenerant waste tank. The application of acid to the cationic column continues until the low level float sensor in the cationic regenerant tank is triggered, after about 300 gallons of acid solution have been passed through the resin bed.

4) *Slow rinse with DI water through the cation column depositing weak acid into the cationic regenerant tank:* At this stage, nearly all of the easily removable metal ions retained in the cationic column have been replaced by hydrogen ions, and most of the bed pore volume is filled with a metal-free acid solution. By flushing this acid out of the column with DI water, Hydromatix is able to create a weak acid solution which is then directed to the now-empty cationic regenerant tank for re-use, rather than to waste. This weak acid will be stored in the cationic regenerant tank and used to prepare acid solution for the next cationic regeneration cycle.

5) *Recirculating rinse through the cation and anion for 20 minutes:* By recirculating DI water through both the cation and anion resin beds, traces of acid remaining in the cation resin bed are removed. Chloride ions present from the hydrochloric acid are removed in the anionic exchange

column; in the process the hydroxide ions released neutralize the excess hydrogen ions from the acid rinse. The resulting DI water is free of metal and chloride ions, and has a neutral pH.

6) *Acid mix cycle:* DI water is added to the cationic regenerant tank until the solution level is at the high set point. Concentrated hydrochloric acid is added until the conductivity probe indicates a six percent solution.

Photo 4. Acid, base and neutralization streams to and from the columns.

7) *Displacement of DI water from the anion bed into the feed tank:* Following the appropriate switching of valves, the caustic regenerant solution is pumped into the anion column, displacing DI water. This DI water is directed to the feed tank, again reducing the amount of waste which will ultimately result from the regeneration processes.

8) *Removal of salts from the anion bed by means of the fresh caustic and depositing the spent caustic into the waste neutralization tank:* Once the waste rinsewater is displaced from the resin bed the process of removing the retained anions can begin. Fresh caustic is passed through the anion bed at a flow rate of 8-10 gpm. As the caustic comes into contact with the resin beads, it exchanges anions from sites within the resin with hydroxide ions supplied by the caustic. As the caustic solution works its way down through the column it gradually becomes concentrated with anions and is ultimately directed through the waste neutralization line into the regenerant waste tank. The application of caustic to the anionic column continues until the low level float sensor in the anionic regenerant tank is triggered, after about 300 gallons of caustic solution have been passed through the resin bed. At the end of this cycle and subsequent valve changes, deionized water is drawn from the DI water storage tank and passed through the anion bed. The resulting rinse water from the anion bed will contain a mixture of salts in a diluted caustic stream. For the first five minutes of this rinse cycle the waste stream is discharged into the waste neutralization tank.

9) *Slow rinse with DI water through the anion column and depositing the weak caustic into the anionic regenerant tank:* DI water is passed through the anion column to remove the excess, unspent caustic from the resin bed, with the excess caustic creating a weak caustic solution. This weak caustic solution is reuseable and is directed to the anionic regenerant tank.

10) *Recirculating rinse through the cation and anion for at least 60 minutes or until desired conductivity is achieved:* To remove sodium ions remaining on the anion resin the solution is passed through the cationic beds, where the sodium is removed and the consequent hydrogen ion produced is used to neutralize the hydroxide ion present. This results in the production of a DI solution with a neutral pH.

11) *Caustic Mix Cycle:* DI water is added to the anionic regenerant tank until the solution level is at the high set point. Concentrated sodium hydroxide is added until the conductivity probe indicates a six percent solution.

The flow directions are controlled primarily by air-actuated diaphragm valves. The PLC sends signals to the solenoids that control the valves. High and low level switches in each of the chemical reuse tanks trigger the switch-over from one cycle to the next.

3.0 Evaluation Approach

The Hydromatix 786E system evaluation required measurements of treatment volumes, generated wastes, a calculation of mass balance, and a determination of the regenerated resin capacity. Hydromatix 786E system documents and diagrams were reviewed to determine the placement of monitoring and sampling equipment. Flow diagrams for plating operations at AEC were studied to determine which waste streams entered the Hydromatix system.

These waste streams were characterized by studying the chemical make-ups for the baths that contributed to these waste flows. Material Safety Data Sheets (MSDSs) were obtained for each plating bath which contributed to the waste stream entering the Hydromatix system. These reference documents showed which chemical species would be present in the rinse wastewater originating from these plating operations. DTSC's Hydromatix Technology Evaluation Workplan (Appendix A) focused on quantifying the primary objectives: regenerant waste volume produced, cation and anion exchange capacities restored, rinse wastewater volume treated, masses of acid and base volume consumed, masses of metal species in the regenerant waste, and product DI water quality.

After review and approval of the Workplan, specification and installation of monitoring and sampling equipment on the Hydromatix 786E system at AEC was implemented. The equipment allowed for monitoring of flows in the feed rinse wastewater, product DI water, regenerant solutions, and regenerant waste streams during actual production operations at AEC. Sampling equipment allowed for the collection of samples from the feed rinse wastewater, product DI water, and regenerant waste streams.

Arrangements were made to have independent chemical analysis of the samples collected during the five test runs. An ion exchange resin sampling method to determine the capacities used and restored was devised, and arrangements were made for resin analysis at the manufacturer's laboratory. Provisions for quality control and data evaluation were devised. Data compilation and evaluation methods were developed, and a peer review team was established. Guidelines for final report preparation were adopted.

Sample collection and detailed measurements of the flowrates were performed for the waste streams and product DI water. This allowed the verification of the waste volumes produced, and the calculation of resin capacity restored. Sampling of the feed rinse wastewater, product DI water, and regenerant wastes was also conducted. Anions and cations for these streams were determined through laboratory analyses. The cationic and anionic resins were sampled and analyzed to determine their capacities restored

by regeneration. These were then compared to those capacities available after the resin's approximately five years of life. The field data collected from each test run is listed in Table 1, Field Monitoring, Sampling, and Analytical Methods.

Table 1. Field Monitoring, Sampling, and Analytical Methods

Type	Parameter(s)	Frequency	Location	Method(s)	Containers (Storage Limits)
Rinse Wastewater	Volume	Each test run	Feed line from collection tank	Inline flow totalizer	Recorded on site
	Flow rate	Daily	Feed line from collection tank	Inline flow totalizer	Recorded on site
	Al, B, Cu, K, Na, Ni, Zn	Each test run	ISCO Automatic Sampler at collection tank	U.S. EPA Methods 3010A, 6010B	1000 ml P or G, HNO_3, pH<2 (6010B 6 months)
	NH_4^+	Each test run	ISCO Automatic Sampler at collection tank	U.S. EPA Method 350.2	1000 ml P or G, H_2SO_4, pH<2 (350.2 28 days)
	Cl^-, F^-, SO_4^{2-}, NO_3^-, PO_4^{3-} Total dissolved solids, Specific conductance, Alkalinity	Each test run	ISCO Automatic Sampler at collection tank	U.S. EPA Methods 300.0, 9050A, 310.1, 160.3	1000 ml P or G, 4 C, no preservative (300.0 48 hours) (310.1 7 days) (9050A 28 days)
	pH	Each test run	ISCO Automatic Sampler at collection tank	U.S. EPA Method 150.1	Measured on site at time of collection
Rinse Wastewater Duplicate	Al, B, Cu, K, Na, Ni, Zn	Each test run	ISCO Automatic Sampler at collection tank	U.S. EPA Methods 3010A, 6010B	1000 ml P or G, HNO_3, pH<2 (6010B 6 months)

Type	Parameter(s)	Frequency	Location	Method(s)	Containers (Storage Limits)
Product DI Water	Al, B, Cu, K, Na, Ni, Zn	Each test run	Grab sample from Product DI water pipe sample port	U.S. EPA Methods 3010A, 6010B	1000 ml P or G, HNO_3, pH<2 (6010B 6 months)
	NH_4^+	Each test run	Grab sample from Product DI water pipe sample port	U.S. EPA Method 350.2	1000 ml P or G, H_2SO_4, pH<2 (350.2 28 days)
	Cl^-, F^-, SO_4^{2-}, NO_3^-, PO_4^{3-} Total dissolved solids, Specific conductance, Alkalinity	Each test run	Grab sample from Product DI water pipe sample port	U.S. EPA Methods 300.0, 9050A, 310.1, 160.3	1000 ml P or G, no preservative (300.0 48 hours) (310.1 7 days) (9050A 28 days)
	pH	Each test run	Grab sample from Product DI water pipe sample port	U.S. EPA Methods 150.1	Measured on site at time of collection
	EC reading	Daily	Sensor is in the effluent pipe; display is on the panel	Rosemont Analytical Solu Comp Model SCL-C-002-M2	Recorded on site
Cationic Regenerant Waste	Volume	Each test run	Line from cationic column to neutralization tank	Inline flow totalizer	Recorded on site
	Al, B, Cu, K, Na, Ni, Zn	Each test run	ISCO Automatic Sampler installed at Regenerant waste collection line	U.S. EPA Methods 3010A, 6010B	1000 ml P or G, HNO_3, pH<2 (6010B 6 months)
	NH_4^+	Each test run	ISCO Automatic Sampler installed at Regenerant waste collection line	U.S. EPA Method 350.2	1000 ml P or G, H_2SO_4, pH<2 (350.2 28 days)
Anionic Regenerant Waste	Volume	Each test run	Line from anionic column to neutralization tank	Inline flow totalizer	Recorded on site
	Cl^-, F^-, SO_4^{2-}, NO_3^-, PO_4^{3-}	Each test run	ISCO Automatic Sampler installed at Regenerant waste collection line	U.S. EPA Method300.0	1000 ml P or G, no preservative (300.0 48 hours)
QA Travel Blank- Metals	Al, Cu, Ni, Zn	Each test run	Prepared at So Cal HML	U.S. EPA Method 6010B	1000 ml P or G, HNO_3, pH<2 (6010B 6 months)

