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## DEMONSTRATION OF REMOVAL, SEPARATION, AND RECOVERY OF HEAVY METALS FROM INDUSTRIAL WASTESTREAMS USING MOLECULAR RECOGNITION TECHNOLOGY (MRT)

### Final Report



by  
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14. ABSTRACT This report validates the performance and life cycle costs of molecular recognition technology (MRT) for selective heavy metal recovery from industrial process waste streams. MRT selectively removes heavy metal ions using synthetic chemical compounds called macrocyclic ligands. These ligands complex with the targeted heavy metal ions while allowing alkaline earth and alkali metal ions to pass through the MRT system. The captured heavy metal ions can be regenerated in a highly purified concentrated form, which can be recycled back to the industrial process or sold to a metal reclaimer. IBC Technologies, Inc., has patented these macrocyclic ligands as <i>Superligs</i> <sup>®</sup> . MRT can recover in a single process Cu, Cd, Cr (Cr VI or Cr III), Ni, Pb, Zn, and Ag to below regulated discharge limits for industrial wastewater treatment plants. For other applications, MRT can be designed to remove single metal ions such as arsenic (As). This report includes all the data from the operational runs of the pilot scale MRT demonstration at Puget Sound Naval Shipyard's industrial wastewater pretreatment facility (IWPF). At the IWPF, two MRT mixed bed columns were used where <i>Superlig</i> <sup>®</sup> 327 recovered copper, lead, silver, nickel, cadmium and zinc from the influent stream. <i>Superlig</i> <sup>®</sup> 307 and <i>Superlig</i> <sup>®</sup> 310 were used to recover chromium (VI) and chromium (III) respectively. The efficiency of MRT system was calculated and cost data presented along with a discussion of regulatory issues.					
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## EXECUTIVE SUMMARY

This Technical Report describes the results of the demonstration/validation of Molecular Recognition Technology (MRT). The MRT project was funded by the Environmental Security Technology Certification Program at Puget Sound Naval Shipyard's (PSNS) Industrial Wastewater Pretreatment Facility (IWPF). It shows that MRT can be utilized to treat the entire industrial wastestreams so that the IWPF effluent can be sewerred to a public owned treatment works (POTW). MRT can also be an effective point source technology to selectively remove one or more heavy metal ions in order to remain in compliance under the Clean Water Act (CWA). The MRT process significantly reduces or eliminates the generation of RCRA F006 heavy metal sludges going to landfill as it allows the user to recover heavy metals ions in a concentrated solution that can be sold to metal recyclers. In 1995, NFESC published the results of feasibility testing of three novel metal adsorption technologies. One of these metal adsorption technologies, patented by IB Advance Technologies, Inc., met the Navy's requirements for future compliance regulations as well as recycling/resale potential. IBC advanced Technologies process is based on the use of one chemical structure, called the host, to recognize specific electronic and spatial features of another chemical called the guest, to form a "host-guest" complex. A guest, the dissolved heavy metal ion species, can be selectively removed from the industrial wastestream and later recovered using various regenerative techniques. The host is chemically called a macrocyclic ligand, which IBC Advanced Technologies, Inc. has patented as *Superlig*<sup>®</sup>. These *Superligs*<sup>®</sup> are bonded to polymer supports and are very stable in the solid form and this allows the *Superligs*<sup>®</sup> to be used in a packed bed column or membrane configuration for process at high flow rates for industrial operations. The cost savings and payback for a complete MRT industrial wastewater treatment facility is largely dependent on future liability costs of land filling RCRA F006 sludge. Five different cost estimates are illustrated using MRT in different recovery, recycle scenarios. For a typical Navy industrial wastewater treatment plant, the annual cost savings of installing and operating a MRT system over the conventional system is \$73K per year. If MRT is used for pretreatment of chelated heavy metals, the annual cost savings is \$40K per year. As a point source MRT system for chromium (VI, III) ion recovery, annual cost savings are estimated to be \$17K per year. For a MRT embedded membrane-polishing system, the annual cost savings would be calculated based on the cost of "out of compliance" episodes during the year.

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## Acronyms

ATMA	Automated Trace Metal Analyzer
Cr (VI)	Hexavalent Chromium Ion
Cr (III)	Reduced Chromium Ion
CWA	Clean Water Act
DoD	Department of Defense
EDTA	Ethylenediaminetetraacetic Acid
ESTCP	Environmental Security Technology Certification Program
F006	Designation of Hazardous Waste Under 40 CFR 261.31
gpm	gallons per minute
ICP	Inductively Coupled Plasma
IWPF	Industrial Wastewater Pretreatment Facility (term used for this report)
IWTP	Industrial Wastewater Treatment Plant (generic term not used in text)
LM <sup>2+</sup>	Ligand-Metal Complex for metals Ag <sup>2+</sup> , Cd <sup>2+</sup> , Cr <sup>3+</sup> , Cu <sup>2+</sup> , Ni <sup>2+</sup> , Pb <sup>2+</sup> , & Zn <sup>2+</sup>
M <sup>2+</sup>	Alkaline Earth Metals (Mg <sup>2+</sup> , Ca <sup>2+</sup> )
M <sup>+</sup>	Alkali Metals (Na <sup>+</sup> , K <sup>+</sup> )
MP&M	Metal Products and Machinery (Rule)
MRT	Molecular Recognition Technology
NFESC	Naval Facilities Engineering Service Center
NSPS	New Source and Performance Standards
O&M	Operations and Maintenance
POTW	Public Owned Treatment Works
ppm	parts per million
PSNS	Puget Sound Naval Shipyard
PSES	Pretreatment Standard for Existing Systems
QA/QC	Quality Assurance/Quality Control
RCRA	Resource Conservation and Recovery Act
WODE	Washington Department of Ecology
USEPA	U.S. Environmental Protection Agency

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# 1. Introduction

## 1.1 Background

The Environmental Security Technology Certification Program (ESTCP) funded a pilot scale demonstration of Molecular Recognition Technology in FY98. The Naval Facilities Engineering Service Center (NFESC) has had prior MRT developmental studies funded by the Office of Naval Research (ONR) and Naval Facilities Engineering Command. With increasing potential for “out of compliance violations” (Ref 1) under the Metal Products and Machinery (MP&M) Rule-40CFR 438, NFESC was tasked by ONR to develop and demonstrate innovative industrial wastewater treatment technologies. This effort was part of a joint Army, Navy and Air Force Tri-Service coordinated program to evaluate advanced techniques to effectively recycle/reclaim metals from industrial wastewaters (Ref 2). An industrial process was sought that would selectively recover heavy metal ions and not retain the benign alkaline earth metal ions ( $\text{Na}^+$  and  $\text{K}^+$ ) or alkali metal ions ( $\text{Mg}^{2+}$  or  $\text{Ca}^{2+}$ ). In 1995, NFESC published the results of feasibility testing of three novel metal adsorption technologies (Ref 3). One of these metal adsorption technologies, of which IBC Advance Technologies, Inc, holds 27 patents met the Navy’s treatment requirements for heavy metal ion recovery from acid/alkali cleaning process wastewaters and chromium plating rinse waters. IBC Advanced Technologies’ metal recovery/recycle process is based on the use of synthetic chemical compounds called macrocyclic polyether ligands (crown ethers), a concept that received the 1987 Nobel Prize in chemistry (Ref 4). These highly selective ligands will complex with heavy metals ions and have very weak interactions with benign alkaline earth or alkali metal ions commonly found in industrial wastewater. “Molecular recognition” has been applied to these macrocyclic ligands that are capable of single metal ion selection. These highly selective macrocyclic ligands are then covalently attached to solid supports such as silica or polyacrylate and the resulting products is trademarked as *Superlig*<sup>®</sup>. The applications for industrial wastewater treatment are numerous from primary metal recovery, removal of impurities such as tramp metals arsenic and mercury, to effluent polishing.

## 1.2 Official DoD Requirement Statements

### 1.2.1 DoD Requirement

This project addresses the Tri-Service EQ Strategic Plan, Requirement 3.I.1.1.e: Reduce Hazardous Waste Generation from IWTP Sludges and 2.II.1.q: Control/Treat Non-Point Source Discharges.

### 1.2.2 How Requirements Were Addressed

The results of the demonstration showed that MRT successfully recovered all heavy metals regulated under the CWA pretreatment standards at PSNS. The metal ion concentration in the influent stream was two orders of magnitude below PSNS monthly regulatory discharge limits. The analytical results showed benign alkaline earth and alkali metals passed through the MRT column as predicted. Due to the passage of these benign metals, the mass balance analysis confirmed the MRT column capacity was five orders of magnitude greater than regular ion exchange columns. For the reduction of infrastructure at DoD facilities, MRT has a small footprint and ancillary equipment is minimal, discounting storage tanks for wastewater and effluent streams. The cost savings for a complete MRT industrial wastewater treatment facility

is dependent on the estimates for future liability costs to land filling F006 sludge. Revenues from metal reclaimer for pure metal concentrates from MRT processing is lacking due to large facility/regional-wide hazardous disposal contracts. Due to lacking information for disposal costs, the cost savings uncertainly for alternative MRT was estimated at  $\pm 30\%$ . Payback varies from less than 1 year to 9 years according to the type of MRT system installed and the particular site requirements.

### **1.3 Objective of the Demonstrations**

The objective of this project is to demonstrate and validate the technical performance and life cycle cost of Molecular Recognition Technology (MRT) at Puget Sound Naval Shipyard (PSNS). This alternative metal recycle/reclaim process, as opposed to the conventional precipitation method, will be evaluated on its capability to 1) ensure DoD's can remain in compliance of Federal, state, and local regulatory changes and 2) reduce or eliminate hazardous sludge. MRT should significantly increase pollution prevention opportunities for recycling metal laden hazardous waste to metal reclaimers.

### **1.4 Regulatory Issues**

Heavy metal recovery technologies must be developed to enable DoD facilities to treat to the discharge levels expected from the proposed changes in the Clean Water Act (CWA). Wastewater discharges into surface waters are governed under the Clean Water Act, which established the National Pollutant Discharge Elimination System (NPDES). Industrial wastewater discharges from DoD IWTPs have specific limits dependent on whether the industrial operation discharges directly to a waterway or indirectly through a sewage treatment facility or publicly owned treatment works (POTW). EPA is proposing effluent limitations guidelines and pretreatment standards for wastewater discharges from metal products and machinery (MP&M) facilities. Since the metal products and machinery industry includes facilities that manufacture, rebuild or maintain metal products, parts and machines, DoD IWTPs may be affected by these changes.

### **1.5 Previous Testing of the Technology**

The Office of Naval Research (ONR), under the 6.2 Environmental Quality Applied Research Program, supported the successful evaluation of bench scale tests for sequential, selective removal of heavy metals from the Navy's acid/alkali and chromium electroplating wastestreams. The samples were from NADEP North Island acid/alkali and chromium wastestream. Prior to bench scale studies by ONR, the bulk of the IBC's experience lies in the recovery of precious metals, removing contaminants in base metals refining applications, and analytical separations. Feasibility testing have been conducted for (1) lead removal from tin plating, zinc electrogalvanizing, and tin refining baths, (2) mercury removal from sulfuric acid, and (3) antimony removal from copper electrolyte. The MRT has been demonstrated at a Department of Energy Resource Recovery Project at an inactive open-pit mine in Butte, Montana.

## 2.0 Technology Description

### 2.1 Description

Heavy metal ions are among the most common toxic components in industrial wastewaters from DoD industrial operation. At PSNS the industrial wastewater pretreatment facility (IWPF) can receive large volumes (> 1 million gallons/year) of metal laden wastewaters. At PSNS, the metal finishing facility generates 90% of the volume distributed as (1) 56 % acid/alkali cleaning wastewaters, (2) 35 % chromium plating rinse waters, and (3) 9 % cyanide process wastewaters. Hydroxide precipitation is the conventional method for removal of heavy metals from these three influent wastestreams. This treatment process generates hazardous sludge, classified as F006 hazardous waste under the CWA, and currently this sludge is sent to a landfill. Section 3.2 discusses PSNS's industrial pretreatment process in more detail.

In order to avoid generation of metal contaminated sludge, an alternative technology must be capable of recycle/reclaim heavy metals such that they are selectively or sequentially segregated from the industrial wastestream. An additional requirement that must be met is the regenerate is amenable to recycle to process or resale to metals recycle vendor. In DoD facilities, the removal of heavy metals below discharge standards will be in the *presence of other dissolved solids*. Besides heavy metal contamination, industrial wastewaters contain large concentrations of alkali metals ( $\text{Na}^+$ ,  $\text{K}^+$ ) and alkaline earth metals ( $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ), which are not regulated and need not be removed from the wastewater. Although ion exchange resins offer heavy metals, they are not selective to that class of metals alone. Thus, both alkali/alkaline metals, as well as heavy metals, may bind to the resin and reduce the efficiency by rapidly loading the binding sites, and thus increasing the number of regeneration cycles.

A major research interest over the last three decades has been identification and investigation of an alternative chemical sorption/desorption process that could selectively bind heavy metals called molecular recognition technology. Molecular recognition uses one chemical structure, called the host, to recognize specific electronic and spatial features of another chemical called the guest, to form a "host-guest" complex. A guest, such as a dissolved ionic species, can be selectively removed from solution by being complexed with the host chemical and thus be isolated for later recovery/recycle. Ligand is a term defined as any molecule or ion that has at least one electron pair that acts as a donor atom, such as oxygen, nitrogen, or sulfur. Figure 2-1 shows oxygen electron pair donors in ethylenediaminetetraacetic (EDTA). The selectivity for a specific contaminant metal ion does not occur with EDTA and both copper and lead are equally removed from the wastestream (ref 5.).

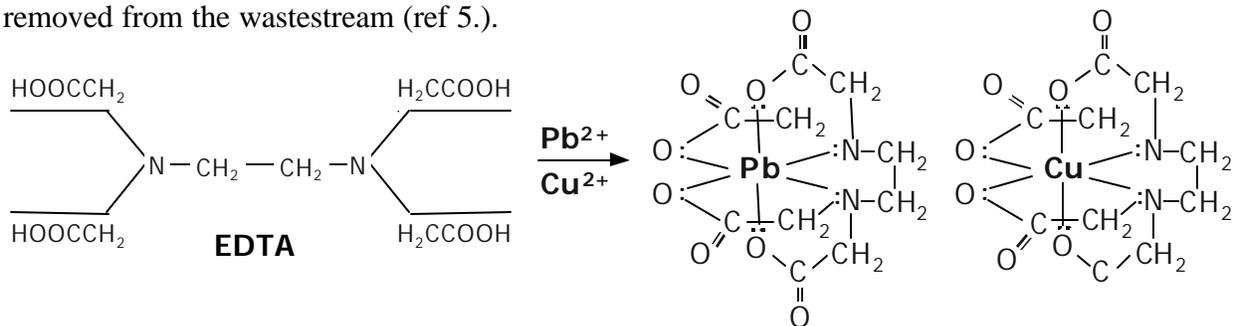
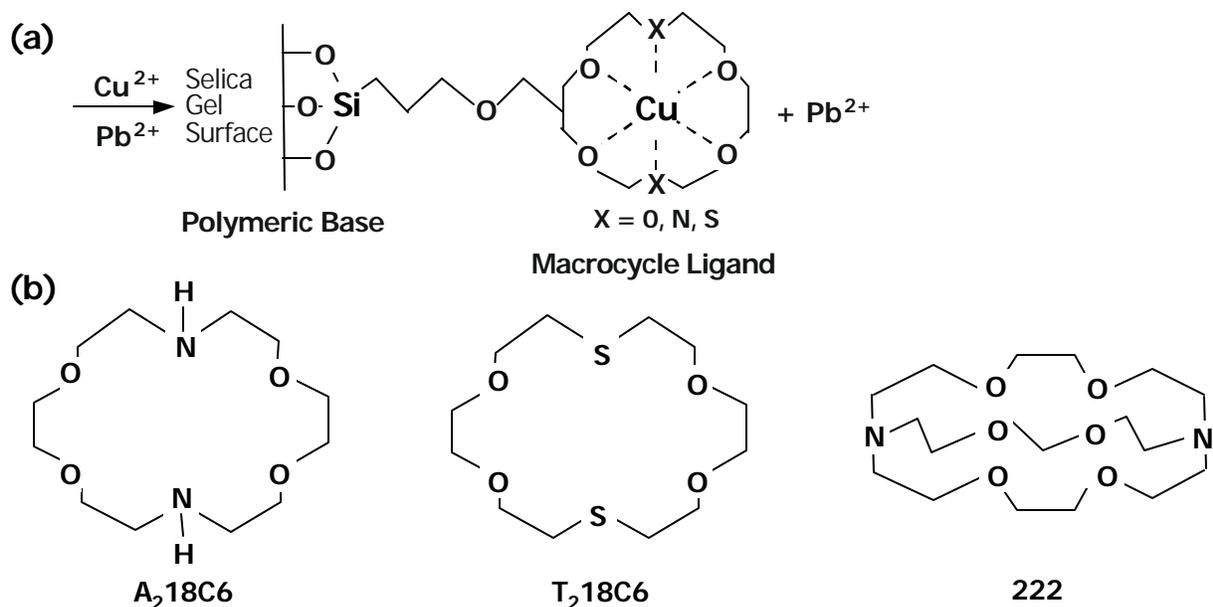


Figure 2-1. Non-Specific Metal Removal from Industrial Wastestreams with EDTA.



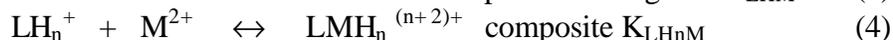
**Figure 2-2. (a) Specific Ion Selectivity for Copper Ion Is Shown Over Lead Ion  
(b) IBC Patented Macrocyclic Ligands for Sequestering Metal Cations & Anions**

In Figure 2-2 (a) copper is selectively removed over lead. Chemically, the macrocyclic ligand process is based on two factors, (1) metal ion-dipole interaction between the heavy metal and the negatively charged donor atoms placed in the macrocyclic ligand and (2) the size and geometry of the macrocycle cavity. This ion-dipole interaction between the heavy metal cation and the negatively charged donor such as oxygen, nitrogen, sulfur. Figure 2-2 (b) shows the wide range of macrocycle ligands that are able to be ion specific for metal cations. The capability to form complexes with heavy metals can be calculated from each ligand's deprotonation and ligand-metal stability constants ( $\log K$ ). The deprotonated form accepts the heavy metal cation and is determined by titration using a dilute base (ref. 5 & 6). The L and  $M^{2+}$  are ligand and metal ion respectively in the following equations.

Deprotonation Calculation:



Step-wise complexations to the bare, or protonated ligand are given by the following equations



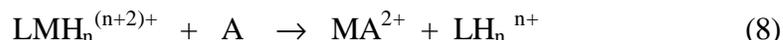
Equation 4 represents overall metal-binding capability. Complexes are formed, not only between the bare ligand and metal ion (equation 2), but also with protonated-ligand species (equation 3). It is the sum of all the metals complexes, which co-exist in solution, that determine the overall ability of macrocycle or similar ligands to sequester divalent metal cations. Table 2-1 gives metal-binding stability constants ( $K$ ) for *Superlig*<sup>®</sup> and for comparison various chelating and ion exchange resins. Chelating resins or ion exchange, normally have binding constants of  $10^{10}$  or  $10^3$ , respectively. MRT has binding constants as high as  $10^{50}$ .

**Table 2-1. Metal-Binding Stability Constants (Log K) for *Superlig*®, Cation Ion Exchange, and Chelating Ion Exchange**

<b>Cation</b>	<b><i>Superlig</i>®</b>	<b>Regular IX</b> Sulfonic Acid as Active Group	<b>Chelating IX</b> Iminodiacetic Acid as Active Group
Mg <sup>2+</sup>	0.02		
Cd <sup>2+</sup>	13.8	<0.7	3.0
Cr <sup>2+</sup>	30.0	<0.7	
Cu <sup>2+</sup>	22.0	<0.7	7.3
Ni <sup>2+</sup>	17.0	<0.7	4.9
Pb <sup>2+</sup>	14.4	<0.7	4.2
Zn <sup>2+</sup>	14.4	<0.7	3.8
Ag <sup>2+</sup>	13.8	<0.7	<0.7

In this case, *Superlig*® regeneration is typically accomplished by elution with 1 to 4 M acid solution. The following equations can be used to calculate dissociation rate of metal cation where:

$$k_{\text{observed}} = k_o + k_A[A] \quad \text{acid} = A$$



The ligand (L) in equation (7) is then protonated as shown in equation (1). That is, below a certain pH the metal cation is competitively displaced by protons that bind to each of the donor atoms (multiple sites per ligand) making the ligand positively charged with a 1<sup>+</sup> charge on each donor atom. When the H<sup>+</sup> or acid concentration is high enough to displace the strongest held metal cation, all of the cations are concurrently displaced. For a mixed bed column, only one elution of all of the metal cations is necessary when the proper acid molarity is chosen. In the case of selective removal of metal ions such as chromium (VI), the elution solution will vary depending on the wastestream matrix. See Section 2.2.1

## 2.2 Hexavalent Chromium (VI) Chemistry

**PSNS Sources of Chromium:** Chromium is regulated as total chromium, not Cr (VI) or Cr (III). Therefore, both species have to be removed from the wastestream. In the conventional precipitation process, the Cr (VI) anion is reduced to the Cr (III) cation by various reducing agents such as ferrous sulfate (PSNS), sulfur dioxide, sodium borohydride, or sodium bisulfite. The Cr (III) cation is then precipitated with NaOH. The hard chrome plating bath in the metal finishing facility contains about 10 to 25 % Cr (III) due to fact that the anode re-oxides most of the Cr (III) back to Cr (VI). At PSNS, all chromium sources were determined by looking at the metal plating facility as a whole. The greatest contributor to the chromium wastestream coming

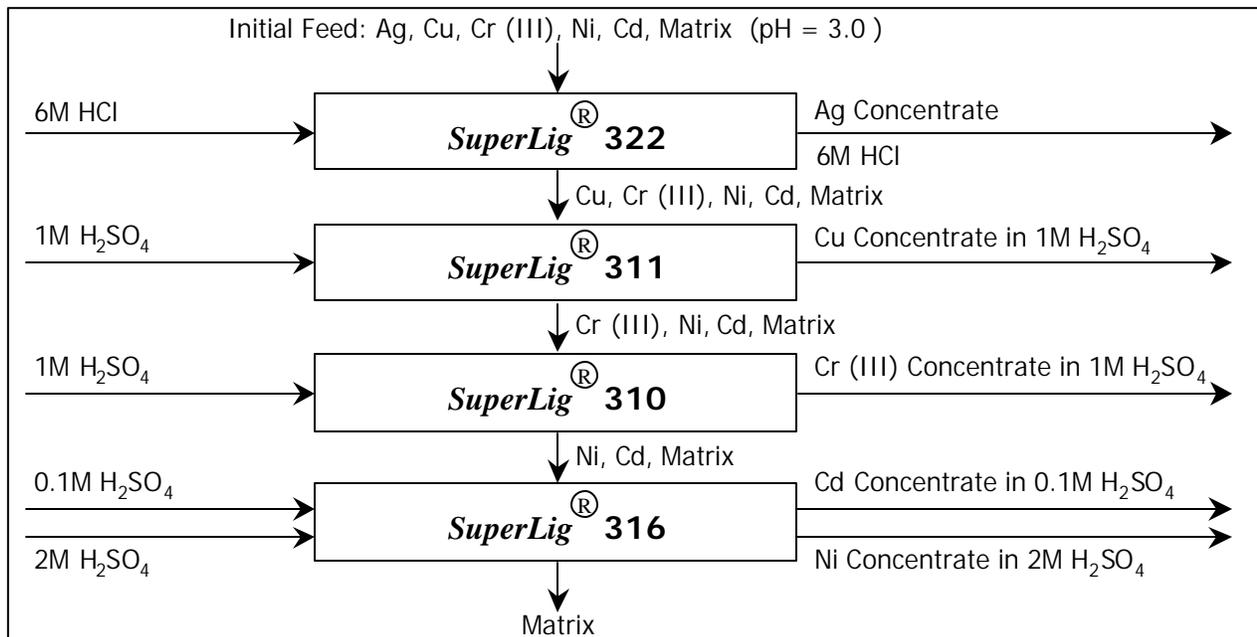
to the IWPF was from the ventilation systems as shown on Table 2-2. The ratio of Cr (VI) to Cr (III) is an average of 75% Cr (VI) and 25% Cr (III).