Type	Parameter(s)	Frequency	Location	Method(s)	Containers (Storage Limits)
QA Travel Blank- Anions	Cl^-, SO_4^{2-}, PO_4^{3-}	Each test run	Prepared at So Cal HML	U.S. EPA Method 300.0	1000 ml P or G, (300.0 48 hours)
QA Spike-Metals	Al, Cu, Ni, Zn	Each test run	Prepared at So Cal HML	U.S. EPA Method 6010B	1000 ml P or G, HNO_3, pH<2 (6010B 6 months)
QA Spike-Anions	Cl^-, SO_4^{2-}, PO_4^{3-}	Each test run	Prepared at So Cal HML	U.S. EPA Method 300.0	1000 ml P or G, (300.0 48 hours)
Cationic Column Resin	Operating capacity remaining, regeneration efficiency	Each test run	Cationic column	Purolite Laboratory methods	1000ml P or G, sample must be kept moist
QA Cation Resin Travel Blank	Operating capacity, regeneration efficiency	One sample	Fresh Cationic resin from Purolite	Purolite Laboratory methods	1000 ml P or G, sample must be kept moist
Anionic Column Resin	Operating capacity remaining, regeneration efficiency	Each test run	Anionic column	Purolite Laboratory methods	1000 ml P or G, sample must be kept moist
QA Anion Resin Travel Blank	Operating capacity remaining, regeneration efficiency	One sample	Fresh Anionic resin from Purolite	Purolite Laboratory methods	1000 ml P or G, sample must be kept moist
Acidic Regenerant	Volume	Each test run	Line from acid tank to cation column	Inline flow meter	Recorded on site
	HCl	Each test run	Grab sample from Acid make-up tank	U.S. EPA Method 305.1	1000 ml P or G (305.1, as short as practically possible)
QA Acid Standard	HCl	One sample	Prepared at So Cal HML	U.S. EPA Method 305.1	1000 ml P or G (305.1, as short as practically possible)
Basic Regenerant	Volume	Each test run	Line from acid tank to cation column	Inline flow meter	Recorded on site
	NaOH	Each test run	Grab sample from Base make-up tank	U.S. EPA Method 310.1	1000 ml P or G (310.1, as short as practically possible)
QA Base Standard	NaOH	One sample	Prepared at So Cal HML	U.S. EPA Method 310.1	1000 ml P or G (310.1, as short as practically possible)

3.1 Sampling Procedure and Equipment

Several pieces of equipment were installed to sample the feed rinse wastewater, regenerant waste, and DI product water streams. Automatic flowmeters and samplers were installed to enable recovery of the flow data when samples were taken. An ISCO (Lincoln, Nebraska) Automatic Sampler Model 6800 was used to take 200 ml samples from the feed rinse wastewater at every 2000 gallons of flow (flow paced sampling). This resulted in one to two gallons of sample collected in a five gallon ISCO sample bottle during the three-day test runs. Flow volume was measured by a flow totalizer that operated when it detected flow. Once treatment was complete and the columns exhausted, the EC would climb and the system would stop. Samples were retrieved from the ISCO sample bottle. Regeneration was then started with acid solution rinsing out the cation column. Flows were measured and samples taken of acid and waste. The ISCO sampler was set to collect 200 ml regenerant waste samples every two minutes (time paced sampling). Corresponding measurements and samples were taken for the anion column. The following specific items of equipment were installed:

Photo 5. Signet flow totalizers and flow sensor (bottom).

Flow Sensors - Signet (El Monte, California) Model 515 Rotor-X Flow Sensors were installed at the following locations: the rinse wastewater feed line between cartridge filter and carbon filter, the line between the acid tank and cation column, the line between the caustic tank and anion column, and the regenerant waste line between the columns and the neutralization tank. Manufacturer's recommendations for equipment installation were followed to achieve the maximum accuracy for the instruments, including pipe run lengths and equipment orientation. Flow sensors and totalizers were calibrated both before and after the sampling events to ensure to collection of accurate flow data.

Flow Totalizers - Signet Flow Totalizers Model 8550 were installed to record readings from each flow sensor.

ISCO Automatic Sampler - The ISCO Automatic Sampler Model 6800 was installed and set to sample periodically at the following locations: at the rinse wastewater feed tank, and the regenerant waste line between the columns and the neutralization tank. The feed tank was sampled directly through the manhole port in the top of the tank. The following equipment was installed on the regenerant waste line: Pressure Reduction Valve (ISCO Model SPA 1081), a Three-way Valve (ISCO Model SPA 1082), and a Relay Contact (ISCO Model SPA 665). Flow paced and time paced sampling regimes were programmed into the sampler to obtain appropriate sample volumes. To prevent sampling during hours

when there was no production the sampling unit was triggered externally using a flow signal generated by the flow transmitter. Once programmed, the ISCO sampler was calibrated with a graduated cylinder by DTSC personnel.

Photo 6. Automatic ISCO Sampler, top view with tube entering pump and keypad for programming the sampling outline.

Resin Sampling Probe - An approximately six foot long one-half inch diameter polyvinyl chloride (PVC) pipe with a removable cap was used for sampling the resin. The PVC pipe was inserted through the top of the columns down through the full 5.5 feet of bed. The top cap was removed during sampling to allow the semi-viscous resin to flow up into the pipe; it was then replaced for withdrawal of the resin sample.

Electrical Conductivity (EC) Meter - The existing Rosemont (Irvine, California) Analytical Model SCL-C-002-M2 electrical conductivity sensor/meter was used to measure the electrical conductivity of the treated rinse water. Rosemont Analytical reports the accuracy and precision at 0.1 microSiemens per centimeter (µS/cm). The existing sensor/meter readings were checked against laboratory analysis, and the results were not used in any calculations.

pH Meter - A Orion (Beverly, Massachusetts) Model 330 pH Meter was used to measure samples directly onsite at the time of sample collection. The unit adhered to the requirements of U.S. EPA Method 150.1, including a provision for two-point calibrations. The unit was calibrated before each set of readings were collected.

3.2 Verification Activities

The Technology Evaluation Workplan specified field testing, sampling and data acquisition from five runs using the Hydromatix 786E system at AEC. Field testing was conducted at AEC from February 28, 2001 through May 3, 2001, as shown in the Chronology of Major Events (Appendix B). Each run included collecting samples from the feed rinse wastewater, product DI water, regenerant solutions, and regenerant waste lines. A detailed list of the test run sampling is included in Table 1.

As shown in Table 1, metals and anion samples were taken from the feed rinse wastewater and product DI water streams. Cations and anions were also taken from the corresponding regenerant waste streams. Flowrates were measured for all streams. Acid, base, and resin samples were collected during each run. Five separate test runs using the same cation and anion resin columns were conducted; each test run consisted of a complete treatment cycle including column exhaustion and regeneration. Installed

in October 1997, AEC has one of the oldest 786E systems in operation. AEC reported that the system had been regenerated more than 200 times with no detectable degradation in resin performance. The system contained 18 cubic feet each of Purolite PFC-100 H cationic and Purolite PFA-300 OH anionic resins. Rinse wastewater flow to the system was typically 35-45 gpm.

The ion samples were produced in each sampling run by the following series of steps. Each sampling run began with the start of the operating train's treatment phase. The ISCO automatic sampler was programmed to turn on with the start of flow through the feed rinse wastewater line, when that flow was directed through the operating train. The signal controlling the ISCO sampler came from the flow meter in the feed line and the GF valves that controlled the flow direction of the waste stream. A flow totalizer which had been previously zeroed began recording as the solution began flowing. The ISCO sampler purged its line and then took its first sample of 200 ml after a volume of 2000 gallons of feed rinse wastewater had passed the flow sensor. The collected sample was directed into a five gallon glass holding bottle within the sampling unit. The ISCO collected 200 ml of sample after each 2000 gallon volume had passed the sensor, or approximately every 45-60 minutes for the duration of the three day treatment run. Conclusion of the treatment run was signaled with the rise in the displayed EC. Bed saturation was indicated by a display EC of approximately ten µS/cm, which would then be followed by a fast rise in the EC. The operating train was set to shut off by the AEC operators at display ECs of about 65-75 µS/cm. As the display EC rose rapidly past that point, the flow to the operating train was redirected to the alternate set of columns, and the ISCO sampler was stopped automatically. This final EC set point differed from that stated in the Workplan: "at an EC set point of about 20 µS/cm...the feed flow is redirected to a fresh set of columns." In the analysis of the product DI water, the quantified EC results were closer to this expected value of approximately 20 µS/cm. This simply shows that a discrepancy exists between the conductivity meter display and specific conductance measured by the analytical laboratory.