**Table 2-2 Metal Finishing Facility Sources\* of Chromium (VI) and Chromium (III) Ions**  
(Concentration for Chromium Species in mg/l)

Chromium Species	Ventilation	Chromium Sump	Chromium Scrubber	Chromium Dip Rinse Tank	Dichromate Rinse Tank
Chromium (VI)	445,000	11.54	0.21	74.2	31.9
Chromium (III)	7,254	85.8	3.9	15.79	4.69
Chromium Total	452,254	97.14	4.11	89.99	36.56

\*Data from PSNS 07/1999

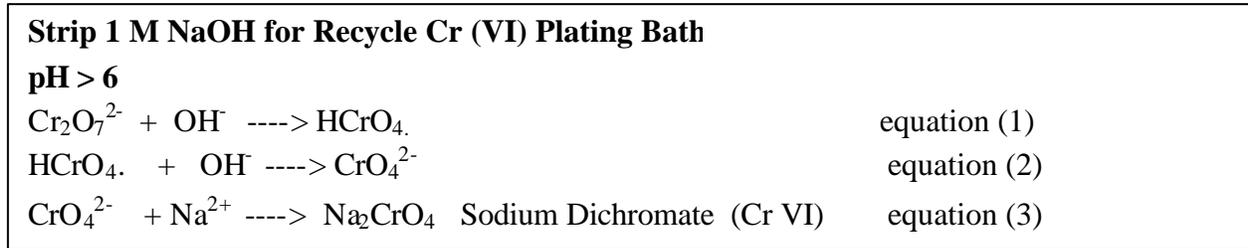
**MRT Approach to the Recovery/Recycle of Chromium Species:** Under funding from ONR, bench scale studies were performed using wastewater from the Naval Air Station (NADEP) North Island. Figure 2-3 shows the selective, sequential steps to recover the various metals from the wastestream. The chromium (VI) does not need to be reduced to Cr (III) but is recovered by hexavalent chromium *Superlig*<sup>®</sup> 307. At PSNS, Cr (III) was recovered by mixing *Superlig*<sup>®</sup> 310 into the mixed *Superlig*<sup>®</sup> 327 packed bed column when processing for the acid alkali metals. See Section 2.2.1 for physical setup.



Note: Wastewaters from Naval Air Station North Island, San Diego, CA

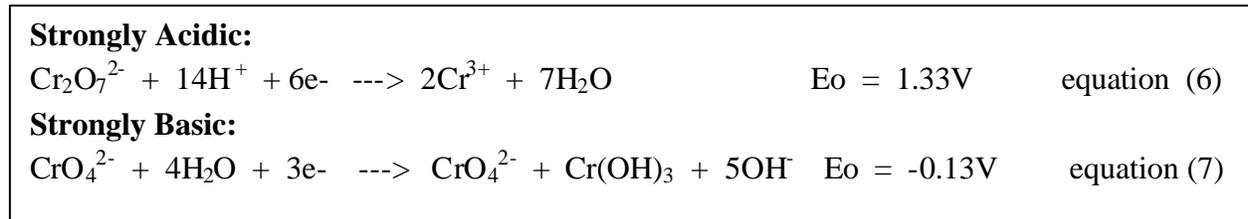
**Figure 2-3. MRT Sequential, Selective Separation of Navy Chromium Wastewater**

The regeneration of the of the Cr (VI) MRT column can be accomplished such that all Cr (VI ) is recovered. Equations 1-3 show the chemistry if the column is stripped with 1 M NaOH so that sodium dichromate Ch (VI) is recovered for recycle.



If it is desired to have the wastestream recovered as all Cr (III) cation for further processing, then 4 M sulfuric acid is used for elution. Cr (III) does not have the same toxicity profile as Cr (VI).

The electron potential for Cr (VI) to Cr (III) reduction is shown in Table 2-3 below. In strongly basic solution as in equation (7), Cr (VI) has a E<sup>o</sup> that is strongly negative (-0.13V) and therefore does not take this reaction pathway. However, in strongly acidic solution the reduction from Cr (VI) to Cr (III) takes place with an E<sup>o</sup> of 1.33V.



**Table 2-3. Potential ( E<sup>o</sup> ) for Cr (VI) to Cr (III) as a Function of Acidic pH**

pH of Solution	Potential (E <sup>o</sup> volts)
0	1.38
1	1.24
2	1.10
3	0.97
4	0.82
5	0.69
6	0.55

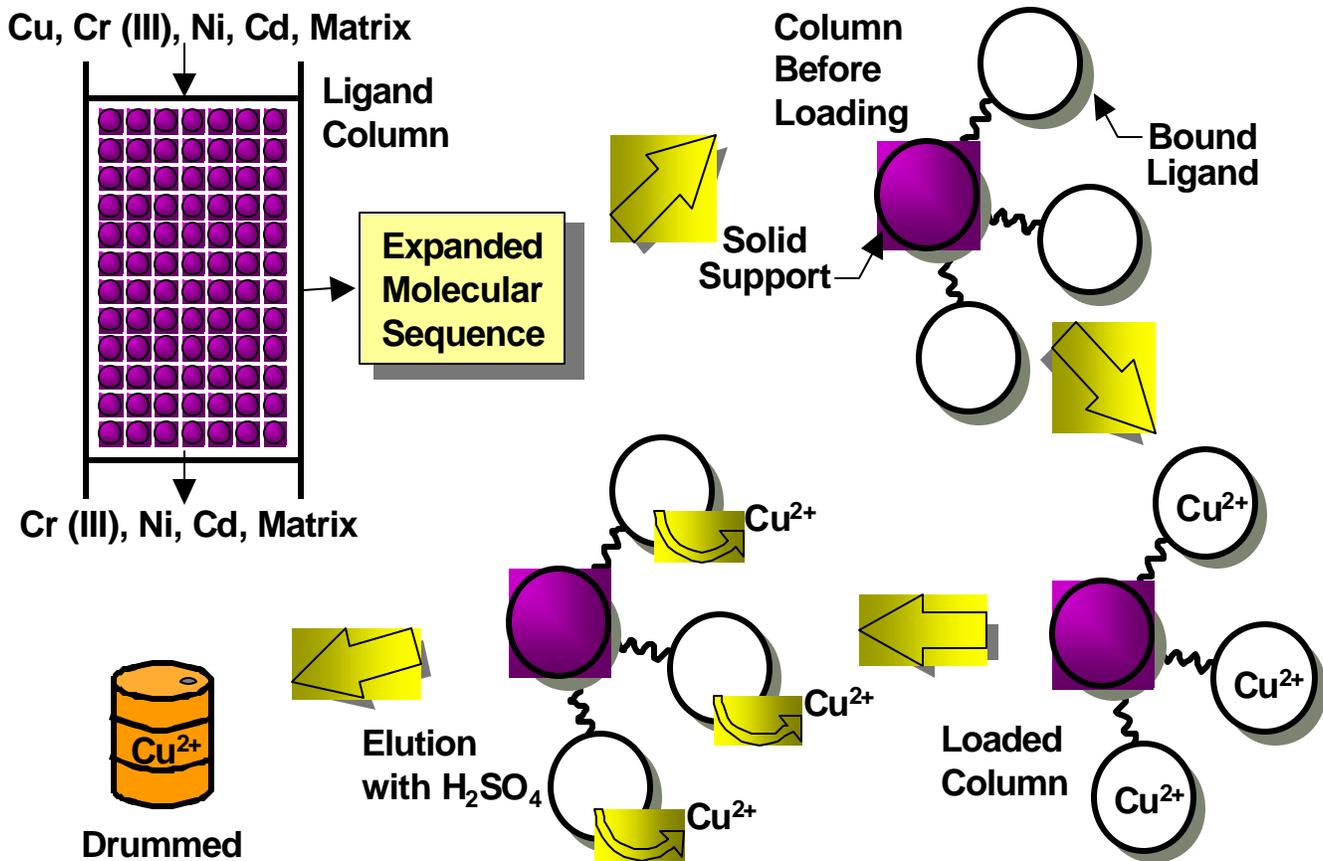


Figure 2-4. MRT Column  $\text{Cu}^{2+}$  Recovery Showing Final Product Concentrate for Recycle

### 2.2.1 Process Description

The *Superlig*<sup>®</sup> materials may be embedded in membranes, replaceable cartridges or as the more traditional packed bed column configuration as demonstrated at PSNS (ref. 7). Figure 2-4 shows graphically how copper would be recovered as a mono metal. The wastestream is passed through the column and copper is adsorbed on the MRT column. The column is then regenerated to obtain a highly purified copper metal concentrate that is drummed. The drummed concentrate may be recycled to process or sold to a metal reclaimer as described in Section 6. In Figure 2-4 other metals Cr(III), Ni, and Cd pass through the column.

However, at PSNS, MRT was demonstrated for acid/alkali wastestream with a mixed packed bed column to capture all regulated heavy metals, Cu, Cd, Cr (VI, III), Ni, Pb, Zn, and Ag. Table 2-4 shows the processing steps. The columns are conditioned in step 1 and 2. In step 3, the feed solution is run through lead-trail columns containing the appropriate *Superlig*<sup>®</sup> to remove the targeted metal ion(s). The metal ion(s) are captured and held by the *Superlig*<sup>®</sup> while the bulk solution passes through the column. After the lead column is saturated with the target metal ion(s), the feed is diverted. The captured metal ion(s) are eluted (or stripped) from the column with 4 M sulfuric acid solution as shown in step 5. The eluate contains an acidic, concentrated, pure metal ion sulfate stream. After regeneration with NaOH in step 1, the column is ready to

receive wastewater feed once again. Table 2-4 shows the final destination of process wastewaters. For the demonstration of Cr (VI), *Superlig*<sup>®</sup> 307 was used to remove this single ion. The same steps were followed on Table 2-4 as for the acid/alkali wastestream.

**Table 2-4. Description of MRT Process Treatment Wastewaters**

Step	Input Stream	Column Action	Output Stream	Final Destination
1	Dilute NaOH	Neutralizing Protonated Bound Ligand	Dilute Na <sub>2</sub> SO <sub>4</sub>	Sewer
2	H <sub>2</sub> O	Wash Out Na <sub>2</sub> SO <sub>4</sub>	H <sub>2</sub> O/Na <sub>2</sub> SO <sub>4</sub>	Sewer
3	Acid/Alkali Feed	Removal of Heavy Metal(s)	Feed Effluent Metals	Sewer
4	H <sub>2</sub> O	Wash Through Remaining Feed	Feed Effluent Metals	Sewer
5	Dilute H <sub>2</sub> SO <sub>4</sub> Elution	Strip Heavy Metal(s)	Small Volume Heavy Metal(s) SO <sub>4</sub> <sup>2-</sup> Concentrate	Collection as Product for Recycle Process or Sell Metals Recycler

### 2.2.2 Column/Membrane Parameters Comparison for Throughput

Throughput can be increased by using membranes as instead of packed bed column as demonstrated at PSNS. Membranes provide large surface area, short diffusion paths, and are amenable to continuous operation. Table 2-5 shows the comparison in configuration of column or membrane throughput parameters in the removal of lead (Pb) using a silica based *Superlig*<sup>®</sup> material (ref. 7).

**Table 2-5. Column/Membrane Parameter Comparison for Removing Lead (Pb) from Phenylsulfonic Acid Tin electrolyte Using a Silica Based *Superlig*<sup>®</sup> Material**

Mole (feed)	Mass of <i>Superlig</i> <sup>®</sup>	System Dimensions	Max Flow Rate (bed Vol/min)	Max Flow Rate (mL/min/g)	Effluent PbConc Range (ppb)	Particle Diameter (micron)
Column 10 ppb Pb	1 g	0.55 cm diameter x 9 centimeter high	0.2	0.4	1-2	250-500
Membrane 10 ppb Pb	0.4 g	3.5 cm diameter x 0.1 cm height	200	200	1-2	9

## 2.3 Strengths, Advantages, and Limitations of the Technology

### 2.3.1 MRT Advantages

Future industrial wastewater treatment facilities will require closed loop systems that discharge little or no pollutants to the environment. MRT has a number of advantages for this application as summarized below:

1. The highly selective ligands give MRT the ability to remove selected metals to extremely low levels, often several orders of magnitude below current discharge limits. These lower limits did not require pH adjustment.

2. The design features of MRT allow creation of ligands selective for *only* the ion of interest in the presence of high concentrations of competing ions.
3. The ability to design selective ligands with targeted stability constants allows a range of elution options. Eluents can be chosen that are compatible with industrial wastewater chemistry and therefore recycle of the eluent will be a possible option.
4. Rapid kinetics are possible, which allows high flow rates. For very low influent metal levels, affinity membranes can be used for even higher flow rates and rapid processing.
5. MRT can be fully automated for continuous operation and has small space requirements.
6. If chelating agents are present in an industrial waste stream, pretreatment prior to bulk precipitation by NaOH must be conducted. The PSNS MRT demonstration showed that pretreatment for surfactants and chelating agents was not required for recovery/recycle of metals. Feasibility studies at NAS North Island showed that the MRT processing broke the chemical bond between the chelating agent and copper.
7. MRT can be used as a polishing system for specific metals out of compliance at an IWPF.
8. Due to the simplicity of the process, and highly efficient elution curves, there is a reduction in the volume of process chemical required for MRT.

### 2.3.2 MRT Limitations

The limitations of this technology are more based on site specific factors than the general technology. The following concerns should be evaluated before procuring an MRT system:

1. Several different MRT systems can be configured to meet the requirements of a DoD facility. At PSNS, feasibility tests with both column and membrane configurations were conducted. The packed bed column configuration showed better results for batch operation of high volumes and metal concentrations greater than 50ppm.
2. If the particulate matter in the wastewater is greater than 15 microns, then it is advised to use a pretreatment filtration system.
3. The technical level of the operators requires training beyond the standard wastewater treatment operator certificate.

## 2.4 Factors Influencing Cost and Performance

### 2.4.1 Cost

The cost of using MRT is based on the number of metals to be recovered, their individual concentrations, and the purity of the metal required. At PSNS, a mixed packed bed *Superlig*<sup>®</sup> column was evaluated. However, separate *Superlig*<sup>®</sup> columns for each metal could have been used. The cost comparison is discussed in Section 6.

### 2.4.2 Performance

The performance of the system will depend on the influent wastestream and its components. A wastestream cannot be processed efficiently with a high concentration of solid particulate matter. If the wastestreams are not bag filtered from the metal finishing facility or other industrial operation, a filtration needs to be installed up-front of the MRT.

## 3.0 Site/Facility Description

### 3.1 Background

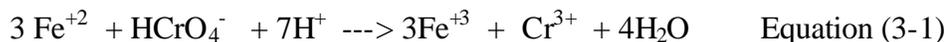
Puget Sound Naval Shipyard was selected because it is typical of other DoD's maintenance and repair facilities. Since 1998, PSNS has been researching the future requirements for wastewater treatment. The ESTCP demonstration/validation project was proposed for PSNS because they were evaluating alternative wastewater treatment technologies that would increase the capability of moving toward a "zero discharge" for a new industrial wastewater pretreatment facility.

### 3.2 Site/Facility Characteristics

Puget Sound Naval Shipyard is engaged in extensive maintenance work on small and large Naval vessels. Work is heavy industrial, including metal plating and cleaning operations such as etching, passivating, plating, galvanizing, and general cleaning. These processes generate rinse water that must be pretreated before discharge to the local sanitary facility. In 1976, PSNS constructed an Industrial Wastewater Pretreatment Facility to treat industrial wastes from several shops throughout the shipyard. IWPF is located on north end of shipyard and is part of the Public Works Department. The waste treatment facility was built in a shipyard central location with piping to transfer the wastewater. Today the only piping remaining is from the largest generator of wastewater, the Metal Preparation Facility. The IWPF receives waste by tank delivery in minimal quantities from the sheet metal shop and the photo laboratory.

The building containing the treatment plant was designed to process wastewater in a two level, heavy concrete structure. The upper level covers a floor area of approximately 9,000 square feet, and is housed in a prefabricated metal structure with masonry walls on the east and south sides. All process equipment is located within the building, and the only external activity is unloading of wastewater from portable tanks and process chemicals, and loading of sludge to be hauled to the Hazardous Waste Containing Storage Area.

There are three wastestreams that constitute the major volume of influent to the PSNS IWPF: 1) chromium electroplating; 2) cyanide rinse and dip; and 3) acid/alkali from cleaning operations. The cyanide wastestream is pretreated for destruction of the free cyanide by oxidation with sodium hypochlorite (NAOCl) and chromium (VI) is reduced with ferrous sulfate to chromium (III) as shown in Equation (1). Ferrous sulfate is used at PSNS because of its low cost with the following reaction:



After a neutralization step, the three latter wastestreams become a single, integrated wastestream where the metals are precipitated as metal hydroxides using caustic soda (NaOH). See Figure 3-1 for treatment processing steps. This metal hydroxide sludge is then de-watered, drummed, and transported to the Treatment, Storage, and Disposal Facility (TSDF). Final disposal of the sludge is in a RCRA Subtitle D landfill. The treated water, after analytical testing, is released to the environment or a municipal sewer, in this case to the city of Bremerton, WA. The PSNS IWPF operates under RCRA "permit by rule" exempting it from requiring a Part B Permit. It now functions under the regulations of the Clean Water Act.

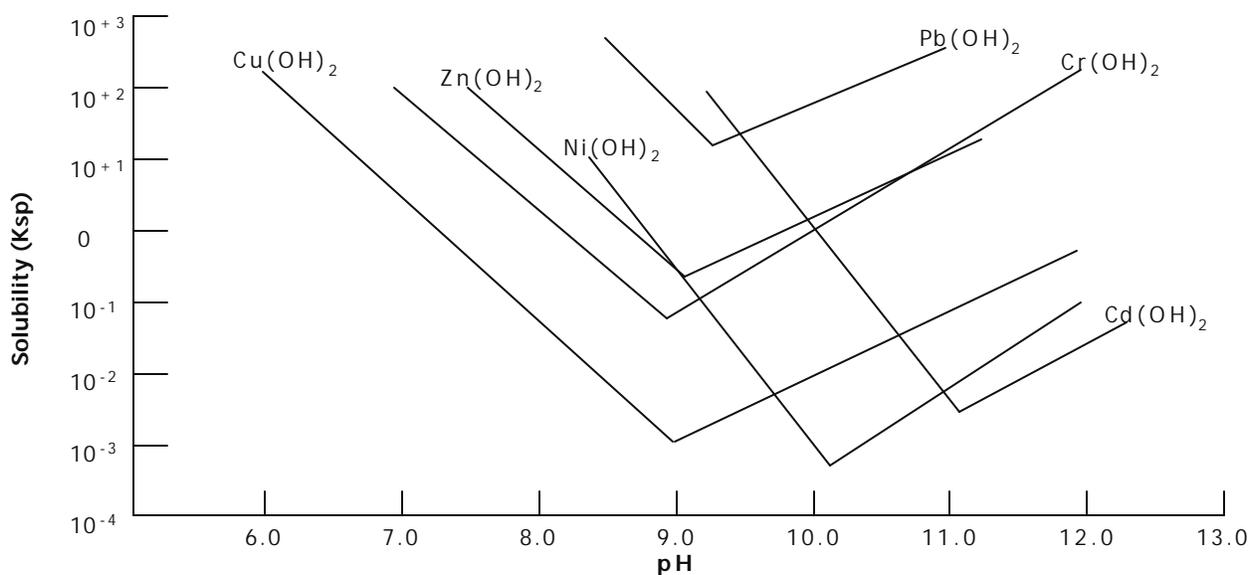
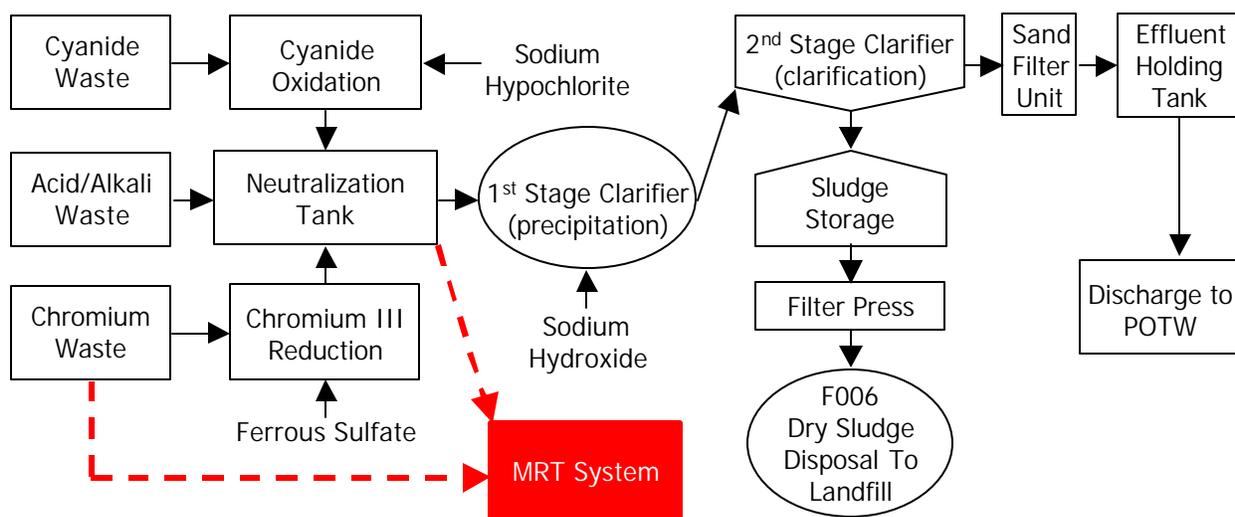


Figure 3-2 Solubility of Metal Hydroxides as a Function of pH (redrawn from ref. 8)

In the conventional method, precipitation of the heavy metals is based on pH of the wastewater. Figure 3-1 shows the maximum pH that each metal will be complexed with NaOH. As the discharge limits are lowered, pH adjustment will have to be more exacting. For example, pH adjustment will need to be raised (> 11) in order to precipitate cadmium to the proposed effluent standards in Table 3-1.

**Table 3-1. PSNS Current and Proposed IWPF Effluent Standards**  
(volume > 1 million gallons discharge/year)

Metal	Daily Maximum Concentration (mg/l)		Maximum Monthly Average (mg/l) *	
	Existing	Proposed	Existing	Proposed
Cadmium	0.17	0.02	0.17	0.01
Chromium	2.77	0.17	1.7	0.07
Copper	3.38	0.44	2.07	0.16
Manganese	none	0.04	none	0.03
Molybdenum	none	0.29	none	0.18
Lead	0.69	0.79	0.43	0.49
Nickel	3.20	1.90	2.38	0.75
Silver	0.43	0.05	0.24	0.03
Sulfide (as S)	none	31	none	13
Tin	none	0.03	none	0.03
Zinc	2.61	0.08	1.48	0.06

The gray shaded rows in Table 3-1 indicate MP&M proposed discharge limits for additional metals. In procuring future IWPF treatment processing, the PSNS must consider future workloads. The proposed effluent standards are for treatment plants with greater than 1 million gallons per year. PSNS volumes for effluent discharge are less than 1 million gallons per year but may change as the workload in the shipyard changes.

## 4.0 Demonstration Approach

### 4.1 Performance Objectives

The objective of this project was to demonstrate the adsorptive metal recycle/reclaim capability of MRT. MRT must meet and exceed the current federal discharge standards under the CWA, as well as local discharge limits to POTWs. These limits were the first primary criteria. MRT must be more cost effective over other adsorptive metal recovery technologies, which were the second primary performance criteria. MRT must also demonstrate metal ion selectivity by showing a 98% extraction of the specific metals from the industrial waste stream. A pollution prevention credit will be gained in reducing or eliminating the metal hydroxide sludges.

**Table 4-1. Performance Objectives**

Performance Objective	Primary Performance Criteria	Expected Performance	Actual Performance
Quantitative	1. Exceed CWA Limits	½ Discharge Limit	Met ½ Discharge Limit
	2. Capital Cost Less Conventional	\$90K	\$120K
	3. Extraction of Specific Metals	98%	98.9%
	4. Efficiency > Related Technology	80%	60%
	5. Sludge Reduction	95%	90%
Qualitative	6. Ease of Use	Minimal Training	Training 1Yr > IWTP Operator

### 4.2 Physical Setup and Operation

The MRT system was installed in the PSNS Industrial Wastewater Pretreatment Facility as shown in Figure 3-1, Section 3.2. The demonstration was “off-line” and performed in batch mode such that current IWPF treatment processing was not disrupted. Figure 4-1 shows graphically the final design of the combined MRT chromium and acid/alkali system at PSNS. The MRT system was skid mounted with dimensions of 15 ft. x 15 ft. x 10 ft.

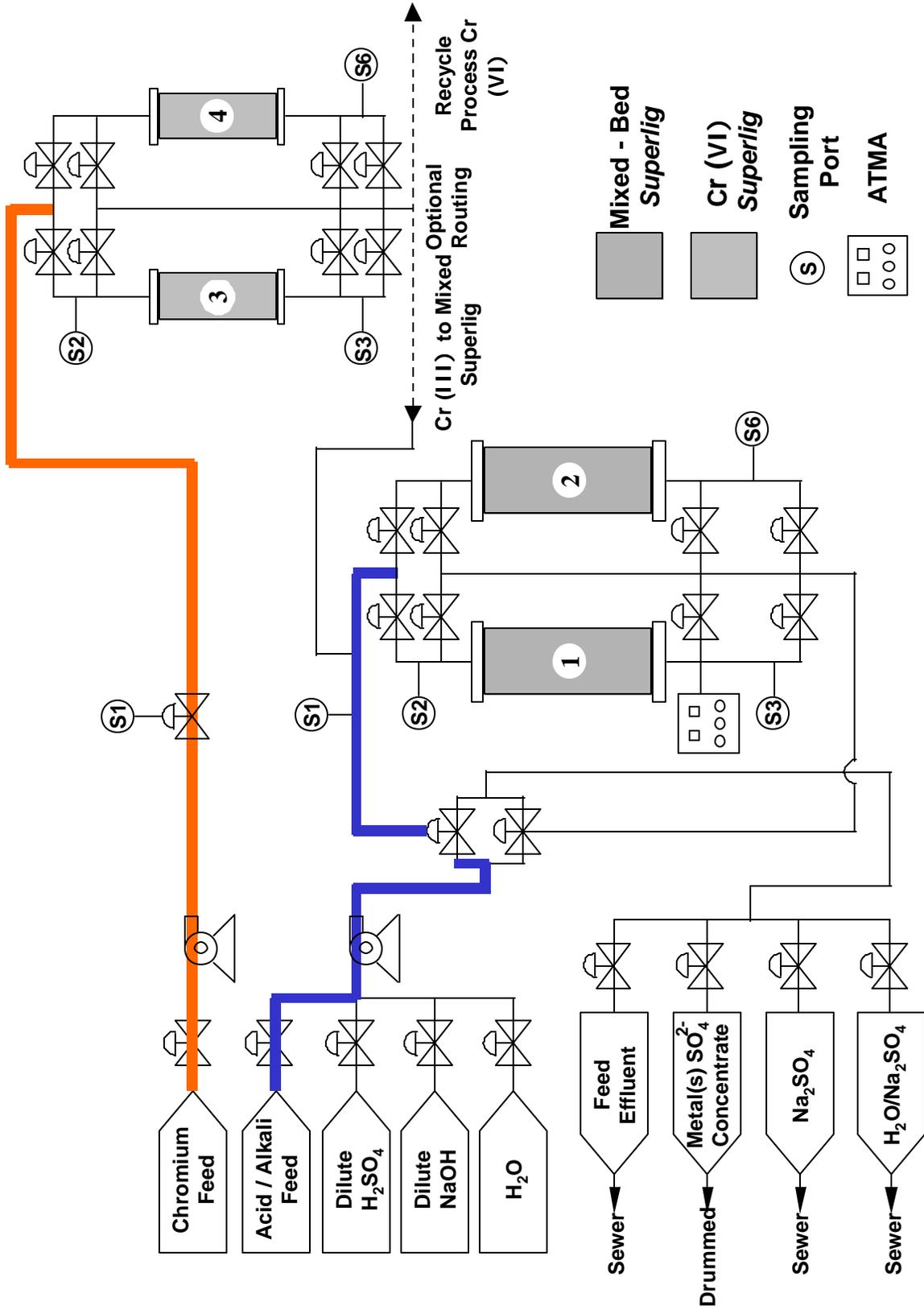


Figure 4-1 MRT for Treating Acid/Alkali and Chromium Waste Stream and Sampling Ports

### 4.2.1 Original Demonstration Skid Design

The original columns designed for the skid were to be full scale to be filled with 173 liter of *Superlig*<sup>®</sup> material. These columns were 1 and 2 as shown on Figure 4-1. Test runs 1, 2, and 3 were conducted with columns 1 and 2.

### 4.2.2 Modified MRT Skid Design

The skid was modified in July 2002 for two smaller columns (3 and 4) as shown on Figure 4-1. All further operational testing was run using columns 3 and 4. See Appendix C for details of modification of the MRT skid.

#### (a) Processing

The acid/alkali operational test runs were processed with wastewater from the PSNS neutralization tank after cyanide oxidation and chromium (VI) reduction to chromium (III). For Cr (VI) a series of operational test runs were performed with only columns 3 and 4 loaded with chromium (VI) *Superlig*<sup>®</sup> 307; the reduction step from Cr (VI) to Cr (III) was not done. An operational test run was defined as completely processing 5,000 gallons of the wastestream, elution of the column, washing the column, and regenerating the column. Columns 3 and 4 were filled with 17.4 liters of *Superlig*<sup>®</sup>, forming a packed bed column with sufficient depth to retain a 2 to 1 aspect ratio to assure that no channeling took place. The IBC patented *Superlig*<sup>®</sup> materials used were *Superlig*<sup>®</sup> 327 for all metals in the acid/alkali waste stream to be recovered (Cu, Pb, Ag, Ni, Cd, Zn) and *Superlig*<sup>®</sup> 310 for chromium (III). For Cr (VI), *Superlig*<sup>®</sup> 307 was used for mono-metal recovery/recycle in later operational test runs 20, 21, 22, and 23. After removing the *Superlig*<sup>®</sup> 327 and *Superlig*<sup>®</sup> 310, the columns 3 and 4 were loaded with the chromium (VI) *Superlig*<sup>®</sup> 307. The chromium operational tests runs for 20, 21, 22, 23 were run on the modified skid without any further design changes. The sequence of operational test runs is given in Table 4-2 and the heavy metals that were recovered.

#### (b) Operational Parameters

After the operational test runs 1,2, and 3, optimal demonstration parameters were 1500 gals/12 hrs with a flow rate of two gal/min with breakthrough estimated +/- 500 gallons. The column-loading rate was 4.06 gal/min/ft<sup>2</sup>. The optimal regeneration flow rate was 0.5 gal/min. The loading flow rate allowed the selected metals to have a single breakthrough so that the trailing column could remain well below compliance levels.

#### (c) Breakthrough Times for Demonstration of Pilot Scale Plant

With the low concentrations at PSNS, it would have taken weeks to do each operational test run to get the breakthrough time if the full sized columns (1 and 2) had been used. Therefore, the lead column breakthrough was calculated and sized to happen between 500 and 1500 gals for the lead column.

**Table 4-2. Sequence of Operational Test Runs and Metal Recovered**

Skid Design	Run	MRT Column	Material <i>Superlig</i> <sup>®</sup>	Metals Recovered	Metals Not Recovered
Original	1	1&2	327 & 310	Cr (III), Ni, Cu, Ag, Pb, Zn, Cd	Cr (VI)*
Original	2	1&2	327 & 310	Cr (III), Ni, Cu, Ag, Pb, Zn, Cd	Cr (VI)*
Original	3	1&2	327 & 310	Cr (III), Ni, Cu, Ag, Pb, Zn, Cd	Cr (VI)*
Modified	4	3&4	327 & 310	Cr (III), Ni, Cu, Ag, Pb, Zn, Cd	Cr (VI)*, Mg
Modified	8	3&4	327 & 310	Cr (III), Ni, Cu, Ag, Pb, Zn, Cd	Cr (VI)*, Mg, Ca, Na, K
Modified	9	3&4	327 & 310	Cr (III), Ni, Cu, Ag, Pb, Zn, Cd	Cr (VI)*, Mg, Ca, Na, K
	10	3&4	327 & 310	Cr (III), Ni, Cu, Ag, Pb, Zn, Cd	Cr (VI)*, Mg, Ca, Na, K
	20	3&4	307	Cr (VI)	Cr (III) K, Mg, Ca, Na, Ni, Cu, Ag, Pb, Zn, Cd
	21	3&4	307	Cr (VI)	Cr (III) K, Mg, Ca, Na, Ni, Cu, Ag, Pb, Zn, Cd
	22	3&4	307	Cr (VI)	
	23	3&4	307	Cr (VI)	Cr (III) K, Mg, Ca, Na, Ni, Cu, Ag, Pb, Zn, Cd

\*Cr (VI) tested using Spectrophotometer DR 2000

Note: Not all test runs analyzed for K, Mg, Ca, Na..

### 4.3 Sampling Procedures

Data collection followed the general guidance in PSNS's NAVSHIPYDPUGETINST P5090.26a. This sampling plan was coordinated with PSNS Code 134, NFESC, and IBC Advanced Technologies. The sampling plan matrix for the acid/alkali wastestream was designed to determine: (1) efficiency in recovery of metals using MRT system compared to other absorbent metal ion technologies and (2) efficiency of MRT *Superlig*<sup>®</sup> columns. The chromium wastestream was analyzed using the same approach.

(1) To determine if the MRT met current compliance limits for discharge and future limits under the anticipate new ruling for Metal Products & Machinery (MP&M) proposed Samples were analyzed at locations shown in Table 4-2 The actual sampling locations are shown Figure 4-1.

(2) To determine efficiency across MRT column, samples were taken at ports as shown in Table 4-2 for capacity. The column capacity was obtained by determining the loading rates (gram metal per kg ligand) at column equilibrium. Breakthrough was determined using the ATMA. The number of bed volumes to strip the columns with sulfuric acid was determined by the metal concentration reaching low ppm levels at ports S3 and S6.

**Table 4-3. MRT Sampling Port Parameters for Acid/Alkali Columns**  
(see Figure 4-1 for locations)

<b>Parameter</b>	<b>Column 1</b>	<b>Column 2</b>
<b>Discharge Limits</b>	S1, S2 & S3	S6
<b>Capacity</b>	S3 & S6	S3 & S6
<b>Breakthrough</b>	S3	S6
<b>Bed Volume</b>	S2 & S3	S3 & S6

#### 4.4 Analytical Procedures

##### 4.4.1 Field Analytical Equipment

An automated trace metal analyzer (ATMA) was used to determine the breakthrough of copper during the test runs of the MRT. The ATMA was developed under a separate ESTCP program by SPAWAR (ref. 9) utilizes potentiometric stripping analysis (PSA). The ATMA was capable of reading copper concentration to 10 ppb. The automated trace metal analyzer was used as a diagnostic tool in determining if the engineering design of the MRT columns was correctly configured. Field analytical equipment included pH meters, conductivity, and oxidation-reduction potential (ORP). The HACH field spectrophotometer DR 2000 was used for copper and hexavalent chromium for breakthrough determination. In addition, EM Quant Test Strips for rapid testing and semi-quantitative screening of ions in the solution were used on site. The following EM Quant Test Strips were used for analyzing Cu, Fe, Pb, Ni, and Zn.

##### 4.4.2 Selection of Analytical Laboratory

The PSNS Analytical Laboratory was selected to perform the analysis for the project. The analytical laboratory is accredited by the State of Washington Department of Ecology, #F001.

##### 4.4.3 Selection of Analytical Method

**(a) Acid/Alkali:** The primary analytical method used by PSNS Analytical Laboratory is Method 200.7. This analysis method is for metal by Inductively Coupled Plasma (ICP) from the 200 Series under the Clean Water Act. This method is documented in "Methods for Chemical Analysis of Water and Waste, EPA-600014-79-020, revised March 1983. The updated version for this project is found in the Federal Register, Title 40 - Part 136 - 136 - Appendix C to Part 136 Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis for Water and Wastes, Method 200.7, August 15, 1990.

**(b) Chromium:** Method 200.7 was used to determine chromium (total). For hexavalent chromium, Method 301B from "Standard Methods for Examination of Water and Wastewater, Vol 16, edition 1985 was used.

## 5.0 Performance Assessment

### 5.1 Performance Data

Due to the high security location within the shipyard, operational test runs could not be continuously run due to staffing requirements. A number of operational test runs were delayed or cut short due operation and maintenance problems that required immediate attention in the IWPF operations. Because of these internal processing conditions within the IWPF, several more operational test runs were not completed within the time frame of the project for both the acid/alkali and chromium system. However, the number of combined acid/alkali and chromium is sufficient to evaluate MRT for specific compliance and pollution prevention needs within DoD and Navy activities. In most of the operational runs, the silver was very low (below ICP detection limits) due to the silver cyanide plating process being off-line for a MILCON upgrade.

#### 5.1.1 General MRT Results

The spreadsheets for analytical results are found in Appendix B of this technical report. Operational test runs 5, 6, and 7 samples were not analyzed due either the test run being cut short or to known problems that the cost of the analysis could not justified.

#### (a) Variation of Influent Wastestreams

Acid/Alkali Metal Concentration and Contaminants: The operational test runs influent wastestream had varying metals concentration as shown in Table 5-1.

**Table 5-1. Concentration of Influent Wastestreams**

Run	Metal	Zn	Pb	Cu	Ag	Cd	Ni	Cr Total	Cr VI	Cr III
4	Acid/Alkali	9.78	0.612	16.08	0.027	0.77	5.05	8.23		
7	Acid/Alkali	9.34	0.65	16.48	0.03	0.74	4.77			5.49
8	Acid/Alkali	178.60	3.99	189.90	0.02	15.93	25.96			22.49
9	Acid/Alkali	76.60	1.78	80.70	0.01	7.15	13.58			8.41
10	Acid/Alkali	172.00	3.96	196.80	0.20	15.43	26.90			22.26
20	Chrome	1.24	0.24	9.70	0.00	0.00	5.44	123.00	70.20	
21	Chrome	0.39	0.08	2.70	0.00	0.02	1.40	36.50	31.73	
22	Chrome	1.31	0.10	8.99	0.00	0.03	7.42	84.46	15.00	
23	Chrome	4.29	< 0.02	51.80	0.03	0.62	7.42	50.40	47.60	

The organic components reflected those that were found in the PSNS Metal Preparation Facility as shown in Appendix D, Table 5. *Wastestream Constituents, Including Incidental Contaminants*. (Ref personal communication with Walter Hunter) Table 5-1 shows the influent wastestream metal concentrations for the acid/alkali for operational test runs 4, 8, 9, and 10. Operational test runs 8, 9, and 10 were from the same batch of wastewater, but operational test

run 9 was diluted by ~43% with shipyard potable water before processing through the MRT. A concentrated pickling bath was added to the IWPF miscellaneous tank prior to filling the MRT storage tanks for runs 8, 9, and 10. For operational test runs 8, 9, and 10, the pickling bath introduced chelating agents, surfactants, and low concentrations of organics. The formulation of the pickling bath is shown in Table 5-2.

**Table 5-2. PSNS Metal Finishing Facility Pickle Bath Formulation**

<b>Pickle Bath Solution</b>	
Hydrochloric Acid (gal)	85.3 F Oz/G
Diethythiorea (lbs)	0.53 Oz/G
Rodine-50 (gal)	0.14 F Oz/G
Detergent (gal)	0.12 F Oz/G
Water	Balance

Chromium Metal Concentration and Contaminants: The concentration of influent hexavalent chromium is shown in Table 5-1. Operational test runs 20, 21, and 22 were all completed from the same batch of wastewater pumped to the MRT storage tanks. Run 21 was diluted with shipyard water to ~30% the original concentration. Operational test run 23 was run several months later with a completely different batch of wastewaters.

**(b) Effluent Data, Extraction, and Mass Balance Data**

Acid/Alkali: In Table 5-3, the maximum monthly discharge limits, influent, effluent, percentage extraction, and the mass balance data are shown for operational test runs 4 and 9. In operation test run 4, the lead (Pb) percentage of extraction from the wastestream was very high, but the mass balance indicated that Pb was retained on the MRT column when eluted with 4 M sulfuric acid. This low mass balance may be due to very low concentrations of lead (Pb) in the wastewater "skewing" the calculations. Alternatively, the solubility of Pb in sulfuric acid may be low due to the formation of insoluble lead sulfate. If this is the case, the column can be eluted with HCl in order to avoid any complex formation. However, in operational test run 9, the percent extraction and mass balance were both considerably higher than in operational test run 4. The percent extraction for the rest of the metals averaged ~ 98%. The low value of 2.5% extraction for Mg is expected with its low Log K for selectivity, see Table 2-1. The mass balances were acceptable with the exception of Pb, which may be an anomaly.

Chromium: The effluent analytical results are shown on Table 5-4 where both chromium (total) and hexavalent chromium for operational test run 23. Cr (III) is calculated by subtracting hexavalent chromium from the chromium (total). There is no analytical test for Cr (III). Since the Federal regulatory maximum monthly limits are 2.77 for chromium (total), both Cr (III) and Cr (VI) must be recovered by the MRT system. *Superlig*<sup>®</sup> 310 for chromium (III) and 307 for chromium (VI) were evaluated in MRT system. Chromium (III) results are shown in the acid/alkali section. The hexavalent chromium operation testing showed that the chromium (VI) ion was preferentially being extracted by the *Superlig*<sup>®</sup> 307. The ICP 2007 Method was used to determine chromium (total) (total) and Cu, Pb, Ni, Zn, Ag, and Cd. The hexavalent chromium was analyzed by Method 301B. See Figure 5-2 for chromium (VI) performance plot. At

equilibrium, calculated amount of Cr (III) ranges between 9 to 14 ppm as shown on Table 5-5. The amount of chromium (III) in the PSNS wastestream is expected to be in this range as found in Table 2-2, Section 2.2.

### (c) Breakthrough Data

Acid/Alkali: Breakthrough and concentration data are shown in Table 5-6 for operational test runs 8, 9, and 10. It is noted that there is a close correlation between concentration and breakthrough. Because this was a pilot demonstration, breakthrough came after much earlier than for a full-scale system as described in Section 4.2.2. Operational test run 9 was diluted with shipyard water to approximately half the concentration. The breakthrough for the more dilute chromium wastewater is also double that of operational test run 8 and 10. Therefore, the linear scale up factor as described in the demonstration plan is validated.

Chromium: For chromium (VI) breakthrough on column 4 occurs at approximately half the volume of column 3. See Table 5-7. In contrast, the acid/alkali breakthrough volume for column 4 was three times the initial breakthrough in column 3. See Table 5-6.

Breakthrough Data Correlation to Log K: Breakthrough is also correlated to the Log K. See Table 5-8. Due to the high Log K for Cu and Cr (III), they showed retention on column 3 more than column 4. For the other metals, concentrations were slightly higher on column 4 than column 3.

Special Breakthrough Considerations for Mixed Bed Columns: For a single metal column, the difference in Log K would not be relevant. For a design of a single *Superlig*<sup>®</sup> column, the number of regenerations would specify the size of the column. For a mixed *Superlig*<sup>®</sup> packed bed column as demonstrated at PSNS, the *Superlig*<sup>®</sup> mixture needs to be increased for zinc as shown in the data from operational runs 8, 9, and 10. The plots for these operation operational test runs show breakthrough for Zn (port 3) before the rest of the metals. In Appendix E Table 2 shows that the highest metal concentrations when averaged over a four-year period are Zn, Cu, and Cr (total). If the wastestream changes over time, new *Superlig*<sup>®</sup> material can be added to the mixed packed bed column to accommodate these changes in metal concentration.

### (d) Regeneration

Acid/Alkali: Figure 5-6 shows for operational run 4 that the column was regenerated with a clean elution curve within two bed volumes.

Chromium: In operational test runs 20 and 21, the columns were eluted with 4 M sulfuric acid. All the chromium (VI) was converted to Cr (III). In operational test run 222 and 23, the columns were eluted with 1 M NaOH, the Cr (VI) ion is maximized in the first two bed volumes as shown in Figure 5-7.

### (e) Non-adsorption of Base Metals (Na, K, Ca, and Mg) on MRT Column

Acid/Alkali: Unlike ion exchange where all the sites are occupied, the *Superlig*<sup>®</sup> materials are selective for specific heavy metals. As shown on Table 5-9, base metals pass through the MRT column as predicted by the affinity constants. Figure 5-1 graphically shows that magnesium, with low affinity constant of 0.02, is passing through the column. Analytical data from operational #10 show that these base metals are passing through the MRT columns 3 and 4.

**Chromium:** For the chromium system, operational test runs 20 and 21 showed that the base metals were not adsorbing onto the Chromium (VI) *Superlig*<sup>®</sup> 307. See Table 5-10. Chromium (VI) *Superlig*<sup>®</sup> 307 is specific for this metal anion. In addition, all other heavy metals (Cu, Ni, Pb, Cd, Zn, Ag, passed through the column as shown in Table 5-4. Operational test runs 22 and 23 were eluted with sodium hydroxide, instead of 4 M sulfuric acid, so the sodium content of the eluent was very high, that is higher than expected from sodium found in the wastestream or shipyard water. The sodium dichromate could be recycled to the process bath.

**(f) Special Tests**

During the demonstration, there were special tests that are discussed in Appendix B. The following tests were performed:

- Fluoride content in the shipyard potable water
- Shipyard potable water for metals concentration
- Metal concentration in processed sludge
- Particle sizing in the wastestream
- Organic carbon in pore water of the MRT columns

**Table 5-3. Acid/Alkali MRT Effluent Data, % Metal Extraction, and Mass Balance Operational Test Runs 4 and 9**

<b>Acid/Alkali Operational Test Run 4</b>					
<b>Metal</b>	<b>PSNS Max Limits Monthly (mg/l)</b>	<b>MRT Influent (mg/l)</b>	<b>Processed Effluent (mg/l)</b>	<b>Extraction (%)</b>	<b>Mass Balance</b>
<b>Mg</b>	NA	18.10	17.600	2.5	98.00
<b>Cd</b>	0.11	0.70	0.005	99.40	71.20
<b>Cr (III)</b>	1.71	6.50	0.068	98.90	73.50
<b>Cu</b>	2.07	16.40	0.010	99.90	103.40
<b>Ni</b>	2.38	4.80	0.002	99.80	91.10
<b>Pb</b>	0.43	0.70	0.099	98.90	26.90
<b>Zn</b>	1.48	9.40	0.099	98.90	91.10
<b>Ag</b>	0.24	ND	NA	NA	NA
<b>Acid/Alkali Operational Test Run 9</b>					
<b>Mg</b>	NA	NA	NA	NA	NA
<b>Cd</b>	0.11	8.10	0.069	41.40	85.60
<b>Cr (III)</b>	1.71	52.00	0.079	91.00	83.00
<b>Cu</b>	2.07	65.30	0.411	80.00	115.00
<b>Ni</b>	2.38	14.17	0.078	53.00	85.90
<b>Pb</b>	0.43	1.87	ND	80.00	68.30
<b>Zn</b>	1.48	79.60	0.724	41.00	93.00
<b>Ag</b>	0.24	NA	NA	NA	NA

**Table 5-4 Chromium (VI) Effluent and Equilibrium Data for Run 23 \*\*\***  
 ICP Method 200.7 for Chromium (total) and Method 301B for Hexavalent Chromium

Identity	Gals	Zn	Pb	Cu	Ag	Cd	Ni	Total Cr	Cr VI
23-3-27-0010	10	1.65	<0.10	1.36	0.02	0.25	2.44	5.31	0.01
23-3-27-0020	20	3.92	<0.10	28.73	0.04	0.54	6.76	8.73	0.01
23-3-27-0040	40	4.03	<0.10	49.65	0.05	0.57	6.92	9.94	0.02
23-3-27-0080	80	3.96	<0.10	51.86	0.04	0.57	6.78	9.57	0.02
23-3-27-0120	120	3.98	<0.10	52.84	0.03	0.56	6.77	9.82	0.03
23-3-27-0160	160	4.02	<0.10	53.09	0.04	0.59	6.87	10.72	0.02
23-3-27-0200	200	4.04	<0.10	53.77	0.03	0.58	6.93	12.20	0.02
23-3-27-0240	240	4.01	<0.10	53.60	0.04	0.58	6.90	13.62	0.03
23-3-27-0480	480	4.00	<0.10	53.67	0.04	0.58	6.87	22.09	6.50
23-3-27-0720	720	4.00	<0.10	53.95	0.04	0.57	6.81	31.53	16.43
23-3-28-1080	1,080	4.04	<0.10	53.76	0.04	0.58	6.89	44.68	30.61
23-3-28-1560	1,560	4.00	<0.10	53.89	0.04	0.57	6.87	52.69	39.50
23-3-28-1920	1,920	4.01	<0.10	53.25	0.04	0.57	6.83	55.11	40.67
23-3-29-2040	2,040	4.00	<0.10	44.22	0.04	0.58	6.83	53.94	41.88
23-3-29-2160	2,160	3.94	<0.10	50.63	0.04	0.57	6.71	52.77	43.70
23-3-29-2400	2,400	4.03	<0.10	53.08	0.04	0.59	6.89	52.95	41.03
23-3-29-2640	2,640	4.00	<0.10	52.64	0.04	0.57	6.82	52.31	40.55