4.0 Data Quality Assurance/Quality Control

A high level of quality assurance and quality control (QA/QC) was required throughout the verification. This was achieved by following QA/QC requirements established in the Technology Evaluation Workplan. The QA/QC requirements ensured that data generated through field test methods and analytical laboratory tests was of sufficient quality to be used in the evaluation of Hydromatix's claims.

The Workplan established that analytical laboratory data would be disregarded if sample contamination were identified, if QA/QC procedures were not followed, or if the results of percent recoveries were not within the established acceptable ranges. A charge balance would be performed to determine if ionic species were undetected in the collected samples. For samples sent to Advanced Technology Laboratories (ATL, Signal Hill, California), QA/QC was to be performed in accordance with internal laboratory procedures. This included protocols for sample preservation and sample holding times, as well as the analysis of spikes, matrix spikes, and blanks. ATL's data integrity was validated by the laboratory chemist, analyst, and supervisor, and was documented in the laboratory data packages. For samples sent to ATL and to Purolite Resin Laboratory, DTSC established a comprehensive set of external QA/QC requirements focusing on control of analytical accuracy and precision, and identification of sample contamination.

For each of five test runs, duplicate, travel blank, spike, and acid and base standards were included for analyses and were sent to ATL as blind control samples. The duplicates consisted of splits from the rinse wastewater samples. DTSC established acceptance criteria of relative percent differences of no greater than 20 percent for the duplicates. The travel blanks consisted of DI water supplied by DTSC's Hazardous Materials Laboratory (HML) in Los Angeles, California. The acceptance criteria for the travel blanks was no appreciable ionic contamination; no concentration values were specified for ionic contamination of the blanks. The spikes were prepared by HML, and consisted of a known synthetic mixture of cations and anions expected to be present in the waste samples. The spikes were prepared at the 25 mg/L level, and acceptance criteria of 80 to 120 percent recovery for metals, and 85 to 115 percent recovery for anions, was established. For the phosphate spikes, laboratory results are presented in terms of phosphorous, so that a conversion to phosphate ion is necessary in order to determine the percent recovery.

The acid and base standards were prepared by HML, and consisted of hydrochloric acid at the six percent weight to volume (w/v) level, and sodium hydroxide at the six percent (w/v) level, with an acceptance criteria for percent recovery of 80 to 120 percent established for each. For each of five test runs, resin travel blanks were sent to Purolite Laboratories as blind control samples. Quality control cation and anion resin samples consisting of fresh unused materials were submitted as the resin travel blanks, along with the actual resin samples collected at the conclusion of each test run. No spike was prepared for the resin samples. QA/QC for the resin samples sent to Purolite's analytical laboratory was performed in accordance with their internal company procedures; the acceptable range for percent recovery was 80 to 120 percent for both cationic and anionic resins. Samples analyzed with associated spike recoveries outside of this range were not used in subsequent calculations of mass balance, acid or base consumption, or resin capacity restored. Fresh resin samples were similarly deemed unacceptable and the associated data rendered unuseable if the recoveries were not within the established range.

An examination of charge balance for the feed rinse wastewater and product DI water samples was performed to indicate if ions were undetected in the analysis. In the examination of charge balance, the concentration of each ionic species is expressed in units of milliequivalents (meq) per liter, and the sum of the cations is compared to that of the anions. An acceptable range for the difference between cation and anion sums is published in Standard Methods for the Examination of Water and Wastewater. This range encompasses one standard deviation, and has been empirically established as:

$$\Sigma \text{ anions} - \Sigma \text{ cations} = \pm (0.1065 + 0.0155\, \Sigma \text{ anions})$$

Values falling outside the limits set by this equation indicate that at least one of the determinations should be rechecked, or that one or more ionic species present in the sample was not detected in the analysis. Complete chemical analyses was performed on only the feed rinse wastewater and product DI water samples.

The examination of charge balance for these samples revealed one sample of feed rinse wastewater, from test run three, met the criteria, while the other four samples had differences exceeding the established range. For the product DI water samples, test runs one and three yielded values within the range, while the other runs did not. These differences can be explained by considering the additional quality control checks performed on the samples. For example, the product DI water sample from test run one had acceptable results for cationic and anionic spike percent recoveries, duplicate sample relative percent differences, and

for the associated blank QA/QC samples. Consequently, the charge balance difference also fell within the acceptable range. By comparison, the product DI water sample from test run two had an unacceptable relative percent difference from the duplicate sample, and had an unacceptable result from the analysis of the associated blank QA/QC sample. Thus, the true value from the duplicate analysis is not known, and the reported sample value associated with the blank analysis is probably inflated. Predictably, the charge balance difference for this analysis fell outside of the acceptable range.

An acceptable level of accuracy for the flow sensors was verified through calibration and re-calibration procedures. The units were certified by the manufacturer at ten flowrates, both prior to installation and after removal from the system. The acceptable range for both the new and used units was defined as no flowrate measurement exceeding one percent deviation. Flow data from the cationic regenerant waste measurement for the second run was lost, probably due to debris in the paddle wheel mechanism. After sensor removal, cleaning, and re-installation, all subsequent data was useable. All units passed the recalibration procedures conducted at the manufacturer's testing facility after the sensors were removed from the system following completion of the test runs.

Data review and validation was conducted by members of the project team to ensure that the procedures and activities conformed to the requirements outlined in the Workplan. Dr. Ruth Chang and Mr. Ed Benelli verified the procedures and data generated by ATL and Purolite Resin Lab. Dr. Bruce La Belle, Mr. Ed Benelli and others from the project team provided qualitative review of survey results to ensure that the data could support the project evaluation. Additionally, the Neptune Company (Los Alamos, New Mexico) was retained by U.S. EPA to review field testing procedures. A physical inspection of the Hydromatix system, and of DTSC's sampling equipment installation and procedures, was conducted at AEC on April 20, and 21, 2001. The U.S. EPA contractor's Field Quality Control Audit is included as Appendix C.

Tables 2 and 3 index DTSC's QA review of ATL analyses for the Hydromatix ETV Project. Table 2 lists Standard, Spike, Duplicate, and Travel Blank QA samples which failed DTSC's acceptance criteria. These QA samples were submitted to ATL as blind controls along with field samples collected from the Hydromatix system at AEC. The listed QA samples failed one or more of three acceptance criteria: standards and spike samples had to exhibit recoveries of between 80 and 120 percent for acids and cations, or between 85 and 115 percent for anions; duplicate samples could not exhibit relative deviations of greater than 20 percent; and travel blanks could not exhibit significant analyte concentrations. Of the five acid standards submitted to ATL, the results of three analyses were rejected due to recoveries of 28, 257, and 196 percent. Two sets of duplicate results were rejected due to relative differences of 41 and 193 percent. Quality assurance samples which failed DTSC's acceptance criteria are denoted in Table 2 with a footnote (1).
Table 3 lists the associated field samples for which the analysis was dependant on acceptable QA sample results. Using the acid standards as an example, for each of the three QA samples that failed, the analytical results from one field sample collected at AEC had to be rejected. In some cases as many as four field sample results were rejected as a consequence of a QA sample failure. Field samples for which the analysis was dependant on acceptable QA sample results, and for which the QA samples that failed are denoted in Table 3 with a footnote (2). The results from these data analyses were not used in subsequent calculations and are presented here only to show the range and variability of data collected in field and through laboratory analysis.

In summary, eleven QA samples failed DTSC's acceptance criteria which resulted in the rejection of 23 field sample results. For the Hydromatix ETV Project overall, 64 species were analyzed for each of the five test runs for a total of 320 analyses. Because of the original provision for excess sample collection, data of sufficient quality and quantity remained to complete the Hydromatix ETV Project evaluation.

Table 2. Quality Assurance Samples Which Failed Acceptance Criteria. Standard, spike, duplicate, and travel blank QA samples were prepared by DTSC's HML Laboratory and were submitted as blind controls to ATL Laboratory along with the field samples collected at AEC.

Test Run	Sample Type	Analyte	Notes
1	QA Acid Standard	Acidity, Total	Percent recovery of 28 percent renders Acid Standard unacceptable.
2	Rinse Wastewater for Metals	Potassium	Relative Percent Difference of 41 percent renders Metals Duplicate unacceptable.
2	Rinse Wastewater Duplicate for Metals	Potassium	
2	QA Travel Blank for Anions	Chloride	Appreciable analyte concentration renders Travel Blank unacceptable.
2	QA Acid Standard	Acidity, Total	Percent recovery of 257 percent renders Acid Standard unacceptable.
3	Rinse Wastewater for Metals	Sodium	Relative Percent Difference of 193 percent renders Metals Duplicate unacceptable.
3	Rinse Wastewater Duplicate for Metals	Sodium	
3	QA Travel Blank for Anions	Chloride	Appreciable analyte concentration renders Travel Blank unacceptable.
3	QA Acid Standard	Acidity, Total	Percent recovery of 196 percent renders Acid Standard unacceptable.
5	QA Travel Blank for Anions	Chloride	Appreciable analyte concentration renders Travel Blank unacceptable.
5	QA Travel Blank for Anions	Sulfate	Appreciable analyte concentration renders Travel Blank unacceptable.