\*\*\* Gray shaded area is where MRT column in equilibrium with Cr ions

**Table 5-5. Chromium (VI) Equilibrium Data for Run 23 in PSNS Equilibrium Data**

Cr (Total)	Cr (VI)	Cr (III)
55.11	40.67	14.44
53.94	41.88	12.06
52.77	43.70	9.07
52.95	41.03	11.92
52.31	40.55	11.76

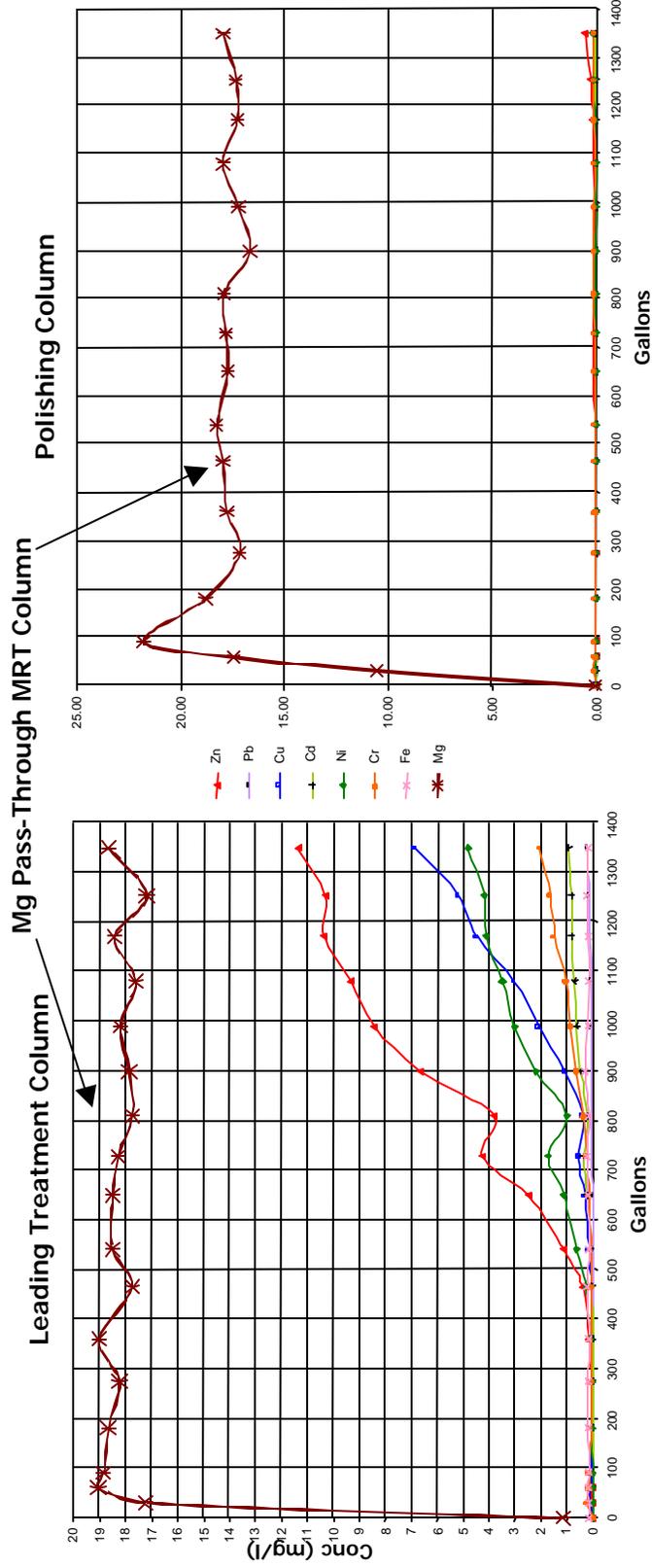


Figure 5-1 Acid/Alkali Performance Plot of Lead/Trail Column for Operational Test Run #4

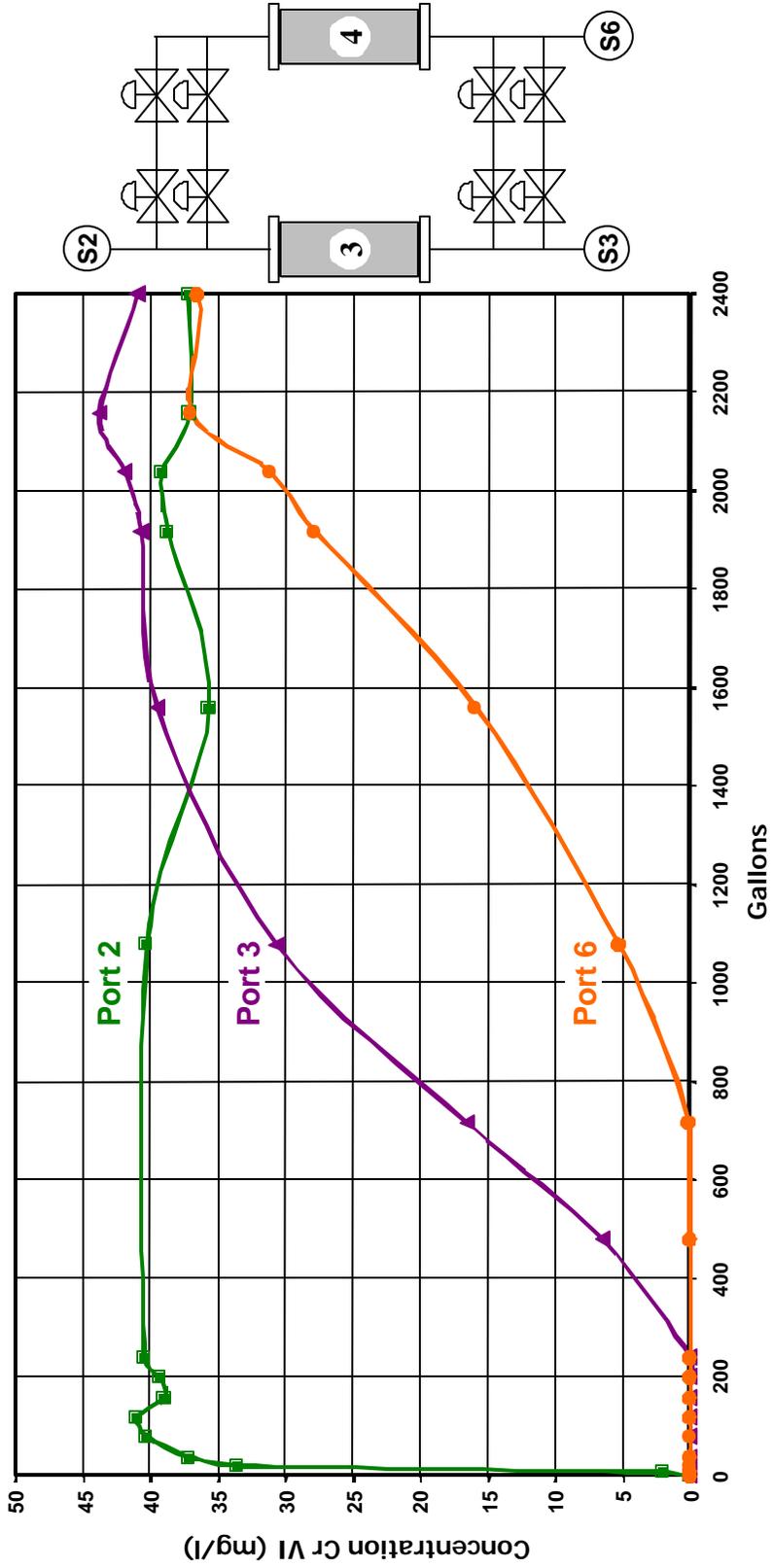


Figure 5-2 Chromium VI Performance Plot for Test Run #23

MRT - Chrome (Total), Chrome (III), and Cr (VI)  
Run 23

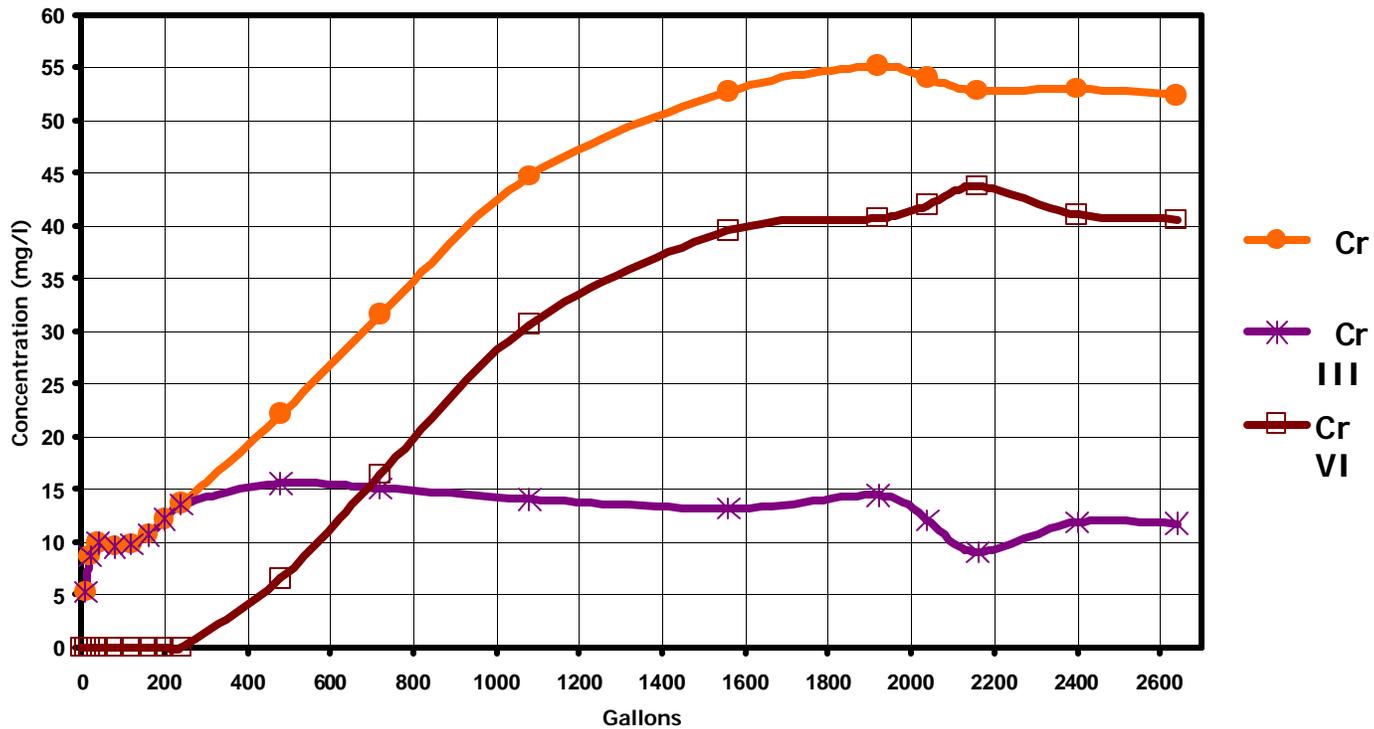


Figure 5-3. Plot of Cr (VI), Cr (III) & Total Cr plotted. (Total Cr<sub>T</sub> - Cr (VI) = Cr (III))

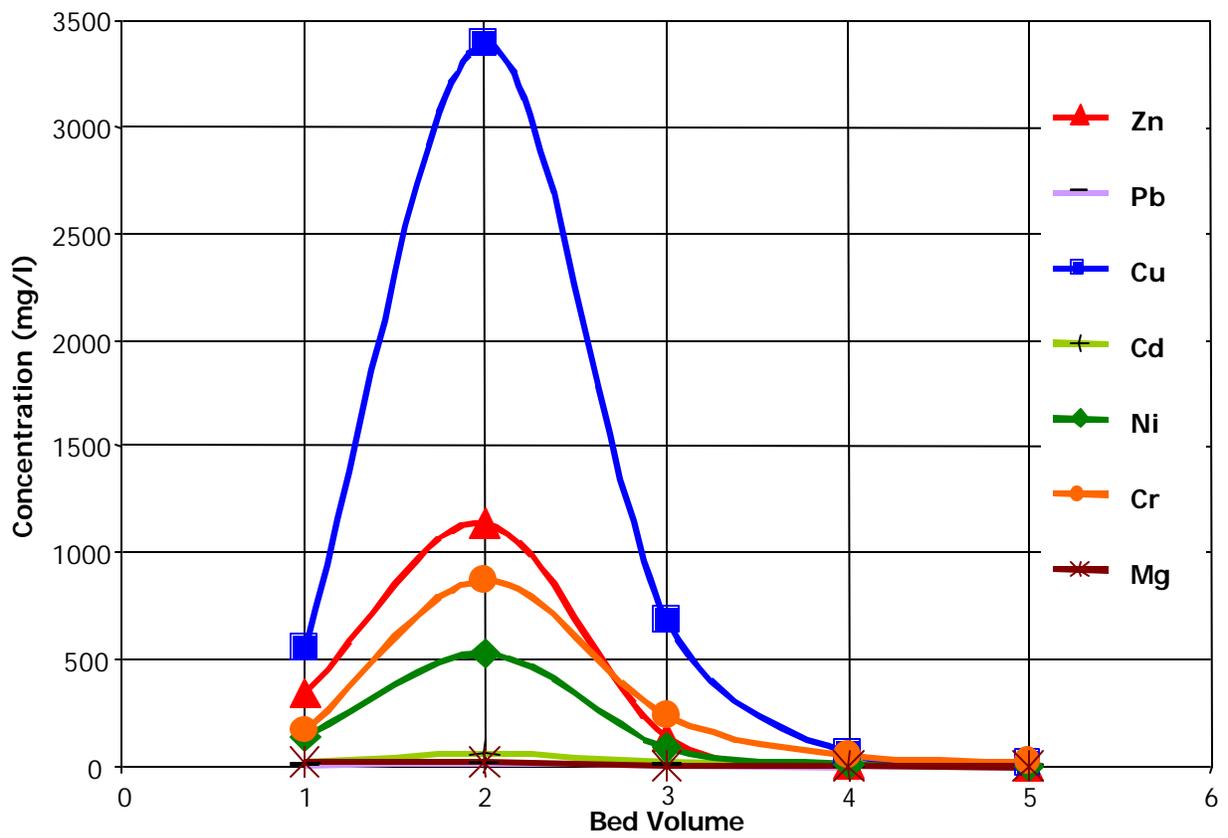


Figure 5-4. Acid/Alkali Regeneration Plot for Operation Test Run 4

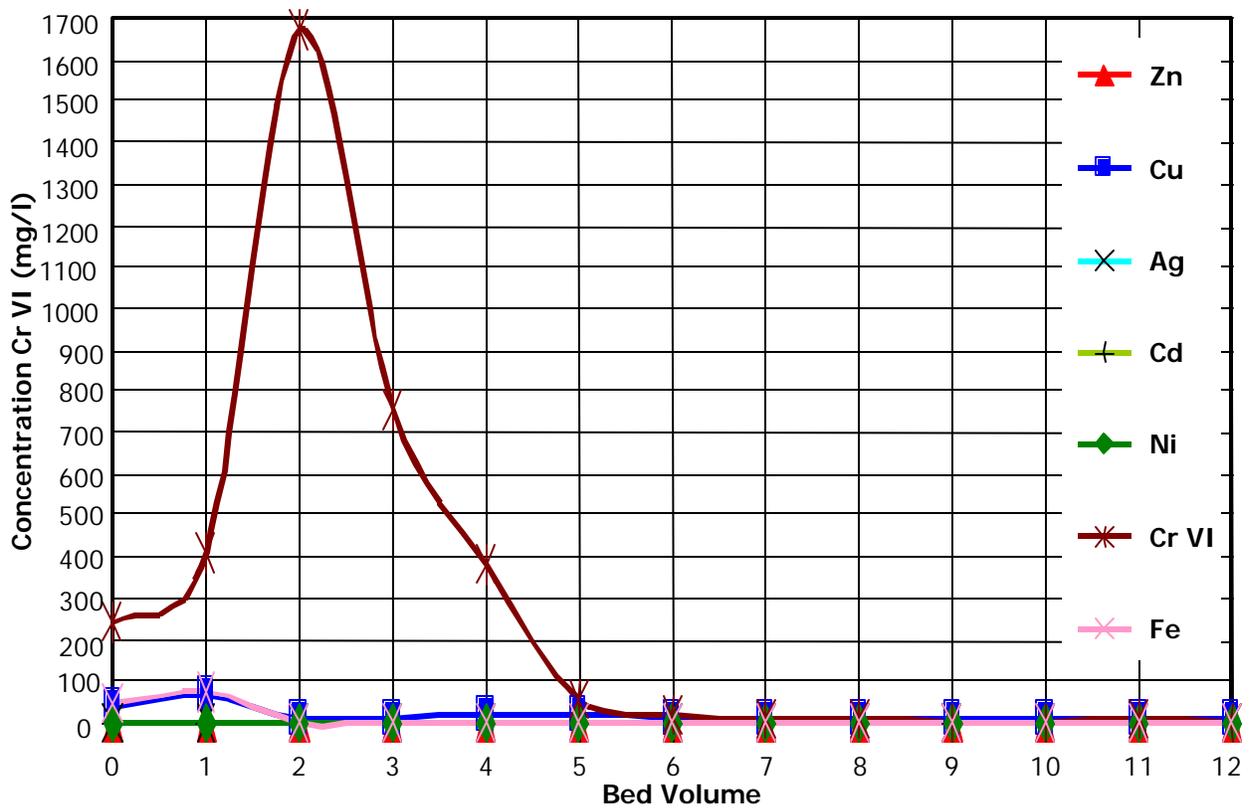


Figure 5-5. MRT 1M NaOH Regeneration for Chromium Recycle to Process, Run 23

**Table 5-6. Acid/Alkali Correlation of Concentration and Breakthrough of the Columns Acid/Alkali Operational Test Runs 8, 9, and 10**

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr (III)
8-2-18-0012	12	178.56	3.99	189.9	0.19	15.43	25.97	22.49
10-2-20-0036	36	172.02	3.96	195.53	0.2	15.43	26.19	22.26
9-2-19-0009	9	76.7	1.78	80.7	0.09	7.75	13.58	8.41
Number of Gallons Processed Before Breakthrough of Column 3 and 4								
Run 8-C3	Run 8-C4	Run 9-C3	Run 9-C4	Run 10-C3	Run 10-C4			
45 gals	150 gals	75 gals	300 gals	45 gals	145 gals			

**Table 5-7 Chromium (VI) Correlation of Concentration and Breakthrough of the Column**

Run	Influent Concentration (avg)	Breakthrough Column 3	Breakthrough Column 4
20	70.2	300	660
21	31.7	410	830
23	47.6	225	480

**Table 5-8 Acid/Alkali Mixed Bed Superlig<sup>®</sup> Adsorption of Metals and Correlation to Log K**

Metals	Ag	Cd	Zn	Pb	Ni	Cu	Cr (III)
Log K	13.80	13.80	14.40	14.40	17.00	22.00	30.00
Run 8 C3	ND	17.00	234.00	11.50	48.60	1440.00	178.00
Run 8 C4	ND	33.00	470.00	10.00	90.00	807.00	68.00
Run 9 C3	ND	38.90	520.00	20.20	10.30	1710.00	164.00
Run 9 C4	ND	46.50	595.00	17.60	11.30	1410.00	93.00
Run 10 C3	ND	47.00	598.00	11.70	98.50	1040.00	91.10
Run 10 C4	ND	55.00	737.00	11.20	98.70	813.00	79.40

**Table 5-9. Alkali/Alkaline Base Metals Non-Retention by MRT Column**

Identity Port 2	Gallons	K	Mg	Ca	Na
10-2-20-0018	18	198.62	25.16	139.39	2,999.18
10-2-20-0108	108	198.82	26.67	139.78	3,030.76
10-2-20-0477	477	203.68	27.39	148.52	3,163.46
Identity Port 3	Gallons	K	Mg	Ca	Na
10-3-20-0018	18	226.15	2.07	183.61	3,177.29
10-3-20-0108	108	213.87	35.91	146.42	3,075.35
10-3-20-0477	477	203.33	38.84	232.67	2,623.29
Identity Port 6	Gallons	K	Mg	Ca	Na
10-6-20-0018	18	27.26	4.00	69.16	3,678.00
10-6-20-0108	108	230.86	13.67	163.21	3,141.61
10-6-20-0477	477	196.60	30.62	165.95	3,180.97
Identity Tank	Gallons	K	Mg	Ca	Na
10-1-20-0423	423	207.08	32.77	190.36	3,110.99
Identity (Eluents)		K	Mg	Ca	Na
10-C3-20-ELU		21.50	8.77	146.97	41.03
10-C4-20-ELU		24.62	14.90	187.04	122.26

**Table 5-10. Base Metals Non-Retention on Hexavalent Chromium *Superlig*<sup>®</sup>**

Identity Port 2	Gallons	K	Mg	Ca	Na
20-2-22-0020	20	238.49	14.12	17.40	1,233.61
20-2-22-0510	510	261.86	15.77	16.04	1,401.72
20-2-23-1020	1020	224.41	13.20	17.72	1,252.90
Identity (Eluents)		K	Mg	Ca	Na
20-3-ELU-3BV		20.81	2.61	5.52	10.44
20-3-ELU-3BV		19.84	3.05	4.92	19.55

## 5.2 Technology Comparison

Some of the data required for comparative costing will be based on efficiency of metal removal from the wastestream relative to ion exchange. There were two kinds of metal removal efficiencies to determine during the MRT demonstration.

### 5.2.1 Efficiency of MRT System to Exceed Discharge Limits

The metal removal efficiency of the MRT to meet regulatory limits is based on its capability to remove metal from the neutralization tank to effluent holding tank as shown on Figure 4-2. Table 5-1 shows that the leading column extracted all metals. The polishing column was maintained well below regulator discharge level even after breakthrough of the lead column.

### 5.2.2 Efficiency Based on MRT Column Capacity

The second metal removal efficiency is across the *Superlig*<sup>®</sup> column itself. It is easy to vary the recycle time, but not the adsorption capacity of the *Superlig*<sup>®</sup> column. Table 5-11 shows that the amount (grams) of ion exchange resin required is double that of MRT *Superlig*<sup>®</sup>.

**Table 5-11 Comparison of *Superlig*<sup>®</sup> Sites vs. Typical Ion Exchange**

Heavy Metals	Metal (mmoles) in Processed Wastewater	Parameter Measured	Bench Scale	Actual PSNS
Cd	33	Mixed Bed <i>Superlig</i> <sup>®</sup> Capacity (mmoles/gram)	1.71	0.8
Cr	637			
Cu	1,321	Typical Ion Exchange (mmoles/gram)	1.0	—
Ni	416			
Pb	16	Grams of <i>Superlig</i> <sup>®</sup> Required	1,552	3,317
Zn	231			
<b>Total: 2,654 mmoles</b>		Grams of Ion Exchange Required	6,448	—
<b>Earth Metals</b>				
Mg	3,794			
Ca	-			
Na	-			
K	-			
<b>Total: 3,794 mmoles</b>				

As stated in Section 2.1, the benign cations would pass through the column thus increasing the number of sites for the contaminant metals. The number of available sites is less due to the Mg taking up sites as well as heavy metal cations, i.e., (2,654 + 3,704 = 6448 number of sites taken on Ion Exchange column). The capacity during operational run 4 was 0.8 mmoles/gm. The metal capacity of typical ion exchange is 1 mmole/gm, but due to the presence of Mg<sup>2+</sup> the metal capacity is reduced to 0.44 mmoles/gm. Other base metal (Ca<sup>2+</sup> Na<sup>+</sup> and K<sup>+</sup>) will reduce capacity linearly. However, these other base cations were not analyzed for operational run 4.

## 6.0 Cost Assessment

### 6.1 Cost Performance

#### 6.1.1 Demonstration Cost

**(a) Environmental Cost Analysis Methodology:** The Environmental Cost Analysis Methodology (ECAM) (Ref 13) was utilized for cost assessment for the MRT pilot demonstration at PSNS. The ECAM analysis calculates the incremental profitability of the MRT technology relative to the conventional precipitation processes (called base process) as presently being used by DoD's IWTPs. During the demonstration period at PSNS, a MILCON was underway at the Metal Finishing Facility. Therefore, a lower volume of wastewaters was being processed at the IWPF. In Table 6-2, the cost estimate used for annual waste management was averaged over 14 DoD facilities to be \$67,000 (Ref 6). The MRT technology does not require a large space for neutralization/precipitation and flocculation/clarification units. Due to smaller footprint (10' x 10' x 15') of the MRT system, cost savings may be realized in lower infrastructure, which is not taken into account in this report. For the pilot demonstration at PSNS, costs for additional chemicals to treat wastewaters non-amenable to precipitation were not estimated. However, the costs incurred for copper chelated wastewaters are illustrated in Table 5.3 for NAS North Island IWTP. The ECAM analysis did not take into account the escalating costs of land disposal for hydroxide sludges or future liability, but a discussion of the matter is found in Section 7.1.1. There were no labor savings due to the fact that two operators are required to be present for safety reasons in the PSNS IWPF regardless of the work performed. Other non-variable cost factors that are not shown include but not limited to such cost items as document maintenance, worker compensation, health exams, compliance audits, and manifesting.

**(b) MRT Cost *Superlig*<sup>®</sup> and Associated Equipment:** The size of the pilot demonstration columns at PSNS was 17.14 liters each as shown in Figure 4-1 for columns 3 and 4. The mixed *Superlig*<sup>®</sup> 327 and *Superlig*<sup>®</sup> 310 material in 1999 cost \$1800/kilogram for the quantities used in the pilot plant demonstration. For larger size MRT systems, the unit cost for the *Superlig*<sup>®</sup> will be less due to economies of scale in production. In addition, depending on the support system for *Superlig*<sup>®</sup> material the conversion factor may vary, i.e.: for silica supported *Superlig*<sup>®</sup> it is 0.45 kilograms/liter and for polyacrylates supported 0.21 kilograms/liter. For the pilot demonstration, the mixed bed *Superlig*<sup>®</sup> 327 and *Superlig*<sup>®</sup> 310 are acrylate based so the cost calculated was \$12,958 (2 columns x 17.14 liters x 0.21 kilograms/liter conversion factor x \$1800/kilogram). In order to scale up to full size MRT system for PSNS, the volume of columns (1 and 2) would be 173 liters each. The cost for the *Superlig*<sup>®</sup> 327 and 310 would be \$130,788 (2 x columns x 173 liters x 0.21 kilograms/liter x \$1800/kilogram). The cost for the chromium (VI) *Superlig*<sup>®</sup> 307 is \$111K for columns 3 and 4 with no scale up for the PSNS MRT demonstration system. (Please note that the cost for the Cr (VI) *Superlig*<sup>®</sup> 307 is significantly less costly than at the time of the PSNS pilot demonstration.)

### 6.1.2 Cost for Full Scale MRT System

The costs for the PSNS demonstration are shown in Table 6-1. Some of the costs in gray were for the demonstration only and would not be incurred for a full-scale installation.

**Table 6-1 MRT Demonstration Costs at PSNS for Acid/Alkali and Chromium Wastestreams**

Direct Environmental Activity				Indirect Environmental Activity Costs		Other Costs	
Start-Up		Operation & Maintenance		Activity	Cost	Activity	Cost
Activity	Cost	Activity	Cost	Activity	Cost	Activity	Cost
Facility preparation and de-mobilization	\$50K	Labor to operate equipment	\$75K	Compliance audits (QA/QC)	\$5K	Overhead assoc. with process	NA
Equipment design	\$15K	Labor to manage hazardous waste		Document maintenance		Productivity/cycle time	NA
Equipment purchase (Hardware/Skid)	\$33K	Utilities	NA	Envr. Mgmt. Plan development and maintenance		Worker injury claims and health costs	NA
Installation	\$10K	Mgmt/treatment of by-products	\$10K	Reporting requirements			
Training of operators	\$9K	Hazardous waste disposal fees	\$5K	Test/analyze waste streams	\$25K		
Rental tanks	\$3K	Raw materials		Medical exams (includes loss of productive labor)	NA		
Modification to Skid	\$45K	Process chemicals	\$20K	Waste transportation (on and off site)			
Superlig <sup>®</sup> Material	\$74	Consumables and supplies	\$15K	OSHA/EHS Training			
Shipping Skid	\$10	Equipment maintenance	\$10K				
		Training of operators	\$3K				

The Environmental Cost Analysis Methodology (ECAM) (Ref 11) was utilized for cost assessment for the pilot demonstration at PSNS. The purpose of ECAM is to compare an existing or base process with the reported innovative technology alternative. See Table 6-2 for the cost assessment of the PSNS base process as compared with the MRT process for a full-scale industrial wastewater treatment plant.

**Table 6-2 ECAM Summary of Full-Scale MRT System Cost Assessment (1999)**  
(Based on 30,000 gallons/24 hours and historic range of metal concentration on Table 6-2)

<b>ECAM Cost Description</b>	<b>Base Process</b>	<b>MRT Process</b>
<b>IWTP Initial Investment Costs</b>		
Miscellaneous Tank	\$234,000	\$234,000
Final Effluent Tank	\$68,000	\$68,000
Cyanide Oxidation Unit	\$146,250	\$146,250
Chromium Reduction Unit	\$105,300	
Chromium (Cr VI) MRT System		\$144,000
Neutralization/Precipitation Unit	\$98,280	
pH Control System	\$15,000	\$15,000
Flocculation/Clarification Unit	\$108,810	
Acid/Alkali & Cr (III) Mixed Bed MRT System		\$340,000
Sludge Storage Tank	\$15,210	
Filter cake/Brine Storage	\$30,000	\$5,000
Belt Filter Press	\$117,000	
Pre-treatment Debris Removal		\$25,000
Post-treatment Polishing Sand Filters	\$30,000	
Installation (30% of equipment cost)	\$281,355	\$291,675
<b>Total Capital Costs</b>	<b>\$1,219,205</b>	<b>\$1,263,925</b>
<b>MRT Process - Initial Investment Costs Above Base Process:</b>		<b>\$44,720</b>
<b>IWTP Annual Operating Costs</b>		
Direct Materials	\$8,850	\$8,030
Utilities	\$55,650	\$55,650
Direct Labor	\$163,390	\$163,390
Waste Management**	\$67,000	\$1,600
Regulatory Compliance	\$27,500	\$27,500
Revenues (By-Product)	\$0	(\$6,800)
<b>Total Annual Operating Costs</b>	<b>\$322,390</b>	<b>\$249,370</b>
<b>MRT Process - Cost Savings Over Base Process</b>		<b>\$73,020</b>
<b>Net Present Value</b>	<b>-\$4,001,118</b>	<b>-\$3,415,748</b>
<b>ECAM Analysis: Discount Rate 2.7%, Lifetime 10 yrs, Payback 9 years</b>		

\*\*Personal Communication from Tinker Air Force Base for DoD statistics average over 14 DoD Industrial Wastewater Treatment Plants (Ref 15). Other data from Refs 10, 12, and 13.

## Point Source Cost for Chromium MRT System

As shown on Table 1 in Appendix D, the chromium wastestream is about ~34% of the total wastewaters being processed by the PSNS IWPF. Therefore, the waste management cost was cut by 1/3 in the estimate in Table 6-3 from those in Table 6.2.

**Table 6-3. Point Source Cost for Chromium MRT System**

Cost Description	Base Process	MRT Process
<b>IWTP Initial Investment Cost</b>		
Chromium Reduction Unit	\$105,300	
Chromium MRT Unit (Total Cr VI and Cr III)		\$144,000
pH Control	\$15,000	\$15,000
Filter Press (1/3 Cr Wastestream)	\$78,000	
Post-Treatment Sand Filters	\$30,000	
Pre-Treatment Debris Removal		\$25,000
Installation (30% Equipment)	\$68,400	\$55,200
Total Capital Costs	\$296,700	\$239,200
<b>MRT Process - Initial Investment Costs Below Base Process:</b>		\$57,500
<b>IWTP Annual Operating Costs</b>		
Direct Materials	\$2,950	\$2,677
Waste Management	\$22,333	\$1,600
Revenues (by-Product)	\$0	-\$2,266
Training (5 days * \$1,500/day)		7,500
Total Operational Costs	\$25,283	\$9,511
<b>MRT Process – Cost Savings Over Base Process</b>		\$15,772

## 6.2 Cost Analysis

The Net Present Value (NPV) of the Base Process was estimated to be -\$4,001,118 and for the MRT Process was -\$3,415,745, which indicates a small cost advantage for the MRT Process as demonstrated at PSNS. If MRT were being considered as a replacement for conventional IWTP precipitation process, the payback period would be 9 years. However, if the MRT system were installed as part of a MILCON project, it would offer additional benefits. The MRT technology does not require as large a floor space as neutralization/precipitation and flocculation/clarification units. Due to the smaller footprint (10' x 10' x 15') of the MRT system, cost savings may be realized in lower infrastructure, which is not accounted for in ECAM. The MRT technology is also able to achieve lower discharge limits than the conventional process, which would avoid the occasional requirement with conventional process to manifest batches off-site to a hazardous waste contractor.

Although the MRT pilot demonstration showed only a small cost advantage for MRT when directly compared with conventional technology, four other alternative scenarios for MRT application are describe below (in addition to 1 and 2, which were demonstrated at PSNS), and associated equipment cost on Table 6.5.

(1) Mixed Bed Acid/Alkali With Cr III: Cost of a full-scale mixed bed for acid/alkali with Cr (III) would be \$340,000 assuming that the *Superlig*<sup>®</sup> 327 and 310 are not discounted for larger quantity purchase. In addition, the actual cost of the MRT would depend on many factors including the utilities available, degree of automation desired, and local site requirements. (Ref. 13)

(2) Single Metal Add-on Mixed Bed Cr (VI): A chromium (VI) MRT lead-trail column system as shown in Figure 4-1 for PSNS is estimated to cost \$144K, assuming that the major pumps, valves, flow meters, etc are already installed on the skid for acid/alkali with Cr (III) mixed bed MRT as described in 1) above (Ref 13).

(3) Pretreatment (Chelated Cu): At NAS North Island IWPF, the influent from certain maintenance operations chelates the copper that is then not amenable to the NaOH precipitation process (Ref. 16). Feasibility testing showed that processing this chelated-copper waste stream through a MRT column containing copper *Superlig*<sup>®</sup> 311 would break the copper-chelated bond (Ref 12). The wastestream would then be treated by the conventional precipitation process with the free, unchelated copper <sup>2+</sup> ion. For a mono-metal recovery MRT system was estimated as \$85K. In this scenario, the MRT is an add-on batch processing system used for 5,000 gal/month the chelated copper wastestream. The MRT capital cost per year is \$11.8 K (at 6.5 interest rate for 10 years with a capital recovery rate of 0.1391). The enhanced savings in labor and disposal cost show a payback of less than 0.5 years. The labor for the base process is high due to the time it takes for operators to do analytical testing and manifesting the chelated copper wastestream

**Table 6-4. MRT Payback for Chelated Copper Treatment**

<b>Category</b>	<b>Present Process (\$K/Yr)</b>	<b>MRT Process (\$K/Yr)</b>
Capital Cost	0.0	11.8*
Labor	41.0	8.0
Materials	2.7	2.0
Disposal	20.0	0.0
Total	63.7	22.8
<b>Cost/Gallon</b>	<b>\$1.06</b>	<b>\$0.38</b>

**Net Savings:** \$0.68/gal x 60,000 gal/yr = **\$40,800/yr**      **Payback:** < 0.5 years

\* Annual Cost of Capital Investment: \$85K is capitalized over 10 years at 6.5% interest rate with Capital Recovery Factor of 0.1391.

(4) Point Source for Total Cr: A chromium (VI) and (III) MRT can be installed in a Metal Finishing Facility such as at PSNS. The cost will have to include both Cr (VI) and Cr (III) *Superlig*<sup>®</sup> because the discharge limit is measured as total chromium. Due to the small footprint and the easy analytical techniques for chromium, the operation and maintenance will be minimal. See Table 6-3 for cost estimate for point source chromium MRT (Ref 12 and 13).

(5) Sequential/Selective for Target Metals: Sequential selective recovery/recycle for mono metals concentrated streams was bench tested using acid/alkali and chromium in 1996 (Ref 3). Figure 5-3 shows sequential selective recovery of metals with samples from NADEP North Island industrial waste stream. The number of columns would be 8 (4 lead and 4 trail). The cost estimate for this MRT system is given in Table 6-5.

(6) Membrane Embedded *Superlig*<sup>®</sup> Polishing: Unlike ceramic membranes, the membrane embedded *Superlig*<sup>®</sup> will select only the desired metals to recover. The throughput is greatly enhanced over the packed bed column. Other metals that may require removal are arsenic (drinking water), tin, molybdenum, and manganese. For a MRT polishing system for > 1 million gallons but < 2 million gallons per year, the cost would be \$86K without automation as shown on Table 6-5 (Ref. 7 and 11).

Table 6-5 shows a summary of cost estimates based on discussion with IBC Advanced Technologies, bench scale studies, feasibility studies and PSNS pilot scale demonstration.

**Table 6-5 MRT Cost Estimates (\$K) for Different Configuration Scenarios**

MRT System	Place	Acid/Alkali <i>Superlig</i> <sup>®</sup>	Skid	Cr VI <i>Superlig</i> <sup>®</sup>	Skid	Cr III	Skid	Cu <i>Superlig</i> <sup>®</sup>	Skid	Total
<b>1. Mixed Bed</b> Acid/Alkali with Cr (III)	PSNS	\$105	\$209			\$6	\$35			\$340
<b>2. Single Column</b> Cr (VI) Addition To Mixed Bed	PSNS			\$111	\$33					\$144
<b>3. Pretreatment</b> Chelated Cu Columns	NAS NI							\$52	\$33	\$85
<b>4. Point Source</b> Plating Shop Total Cr Columns	PSNS			\$111	\$50	\$26	\$35			\$222
<b>5. Sequential</b> Selective Columns	NADEP NI	\$228	\$242			\$26	\$36			\$532
<b>6. Membrane</b> Embedded <i>Superlig</i> <sup>®</sup> Polishing System 10 Year	PSNS	\$25	\$30	\$5	\$10	\$6	\$10			\$86

### 6.3 Cost Comparison

Ion exchange is probably the closest technology for comparison with MRT. In 1995, A.D. Little (Ref 4) investigated ion exchange/electrolysis and ion exchange/electrodialysis as potential chromium recover/recycle systems. The ion exchange/electrolysis system used both cationic and anionic columns for the Cr (VI) and Cr (III) ions respectively with electrolysis unit to recover the chromium ions. The capital costs for a 30 gal/min exchange/electrolysis system was \$259,740. The process is commercially available, but its use on DoD facilities has not been documented. The cost estimate for an ion exchange/electrodialysis system with a flow rate of 30 gal/min was \$251,000 (Ref 10 ). In 1997, A.D. Little (Ref 3) bench scaled tested an ion exchange/ electro dialysis system with a flow rate of 1 gpm. This latter system recycled water, chromic acid, and sulfuric acid. The residual metallic sludge was treated using a hydrometallurgical method to purify the metals for resale to vendors. The capital cost for the 1gpm system was \$81,000. As noted from Table 6-5, MRT cost estimates compare favorably with these two ion exchange systems. The advantage of MRT is a lower infrastructure, i.e., one component rather than several in-line processing units. In addition, MRT can reach lower metal concentration levels than ion exchange as described in Section 2.1.

## **7.0 Regulatory Issues**

### **7.1 Approaches to Regulatory Compliance and Acceptance**

#### **7.1.1 Reauthorization of Clean Water Act**

Treatment of the wastewater using the conventional hydroxide precipitation method generates large volumes of hazardous sludge. For the Navy as well as other DoD agencies, generation of these large volumes of industrial processing sludge will be restricted by Executive Order 12856 as stated in the Federal Compliance with Right to Know Laws and Pollution Prevention Requirements. The "Federal Government should voluntarily set goals to reduce their agency's total releases of toxic chemicals to the environment and off-site transfers of such toxic chemicals by 50 percent by December 31, 1999, to the maximum extent practicable through source reductions. Heavy metal removal/recycle technology must enable DoD facilities to treat to the levels expected from the re-authorization of the Clean Water Act (CWA). Under CWA, DoD agencies will be required to meet the National Pollutant Discharge Elimination System (NPDES) if discharging to a natural body of water, and the General and Categorical Pretreatment Standards under Section 307(b) and 307(c) of the CWA. See Table 3-1.

#### **7.1.2 Local and State Regulations**

Every DoD IWTP must meet the Federal Categorical Pretreatment Standards for discharge under the CWA. However, the IWTP must also meet the standards set by the local POTW if the activity is sewerage of the pretreated wastewaters. The POTWs standards are typically lower than the Federal Standards. There are various scenarios to accomplish regulatory compliance. If the MRT is used as a polishing unit, then this modification of the existing treatment system must be reported to regulators. If the MRT is used as a source recycling technology, say for the removal of Cr (VI) in the metal finishing facility, then there is not a permitting issue. MRT can qualify for exemption to the RCRA permit requirements because it is a close-looped recycling process that produces products for use, reuse, or reclamation.

#### **7.1.3 Holding Times Hazardous Waste--Impact Modification 40 CFR Part 262**

The modification of the holding times allows for accumulation of F006 hazardous waste from 90 days up to 270 days so recycle vendors will accept this waste. Previously, recycle vendors do not like to accept small quantities of F006 waste. Thus, by allowing longer holding times for the generator, large quantities of F006 can be sold for recycle.

## 8.0 Technology Implementation

### 8.1 DoD Need

The MRT system of metal removal will provide an alternative to the conventional precipitation treatment process in DoD facilities. Previous efforts by DoD have been to reduce the volume of IWTP sludge, and not to eliminate sludge going to landfill by either recycling to process or making the heavy metals amenable for selling the metal reclaimers. Secondly, MRT can be selective for only the regulated metals produced by the activity's industrial operation. For example, only copper from hydroblasting during submarine antifouling stripping operations, can be selectively removed and the effluent can then be sewerred or discharged to natural waters. Thirdly, many DoD activities use surfactant based alkaline cleaners or chelating agents as solvent replacements, which cannot be easily treated by precipitation. Cost estimates range from \$35K to over \$100K for shipping non-amenable batches to the NaOH precipitation process out to commercial treatment facilities. Examples of such compounds that may foul the precipitation of heavy metals are citric acid in Navy ship's pipe flushing and EDTA in Army ammunition plants. The usage of alkaline cleaners is expected to increase due to the restrictions on organic solvent cleaners, particularly those containing ozone depleting substances (ODS). Ultraviolet irradiation treatment has been ineffective with these chelated metals due to the spectral interference of dissolved materials in the industrial wastestream. Feasibility testing with wastewaters from Naval Air Station North Island, San Diego, on chelated wastewaters, showed that the MRT system could separate the heavy metals (copper, lead, and zinc) from the chelating complex.

### 8.2 Transition

#### 8.2.1 Steps to Full Scale MRT System

This technology is currently available for off-the-shelf procurement. However, it must be "customized" for each site and the customer's specific requirements as to the hours of operations, etc. The design a MRT system for a particular site requires a treatability test for the particular wastewater stream. The MRT system pilot-scale demonstration at PSNS can be modified to a "full scale system" for the current PSNS acid/alkali wastestream entering the IWPF. The costs that will be incurred will be for larger pumps and additional *Superlig*<sup>®</sup> material for the columns. Prior feasibility tests to the ESTCP pilot demonstration have determined that the design parameters scale linearly with flow, cycle time, and regeneration requirements by the customer. Table 8-1 shows the design parameters for two MRT Systems based on the time requirements of 24 hours per day versus business hours only processing. The amount of *Superlig*<sup>®</sup> in the column must be increased for the business hours only processing. The *Superlig*<sup>®</sup> column sites can be further adjusted to handle increased flow rates.

**Table 8-1. MRT Design Parameters for Scale-Up**

<b>System Parameter</b>	<b>Units</b>	<b>Quantity in 24 Hours</b>	<b>Business Hours Only</b>
Feed Flow Total (per year)	gallons	1,638,600	1,638,600
Feed Flow Rate (average)	l/min.	11.88	49.9
Feed Flow Cycle	gallons	39.014	163,860
Feed Time Cycle	hours	204	204
Cycles Per Year	number	42	10
<i>Superlig</i> <sup>®</sup> Per Column	pounds	26	109
Eluent Flow Rate	l/min.	8.58	36
Eluent Cycle Time	min.	6.06	6.06
Total Cycle Time	hours	204	204

**8.2.2 Deficiencies Identified and Corrective Action**

The main factor affecting performance of metal ion removal is kinetics (speed of flow versus amount of *Superlig*<sup>®</sup> present) for the packed bed column. Other configuration options have been suggested under Section 2.1.1. The efficiency of the up front oil/water separation and filtration system will greatly enhance efficiency and the long-term usage of the MRT columns. The number of regenerative cycles could not be tested in the time span of the demonstration time period. Previous experience in other industries indicates a multi-cycle lifetime in the thousands.

**8.2.3 Best Implementation Pathway**

Regulatory Basis for Selected Pathway

**MP&M Rule:** If the MP&M rule is changed as anticipated to lower discharge limits (see Table 3-1), conventional precipitation treatment will require pH to be close to the maximum range for each metal in order achieve the lower discharge limits. See Section 3 for pH adjustment. For example, cadmium will require a high pH of 11. The additional costs of O&M of the conventional precipitation system due to re-batching to meet compliance limits may make the cost of installing an MRT system more attractive. For this latter case embedded *Superlig*<sup>®</sup> material in a membrane format for fast flow and achievement of low part per million (ppm) metal concentration levels.

**Landfill Cost and Liability Factors:** Secondly, landfill disposal costs are increasing due to the loss of capacity, impact of “land ban” restrictions, and increased disposal taxes. From 1980 to 1990, disposal fees increased by 160% and the Superfund Waste Tax was increased by 27%. The liability factor adds consideration of potential future cleanup sites that would be incurred if a disposal site became a Superfund site. Because commercial reclaimer/recycle vendors waste material they accept is treated on site, presumably the liability factor for this option would be considerably lower than the land disposal option (Ref 17).

## Strategies for Recycle RCRA F006 Waste Using MRT

Due to this increasing cost to landfill and future liability, potential recycle scenarios are needed to increase DoD options for cost effective recycling. NFESC is currently researching the cost benefit of selling the IWPF's hazardous metal sludge or as a MRT concentrate to commercial recycle vendors (Ref 18). There are 10 or more established recycle companies in the U.S. that accept F006 waste as shown in Table 8-2. Table 8-3 shows the preferences for certain types of metal waste stream concentrates or sludges. For example, a metal waste stream with high chromium and nickel containing less than 2% copper is marketable. In addition, copper alone is a valued metal wastestream. By looking at the historical influent wastestream at PSNS in Table 8-4, we see that there is a high content of copper, chromium, and nickel. The wastestream does not contain tramp metals nor is the phosphorus content high. In order to recycle with a cost benefit at PSNS, an MRT system could be installed to take out the copper from the influent wastestream, allowing the remaining feed to be processed by the conventional precipitation method. The copper concentrate would be manifested separately to the recycle vendor. The sludge that remained from conventional processing could be recycled to a vendor accepting metal sludge for the stainless steel manufacturing industry. A non-historical wastestream occurred as shown in Table 8-3 due to an addition of a concentrated pickling bath dump into the IWPF. If the sludge from this wastestream was sent to a reclaimer, there would be a surcharge for the high content of cadmium.