Table 3. Field Samples Which Failed Acceptance Criteria. The acceptance of the Field samples was predicated on successful analysis of the DTSC Quality Assurance samples submitted as blind controls to ATL Laboratories. Due to associated standard, spike, duplicate, and travel blank QA sample failures, the field sample results were unusable.

Test Run	Sample Type	Analysis	Notes
1	HCl Sample	Acidity, Total	QA Acid Standard failed acceptance criteria (Client ID 4215, ATL Lab ID 050406-010A). Percent recovery of 28 percent renders Field sample result unuseable.
2	Rinse Wastewater for Metals	Potassium	Relative Percent Difference of 41 percent renders Field sample results unuseable.
2	Rinse Wastewater Duplicates for Metals	Potassium	
2	Product DI Water for Metals	Potassium	Duplicates for Metals failed acceptance criteria (Client ID's 46011 and 46012, ATL Lab ID's 050485-001A and 050485-002A). Relative Percent Difference of 41 percent renders Field sample result unuseable.
2	Cationic Regenerant Waste for Metals	Potassium	Duplicates for Metals failed acceptance criteria (Client ID's 46011 and 46012, ATL Lab ID's 050485-001A and 050485-002A). Relative Percent Difference of 41 percent renders Field sample result unuseable.
2	Rinse Wastewater for Anions	Chloride	QA Travel Blank for Anions failed acceptance criteria (Client ID 46017, ATL Lab ID 050485-007A). Travel Blank showing appreciable analyte concentration renders Field sample result unuseable.
2	Product DI Water for Anions	Chloride	QA Travel Blank for Anions failed acceptance criteria (Client ID 46017, ATL Lab ID 050485-007A). Travel Blank showing appreciable analyte concentration renders Field sample result unuseable.
2	Anionic Regenerant Waste for Anions	Chloride	QA Travel Blank for Anions failed acceptance criteria (Client ID 46017, ATL Lab ID 050485-007A). Travel Blank showing appreciable analyte concentration renders Field sample result unuseable.
2	HCl Sample	Acidity, Total	QA Acid Standard failed acceptance criteria (Client ID 4090121, ATL Lab ID 050537-007A). Percent recovery of 257 percent renders Field sample result unuseable.
3	Rinse Wastewater for Metals	Sodium	Relative Percent Difference of 193 percent renders Field sample results unuseable.
3	Rinse Wastewater Duplicate for Metals	Sodium	
3	Product DI Water for Metals	Sodium	Duplicates for Metals failed acceptance criteria (Client ID's 416011 and 416012, ATL Lab ID's 050634-001A and 050634-002A). Relative Percent Difference of 193 percent renders Field sample result unuseable.

Test Run	Sample Type	Analysis	Notes
3	Cation Regenerant Waste for Metals	Sodium	Duplicates for Metals failed acceptance criteria (Client ID's 416011 and 416012, ATL Lab ID's 050634-001A and 050634-002A). Relative Percent Difference of 193 percent renders Field sample result unuseable.
3	Rinse Wastewater for Anions	Chloride	QA Travel Blank for Anions failed acceptance criteria (Client ID 416017, ATL Lab ID 050634-007A). Travel Blank showing appreciable analyte concentration renders Field sample result unuseable.
3	Product DI Water for Anions	Chloride	QA Travel Blank for Anions failed acceptance criteria (Client ID 416017, ATL Lab ID 050634-007A). Travel Blank showing appreciable analyte concentration renders Field sample result unuseable.
3	Anionic Regenerant Waste for Anions	Chloride	QA Travel Blank for Anions failed acceptance criteria (Client ID 416017, ATL Lab ID 050634-007A). Travel Blank showing appreciable analyte concentration renders Field sample result unuseable.
3	HCl Sample	Acidity, Total	QA Acid Standard failed acceptance criteria (Client ID 4160115, ATL Lab ID 050634-015A). Percent recovery of 196 percent renders Field sample result unuseable.
5	Rinse Wastewater for Anions	Chloride	QA Travel Blank for Anions failed acceptance criteria (Client ID 53017, ATL Lab ID 050942-003A). Travel Blank showing appreciable analyte concentration renders Field sample result unuseable.
5	Rinse Wastewater for Anions	Sulfate	QA Travel Blank for Anions failed acceptance criteria (Client ID 53017, ATL Lab ID 050942-003A). Travel Blank showing appreciable analyte concentration renders Field sample result unuseable.
5	Product DI Water for Anions	Chloride	QA Travel Blank for Anions failed acceptance criteria (Client ID 53017, ATL Lab ID 050942-003A). Travel Blank showing appreciable analyte concentration renders Field sample result unuseable.
5	Product DI Water for Anions	Sulfate	QA Travel Blank for Anions failed acceptance criteria (Client ID 53017, ATL Lab ID 050942-003A). Blanks showing appreciable concentrations renders Field sample result unuseable.
5	Anionic Regenerant Waste for Anions	Chloride	QA Travel Blank for Anions failed acceptance criteria (Client ID 53017, ATL Lab ID 050942-003A). Travel Blank showing appreciable analyte concentration renders Field sample result unuseable.
5	Anionic Regenerant Waste for Anions	Sulfate	QA Travel Blank for Anions failed acceptance criteria (Client ID 53017, ATL Lab ID 050942-003A). Travel Blank showing appreciable analyte concentration renders Field sample result unuseable.

5.0 Results of Sampling

5.1 Feed Rinse Wastewater Results

Field testing was conducted by DTSC personnel at AEC following the steps shown on the Test Run Checklist (Appendix D). Each test run required collecting the samples shown in Table 1 from the feed rinse wastewater, product DI water, and regenerant waste lines.

Photo 6. Flow sensors installed in feed lines. Valve and sampling line for regenerant waste is shown at rear.

Five separate test runs using the same cation and anion resin columns were conducted at AEC. Each test run consisted of a complete treatment cycle including column exhaustion and regeneration. Each sampling run began with the start of the treatment phase using Train 1. The ISCO automatic sampler was programmed to turn on with the start of flow through the feed rinse wastewater line. The signal came from the flow sensor in the feed line; previously zeroed flow totalizers also began recording the volume of rinse wastewater treated. The ISCO sampler purged its line and then took its first sample of 200 ml after a volume of 2000 gallons of feed rinse wastewater had been processed. The collected sample was stored in a five gallon glass bottle within the ISCO unit. Based on a flow rate of 35-45 gpm, this would occur every 45-60 minutes for the duration of the three day treatment run. The run conclusion was signaled with the rise in the displayed EC. Once the display EC reached approximately ten µS/cm, bed saturation and a fast rise in display EC was imminent. The operating train was set to shut off by the AEC operators at display ECs of about 65-75 µS/cm. As the display EC rose rapidly past that point, the flow to Train 1 was redirected to Train 2, and the ISCO sampling unit was stopped automatically. The product DI water analyses showed EC results closer to the expected value of 20 µS/

Photo 7 (left). ISCO 5-gallon bottle, after a sampling run.

Photo 8 (below). Reading the Signet Totalizers.

cm, showing that a discrepancy exists between the conductivity meter display and the specific conductance measured by the analytical laboratory.

The feed rinse wastewater sample collected in the ISCO's five gallon bottle was transferred to labeled one-liter sample bottles containing the appropriate preservative. The transfer, labeling and delivery of the feed rinse wastewater samples was usually conducted on the final day of the treatment phase. The regeneration steps including sampling of the regenerant waste, acid and base regenerant solutions, and column resins, was usually completed the following day. The resins were sampled with a 5½ foot long, one-half inch PVC tube inserted down through the top of the column to a resin depth of 5½ feet. Original Field Notes are included in Appendix E.

Table 4 presents the Feed Rinse Wastewater analysis results and the associated quality control samples. Original Laboratory Reports are included in Appendix F. QA samples which were rejected are denoted with a footnote (1); dependant field samples which were unuseable because of QA sample rejection are denoted with a footnote (2). Non-available data is shown as N/A, while non-detectable values are indicated by ND, with the detection limits included in parentheses. All volumes are in gallons, and concentrations are reported in units of mg/L.