**Table 8-2. U. S. Metal Reclaimers Processing > 1.1 Million Tons /Year**

Company	Years in Businesses	Waste Types	Metals Accepted	Process	Process Capacity Tons/Year	#Plating Shops Clients
Horsehead Resource Development Co.	1993	F006 F019	Zn, Pb, Cd, Fe	Rotary Kiln	270,000	100
Inmetco	1978	F006	Cr, Ni, Fe, Mo, Cu	Pyrometallurgical	56,000	150
RECONTEK		F006	Zn, Cu, Precious Metals	Hydrometallurgical	33,000	
CP Chemicals	1950	F006 D002&4 D007&8	Pickeling Solutions, Spent Plating Baths, Strippers	Hydrometallurgical	120,000	1,000
World Resources Company	1980			Hydrometallurgical Pyrometallurgical		800
Encycle/Texas, Inc	1988	F006	Cu, Pb, Zn, Ni	Chemical / Hydrometallurgical	25,000	150
Alpha Omega Recycling		F006	Cr, Cr-Ni, Cu	Acid Leaching / Selective Precipitation	5,500	100
Cyano Corp. Michagan		F007 F008 F009	Cyanide Waste	Electrowinning	2,200	50

10-15 Metal Recyclers with ~ 1.1 million tons/yr  
1,064,130 tons/yr

13,470 Plating Shops each generating 79 tons/yr for a total of

**Table 8-3. Metal Reclaimer Marketability of Industrial F006 Waste from IWTPs**

<ul style="list-style-type: none"> <li>• Potential Recycle</li> <li>• Nickel &amp; Copper with Chromium &lt;2.0%</li> <li>• Chromium &amp; Nickel With Copper &lt;2.0%</li> <li>• Copper Only</li> <li>• Nickel Only &gt;10%</li> <li>• NaOH Sludges</li> <li>• Flocculation Anionic Polymers</li> </ul>	<ul style="list-style-type: none"> <li>• Limited / Surcharge</li> <li>• Chromium Only</li> <li>• Phosphorous &lt;0.05%</li> <li>• Moisture &lt;35%</li> <li>• Tramp Metals (Arsenic and Mercury)</li> <li>• Sulfide Sludges</li> <li>• Flocculation Alum and Ferric Compounds</li> </ul>
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**Table 8-4. Analysis of PSNS Influent Wastestreams and Sludge Samples**

Metal	Historical* Averaged Influent (mg/l)	Run #1 (mg/l)	Run #4 (mg/l)	Run #20 (mg/l)	Treated Sludge #20 (mg/kg)
Zinc	23.0	30	9.8	224	18,800
Lead	1.30	0.52	0.61	6.28	759
Copper	31.0	29	16.08	263	27,600
Silver	0.44	0.07	0.027	1.15	184
Cadmium	2.61	1.07	0.772	19.1	1,510
Nickel	6.61	3.36	5.05	35.8	3,870
Chromium	60.0	16.1	8.23	65.8	12,600
Phosphorous	Unknown				3,800

\* Walter Hunter supplied data collected from PSNS Industrial Wastewater Pretreatment Facility. See Appendix D.

### 8.2.4 Transition to Industry of MRT

In order to consider the use of MRT, as an alternative technology for industrial wastewater treatment, the following factors need to be analyzed: a) regulatory changes b) the capability of the alternative technology to make the F006 waste more amenable to recycling and c) wide acceptance of the MRT technology to bring down the cost by such commercialization.

### 8.2.5 Actions Required for Implementation

It is important that the end-user provide an accurate picture of the intent for the MRT application. By application, is the MRT going to be designed for point-source treatment (a chromium rinse bath), an entire industrial processes (an IWTP facility), or a polishing system to an existing system. The end-user must provide the following information at a minimum in order to correctly size a MRT system: 1) concentration range of influent wastestream to be treated; 2) pH of wastestream; 3) metals to be removed to what level for discharge; 4) average flow rate for processing; and 5) the general application.

## **9.0 Lessons Learned**

### **9.1 Bench Scale Studies**

The initial MRT RDT&E was to be able selectively to recycle metal ions back to the industrial process as described in Section 1. Studies were conducted that showed that the metals from the industrial wastestream could be sequentially and selectively removed as concentrated mono-metal streams (ref 3).

### **9.2 Pilot Scale Demonstration**

1. Recycling to process may not be allowed due to strict military specifications.
2. If metals are recycled to process, say in a plating facility, the vendor's warranty of the plating bath may be invalid.
3. As shown in Section 8.2.3.2, cost effectiveness may be found in selling F006 waste to a metal reclaimer.
4. It is of great importance that the range of metal concentrations to be recovered/recycled be determined as accurately as possible. This range is important so that less regeneration are required and therefore use of less chemicals and associated O&M labor costs.

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# APPENDIX A

## POINTS OF CONTACT

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# **APPENDIX B**

## **Operational Parameters and Analytical Data**



## Test Run # 1 Acid/Alkali Operational Parameters

Run #1 Objective	Start Date/End Date	Work Order for Analysis	Superlig Matrix	Total Gallons Processed	Flow Rate	Flow Rate	Change in Delta Pressure
Establish Maximum Parameters as Flow Rate, Regeneration Rate,	04/05/1999 04/06/1999	99PS02561 99PS02577 99PS02608 99PS02610	Mixed Bed Superlig 310 SuperLig 327	1400	4 gal/min	2.5 gal/min	Remained Acceptable Range

pH	Start pH	Final pH	Regeneration Solution	Special Operations	Remarks
7.49	3.5	4M Sulfuric Acid	1 M NaOH wash solution was light yellow and tested positive for Cr (VI) ion using field spectrophotometer in IWPF chemistry laboratory.	The sulfuric acid was tested for metals to determine if stainless steel was leaching metals. The industrial sulfuric acid was from the IWPF and contained 16.8 mg/l nickel and 22.1 mg/l total chromium.	
			A high content of sodium was in the samples from the special oxidation processing of the cyanide wastestream in the IWPF. The IWPF used sodium hypochlorite instead of calcium hypochlorite in order to instead the oxidizing power to break up the chelated cyanide.	For future work, reagent sulfuric acid was ordered.	
			The high content of sodium created difficulties in the prep step for the ICP using EPA Method 200.7. The high content of sodium was noted as a yellowish color in the ICP flame.		

## Test Run # 1 Acid/Alkali Analytical Data

Trace #	Description	Gals.	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Fe
1-S1-95-1000-M	Feed	0	35.2	0.55	33.2	0.18	1.24	3.9	17.9	7.35
1-S2-95-1100-M	Process	350	0.15	0	0.058	0	0	0.11	0.83	1.51
1-S2-95-1230-M	Process	700	4.14	0	0.68	0	0.18	0.28	1.51	0.13
1-S2-95-1420-M	Process	1050	12.1	0	2.73	0.021	0.47	0.73	1.95	0.16
1-S2-95-1600-M	Process	1600	28.1	0.058	7.51	0.033	1.07	1.97	5.21	
1-S2-95-1650-M	Process	1800	33.5	0.085	9.62	0.039	1.25	2.52	6.26	
1-S2-95-1750-M	Process	2100	35	0.12	11.8	0.049	1.28	2.9	7.08	
1-S2-95-1900-M	Process	2400	39.2	0.26	15.7	0.07	1.41	3.62	8.59	
1-S2-95-2010-M	Process	2800	40.4	0.23	19	0.063	1.40	3.97	9.58	
1-S2-95-1025-M	Process	3100	41.4	0.295	18.3	0.063	1.45	4.02	9.69	
1-S2-96-1200-M	Process	3500	39.6	0.48	22.9	0.064	1.37	4.27	10.5	2.95
1-S2-96-1330-M	Process	4000	38.9	0.52	27	0.067	1.34	4.58	11.6	2.69
1.S2-96-1630-M-SA	4 M Sulfuric Acid		146	2.8	714	0	3.75	41.8	2.55	2.69
	Regenerate									
1-S2-97-0855-M-CR6	Chrome (VI) Test		0.98	5.97	2.63	0.15	0.02	0.33	3.57	6.9
SI = influent freed after holding tank but before entering MRT skid										
S2 = effluent freed after processing wastestream through column 1 of MRT skid										
Note: S2 is called S3 in all subsequent runs										

Run #2	Start Date/ End Date	Work Order for Analy	Superlig Materie	Total Gall	Flow Rate	Flow Rate	Change in Delta Pressu
				Processer	Processin	Regeneratio	
Operation run	04/07/1999	99PS02609	Mixed Bed	1000	5 gals/min	0.2 gals/min	Remain
	04/15/1999	99PS02618	SuperLig				Acceptable
		99PS02625					Range
		99PS02667	SuperLig 310				
			SuperLig 327				
Fluoride Test		87114-84000-001					
1 M NaOH & 4 M Sulfuric Acid		99PS02655					

### Test Run #2 Acid/Alkali Operational Parameters

pH Start	pH Final	Regenerate Solution	Special Operations/Tests	Remarks/Results
11.9	6.6	4 M Sulfuric Acid	A fluoride test was requested during ESTCP briefing Spring 1999.	Test showed that there was 16.8 ppm Nickel and 22.1 ppm of chromium in the industrial grade sulfuric acid.
		60 gallons	Due to the mass balance calculations showing that more chromium and nickel came out of the MRT system than went in from the holding tanks, tests done on the IWPF concentrated sodium hydroxide and and the industrial grade sulfuric acid	There was also 218 ppm iron in the sulfuric acid.
			A high content of sodium was in the samples from the special oxidation processing of the cyanide wastestream in the IWPF.	
			The IWPF used sodium hypochloite instead of calcium hypochlorite in order to instead the oxidizing power to break up the chelated cyanide.	
			The high content of sodium created difficulties in the prep step for the ICP using EPA Method 200.7. The high content of sodium was noted as a yellowish color in the ICP flame.	

## Test Run #2 Acid/Alkali Analytical Data

sample	Trace #	Description	Gals.	Zn	Pb	Cu	Ag	Cd	Ni	Cr
2609	2-S1-97-0840-M	influent feed	0	30.2	0.25	29	0.07	1.07	3.36	16.1
2618-1	2-S2-97-1000-M	column 1 process	125	0.04	0.029	0.051	0.046	0	0	1.09
2618-2	2-S2-97-1050-M	column 1 process	250	0.032	0	0.076	0.022	0	0	1.03
2618-3	2-S2-97-1140-M	column 1 process	371	0.051	0	0.053	0	0	0	0.89
2618-4	2-S2-97-1230-M	column 1 process	500	0.34	0	0.14	0.023	0	0.03	0.98
2618-5	2-S2-97-1320-M	column 1 process	625	1.9	0	0.88	0.025	0.082	0.17	1.36
2625-2	2-S2-97-1420-M	column 1 process	776	7.65	0	4.39	0.037	0.27	0.26	2.61
2625-1	2-S2-97-1500-M	column 1 process	875	10.1	0.037	6.75	0.041	0.4	1.03	3.03
2654	2-S2-97-1550M	column 1 process	1000	16	0.073	10.6	0.035	0.58	1.54	4.97
SI = influent freed after holding tank but before entering MRT skid										
S2 = effluent freed after processing wastestream through column 1 of MRT skid										
Note: S2 is called S3 in all subsequent runs										

### Test Run #3 Acid/Alkali Operational Parameters

Run #3	Start Date/ End Date	Work Order for Analy:	Superlig Material	Total Gallons Processed	Flow Rate	Flow Rate Regeneration	Change in Delta Pressure
Operational Test #3	04/19/1999	99PS02891	Mixed Bed	8000	2.5	0.2	Remained
was run to check the	04/20/1999	99PS03054	SuperLig				within
parameters from		99PS03015					acceptable
Test #2.		99PS03134	SuperLig				range
		99PS03135					
		99PS03179					
		99PS03201					
		99PS03495					

pH Start	pH Final	Regenerate Solution	Special Operations/Testing	Remarks/Results
2.4	9.01	4 M Sulfuric Acid	A high content of sodium was in the samples from the special oxidation processing of the cyanide wastestream in the IWPF. The IWPF used sodium hypochloite instead of calcium hypochlorite in order to instead the oxidizing power to break up the chelated cyanide.	It was found that one of the operators accidently filled one of the rental tanks with more wastewater from the miscellaneous tank.
			The high content of sodium created difficulties in the prep step for the ICP using EPA Method 200.7. The high content of sodium was noted as a yellowish color in the ICP flame.	The results show that there is a break in concentration of the influent wastewater at 2150 gallons. There is dilution of the influent wastewater across all the metals.
				Therefor the data for this run was compromised.

### Test Run #3 Acid/Alkali Analytical Data

Tracking Number	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Fe	Ca	Mg
3-108-1240-25	25	0.13	0	0.18	0	0	0.057	0.1	0.27	0	0
3-108-1320-64	64	2.11	0	0.38	0.02	0.54	1.41	0.051	0.24	0	0
3-108-1300-90	90	0.1	0	0.13	0.023	0	0.081	0.072	0.21	0	0
3-108-1345-140	140	0.069	0	0.088	0.023	0	0.083	0.064	0.13	0	0
3-108-1430-265	265	0	0	0.036	0.023	0	0.062	0.046	0.21	0	0
3-109-1010-1020	1020	0	0	0	0	0	0.051	0	0.05	3.94	2.56
3-109-1125-1200	1200	0	0	0	0	0	0.056	0	0.053	5.79	4.78
3-109-1202-1340	1340	0	0	0	0	0	0.051	0	0.051	7.55	6.66
3-109-1320-1470	1470	0	0	0	0	0	0.053	0	0.044	33.6	12
3-109-1410-1600	1600	0	0	0	0	0	0.05	0	0.044	12.3	11.6
3-109-1520-1780	1780	0	0	0	0	0	0.053	0	0.14	15.5	13.3
3-109-1615-1910	1910	0	0	0	0	0	0	0	0.13	0	0
3-110-1435-2150	2150	0	0	0	0	0	0.05	0	0.16	0	0
3-110-1550-2350	2350	0	0	0.027	0.023	0	0.054	0.34	0.11	56.8	24.7
9-110-1650-2500	2500	0	0	0.027	0.023	0	0.048	0.39	0.35	57.9	20.4
3-110-1750-2650	2650	0	0	0.026	0.023	0	0.053	0.4	0.19	59.2	17.2
3-110-1855-2800	2800	0	0	0.024	0.024	0	0.044	0.41	0.16	60	15
3-110-2000-2980	2980	0	0	0.022	0.025	0	0.045	0.42	0.23	64.2	13.8
3-111-0815-4000	4000	0	0	0	0	0	0.054	0.29	0.044	0	0
3-112--0835-4900	4900	0	0	0.039	0	0	0.054	0.44	0.15	0	0
3-124-1102-5281	5281	0	0	0	0	0	0.059	0.42	0.046	0	0
3-124-1035-7230	7230	0	0	0.021	0	0	0.058	0.24	0.095	66	12.4
3-124-1546-8000	8000	0	0	0	0	0	0.06	0.32	0.25	71	12.5

## Test Run #4 Acid/Alkali Operational Parameters

Run #4	Start Date/ End Date	Work Order for Analysis	Superlig Material	Total Gallor Processed	Flow Rate Processing	Flow Rate Regeneration	Change in Delta Pressure
Establish new	08/17/1999	99PS06462	Mixed bed	1350	2 gal/min	1 gals/min	Within
parameters after	08/18/1999	99PS06676	SuperLig				acceptable
modification of skid.			SuperLig				range
The upgrade included			310 for Cr (III)				
automated valves and							
two smaller columns			SuperLig				
(3&4) such that the			327 for rest of				
SuperLig material would			metals				
be the appropriate depth							
to prevent channeling.							

pH Start Column 3	pH Final Column 3	Regenerate Solution	Special Operations	Remarks/Results
9.78	5.55	4 M Sulfuric	An excess of regenerate solution	Port 2 samples consistently
		225 gals	was run to make sure that all the	showed a pH of ~ 4.36.
			metals had been removed from	Port 3 samples showed a
			column 3&4. This operation was	lowering of the pH from 9.78
			due to ensure that the mass	to 5.55.
			balance calculations were correct.	
				Port 6. Started at pH 11.0 and
				lowered to 8.67.
				The elution was completed
				within two bed volumes.
				It was found that lead had a high
				extraction rate but the mass
				balance was low compared to
				the other metals of regulatory
				interest.

## Test Run #4 Acid/Alkali Analytical Data

Table Acid/Alkali Data for Operational Test Run #4  
Ports 2, 3 & 6; Tank 1; and Eluent Samples

Notes: 1) Values are from samples analyzed undigested; 2) Samples preserved prior to analysis;  
3) Detection limit is 0.100 ppm; 4) Values below should be considered estimates.

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Mg
4-1-17-0000	0	9.353	0.570	16.005	0.022	0.744	4.728	6.459	17.942
4-1-17-0360	360	9.247	0.624	16.361	ND	0.734	4.690	6.337	17.801
4-1-18-0900	900	9.589	0.625	17.009	0.021	0.756	4.883	6.553	18.483
4-1-18-1170	1170	8.322	0.599	14.813	0.024	0.667	4.265	5.698	16.168
4-1-18-1350	1350	9.110	0.691	16.166	0.022	0.725	4.648	6.227	17.621
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0	24.3
<b>mg/l (avg)</b>		9.124	0.622	16.071	0.022	0.725	4.643	6.255	17.603
<b>mmol/l (avg)</b>		0.140	0.003	0.253	0.000	0.006	0.079	0.120	0.724

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Mg
4-2-17-0000	0	9.783	0.612	16.082	0.027	0.772	5.053	8.232	19.566
4-2-17-0030	30	8.757	0.660	15.571	0.023	0.707	4.472	6.020	17.015
4-2-17-0060	60	9.696	0.665	17.156	0.023	0.770	4.982	6.616	18.705
4-2-17-0090	90	9.098	0.589	15.175	0.022	0.720	4.615	6.249	17.091
4-2-17-0180	180	10.002	0.743	17.809	0.023	0.797	5.119	6.867	19.308
4-2-17-0275	275	9.144	0.656	16.108	0.022	0.726	4.668	6.223	17.607
4-2-17-0360	360	9.118	0.626	16.027	0.022	0.724	4.649	6.226	17.513
4-2-17-0465	465	9.440	0.659	16.528	0.021	0.755	4.861	6.600	17.999
4-2-17-0540	540	9.306	0.641	16.555	0.023	0.732	4.734	6.317	18.029
4-2-17-0650	650	9.273	0.653	16.201	0.021	0.745	4.727	6.296	17.734
4-2-17-0729	729	8.976	0.631	15.821	0.022	0.712	4.577	6.098	17.329
4-2-18-0810	810	9.165	0.637	16.157	0.021	0.722	4.673	6.242	17.725
4-2-18-0900	900	9.005	0.588	15.916	0.021	0.707	4.605	6.147	17.461
4-2-18-0990	990	9.760	0.692	17.184	0.024	0.778	5.009	6.656	18.677
4-2-18-1080	1080	9.446	0.638	16.734	0.023	0.753	4.812	6.474	18.250
4-2-18-1170	1170	9.663	0.662	16.978	0.023	0.771	4.945	6.631	18.440
4-2-18-1253	1253	9.719	0.702	17.350	0.023	0.766	4.986	6.667	18.895
4-2-18-1350	1350	9.209	0.658	16.308	0.023	0.732	4.669	6.256	17.727
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0	24.3
<b>Avg. Feed (mg/l)</b>		9.364	0.651	16.426	0.023	0.744	4.786	6.490	18.060
<b>Avg. Feed (mmol/l)</b>		0.143	0.003	0.259	0.000	0.007	0.082	0.125	0.743
<b>Feed (mmol)</b>		731.65	16.05	1,321.74	1.07	33.81	416.65	637.72	3,797.51

## Continued Test Run #4

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Mg
4-3-17-0000	0	ND	ND	ND	ND	ND	ND	ND	1.175
4-3-17-0030	30	0.045	ND	0.133	ND	ND	ND	0.237	17.204
4-3-17-0060	60	0.057	ND	0.080	ND	ND	ND	0.146	19.016
4-3-17-0090	90	0.106	ND	0.084	ND	ND	ND	0.139	18.831
4-3-17-0180	180	0.072	ND	0.063	ND	ND	0.034	0.087	18.643
4-3-17-0275	275	0.072	ND	0.051	ND	ND	0.049	0.063	18.221
4-3-17-0360	360	0.169	ND	0.066	ND	ND	0.104	0.063	19.006
4-3-17-0465	465	0.372	ND	0.095	ND	0.041	0.285	0.067	17.727
4-3-17-0540	540	1.113	ND	0.147	ND	0.093	0.629	0.080	18.504
4-3-17-0650	650	2.490	ND	0.308	ND	0.243	1.121	0.147	18.490
4-3-17-0729	729	4.293	0.021	0.558	ND	0.363	1.693	0.227	18.275
4-3-18-0810	810	3.797	0.021	0.343	ND	0.248	1.015	0.354	17.704
4-3-18-0900	900	6.642	0.029	1.082	ND	0.469	2.189	0.611	17.832
4-3-18-0990	990	8.411	0.058	2.077	ND	0.634	2.995	0.835	18.178
4-3-18-1080	1080	9.317	0.086	2.999	ND	0.712	3.475	1.030	17.606
4-3-18-1170	1170	10.357	0.127	4.470	ND	0.810	4.096	1.451	18.430
4-3-18-1253	1253	10.292	0.173	5.164	ND	0.812	4.171	1.620	17.169
4-3-18-1350	1350	11.366	0.237	6.825	ND	0.909	4.779	2.059	18.656
<b>M.W.</b>	<b>65.4</b>	<b>207.2</b>	<b>207.2</b>	<b>63.5</b>	<b>107.9</b>	<b>112.4</b>	<b>58.7</b>	<b>52.0</b>	<b>24.3</b>
<b>mmol</b>	<b>356.46</b>	<b>1.24</b>	<b>1.24</b>	<b>131.07</b>	<b>0.00</b>	<b>16.12</b>	<b>154.15</b>	<b>58.10</b>	<b>3,824.80</b>

Liters Processed: 5,110

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Mg
4-6-17-0000	0	0.020	ND	ND	ND	ND	ND	ND	0.050
4-6-17-0030	30	0.034	ND	0.023	ND	ND	0.034	0.095	10.588
4-6-17-0060	60	ND	ND	ND	ND	ND	ND	0.068	17.461
4-6-17-0090	90	0.023	ND	ND	ND	ND	ND	0.061	21.777
4-6-17-0180	180	0.031	ND	ND	ND	ND	ND	0.046	18.789
4-6-17-0275	275	ND	ND	ND	ND	ND	ND	0.044	17.125
4-6-17-0360	360	0.025	ND	ND	ND	ND	ND	0.044	17.751
4-6-17-0465	465	0.043	ND	ND	ND	ND	ND	0.042	17.974
4-6-17-0540	540	0.046	ND	ND	ND	ND	ND	0.044	18.248
4-6-17-0650	650	0.101	ND	ND	ND	ND	ND	0.058	17.690
4-6-17-0729	729	0.112	ND	ND	ND	ND	ND	0.066	17.810
4-6-18-0810	810	0.029	ND	ND	ND	ND	ND	0.076	17.888
4-6-18-0900	900	0.037	ND	ND	ND	ND	ND	0.075	16.669
4-6-18-0990	990	0.059	ND	ND	ND	ND	ND	0.074	17.238
4-6-18-1080	1080	0.073	ND	ND	ND	ND	ND	0.070	17.944
4-6-18-1170	1170	0.127	ND	0.036	ND	ND	0.044	0.096	17.258
4-6-18-1253	1253	0.242	ND	0.051	ND	0.020	0.078	0.109	17.350
4-6-18-1350	1350	0.520	0.022	0.059	ND	0.050	0.158	0.110	17.954
<b>M.W.</b>	<b>65.4</b>	<b>207.2</b>	<b>207.2</b>	<b>63.5</b>	<b>107.9</b>	<b>112.4</b>	<b>58.7</b>	<b>52.0</b>	<b>24.3</b>
<b>Avg. Effluent (mg/l)</b>		<b>0.095</b>	<b>0.022</b>	<b>0.042</b>	<b>N/A</b>	<b>0.035</b>	<b>0.079</b>	<b>0.069</b>	<b>16.531</b>
<b>Effluent (mmol/l)</b>		<b>507.43</b>	<b>8.08</b>	<b>52.56</b>	<b>0.00</b>	<b>24.64</b>	<b>101.36</b>	<b>349.38</b>	<b>90,009.52</b>
<b>Effluent (mmol)</b>		<b>7.76</b>	<b>0.04</b>	<b>0.83</b>	<b>0.00</b>	<b>0.22</b>	<b>1.73</b>	<b>6.72</b>	<b>3,704.10</b>

Liters Processed: 5,110

## Continued Test Run #4

Identity	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Mg
229-4MH2SO4-1	ND	0.106	ND	ND	ND	0.254	0.299	1.043
229-4MH2SO4-2	ND	0.131	ND	ND	ND	0.242	0.256	1.033
30C3-1BV-E1	345.538	4.682	549.945	ND	19.805	139.593	170.737	16.052
30C3-2BV-E1	1,145.220	13.624	3,395.040	ND	56.641	532.703	879.321	15.071
30C3-3BV-E1	134.313	3.782	690.331	ND	19.290	85.560	233.218	4.127
30C3-4BV-E1	13.950	3.253	56.768	ND	2.782	9.377	51.527	1.713
30C3-5BV-E1	3.217	2.046	14.579	ND	0.374	2.445	18.883	1.231

30C3-E1-0225	mg/l	345.56	8.19	957.65	ND	18.54	142.57	257.35	6.78
Composite (l)	M.W.	65.4	207.2	63.5	107.9	112.4	58.7	52.0	24.3
85.16	mmol	450.0	3.4	1,284.3	N/A	14.0	206.8	421.5	23.8

30C4-E2-0225	mg/l	160.48	2.21	60.44	ND	12.96	65.04	24.73	5.69
Composite (l)	M.W.	65.4	207.2	63.5	107.9	112.4	58.7	52.0	24.3
85.16	mmol	209.0	0.9	81.1	N/A	9.8	94.4	40.5	19.9

Recovery (%):	90.1	26.6	103.3	N/A	70.6	72.3	72.4	1.2
Mass Balance (%):	91.1	26.9	103.4	N/A	71.2	72.7	73.5	98.7

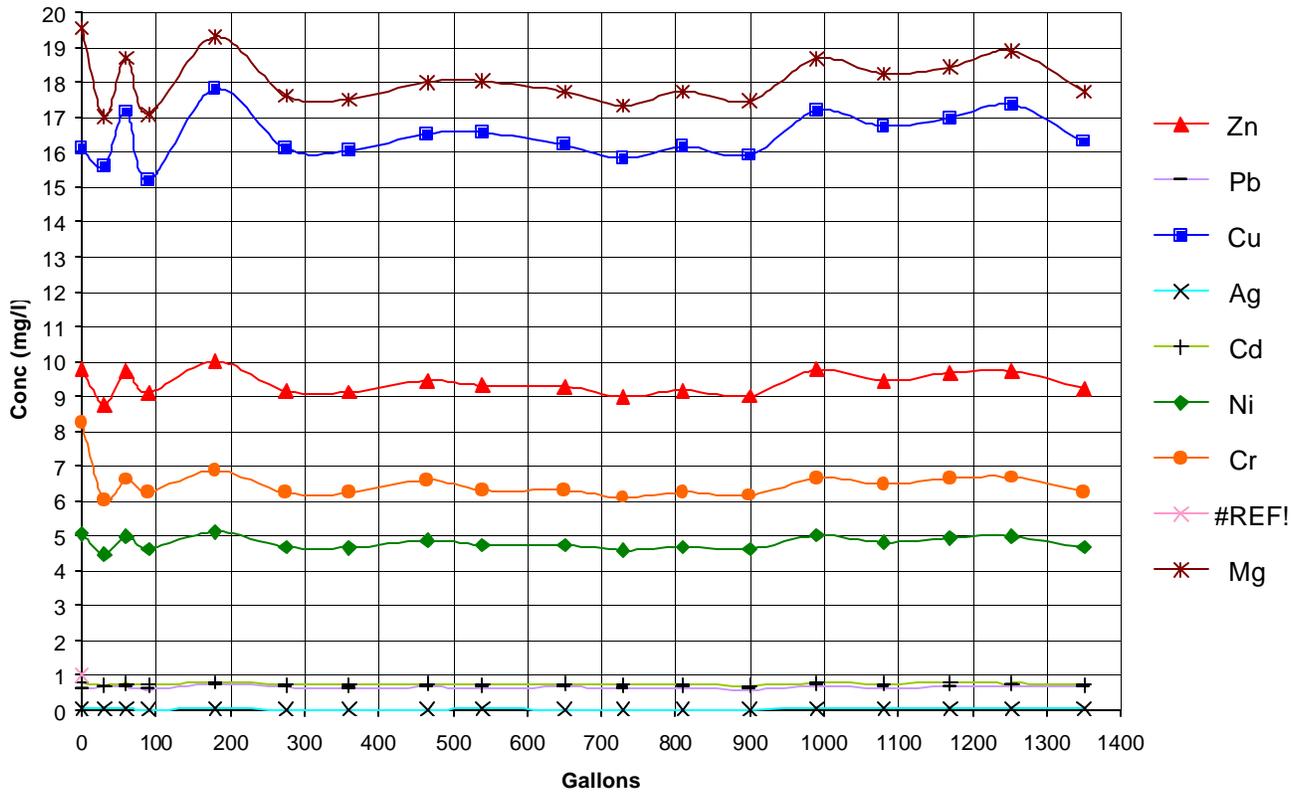
### Mass Balance, Run #4

	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Mg
Feed (mmol)	731.7	16.0	1,321.7	1.1	33.8	416.7	637.7	3,797.5
Feed (mg/l)	9.4	0.7	16.4	0.0	0.7	4.8	6.5	18.1
Effluent (mmol)	7.8	0.0	0.8	0.0	0.2	1.7	6.7	3,704.1
Effluent (mmol/l)	0.099	0.002	0.010	N/A	0.005	0.020	0.068	17.615
Eluate (mmol)	659.0	4.3	1,365.4	N/A	23.9	301.2	462.0	43.7
Extraction (%)	98.9	99.8	99.9	N/A	99.4	99.6	98.9	2.5
Mass Balance (%)	91.1	26.9	103.4	N/A	71.2	72.7	73.5	98.7

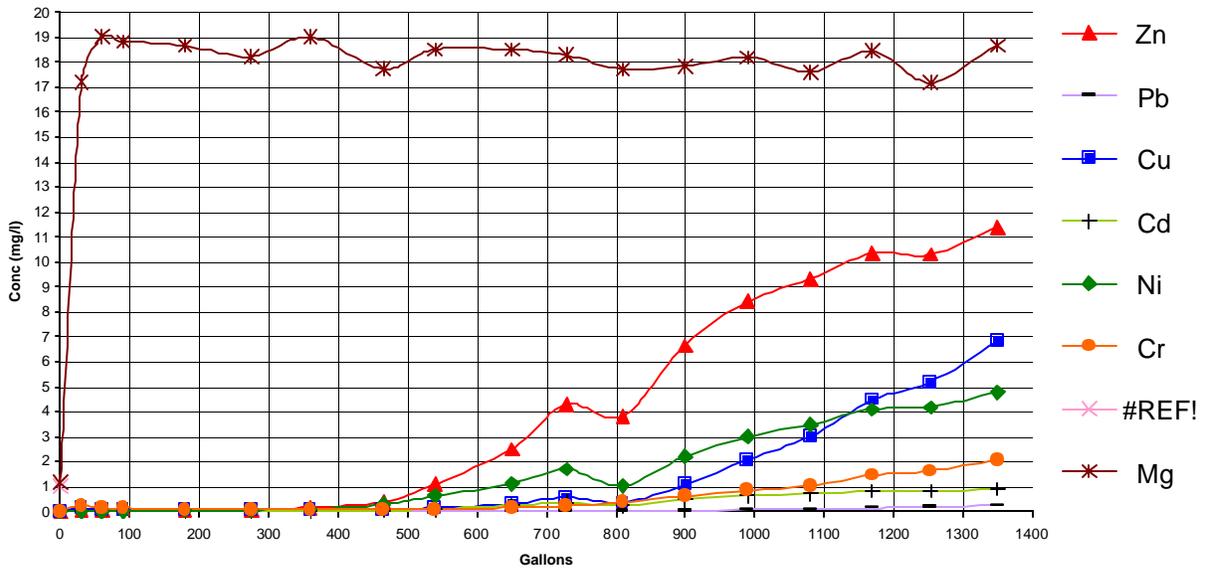
Note: Effluent Concentrations calculated on metal present in 1,350 gal processed. Assumes ND=0.0.

Add PQL value for worst case. Extraction calculated from (Feed-Eff.)/Feed x100.

MRT - Data Run #4, Acid/Alkali, Port 2



MRT - Data Run #4, Acid/Alkali, Port 3



## Test Run #8 Acid/Alkali Operational Parameters

	End Date			Processed	Processing	Regeneration	Delta Pressure
To Duplicate Run #4	07/18/2000	PS#00PS05011	SuperLig 327 Mixed	454	2 gal/min	0.5 gal/min	2.5-3.0
With Different Batch of Wastewater	07/18/2000	PS#00PS06257	SuperLig 310 Cr(III)				
		PS#00PS05165					
		PS#00PS05058					

pH Port 2	pH Port 3	pH Port 6	Regenerate Solution	Concentration of Wastestream	Operational Problems
2.65-3.15	9.63-4.08	2.66-3.68	1 M NaOH	Zn 224 Cu 263 Cd 19.1 Ni 35.8 Cr 65.8 Fe 78	Concentration Range of Metals High than expected for column size High concentrations due to Pickeling bath being
				The concentration of the wastestream was very high in the concentration of metals. This change was due to the dumping of a pickle bath from the PSNS Metal Preparation Facility.	
				Pickle solution: Data Andy Greene hydrochloric acid (gal) 85.3 Foz/G diethylthiorea (lbs) 0.53 Foz/G Rodine-50 (gal) 0.14 Foz/G detergent (gal) 0.12 Foz/G water to balance	
				Code 134, Analytical Lab PSNS	

## Test Run #8 Acid/Alkali Analytical Data

### Test Run # 8: Acid/Alkali Ports 2, 3 & 6; Tank 1; and Eluent Samples

Notes: 1) Values are from samples analyzed undigested; 2) Samples preserved prior to analysis;  
3) Detection limit is 0.100 ppm; 4) Values below should be considered estimates.

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
8-2-18-0012	12	178.560	3.987	189.900	0.191	15.432	25.969	22.494
8-2-18-0060	60	159.947	3.994	185.754	0.117	15.989	25.984	21.342
8-2-18-0084	84	157.378	4.026	183.560	0.156	16.186	26.251	21.637
8-2-18-0108	108	172.754	4.073	195.455	0.195	15.702	26.483	22.671
8-2-18-0192	192	165.163	4.085	185.236	0.193	15.709	26.344	22.722
8-2-18-0372	372	163.357	4.064	184.629	0.193	15.686	26.422	22.711

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
S3	12	1.414	0.018	0.183	0.008	0.033	0.038	0.142
8-3-18-0024	24	2.090	0.002	0.177	0.010	0.034	0.042	0.110
8-3-18-0036	36	5.940	-0.012	0.189	0.008	0.093	0.093	0.156
8-3-18-0048	48	10.935	0.023	0.242	0.003	0.506	0.451	0.168
8-3-18-0060	60	26.082	0.037	0.502	0.011	3.266	3.069	0.133
8-3-18-0072	72	67.410	0.031	1.773	0.010	6.981	8.152	0.182
8-3-18-0132	132	183.528	1.195	51.802	0.022	15.280	24.122	3.410
8-3-18-0180	180	192.152	2.383	90.192	0.029	16.585	26.549	7.089
8-3-18-0228	228	188.851	3.024	111.749	0.040	16.977	27.352	9.948
8-3-18-0300	300	203.966	3.783	146.036	0.074	17.251	28.192	13.403
8-3-18-0454	454	189.756	4.359	168.738	0.180	16.915	28.310	17.907

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
8-6-18-0012	12	0.125	0.028	0.154	0.081	0.013	0.030	0.079
8-6-18-0072	72	0.088	0.012	0.029	0.028	0.001	0.013	0.036
8-6-18-0096	96	0.334	0.004	0.024	0.033	0.007	0.014	0.044
8-6-18-0108	108	0.511	0.029	0.016	0.016	0.009	0.013	0.033
8-6-18-0120	120	0.752	-0.007	0.022	0.016	0.034	0.031	0.043
8-6-18-0132	132	1.202	0.026	0.007	0.014	0.045	0.036	0.034
8-6-18-0180	180	30.161	0.030	0.089	0.011	3.131	1.869	0.050
8-6-18-0228	228	126.733	0.115	1.058	0.028	12.043	9.505	0.258
8-6-18-0300	300	205.108	0.901	14.306	0.053	18.693	22.633	1.775
8-6-18-0454	454	208.696	3.372	75.866	0.041	19.311	30.495	7.747

### Continued Test Run #8

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
8-1-18-0180	180	178.413	3.896	194.943	0.182	14.813	25.013	21.999
8-1-18-0360	360	182.081	3.893	200.913	0.182	14.713	24.928	22.030
8-1-18-0454	454	171.692	3.976	190.747	0.182	15.196	25.560	22.570

Identity (Eluents)	Zn	Pb	Cu	Ag	Cd	Ni	Cr
8-C4-18-ELU	414.0	10.0	729.0	ND	31.7	90.0	64.0
8-C4-18-ELU	470.0	4.0	807.0	ND	33.9	96.0	68.0
8-C3-18-ELU	247.7	3.9	1,483.0	ND	18.0	50.0	184.7
8-C3-18-ELU	235.0	11.5	1,440.0	ND	17.0	48.6	178.0

Identity	Gallons	K	Mg	Ca	Na
8-2-18-0012	12	193.28	25.88	147.23	3,100.17
8-2-18-0372	372	212.00	25.16	148.33	3,125.06

Identity	Gallons	K	Mg	Ca	Na
8-3-18-0012	12	210.82	23.02	153.84	2,825.29
8-3-18-0454	454	214.56	29.18	244.76	2,646.31

Identity	Gallons	K	Mg	Ca	Na
8-6-18-0012	12	150.56	2.53	2.60	3,352.63
8-6-18-0454	454	210.54	28.68	147.07	3,123.42

Identity (Eluents)	K	Mg	Ca	Na
8-C3-18-ELU	25.18	5.21	46.70	174.22
8-C4-18-ELU	21.29	3.24	15.66	17.72



**Test Run #9 Acid/Alkali Analytical Data**  
**Acid/Alkali Test Run #9: Ports 2, 3 & 6; Tank 1; and Eluent Samples**

Notes: 1) Values are from samples analyzed undigested; 2) Samples preserved prior to analysis;  
 3) Detection limit is 0.100 ppm; 4) Values below should be considered estimates.

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Fe
9-2-19-0009	9	76.698	1.776	80.7	0.088	7.751	13.58	8.414	39.504
9-2-19-0018	18	78.364	1.799	78.9	0.08	7.912	13.857	8.584	40.338
9-2-19-0027	27	78.575	1.86	80.2	0.08	7.971	13.98	8.632	40.611
9-2-19-0081	81	78.52	1.816	82.9	0.082	7.938	13.92	8.633	40.509
9-2-19-0135	135	80.403	1.925	81.8	0.091	8.187	14.302	8.815	41.457
9-2-19-0189	189	79.181	1.862	81.3	0.089	8.037	14.048	8.664	40.703
9-2-19-0243	243	80.382	1.893	81.9	0.094	8.181	14.272	8.794	41.371
9-2-19-0297	297	79.633	1.831	80.4	0.084	8.116	14.198	8.738	41.25
9-2-19-0351	351	79.602	1.858	81.1	0.092	8.093	14.192	8.749	41.101
9-2-19-0405	405	80.256	1.925	82	0.086	8.167	14.295	8.871	41.487
9-2-19-0459	459	80.114	1.846	80	0.091	8.155	14.226	8.806	41.28
9-2-19-0567	567	80.139	1.905	78.7	0.095	8.154	14.217	8.748	41.44
9-2-19-0675	675	80.532	1.885	83.2	0.091	8.189	14.297	8.894	41.466
9-2-19-0783	783	80.465	1.925	79.6	0.091	8.2	14.286	8.8	41.632
9-2-19-0837	837	81.766	1.933	79.6	0.096	8.368	14.572	8.999	42.372
9-2-19-0891	891	80.162	1.871	79.5	0.091	8.162	14.272	8.822	41.94
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0	55.8
<b>Avg. Feed (mg/l)</b>		79.675	1.869	80.738	0.089	8.099	14.157	8.748	41.154
<b>Avg. Feed (mmol/l)</b>		1.218	0.009	1.271	0.001	0.072	0.241	0.168	0.738
<b>Feed (mmol)</b>		4,108.52	30.43	4,287.91	2.78	243.00	813.36	567.33	2,487.25
<b>Liters Processed:</b>		<b>3,372</b>							

## Continued Test Run #9

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Fe
9-3-19-0009	9	0.084	-0.019	0.081	-0.009	0.016	0.059	0.112	0.176
9-3-19-0018	18	0.036	-0.052	0.045	-0.01	0.014	0.031	0.058	0.108
9-3-19-0027	27	0.046	-0.031	0.05	-0.009	0.012	0.028	0.041	0.104
9-3-19-0054	54	0.724	-0.002	0.411	-0.009	0.068	0.078	0.079	0.214
9-3-19-0081	81	2.708	-0.022	1.202	-0.008	0.169	0.195	0.095	0.383
9-3-19-0108	108	24.599	0.145	16.775	0.003	2.177	3.489	0.366	5.143
9-3-19-0135	135	30.989	0.341	22.671	0.007	3.169	5.043	1.175	9.202
9-3-19-0162	162	44.219	0.49	26.698	0.008	4.48	6.817	1.81	11.567
9-3-19-0189	189	63.951	0.629	31.675	0.013	5.723	8.566	2.479	14.267
9-3-19-0216	216	60.235	0.542	31.786	0.014	5.811	8.981	2.198	13.341
9-3-19-0243	243	67.669	0.649	36.529	0.02	6.411	9.971	2.587	15.513
9-3-19-0270	270	73.089	0.713	39.808	0.022	6.789	10.587	2.8	17.311
9-3-19-0297	297	84.132	0.806	44.183	0.024	7.236	11.026	3.212	20.008
9-3-19-0324	324	71.071	0.812	46.224	0.028	6.878	11.063	3.053	19.87
9-3-19-0351	351	75.568	0.86	48.187	0.03	7.056	11.243	3.133	21.132
9-3-19-0378	378	76.995	0.916	50.124	0.032	7.318	11.674	3.495	22.851
9-3-19-0405	405	78.186	0.974	52.688	0.036	7.567	12.059	3.77	24.524
9-3-19-0432	432	76.893	1.083	54.207	0.04	7.635	12.302	3.867	24.992
9-3-19-0459	459	79.426	1.107	55.627	0.042	7.848	12.596	4.103	26.251
9-3-19-0513	513	78.907	1.21	58.387	0.047	7.957	12.81	4.209	27.273
9-3-19-0567	567	84.051	1.326	61.304	0.05	8.163	13.075	4.875	30.025
9-3-19-0621	621	80.913	1.373	62.065	0.055	8.195	13.26	4.959	29.756
9-3-19-0675	675	81.638	1.449	63.766	0.059	8.296	13.426	5.284	31.12
9-3-19-0729	729	83.143	1.57	67.005	0.066	8.48	13.808	5.63	32.617
9-3-19-0783	783	82.057	1.55	67.141	0.068	8.412	13.727	5.577	32.304
9-3-19-0837	837	82.542	1.558	67.145	0.069	8.43	13.796	5.652	32.369
9-3-19-0891	891	82.904	1.629	68.149	0.071	8.446	13.862	5.76	32.668
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0	55.8
<b>mmol</b>		3,473.41	16.44	2,556.93	1.20	199.21	611.53	240.13	1,360.70

**Liters Processed: 3,372**

**Continued Test Run #9**

	<b>Gallons</b>	<b>Zn</b>	<b>Pb</b>	<b>Cu</b>	<b>Ag</b>	<b>Cd</b>	<b>Ni</b>	<b>Cr</b>	<b>Fe</b>
9-6-19-0009	9	0.019	-0.033	0.043	-0.008	0.003	0.010	0.032	0.106
9-6-19-0018	18	0.017	-0.002	0.038	-0.007	0.002	0.008	0.028	0.044
9-6-19-0027	27	0.010	0.006	0.031	-0.008	0.002	0.007	0.029	0.032
9-6-19-0054	54	0.011	0.019	0.017	-0.008	0.001	0.006	0.022	0.032
9-6-19-0081	81	0.010	-0.005	0.012	-0.009	0.002	0.011	0.018	0.023
9-6-19-0108	108	0.017	-0.027	0.019	-0.011	0.001	0.008	0.020	0.023
9-6-19-0135	135	0.017	-0.031	0.028	-0.010	0.003	0.005	0.017	0.045
9-6-19-0162	162	0.210	-0.021	0.080	-0.011	0.006	0.001	0.023	0.073
9-6-19-0189	189	0.610	-0.013	0.155	-0.011	0.009	0.008	0.032	0.103
9-6-19-0216	216	0.921	-0.028	0.154	-0.011	0.017	0.024	0.028	0.107
9-6-19-0243	243	1.543	0.005	0.166	-0.010	0.088	0.094	0.045	0.135
9-6-19-0270	270	4.197	-0.006	0.215	-0.010	0.430	0.433	0.034	0.176
9-6-19-0297	297	13.941	-0.014	0.400	-0.009	1.444	1.414	0.024	0.428
9-6-19-0324	324	25.478	-0.003	0.688	-0.010	2.450	2.409	0.032	0.827
9-6-19-0351	351	37.102	-0.024	1.413	-0.009	3.470	3.722	0.041	1.669
9-6-19-0378	378	47.656	0.032	2.797	-0.009	4.471	5.062	0.047	3.081
9-6-19-0405	405	54.212	0.016	4.646	-0.008	5.122	6.142	0.073	4.727
9-6-19-0432	432	59.450	0.100	7.141	-0.008	5.714	7.125	0.133	6.881
9-6-19-0459	459	63.075	0.098	9.930	-0.009	6.152	8.054	0.186	8.612
9-6-19-0513	513	69.215	0.260	16.499	-0.007	6.913	9.509	0.394	12.497
9-6-19-0567	567	74.199	0.394	21.546	-0.007	7.444	10.420	0.657	15.565
9-6-19-0621	621	76.575	0.524	25.358	-0.003	7.802	11.068	0.881	17.127
9-6-19-0675	675	77.565	0.665	30.609	-0.001	7.942	11.510	1.272	19.439
9-6-19-0729	729	80.009	0.863	35.833	0.009	8.266	12.253	1.665	21.741
9-6-19-0783	783	79.853	0.917	38.360	0.013	8.257	12.366	1.979	22.770
9-6-19-0837	837	80.617	1.144	40.625	0.018	8.344	12.480	3.015	26.921
9-6-19-0891	891	82.834	1.192	43.457	0.024	8.654	12.942	2.640	26.340
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0	55.8
<b>Avg. Effluent (mg/l)</b>		34.421	0.223	10.380	-0.005	3.445	4.707	0.495	7.019
<b>Effluent (mmol/l)</b>		158,422.6	1,227.0	54,416.0	-7.0	16,006.4	22,444.3	2,637.7	35,952.5
<b>Effluent (mmol)</b>		2,422.36	5.92	856.95	-0.07	142.41	382.36	50.73	644.31

**Liters Processed: 3,372**

**Continued Test Run #9**

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
9-1-19-0000	0	71.851	1.664	80.562	0.079	7.112	12.549	7.823
9-1-19-0351	351	72.672	1.752	81.620	0.084	7.208	12.710	7.916
9-1-19-0513	513	71.414	1.697	80.023	0.081	7.081	12.483	6.996
9-1-19-0783	783	72.818	1.749	81.681	0.084	7.209	12.684	7.975
	<b>M.W.</b>	65.4	207.2	63.5	107.9	112.4	58.7	52.0
	<b>mg/l (avg)</b>	72.189	1.716	80.972	0.082	7.153	12.607	7.678
	<b>mmol/l (avg)</b>	1.104	0.008	1.275	0.001	0.064	0.215	0.148

Identity		Zn	Pb	Cu	Ag	Cd	Ni	Cr
9-C3-19 ELU	mg/l	496.486	16.798	1,682.424	0.043	40.306	105.398	164.464
9-C3-19-ELU	mg/l	595.000	17.600	1,410.000	ND	46.500	113.000	93.000
Composite (l)	<b>M.W.</b>	65.4	207.2	63.5	107.9	112.4	58.7	52.0
85.16	<b>mg/l (avg)</b>	545.743	17.199	1,546.212	0.043	43.403	109.199	128.732
	<b>mmol</b>	710.66	7.07	2,073.69	0.03	32.89	158.43	210.83

Identity		Zn	Pb	Cu	Ag	Cd	Ni	Cr
9-C4-19-ELU	mg/l	537.360	17.757	1,322.848	0.027	47.270	114.638	92.169
9-C4-19-ELU	mg/l	520.000	20.200	1,710.000	ND	38.900	103.000	164.000
Composite (l)	<b>M.W.</b>	65.4	207.2	63.5	107.9	112.4	58.7	52.0
85.16	<b>mg/l (avg)</b>	528.680	18.979	1,516.424	0.043	43.085	108.819	128.085
	<b>mmol</b>	688.44	7.80	2,033.74	0.03	32.64	157.88	209.77

<b>Recovery (%):</b>	1,773.1	1,228.9	7,034.8	144.4	823.5	2,469.2	9,992.8
<b>Mass Balance (%):</b>	93.0	68.3	115.8	0.1	85.6	85.9	83.1

**Mass Balance, Run #9**

	Zn	Pb	Cu	Ag	Cd	Ni	Cr
<b>Feed (mmol)</b>	4,108.5	30.4	4,287.9	2.8	243.0	813.4	567.3
<b>Feed (mg/l)</b>	79.7	1.9	80.7	0.1	8.1	14.2	8.7
<b>Effluent (mmol)</b>	2,422.4	5.9	856.9	-0.1	142.4	382.4	50.7
<b>Effluent (mmol/l)</b>	46.976	0.364	16.136	-0.002	4.746	6.655	0.782
<b>Eluate (mmol)</b>	1,399.09	14.87	4,107.43	0.07	65.53	316.30	420.60
<b>Extraction (%)</b>	41.0	80.5	80.0	N/A	41.4	53.0	91.1
<b>Mass Balance (%)</b>	<b>93.0</b>	<b>68.3</b>	<b>115.8</b>	<b>0.1</b>	<b>85.6</b>	<b>85.9</b>	<b>83.1</b>