Table 4. Feed Rinse Wastewater Analysis Results

Rinse Wastewater Samples					
Parameter	***Run 1***	***Run 2***	***Run 3***	***Run 4***	***Run 5***
Volume (gal)	66845	64354	67127	92474	87025
Flow Rate (gpm)	N/A	41-48	N/A	33-40, 43-49	N/A
Al (mg/L)	0.50	0.56	0.70	0.47	0.36
B	1.2	2.0	1.9	1.8	2
Cu	0.31	0.12	0.30	0.50	0.83
K	5.4	(1) (2) 5.1	7.1	4.8	8.1
Na	33	33	(1) (2) 9.3	23	31
Ni	0.74	1.0	1.4	0.94	0.55
Zn	2.9	3.3	1.4	1.4	2.8
NH_4^+ (as N)	0.66	0.85	1.5	1.2	1.1
Cl^-	26	(2) 23	(2) 4.0	4.0	(2) 9.7
F^-	1.3	2.1	3.1	2.1	1.8
SO_4^{-2}	20	27	35	20	(2) 21
NO_3^- (as N)	1.9	3.6	5.3	3.1	2.7
PO_4^{3-} (as P)	ND (<2.5)	ND (<2.5)	3.3	ND (<2.5)	3.6
TDS	140	160	130	140	N/A
Conductivity	180	190	180	130	180
Alkalinity	26	9.8	ND	13	40
pH	7.4	4.24	3.88	6.67	N/A
Rinse Wastewater Duplicate					
Al	0.46	0.55	0.72	0.46	0.36
B	1.7	1.9	1.9	1.8	2
Cu	0.33	0.12	0.30	0.49	0.85
K	6.1	(1) (2) 7.7	7.5	4.8	7.9
Na	34	32	(1) (2) 18	27	31
Ni	0.76	0.96	1.4	0.92	0.58
Zn	2.9	3.2	1.4	1.4	2.9
QA Travel Blank - Metals					
Al	ND (<0.10)	ND (<0.10)	ND (<0.10)	ND (<0.10)	ND (<0.10)
Cu	0.06	ND (<0.0030)	0.0059	ND (<0.0030)	ND (<0.0030)
Ni	0.0044	ND (<0.0030)	ND (<0.0030)	ND (<0.0030)	ND (<0.0030)
Zn	0.01	ND (<0.010)	0.020	ND (<0.010)	ND (<0.010)
QA Travel Blank - Anions					
Cl^-	ND (<0.50)	3.3	3.5	ND (<0.50)	5.1
SO_4^{-2}	ND (<2.5)	ND (<2.5)	ND (<2.5)	ND (<2.5)	(1) 3.5
PO_4^{-3} (as P)	ND (<2.5)	ND (<2.5)	ND (<2.5)	ND (<2.5)	ND (<2.5)
QA Spike - Metals					
Al	24	24	25	23	27
Cu	25	24	25	24	26
Ni	24	24	26	24	27
Zn	25	26	29	20	28
QA Spike - Anions					
Cl^-	22	27	28	23	27
SO_4^{-2}	24	26	27	27	27
PO_4^{-3} (as P)	7.3	7.6	7.8	7.5	7.2

5.2 Regenerant Waste Volume Produced

The regenerant waste volume produced was measured with inline flow sensors and totalizers during each test run; no flow sensors were found to be out of calibration. In test run 2, the flow sensor was plugged with debris, and no volume data was collected. Each of the other test runs yielded useable data; the average and range for the regenerant waste volume produced were determined from the four useable test runs. The volume produced is shown on the first line of Tables 5 and 6 for the cationic and anionic regenerant wastes, respectively. QA samples which were rejected are denoted with a footnote (1); dependant field samples which were unuseable because of QA sample rejection are denoted with a footnote (2). The measured volumes varied by less than 10 gallons due to the constant delivery of regenerant controlled by float level switches.

Table 5. Cationic Regenerant Waste Results

Cationic Regenerant Waste Samples					
Parameter	***Run 1***	***Run 2***	***Run 3***	***Run 4***	***Run 5***
Volume (gal)	301	N/A	308	299	300
Al (mg/L)	120	150	160	120	65
B	0.4	ND (<1.0)	2.3	2.5	1.1
Cu	77	30	60	22	240
K	1100	(2) 1300	1600	1400	2200
Na	7500	7700	(2) 3800	3500	14000
Ni	200	270	190	42	180
Zn	820	1000	210	58	950
NH_4^+(as N)	28	1.3	16	180	200

Table 6. Anionic Regenerant Waste Results

Anionic Regenerant Waste					
Parameter	***Run 1***	***Run 2***	***Run 3***	***Run 4***	***Run 5***
Volume (gal)	318	310	313	313	311
Cl^- (mg/L)	6500	(2) 5700	(2) 2000	2500	(2) 3900
F	280	520	1100	600	460
SO_4^{-2}	4200	7500	10000	6500	(2) 5700
NO_3^- (as N)	520	480	320	710	810
PO_4^{-3} (as P)	<300	470	320	500	940

As shown in Tables 5 and 6, the cationic regenerant waste produced averaged 302 gal for 18 ft^3 of resin, yielding a specific volume of 16.8 ± 0.2 gal/ft^3. The anionic regenerant waste produced averaged 313 gal for 18 ft^3 of resin, yielding a specific volume of 17.4 ± 0.1 gal/ft^3. Therefore, the regenerant waste volumes produced averaged 17.1 ± 0.2 gal/ft^3 resin. These were higher than the 10-12.5 gal/ft^3 estimated in the Workplan.

5.3 Cation and Anion Exchange Capacities Restored

Direct sampling of the cation and anion resins was used to determine the exchange capacities restored during regeneration, and the total exchange capacities remaining after several years of service. The 786E system at AEC contained 18 cubic feet each of Purolite PFC-100 H cationic and Purolite PFA-300 OH anionic resins. Samples of the resins were collected using standard industry methodology, generally following the procedures outlined in ASTM Method D-2687-95. The procedures involved inserting a one-half inch PVC pipe into the resin beds from above; when withdrawn the tube retained a core sample of the resin material. To ensure a representative sample through the bed, the pipe was inserted through the full 5½ foot bed depth.

The capacities restored and the total capacities remaining were determined analytically at the resin manufacturer's laboratory using proprietary methods. These methods generally involve eluting the cation resin with a brine solution, followed by a titration of the eluent with base. The anion resin is similarly exposed to brine; the eluent is then titrated with acid and silver nitrate.

The capacities restored to the columns were also checked by performing a mass-balance on incoming feed rinse wastewater, product DI water, and regenerant waste streams. Using volume measurements, the concentrations of ions found in these streams were converted to equivalents, and totaled, providing the number of equivalents entering and leaving the system.

Photo 9. Extracting column resin sample.

The direct resin sampling results in Table 7 show the total capacity remaining and the percent regeneration for each of the five resin sample collections. The total capacities remaining for the cationic resin averaged 96.0 ± 2.1 percent, while the QA sample of fresh resin showed a recovery of 96.3 percent using the same testing procedure. The total capacities remaining for the anionic resin averaged 79.9 ± 1.8 percent, while the QA sample of fresh anionic resin showed a recovery of 94.3 percent using the same testing procedure. The operating capacity is a measure of the quantity of ions, acids, or bases adsorbed or exchanged under the conditions existing during the operation of the column. Because the adsorption

process is terminated before all of the functional groups have been utilized, the operating capacity is always less than the total capacity. There is a relationship between the operating capacity of the resin bed and the quantity of regenerant employed. The operating capacity is based on the resin utilization and the regeneration efficiency. Resin utilization is defined as the ratio of ions removed during treatment to the total ions that could be removed at 100 percent efficiency. The regenerant efficiency is the ions removed from the resin compared to the ions present in the volume of regenerant used. The resin utilization will increase as the regenerant efficiency decreases.

Table 7. Column Resin Analysis Results

Cationic Column Resin						
Parameter	***Run 1***	***Run 2***	***Run 3***	***Run 4***	***Run 5***	***QA Cation Resin Travel Blank***
Moisture Capacity, %	56.0	56.7	55.1	55.8	55.8	52.0
Total Capacity, meq/g	4.82	4.99	5.08	5.05	4.86	4.56
Total Capacity, meq/ml	1.77	1.80	1.90	1.86	1.79	1.83
Bead Integrity, % (Whole-Cracked-Broken)	98-1-1	95-4-1	98-1-1	99-1-0	96	97-1-2
Cation Exchange Capacity Restored (Purolite, Regeneration Efficiency, H)	99.0	98.0	98.0	78.0	99.5	99.5
Total Capacity Remaining, %	93.2	94.7	100	97.9	94.2	96.3
Anionic Column Resin						
Parameter	***Run 1***	***Run 2***	***Run 3***	***Run 4***	***Run 5***	***QA Anion Resin Travel Blank***
Moisture Holding Capacity, %	46.0	46.5	45.0	45.2	45.2	45.0
Total Capacity, meq/g	2.96	2.92	3.05	3.03	2.96	3.49
Total Capacity, meq/ml	1.10	1.07	1.16	1.14	1.12	1.32
% Strong Base	85.0	83.3	80.0	84.3	80.2	88.5
Bead Integrity, % (Whole-Cracked-Broken)	99-1-0	98-1-1	99-1-0	99-1-0	99-0-1	96-4-0
Extractable Organics, mg C/g resin	9.7	9.6	10.1	10.8	10.9	3.8
Anion Exchange Capacity Restored (Purolite, % Regeneration, OH)	90.48	85.56	90.92	89.40	87.28	95.07
% Regeneration, CO3	3.17	6.67	3.47	3.48	6.94	3.52
% Regeneration, Cl	5.82	7.22	4.63	5.81	4.62	1.41
% Regeneration, SO4	0.0	0.0	0.0	0.0	0.0	0.0
% Regeneration, SIO2	0.53	0.55	0.98	1.31	1.16	0.0
Total Capacity Remaining	78.6	76.4	82.9	81.4	80.0	94.3

To determine the resin utilization, the ions retained by the resin before column exhaustion and ion breakthrough were quantified using a mass balance. Ions in the feed rinse wastewater applied to the column, less those ions found exiting the column in the product DI water, equal those retained within the resin. From Table 7, the PFC-100 H cationic resin was found to have an average remaining total capacity of 1.82 meq/ml (96.0 percent of its original). Considering a column containing 18 ft^3 of resin, this yields a column capacity of 927 equivalents. Test runs one, four, and five resulted in useable data for both the feed rinse wastewater volume and cation analysis (Table 4), and for the analysis of product DI water cations (Table 9). For test run one, 547.2 cationic equivalents were applied to the resin in the feed rinse wastewater, while 63.57 were passed through the resin bed and were quantified in the product DI water. The difference, 483.53 equivalents, was retained by the cationic resin; compared to the column capacity of 927 equivalents, the cationic column shows a resin utilization of 52.2 percent. For run four, 678.27 equivalents were applied, and 267.44 were passed through, resulting in a resin utilization of 44.3 percent. For run five, 777.45 equivalents were applied, and 374.95 were found in the product DI water, resulting in a resin utilization of 43.4 percent. Therefore, the average resin utilization measured in the three useable test runs was 46.6 ± 4.6 percent.

Photo 10. Packaged Cation/Anion Resin samples just after extraction from columns.

To determine the regenerant efficiency, data from the Cationic Regenerant Waste Results (Table 5) and the Regenerant Analysis Results (Table 8) was used to compare the ions removed to the number applied. Test runs four and five yielded useable data for both cation species and acidity analysis. For test run four, 246.07 equivalents were measured in the cationic regenerant waste stream. Using a volume of 299 gallons, and an acid concentration of 88,000 mg/L as $CaCO_3$ measured for the regenerant, it can be seen that 1991.8 equivalents were applied to the column in the acid regenerant solution. This results in a regenerant efficiency of 12.4 percent. For test run five, 827.89 equivalents were measured in the cationic regenerant waste stream. Using a volume of 265 gallons, and an acid concentration of 87,000 mg/L as $CaCO_3$ measured for the regenerant, it can be seen that 1745.3 equivalents were applied to the column in the regenerant solution, yielding a regenerant efficiency of 47.4 percent. The average regenerant efficiency measured in the two useable test runs was therefore 29.9 ± 28.8 percent.

Resin utilization for the anionic resin was also determined using a mass balance calculation. The PFA-300 OH anionic resin was found to have an average remaining total capacity of 1.12 meq/ml (79.9 percent of its original). Considering a resin volume of 18 ft^3, this yields a column capacity of 570.8 equivalents. Test runs one and four yielded useable data for anions found in the feed rinse wastewater (Table 4), and the Anionic Regenerant Waste Results (Table 6). To determine the resin utilization for test run one, the volume of 66,845 gallons was used along with the analytical data and a total of 504.9 equivalents were calculated to have been applied to the column, while 51.9 equivalents were seen to have passed through in the product DI water. The difference of 453.0 equivalents was retained by the

anionic resin; compared to the column capacity of 570.8 equivalents, the cationic column shows a resin utilization of 79.4 percent. For run four, 434.9 equivalents were applied, and 235.1 were found in the product DI water, resulting in a resin utilization of 35.0 percent. Therefore, the average resin utilization measured in the two useable test runs was 57.2 ± 36.5 percent.

To determine the regeneration efficiency, data from the Anionic Regenerant Waste Results (Table 6) and the Regenerant Analysis Results (Table 8) were used to compare the ions removed to the number applied. Test runs one and four yielded useable data for both anion species and basic regenerant analysis. For test run one, 423.5 equivalents were measured in the cationic regenerant waste stream. Using a volume of 281 gallons, and a base concentration of 67,000 mg/L as $CaCO_3$ measured for the regenerant, it can be seen that 1425.2 equivalents were applied to the column in the basic regenerant solution. This results in a regeneration efficiency of 29.7 percent. For test run four, 424.4 equivalents were measured in the cationic regenerant waste stream. Using a volume of 260 gallons, and a base concentration of 63,000 mg/L as $CaCO_3$ measured for the regenerant, it can be seen that 1239.4 equivalents were applied to the column in the regenerant solution, yielding a regeneration efficiency of 34.2 percent. The average regeneration efficiency measured in the two useable test runs was therefore 32.0 ± 3.7 percent.

5.4 Rinse Wastewater Volume Treated

The volume of rinse wastewater treated was measured with an inline flow sensor and totalizer. The recorded values are found in Table 4. Feed Rinse Wastewater Analysis Results. The totalizer read both current flowrates and the total flow over the period of operation. Upon re-certification of the flow sensors at the manufacturer's testing facility, no units were found to be out of calibration. Based in the five runs detailed in Table 4, the rinse wastewater volume treated averaged 75,565 ± 9,663 gallons. The first three runs were all around 66,100 gallons; the last two just under 90,000 gallons.

Photo 11. Extracting acid tank sample with glass Coliwasa tube.

5.5 Masses of Acid and Base Consumed

The masses of acid and base consumed were determined by monitoring the volumes of acid and base solutions applied to the columns during regeneration, combined with measurements of those solutions for concentration. The flows from the acid and base tanks were each measured with an inline flow totalizer. Table 8 shows the results of the Regenerant Analysis. QA samples which

were rejected are denoted with a footnote (1); dependant field samples which were unuseable because of QA sample rejection are denoted with a footnote (2).

Table 8. Regenerant Analysis Results

Acidic Regenerant					
Parameter	***Run 1***	***Run 2***	***Run 3***	***Run 4***	***Run 5***
Volume (gal)	266	265	260	299	265
HCl (mg/L as $CaCO_3$)	(2) 12,000	(2) 100000	(2) 92000	88000	87000
QA Acid Standard					
6% HCl (mg/L as $CaCO_3$)	(1) 10000	(1) 93000	(1) 71000	36000	34000
Basic Regenerant					
Volume (gal)	281	280	279	260	272
NaOH (mg/L as $CaCO_3$)	67000	65000	67000	63000	65000
QA Base Standard					
6% NaOH (mg/L as $CaCO_3$)	76000	76000	77000	78000	74000

The masses and volumes of acid and base used per regeneration were determined by measuring the volumes of acid and base solutions applied to the columns during each test run. These volumes were combined with analysis of those solutions for concentration to determine the masses used. The flows from the acid and base tanks were measured with an inline flow sensor and recording totalizer. Each of the five values for acid and base regenerant volumes recorded at AEC were useable. The acid volume averaged 271 ± 11.6 gallons, ranging from 260 to 299. The base volume averaged 274.4 ± 6.5 gallons, with a range of 260 to 281. The mass was calculated using the average of the five volumes recorded, and the two concentrations which were acceptable, those from runs four and five. The average for acid volume of 271 gallons and concentration of 87,500 mg/L as $CaCO_3$ yielded a mass of 144.3 lbs HCl, which corresponds to 38.9 gallons of concentrated HCl solution (37 percent w/v). Thus, each regeneration cycle for the cationic column was found to require slightly less than 40 gallons of concentrated HCl. As described in Section 2.2 Regeneration, a portion of the acidic regenerant solution was reused from the previous regeneration cycle, but that fraction was not determined in this study.

Each of the volume measurements, and each of the QA samples associated with the base regenerant study were useable, therefore the reported data is an average of all five test runs. The average base regenerant used was 274.4 ± 6.5 gallons. The average base concentration was 65,400 mg/L as $CaCO_3$, which yields an average mass of 119.7 pounds NaOH. This corresponds to 18.7 gallons of concentrated NaOH solution (50 percent w/v) used per anionic column regeneration. As with the acidic regenerant, a portion of the basic regenerant solution was reused from the previous regeneration cycle, but that fraction was not determined in this study.

5.6 Masses of Metal Species in the Regenerant Waste

The concentrations of cations in the regenerant waste were determined for mass balance calculations and to provide information for potential end-users and metal reclaimers. The concentrations were used with regenerant waste volume measurements to calculate the masses of metal species in the regenerant waste. The average and range for the masses of the representative metal species copper, nickel, and zinc were determined.

Each of the five test runs provided useable concentration data for metals, and all but test run two yielded useable data for waste volumes. The average masses and ranges were found to be 113.8 ± 89.7 g and 24.9 to 272.5 g for copper, 175.3 ± 70.5 g and 47.5 to 227.9 g for nickel, and 580.8 ± 411.5 g and 65.6 to 1,078.7 g for zinc.

5.7 Product DI Water Quality

EC readings were recorded daily from the panel display. Before the evaluation, the EC of the product DI water was monitored using the existing conductivity sensor/meter in the effluent pipe. Hydromatix reported that at the beginning of each treatment, the EC was about 0.3 µS/cm, and that the set point for the end of the treatment was normally chosen to be about 20 µS/cm. Although the displayed EC at the start of treatment was about 0.5-1 µS/cm, the end of treatment set point was actually set at 65-75 µS/cm by the operator. When the sensor/meter indicates the EC was greater than or equal to the set point, the treated water EC indicator light on the panel illuminated and the rinse wastewater flow was redirected to a fresh set of columns.

Photo 12. Labeled water samples sealed in plastic bags.

Five product DI water samples were collected, one at the end of each test run, when the set point had been reached. These samples were measured for the same constituents as the feed rinse wastewater, with an additional analysis for electrical conductivity. The average and range for the EC of the product DI water was determined from the five runs. The reported values for maximum ionic concentrations in the product DI water were determined from useable test runs only.

Product DI Water Analysis Results are presented in Table 9. QA samples which were rejected are denoted with a footnote (1); dependant field samples which were unuseable because of QA sample rejection are denoted with a footnote (2). As measured by ATL, the EC of the DI water averaged 36 µS/cm at the end of a run, with extreme values of 13 and 78 µS/cm noted. The EC

values reported on the control panel often exceeded 100 µS/cm at the end of a run. Other water quality indicators measured included pH averaging 4.5, TDS averaging 36 mg/L, and non-detectable alkalinity.

The presence of elevated chloride concentrations, and decreased pH levels, are associated with end of run samples. This is an indication that the anion column resin is exhausted first, which is by design. If the cationic column was exhausted prior to the anionic, metals would pass through and enter the downstream anionic column. In the anionic column the pH is raised by the release of hydroxide ions; these would form hydroxide precipitates with any metals present, which would tend to foul the resin bed. By design, the anion column is exhausted before the cation. Metals are still removed from the waste stream, but anions are not exchanged for hydroxide ions. Hence, the anions are present in the product water and no hydroxide ions have been released to react with the hydrogen ions, resulting in a lower pH.

Table 9. Product DI Water Analysis Results

Product DI Water Analysis					
Parameter	***Run 1***	***Run 2***	***Run 3***	***Run 4***	***Run 5***
Al	ND (<0.10)	ND (<0.10)	ND (<0.10)	ND (<0.10)	0.1
B	0.63	0.71	0.7	2.4	3.8
Cu	0.0075	0.01	0.003	ND (<0.0030)	0.02
K	ND (<2.0)	(2) 2.4	2.7	ND (<2.0)	ND (<2.0)
Na	ND (<2.0)	ND (<2.0)	(2) ND (<2.0)	ND (<2.0)	ND (<2.0)
Ni	0.01	ND (<0.0030)	0.0058	0.0079	ND (<0.0030)
Zn	0.010	0.010	0.010	ND (<0.010)	0.01
NH_4^+ (as N)	ND (<0.030)	0.058	0.12	0.41	0.14
Cl^-	ND (<0.5)	(2) 6.4	(2) 3.8	0.56	(2) 5.1
F^-	ND (<0.5)	1.4	1.5	5.5	1.1
SO_4^{-2}	ND (<2.5)	ND (<2.5)	ND (<2.5)	ND (<2.5)	(2) ND (<2.5)
NO_3^- (as N)	ND (<0.50)	ND (<0.50)	ND (<0.50)	ND (<0.50)	ND (<0.50)
PO_4^{-3} (as P)	ND (<2.5)	ND (<2.5)	ND (<2.5)	ND (<2.5)	ND (<2.5)
TDS	15	23	21	86	N/A
Conductivity	13	36	31	78	23
alkalinity	ND (<2.0)	ND (<2.0)	ND (<2.0)	ND (<2.0)	N/A
pH	5.48	4.3	4.3	4.04	N/A

Two DI water samples were collected at the start and midpoint of a treatment run, in addition to the product DI water sample collected at the end of the run. The start and midpoint samples were collected to show the performance of the system during normal treatment operations, whereas the sample collected at the end of the run shows the product DI water in the worst case condition, just prior to column exhaustion. For the first sample collected at the start of the run, chloride was detected at 4.5 mg/L, ammonia (as N) at 0.28, and boron at 0.05 mg/L. All other ions from the analytical suite were not found above the detection limit. The sample conductivity was 0.5 µS/cm after 7657 gallons had been processed, with an alkalinity of 2.1 mg/L and a pH of 7.1. The midpoint sample was collected after 35,790 gallons had been treated. Only ammonia (as N) was detected at 0.77 mg/L; all other ions were below

the detection limits. The alkalinity was 2.1mg/L, the TDS non-detectable, and the pH was 8.0. Typical panel readouts during the start and midpoints of runs were from 0.5-2 µS/cm, and these values agreed with analytical results determined by ATL.

5.8 Worker Health and Safety

Onsite observations at AEC, end-user interviews, and reviews of Hydromatix documentation were used to assess the risks posed to worker health and safety posed by the 786E system. These observations and inquiries indicate that accidental releases due to the failure of piping, valves, or pumps, appear to be unlikely. Routine contact with the system should not result in worker exposure because the waste and regeneration solutions are entirely contained within sealed pipes. Routine maintenance operations such as filter cartridge removal and acid and base concentrate replenishment may involve contact with hazardous solutions and could therefore pose a risk. Non-routine operations such as resin and carbon change-outs would similarly involve hazardous conditions. However, the risk from exposure can be minimized by operators following established operating procedures including adherence to the individual plant's health and safety plan.

The Hydromatix system is typically located in plating shops where potential chemical hazards exist. Such sites should have eye wash stations and safety showers, first aid kits, and all applicable Material Safety Data Sheets (MSDS) available. Workers should have training appropriate for operations involving hazardous chemicals. The training should ensure that employers are complying with federal and state regulations. The Hazardous Waste Operations and Emergency Response (HAZWOPER) standard located in 29 CFR 1910.120 sets forth training requirements for employers and workers involved in hazardous waste clean up, treatment, and emergency response operations. Personal protective equipment including eye protection, gloves, boots, coveralls, and ear protection should be required when physical or chemical hazards are present. An on-going program of continued hazard monitoring, health and safety plan review and modification, re-training, and frequent inspection should be established.

While using the 786E system end-users should follow the recommended safety practices as outlined in the Hydromatix Installation, Operation, and Maintenance Manual (Appendix G). The Hydromatix system itself does not appear to pose a danger to workers during normal operation, although proper precautions should be taken around acidic and basic solutions, holding tanks, and piping and pumping systems. Secondary containment around the tank and piping systems, and good housekeeping procedures should provide protection against spills and leaks.

Photo 13. Sampling air over acid tank porthole for HC1.

At AEC, the Hydromatix 786E Ion Exchange Recycling System has been operating for five years without a significant safety incident. As a precaution before beginning sampling of the hydrochloric acid tank, DTSC personnel took measurements of air quality above the sample port. A Draeger CMS pump and hydrogen chloride chip were used to detect acid gas emissions from above the acid make-up tank sample port. During multiple sampling events, DTSC personnel detected no measurable hydrogen chloride concentrations in the air above the sample port of the hydrochloric acid tank. OSHA has established a permissible exposure limit for hydrogen chloride gas of no greater than 5 ppm maximum at any time. The monitoring equipment utilized has a detection limit of 1 ppm HCl; thus, the initial concern of gas generation from the acid storage tank and consequent exposure to workers was shown to be of reduced concern.

5.9 End-user Data Collection

Early in the evaluation, DTSC staff had contacted, by phone, several Hydromatix end-users. Sets of questions on the following main subjects were asked: system information, process information, volume of regenerant, waste generation/management, system performance, reliability, and user health and safety. The purpose of the phone questionnaires was to provide supportive information to the evaluation of this technology and to develop a database of information from which to select end-users for onsite visits.

Three end-user questionnaires were completed (Appendix H); questionnaire topics were chosen to characterize system performance:

System Information

- systems:
- chosen because capable of zero discharge
- were evaluated/compared before purchase
- were not modified.

Process Information

- process:
- used for Ni & Cu
- 11-12 gal regenerant/ft^3 resin (key objective)
- have been operating for 2-3 yrs
- 3 + regenerations/wk.

Waste Generation and Waste Management Information

- ~2 regeneration cycles collected
- use evaporation
- waste filter cakes are sent for reclamation
- average 90 lbs/day of filter cake
- disposal cost - $0.24/lb.
- air permit needed
- metals analysis available
- used precipitation previously

System Performance (cleaning, regenerant has not increased, maintenance)

- consistent regenerant volumes
- easy system operation

- low maintenance
- increased water quality
- reduced operating cost

Reliability

- repairs can easily be made by operators
- very reliable system
- resin lasts approximately 1 year

User Health and Safety

- protective clothing is not required
- no major leaks
- no health and safety issues

Operator Comments

- system works well and is easy to use

6.0 Hazardous Waste Management and Hazardous Waste Regulations

Ion exchange systems such as the Hydromatix 786E concentrate and separate dilute wastewaters from metal finishing operations, producing a metal-free treated water which is reused onsite, but also producing a concentrated regenerant waste which requires further treatment. The concentrated regenerant waste is typically a hazardous waste, and in accordance with 40 CFR Part 261, is subject to permit requirements and other restrictions at the Federal, State, and local levels. Since a significant portion of the wastewater treated by the Hydromatix system is reused onsite, the ion exchange system is recognized as a recycling unit, and may be exempt from permit requirements. Downstream processes apart from the ion exchange system which change the physical or chemical characteristics of the regenerant waste may still constitute hazardous waste treatment. Hazardous waste treatment processes could include neutralization of the regenerant waste (physical/chemical treatment), and evaporation of the neutralized regenerant wastes (volume reduction). California's Permit-by-Rule regulations found in California Code of Regulations (CCR) 45 §67450.1 should be consulted with relevant Federal, State, and local regulatory requirements to determine the permit requirements and other restrictions for treatment of hazardous wastes. For wastewater treatment systems in California, requirements found in Health and Safety Code (HSC) §25143.2 (c)(2) and (d)1 should be consulted for recycling provisions. Questions may be directed to the Waste Identification and Recycling Section (WIRS) of DTSC.

7.0 Summary of Verification Activities and Sampling Results

This ETV report documented the Hydromatix 786E Ion Exchange Rinsewater Recycling System evaluated by DTSC at Aero-Electric Connectors in Spring 2001. The primary objectives of regenerant waste specific volume and cation and anion exchange capacities were determined. This determination allowed DTSC to state how much waste is generated by the Hydromatix system while documenting the amount of exchange capacity restoration achieved. Secondary objectives included providing information for potential end-users and metal reclaimers, and observing worker health and safety conditions during normal operation of the system. Because no pass or fail criteria were established for this evaluation, the results of the verification performance form the basis for the conclusions of this report.

The central claim made by Hydromatix involved the ratio of gallons of waste produced per cubic foot of resin regenerated. DTSC was able to quantify these values over five test runs and determine the regenerant waste specific volume. A reduced specific volume allows more waste management options and assists metal plating facilities in achieving zero wastewater discharge. The evaluation verified, through independent testing, the following performance results:

Regenerant waste volume produced: Measured over four test runs, the regenerant waste volume produced averaged 17.1 ± 0.2 gal/ ft^3 resin. The cationic regenerant waste produced averaged 302 gal for 18 ft^3 of resin, yielding a specific volume of 16.8 ± 0.2 gal/ft^3. The anionic regenerant waste produced averaged 313 gal for 18 ft^3 of resin, yielding a specific volume of 17.4 ± 0.1 gal/ft^3. These regenerant waste values were different from those predicted in the Workplan.

Cation and anion exchange capacities restored: The percentages of the resin capacities restored were measured through direct resin sampling and laboratory analysis. Cation and anion exchange capacities restored were 94.5 ± 6.8 and 88.7 ± 1.7 percent, respectively. Compared to new resin material, the remaining cationic resin capacity averaged 96.0 ± 2.1 percent, and the remaining anionic resin capacity averaged 79.9 ± 1.8 percent. Mass balances were used to determine the resin utilization and regenerant efficiencies. For the cation resin, the resin utilization was found to be 46.6 ± 4.6 percent, and the regenerant efficiency was 29.9 ± 28.8 percent. For the anion resin, the resin utilization was found to be 57.2 ± 36.5 percent, while the regenerant efficiency was 32.0 ± 3.7 percent. No prediction of values for resin capacity restoration, utilization, or regeneration efficiency were stated in the Workplan; these values serve as a baseline for future comparisons of ion exchange technologies.

Rinse wastewater volume treated: The rinse wastewater volumes treated averaged 75,565 ± 9,663 gallons per test run.

Masses of acid and base consumed: The acid volume averaged 271 ± 11.6 gallons ranging from 260-299 gallon. The base volume averaged 274 gal ranging from 260-281 gallon. The masses of acid and base used were 144.3 pounds of HCl and 119.7 pounds of NaOH per regeneration cycle. The volumes of concentrated acid and base were 38.9 gallons 37 percent HCl, and 18.7 gallons 50 percent NaOH.

Masses of metal species in the regenerant waste: The averages and ranges for the masses of the representative metal species copper, nickel, and zinc the regenerant waste were determined. The masses and ranges were found to be: 113.8 ± 89.7 g and 24.9 to 272.5 g for copper, and 175.3 ± 70.5 g and 47.5 to 272.5 g for nickel, and 580.8 ± 411.5 g and 65.6 to 1,078.7 g for zinc. These masses were contained in an average cationic regenerant waste volume of 302 gallons per test run.

Product DI water quality: The ECs in the product DI water collected at the end of the treatment runs averaged 36 µS/cm, with extreme values of 13 and 78 µS/cm noted. Typical panel readouts during the start and midpoints of runs were from 0.5 to 2 µS/cm. Maximum ionic contamination of the product DI water was 2.7 mg/L potassium (K) and 6.4 mg/L chloride (Cl). Values for pH ranged from 4.04 to 5.48, with an average pH of 4.5.

User opinion responses on system performance, reliability, and waste generation were collected; all were generally favorable, with no negative comments noted. The data resulting from field testing was reviewed by the Project Review Team. The collected data and supporting information were sufficient to verify the technology, and DTSC has prepared a Verification Decision. Additional field tests are not necessary, and the scope of the Environmental Technology Verification is representative of a potential commercial application.

8.0 Vendor's Comments

The following information was provided by Hydromatix. The purpose is to provide the vendor with the opportunity to share additional information on their technology. This information does not reflect agreement or approval by U.S. EPA and Cal/EPA.

Target Market Applications: The features of the technology offer tremendous advantages in the following areas:

a. Metal Finishing Rinsewater
b. Printed Wiring Board Rinsewater
c. Semiconductor Liquid Abatement
d. Groundwater Remediation

Competitive Advantage: The minimized waste volume produced by the 786E can be up to 93% less than conventional ion exchange base recycling system. This makes evaporation of the residual liquid waste feasible. Thus the 786E provides the bridge to zero discharge. Regardless if the user wishes to be zero discharge or not, the cost for wastewater treatment is reduced substantially.

System Operating Costs: The Hydromatix 786E System costs approximately $5.00 per 1,000 gallons treated to operate. This takes into consideration the present value of 5 years worth of consumables (resins included), sludge haul off, and labor. This compares with treat and discharge systems based on conventional precipitation with an operation cost between $20.00 and $35.00 per 1,000 gallons treated. These costs include the cost of City water and sewage treatment. The variance is due to the differences in plating operations and municipal water and sewer charges. The typical payback period, when using a Hydromatix 786 System is under two years, when compared to conventional treatment.

Compliance: With a minimized waste treatment system, the final waste volume can be either evaporated or batch treated. If a user opts for batch treatment, they have the option to be zero discharge at any time in the future. In a batch treatment mode, the user releases the batch to the POTW when their in-house tests indicate that compliance targets are within limits. The batch treater, typically the chemical precipitation unit that was previously used on a continuous basis as the treat and discharge treatment system. When a 786E System is installed, the [regenerant] waste can be treated by this same precipitation unit, but unlike the continuous mode, in a batch, target contaminants can be precipitated to their optimum levels. Since pH plays a significant role in optimizing the precipitation of various mixed metals, the batch allows for the ability to vary the pH during the batch process, thus insuring optimum separation and removal.

Environmental benefits of a closed loop water system include:

a. Substantially reduced water requirements. This is particularly beneficial in arid areas or those that experience water shortages, but is of value anywhere, because it means that less water is diverted from municipal supplies for the manufacturing process.
b. Zero discharge. This alleviates the environmental risks and challenges of water treatment. Removing the potential for heavy metal contamination of water supplies is an obvious advantage. Lowering the demand on energy and resource intensive waste treatment processes is also a significant benefit.
c. Other factors. A number of environmental and ecological problems are associated with industrial water use, even with proper water treatment facilities. These include such local disturbances as elevated temperatures in streams and rivers.

The economic and business benefits also include:

a. Recycles DI water. By putting DI water back into the rinsewater systems, the expense of and infrastructure needs for city water purification or in the case of the electronics industries, ultrapure water, are diminished.
b. Avoids water and sewer costs. These can be particularly high in specific metropolitan area. Such cost avoidance can mean that the zero discharge system is considerably less expensive over time than continuing to treat and discharge water.
c. Avoids regulatory procedures. The closed loop system requires little or no regulation.
d. Alleviates risk. As a more environmentally sound method, the process lessens the likelihood of liability claims.

Hydromatix Contact: The latest information about Hydromatix products can be obtained from Hydromatix at:

Greg White
BOC Edwards Hydromatix
10450 Pioneer Blvd., Santa Fe Springs, CA 90670
Telephone (800) 2215152

Systems Costs: The Hydromatix 786E System costs approximately $5.00 per 1,000 gallons treated to operate. This takes into consideration the present value of 5 years worth of consumables (resins included), sludge haul off, and labor. This compares with treat and discharge systems based on conventional precipitation with an operation cost between $20.00 and $35.00 per 1,000 gallons treated. These costs include the cost of City water and sewage treatment. The variance is due to the differences in plating operations and municipal water and sewer charges. The typical payback period, when using a Hydromatix 786 System is under two years, when compared to conventional treatment.

Compliance: With a minimized waste treatment system, the final waste volume can be either evaporated or “batch treated”. If a user opts for batch treatment, they have the option to be zero discharge at any time in the future. In a batch treatment mode, the user releases the batch to the POTW when their in-house tests indicate that compliance targets are within limits. The batch treater, typically the chemical precipitation unit that was previously be used on a continuous basis as the “treat and discharge” treatment system. When a 786E System is installed, the regenerate waste can be treated by this same

precipitation unit, but unlike the continuous mode, in a batch, target contaminants can be precipitated to their optimum levels. Since pH plays a significant role in optimizing the precipitation of various mixed metals, the batch allows for the ability to vary the pH during the batch process, thus insuring optimum separation and removal.

The latest information about Hydromatix products can be obtained from Hydromatix at:

Greg White - Telephone (800) 221-5152

6.0 Availability of Verification Statement and Report

Copies of the public Verification Statement and Verification Report are available from the following:

(NOTE: Appendices A - H are included as separate volumes, and are available from DTSC upon request.)

1. United States Environmental Protection Agency
   P.O. Box 42419
   Cincinnati, Ohio 45242-2419

Web Site: http://www.epa.gov/etv/library.htm *(electronic copy)*