**Note: Effluent Concentrations calculated on metal present in 891 gal processed. Assumes ND=0.0. Add PQL value for worst case. Extraction calculated from (Feed-Eff.)/Feed x100.**

### Continued Test Run #9

Identity	Gallons	K	Mg	Ca	Na
9-2-19-0009	9	41.68	0.58	0.12	1,814.45
9-2-19-0081	81	86.13	14.38	67.40	1,306.75
9-2-19-0351	351	94.11	13.15	69.04	1,302.89
9-2-19-0567	567	93.62	14.46	69.65	1,327.27
9-2-19-0891	891	94.46	13.68	67.66	1,390.26

Identity	Gallons	K	Mg	Ca	Na
9-3-19-0027	27	102.78	0.58	0.20	1,774.12
9-3-19-0081	81	104.03	16.92	79.50	1,503.70
9-3-19-0351	351	86.55	14.38	69.05	1,296.23
9-3-19-0567	567	94.04	13.39	71.24	1,303.55
9-3-19-0891	891	93.97	13.26	70.04	1,328.59

Identity	Gallons	K	Mg	Ca	Na
9-6-19-0009	9	20.39	0.74	0.36	1,517.84
9-6-19-0081	81	109.92	0.63	0.31	1,639.13
9-6-19-0351	351	99.03	13.62	76.11	1,446.85
9-6-19-0567	567	89.12	14.58	82.24	1,756.41
9-6-19-0891	891	91.33	14.94	68.79	1,315.82

Identity	Gallons	K	Mg	Ca	Na
9-1-19-0000	0	96.26	14.22	41.80	954.11
9-1-19-0783	783	97.09	13.15	66.93	1,343.63

Identity (Eluents)		K	Mg	Ca	Na
9-C3-19-ELU		24.76	8.25	54.30	81.71
9-C4-19-ELU		18.80	3.69	18.92	31.14

## Test Run #10 Acid/Alkali Operational Parameters

Acid/Alkali #10							
<b>Objective</b>	<b>Start Date/ End Date</b>	<b>Work Order for Analysis</b>	<b>Superlig Material</b>	<b>Total Gallon Processed</b>	<b>Flow Rate Processing</b>	<b>Flow Rate Regeneration</b>	<b>Change in Delta Pressure</b>
Repeat Run #8	10/20/2000	PS#00PS05306	Superlig 327	477	2 gal/min	0.5 gal/min	2.3-15.2
Identical	10/20/2000	PS#00PS05242	Superlig 310				
		PS#00PS06007					

pH Port 3	pH Port 6	Regenerate Solution	Gals Elution	Special Operations
11.79-3.75	12.00-4.54	4 M Sulfuric Acid	30 C3 30 C4	Zn 172.01 Pb 3.96 Cu 195.53 Ag 0.20 Cd 15.43 Ni 26.19 Cr 22.26 Fe 77.46
				The concentration of the wastestream was very high in the concentration of metals. This change was due to the dumping of a pickle bath from the PSNS Metal Preparation Facility.
				Pickle solution: Data Andy Greene hydrochloric acid (gal) 85.3 Foz/G diethylthiorea (lbs) 0.53 Foz/G Rodine-50 (gal) 0.14 Foz/G detergent (gal) 0.12 Foz/G water to balance
				Code 134, Analytical Lab PSNS

## Test Run #10 Acid/Alkali Analytical Data

Acid/Alkali: Test Run #10 Ports 2, 3 & 6; Tank 1; and Eluent Samples

Notes: 1) Values are from samples analyzed undigested; 2) Samples preserved prior to analysis;  
3) Detection limit is 0.100 ppm; 4) Values below should be considered estimates.

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
10-2-20-0036	36	172.016	3.956	195.529	0.203	15.426	26.19	22.262
10-2-20-0090	90	174.712	3.966	199.908	0.201	15.247	26.033	21.988
10-2-20-0315	315	175.433	3.992	200.068	0.198	15.23	25.887	21.951
10-2-20-0477	477	177.591	4.088	198.749	0.205	15.658	26.421	22.389

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
10-3-20-0018	18	0.118	-0.029	0.099	0.011	0.022	0.051	0.053
10-3-20-0036	36	0.390	0.008	0.320	0.010	0.051	0.093	0.069
10-3-20-0054	54	35.341	0.462	26.049	0.032	3.430	5.351	1.397
10-3-20-0072	72	89.262	1.450	76.022	0.060	7.012	12.141	6.744
10-3-20-0090	90	149.438	2.174	80.336	0.074	11.788	21.888	11.583
10-3-20-0108	108	108.402	1.952	98.394	0.101	9.235	15.657	10.881
10-3-20-0126	126	108.482	2.278	87.716	0.101	10.348	17.140	12.382
10-3-20-0153	153	111.554	2.414	91.829	0.122	10.718	17.785	12.955
10-3-20-0180	180	131.246	2.545	98.164	0.121	11.591	18.612	14.797
10-3-20-0207	207	135.740	2.752	118.958	0.146	12.232	20.022	14.894
10-3-20-0261	261	129.517	2.709	124.415	0.143	12.855	20.249	13.643
10-3-20-0315	315	150.291	2.961	137.993	0.159	12.942	20.962	15.645
10-3-20-0369	369	145.189	3.336	136.352	0.151	14.093	22.145	16.882
10-3-20-0423	423	158.472	3.264	150.985	0.172	14.240	22.762	17.287
10-3-20-0477	477	158.134	3.331	147.326	0.177	14.830	23.254	16.872

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
10-6-20-0018	18	0.090	0.005	0.088	0.011	0.005	0.015	0.049
10-6-20-0036	36	0.030	-0.034	0.037	0.009	0.003	0.013	0.029
10-6-20-0054	54	5.145	0.020	3.108	0.012	0.483	0.717	0.168
10-6-20-0072	72	0.014	-0.021	0.016	0.008	-0.013	-0.017	0.022
10-6-20-0090	90	0.052	0.003	0.045	0.011	0.005	0.017	0.009
10-6-20-0108	108	0.612	0.063	0.364	0.010	0.032	0.041	0.026
10-6-20-0126	126	21.054	0.099	10.119	0.022	2.375	3.147	0.239
10-6-20-0153	153	52.497	0.725	38.228	0.038	5.005	8.024	3.796
10-6-20-0180	180	68.731	1.133	47.676	0.048	6.837	10.543	5.285
10-6-20-0207	207	77.804	1.359	60.552	0.062	7.501	11.951	6.669
10-6-20-0261	261	87.427	1.654	61.878	0.071	9.027	13.598	6.951
10-6-20-0315	315	91.286	1.744	68.669	0.082	8.749	13.622	8.405
10-6-20-0369	369	88.303	1.709	62.137	0.071	9.076	13.394	8.537
10-6-20-0423	423	109.459	2.068	78.887	0.095	10.630	15.858	10.724
10-6-20-0477	477	112.468	2.269	85.264	0.099	11.232	16.580	13.619

**Continued Test Run #10**

<b>Identity</b>	<b>Gallons</b>	<b>Zn</b>	<b>Pb</b>	<b>Cu</b>	<b>Ag</b>	<b>Cd</b>	<b>Ni</b>	<b>Cr</b>
10-1-20-0000	0	173.177	3.943	186.130	0.194	15.264	26.531	23.000
10-1-20-0090	90	197.158	3.973	211.710	0.197	15.447	26.503	22.837
10-1-20-0153	153	183.784	4.037	200.481	0.204	15.447	26.586	22.911
10-1-20-0423	423	173.688	3.951	191.895	0.200	15.287	26.214	22.622

<b>Identity (Eluents)</b>	<b>Zn</b>	<b>Pb</b>	<b>Cu</b>	<b>Ag</b>	<b>Cd</b>	<b>Ni</b>	<b>Cr</b>
10-C3-20-EL1	598	11.7	1,040	ND	47.0	98.5	91.1
10-C3-20-EL2	601	11.6	1,050	ND	46.6	97.5	90.3
10-C4-20-EL1	735	11.0	813	ND	54.6	97.8	78.9
10-C4-20-EL2	737	11.2	818	ND	55.1	98.7	79.4

<b>Identity (Eluents)</b>	<b>Zn</b>	<b>Pb</b>	<b>Cu</b>	<b>Ag</b>	<b>Cd</b>	<b>Ni</b>	<b>Cr</b>
10-BF-3-W	82.318	2.108	38.348	0.022	6.605	8.807	23.907
10-BF-4-W	53.947	1.667	39.476	0.014	4.823	6.091	13.734
10-BF-3-CON	125.167	25.080	206.717	0.092	7.132	31.382	319.584
10-BF-4-CON	66.640	13.616	133.684	0.055	5.051	14.757	161.565

<b>Identity</b>	<b>Gallons</b>	<b>K</b>	<b>Mg</b>	<b>Ca</b>	<b>Na</b>
10-2-20-0018	18	198.62	25.16	139.39	2,999.18
10-2-20-0108	108	198.82	26.67	139.78	3,030.76
10-2-20-0477	477	203.68	27.39	148.52	3,163.46

<b>Identity</b>	<b>Gallons</b>	<b>K</b>	<b>Mg</b>	<b>Ca</b>	<b>Na</b>
10-3-20-0018	18	226.15	2.07	183.61	3,177.29
10-3-20-0108	108	213.87	35.91	146.42	3,075.35
10-3-20-0477	477	203.33	38.84	232.67	2,623.29

<b>Identity</b>	<b>Gallons</b>	<b>K</b>	<b>Mg</b>	<b>Ca</b>	<b>Na</b>
10-6-20-0018	18	27.26	4.00	69.16	3,678.00
10-6-20-0108	108	230.86	13.67	163.21	3,141.61
10-6-20-0477	477	196.60	30.62	165.95	3,180.97

<b>Identity</b>	<b>Gallons</b>	<b>K</b>	<b>Mg</b>	<b>Ca</b>	<b>Na</b>
10-1-20-0423	423	207.08	32.77	190.36	3,110.99

<b>Identity (Eluents)</b>	<b>K</b>	<b>Mg</b>	<b>Ca</b>	<b>Na</b>
10-C3-20-ELU	21.50	8.77	146.97	41.03
10-C4-20-ELU	24.62	14.90	187.04	122.26
10-BF-3-W	38.98	8.39	84.30	396.67

## Test Run #20 Chromium Operational Parameters

Objective	Start Date/ End Date	Work Order for Chemical A Analysis	Superlig Material	Special Characteristics of Industrial Wastestream	Total Gallons Processed	Flow Rate Processing	Flow Rate Regeneration	Change in Delta Pressure
Test to Determine that Superlig Selectively Removed Cr VI		PS#00PS05838 PS#00PS06081	Superlig 307	The wastestream contained surfactant from unknown source	1080	2 gal/min	0.55 gal/min	2.0-6.0
Allowing heavy and alkaline earth metals to pass through column			Chromium VI	Unit mg/l Zn 1.24 Pb 0.24 Cu 9.7 Ag ND Cd ND Ni 5.44			every 5.8 gallons	
Columns open and samples taken end of run to determine if precipitation taking place in the columns				Total Cr 123 Cr VI 70.2 Fe 14.4 Phosphous 2.60				

pH Port 2	pH Port 3	pH Port 6	Regenerate Solution	Start Conditioning of Column	Conditionomg for Regeneration	Concerns During Run	Remarks
6.73	2.26 to 3.47	2.63-2.50	4 M Sulfuric Acid	Column conditioned with water flush at 2 gal/min	9.6 gallons of water run	Would 1 M Sulfuric Acid reduce the all the Cr (VI) to Cr (III)?	Port 3 showed high Cr VI than Port 2 Concentration

## Test Run # 20 Chromium Analytical Data

Test #20 Chromium Wastestream Ports 2, 3 & 6; Tank 1; and Eluent Sample

Notes: 1) Values are from samples analyzed undigested; 2) Samples preserved prior to analysis;  
3) Detection limit is 0.100 ppm; 4) Values below should be considered estimates.

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
20-2-22-0010	10	0.060	-0.046	0.431	0.015	0.002	1.376	70.828
20-2-22-0210	210	0.354	-0.001	2.824	0.014	0.007	2.590	85.709
20-2-22-0450	450	0.028	-0.033	0.279	0.014	0.015	1.387	71.718
20-2-22-0900	900	0.053	-0.019	0.537	0.025	0.004	1.523	72.686
20-2-23-1080	1080	0.217	-0.016	1.748	0.015	0.006	2.149	79.765

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
20-3-22-0030	30	0.034	-0.015	0.214	0.001	0.001	1.279	0.414
20-3-22-0210	210	0.093	0.010	0.278	0.003	0.002	1.085	0.631
20-3-22-0480	480	0.044	0.004	0.257	0.001	0.000	0.943	5.041
20-3-22-0900	900	0.017	-0.079	0.103	0.002	-0.003	0.656	72.291

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
20-6-22-0210	210	0.098	-0.011	0.23	0.002	0.004	1.06	0.305
20-6-22-0900	900	0.026	-0.027	0.091	0.002	-0.001	0.644	1.416

Identity	Gallons	K	Mg	Ca	Na
20-2-22-0020	20	238.49	14.12	17.40	1,233.61
20-2-22-0510	510	261.86	15.77	16.04	1,401.72
20-2-23-1020	1020	224.41	13.20	17.72	1,252.90

Identity	Gallons	K	Mg	Ca	Na
20-3-22-0510	510	222.54	13.75	17.03	1,242.63
20-3-23-1020	1020	235.72	13.32	17.29	1,197.51

Identity	Gallons	K	Mg	Ca	Na
20-6-22-0510	510	21.64	2.60	5.38	15.61

Identity (Eluents)	K	Mg	Ca	Na
20-3-ELU-3BV	20.81	2.61	5.52	10.44
20-4-ELU-3BV	19.84	3.05	4.92	19.55
20-HUD-23-4	22.61	0.54	0.07	6.94

**Continued Test Run #20**

<b>Identity</b>	<b>Cr VI</b>
20-1-23-001	53.73
20-Tank-1-23-002	57.19
20-Tank-1-23-003	55.95
20-1-23-004	55.46
20-Tank-1-23-Hud-Col3	0.43
20-Tank-1-23-Hud-Col4	2.66

<b>Identity</b>	<b>Gallons</b>	<b>Cr VI</b>	<b>Cr</b>	<b>Cr III</b>
20-2-22-0010	10	55.71		
20-2-22-0030	30	34.93		
20-2-22-0060	60	35.42		
20-2-22-0090	90	38.40		
20-2-22-0120	120	30.86		
20-2-22-0150	150	26.78		
20-2-22-0180	180	33.57		
20-2-22-0210	210	55.71	85.71	30.00
20-2-22-0330	330	53.73		
20-2-22-0450	450	54.22	71.72	17.49
20-2-22-0600	600	57.19		
20-2-22-0840	840	56.45		
20-2-23-1080	1080	57.93	79.77	21.83

<b>Identity</b>	<b>Gallons</b>	<b>Cr VI</b>	<b>Cr</b>	<b>Cr III</b>
20-3-22-0010	10	0.00		
20-3-22-0020	20	0.00		
20-3-22-0030	30	0.00		
20-3-22-0060	60	0.00		
20-3-22-0120	120	0.00		
20-3-22-0180	180	0.00		
20-3-22-0240	240	0.03		
20-3-22-0300	300	0.27		
20-3-22-0360	360	0.47		
20-3-22-0420	420	1.99		
20-3-22-0480	480	3.03	5.04	2.01
20-3-22-0540	540	3.90		
20-3-22-0720	720	8.98		
20-3-22-0780	780	18.34		
20-3-22-0840	840	17.39		
20-3-22-0900	900	43.55	72.29	28.74
20-3-22-0960	960	88.80		
20-3-22-1020	1020	91.52		
20-3-23-1080	1080	93.86		

**Continued Test Run #20**

<b>Identity</b>	<b>Gallons</b>	<b>Cr VI</b>	<b>Cr</b>	<b>Cr III</b>
20-6-22-0010	10	0.00		
20-6-22-0020	20	0.00		
20-6-22-0030	30	0.00		
20-6-22-0060	60	0.00		
20-6-22-0120	120	0.00		
20-6-22-0210	210	0.00	0.31	0.30
20-6-22-0240	240	0.00		
20-6-22-0300	300	0.00		
20-6-22-0360	360	0.00		
20-6-22-0420	420	0.00		
20-6-22-0480	480	0.00		
20-6-22-0540	540	0.01		
20-6-22-0660	660	0.26		
20-6-22-0780	780	0.43		
20-6-22-0900	900	0.70	1.42	0.72
20-6-22-1020	1020	1.11		
20-6-23-0960	1080	1.15		

<b>Identity (Eluents)</b>	<b>Cr VI</b>
20-4-ELU-5BV	-0.09
20-4-ELU-4BV	-0.09
20-4-ELU-3BV	-0.09
20-4-ELU-2BV	-0.07
20-4-ELU-1BV	-0.04
20-3-ELU-5BV	-0.09
20-3-ELU-4BV	-0.09
20-3-ELU-3BV	-0.09
20-3-ELU-2BV	-0.07
20-3-ELU-1BV	-0.07
20-4-ELU-COMP	-0.09
20-3-ELU-COMP	-0.07

## Test Run #21 Chromium Operational Parameters

Chrome #21								
Objective	Start Date/ End Date	Work Order for Analytical Analysis	Superlig Material	Total Gallons Processed	Flow Rate Processing	Flow Rate Regeneration	# Gallons Eluant	Change in Delta Pressure
Increase the number of gallons processed over Run #20	08/24/2000 08/25/2000	PS#00PS06258 PS#00PS05838	Superlig 307 Chrome VI	1630	2 gal/min	0.5 gal/min	30 gallons Column #3 30 gallons Column #4	1.6-2.8
Chrome wastewater was diluted to approx half of Run #20								

Port 3	Port 6	Regenerate Solution
2.4-2.6	2.97-3.00	4M Sulfuric Acid

## Test Run #21 Chromium Analytical Data

Chromium Test #21 Ports 2, 3 & 6; Tank 1; and Eluent Samples

Notes: 1) Values are from samples analyzed undigested; 2) Samples preserved prior to analysis;  
3) Detection limit is 0.100 ppm; 4) Values below should be considered estimates.

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
21-2-24-0060	60	0.387	0.083	2.730	0.005	0.017	1.403	27.563
21-2-24-0230	230	0.425	0.003	3.065	0.002	0.011	1.629	33.004
21-2-24-0650	650	0.464	0.032	3.422	0.005	0.012	1.754	36.650
21-2-24-0950	950	0.463	-0.051	3.409	0.004	0.012	1.741	36.384
21-2-25-1730	1730	0.469	-0.011	3.426	0.004	0.011	1.779	36.289
21-2-25-2590	2590	0.477	-0.058	3.438	0.007	0.012	1.838	36.498

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
21-3-24-0010	10	0.466	0.025	3.058	0.003	0.012	1.770	2.313
21-3-24-0230	230	0.472	0.010	3.166	0.004	0.013	1.818	2.311
21-3-24-0650	650	0.457	-0.032	4.034	0.003	0.011	1.752	3.661
21-3-25-1730	1730	0.467	-0.002	3.381	0.009	0.019	1.758	6.635
21-3-25-2590	2590	0.476	0.014	3.321	0.006	0.014	1.828	17.124

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
21-6-24-0010	10	0.378	0.029	2.379	0.001	0.013	1.398	2.127
21-6-24-0030	30	0.463	0.018	3.005	0.001	0.013	1.769	2.128
21-6-24-0650	650	0.459	0.022	3.969	0.002	0.010	1.740	2.288
21-6-24-0950	950	0.458	-0.017	3.041	0.004	0.010	1.745	2.609
21-6-25-1730	1730	0.477	-0.012	3.257	0.002	0.012	1.791	3.724
21-6-25-2590	2590	0.481	0.017	3.288	0.007	0.012	1.867	5.273

Identity (Regenerates)		Zn	Pb	Cu	Ag	Cd	Ni	Cr
21-3-1-BV2		0.194	-0.261	0.758	0.002	0.026	0.161	2330.017
21-3-1-BV5		0.182	0.317	0.666	-0.005	0.025	0.105	128.748
21-4-1-BV2		0.193	0.219	0.670	-0.002	0.021	0.100	398.301
21-4-1-BV5		0.183	0.368	0.659	-0.002	0.022	0.098	26.297

**Continued Test Run #21**

<b>Identity</b>	<b>Gallons</b>	<b>K</b>	<b>Mg</b>	<b>Ca</b>	<b>Na</b>
21-2-24-0110	110	105.41	7.62	23.70	595.21
21-2-24-0530	530	109.92	7.85	19.47	552.95
21-2-24-1250	1250	106.45	8.27	20.08	586.36
21-2-25-2450	2450	109.64	8.08	25.01	535.50

<b>Identity</b>	<b>Gallons</b>	<b>K</b>	<b>Mg</b>	<b>Ca</b>	<b>Na</b>
21-3-24-0020	20	105.62	8.23	25.68	574.42
21-3-24-0530	530	101.18	7.49	25.24	603.29
21-3-24-1250	1250	113.94	7.76	24.12	614.70
21-3-25-2450	2450	107.98	7.53	24.41	564.24

<b>Identity</b>	<b>Gallons</b>	<b>K</b>	<b>Mg</b>	<b>Ca</b>	<b>Na</b>
21-6-24-0020	20	109.23	8.49	25.95	616.63
21-6-24-0530	530	103.82	7.86	25.01	612.61
21-6-24-1250	1250	106.38	8.47	24.63	617.84
21-6-25-2450	2450	108.53	7.64	25.59	608.14

<b>Identity</b>	<b>Cr VI</b>
21-01-24-001 Tank 2	31.44
21-01-24-002 Tank 2	32.18
21-01-24-003 Tank 2	32.67
21-01-24-004 Tank 2	31.19

**Continued Test Run #21**

<b>Identity</b>	<b>Gallons</b>	<b>Cr VI</b>	<b>Cr</b>	<b>Cr III</b>
21-2-24-0020	20	32.71	33.00	0.29
21-2-24-0230	230	23.57		
21-2-24-0590	590	33.21		
21-2-24-0950	950	32.22	36.38	4.16
21-2-24-1370	1370	31.73		
21-2-25-1850	1850	32.47		
21-2-25-2210	2210	31.23		
21-2-25-2590	2590	31.73	36.50	4.77

<b>Identity</b>	<b>Gallons</b>	<b>Cr VI</b>	<b>Cr</b>	<b>Cr III</b>
21-3-24-0010	10	0.00	2.31	2.31
21-3-24-0030	30	0.00		
21-3-24-0110	110	0.00		
21-3-24-0290	290	0.07		
21-3-24-0410	410	0.38		
21-3-24-0530	530	0.78		
21-3-24-0650	650	1.07	3.66	2.59
21-3-24-0830	830	1.99		
21-3-24-0101	1010	2.43		
21-3-24-1250	1250	3.17		
21-3-25-1610	1610	5.50		
21-3-25-1970	1970	8.09		
21-3-25-2330	2330	10.05		
21-3-25-2590	2590	13.02	17.12	4.10

<b>Identity</b>	<b>Gallons</b>	<b>Cr VI</b>	<b>Cr</b>	<b>Cr III</b>
21-6-24-0010	10	0.00	2.13	2.13
21-6-24-0030	30	0.00	2.13	2.13
21-6-24-0110	110	0.00		
21-6-24-0230	230	0.00		
21-6-24-0350	350	0.00		
21-6-24-0470	470	0.00		
21-6-24-0650	650	0.00	2.29	2.28
21-6-24-0830	830	0.10		
21-6-24-1010	1010	0.35		
21-6-25-1610	1610	1.22		
21-6-25-1970	1970	1.63		
21-6-25-2450	2450	2.04		
21-6-25-2590	2590	2.25	5.27	3.02

## Test Run #22 Chromium Operational Parameters

Chrome #22						
Objective	Start Date/	Work Order for Analysis	Superlig Material	Total Gallon	Flow Rate	Flow Rate
	End Date				Processed	Processing
Regenerate Column	09/05/2001	PS#00PS06463	Superlig 307	3652	2 gallon/min	0.5 gal/min
with NaOH	09/07/2001	PS#00PS07523	Chrome VI			
		PS#00PS07720				
Strip with NaOH and		PS#00PS08429				
then 0.2 M Sulfuric		PS#00PS07575				
		PS#00PS07576				

pH Start	pH Final	Regenerate
		Solution
2.3	3.1	NaOH

## Test Run # 22 Chromium Analytical Data

Chromium Test Run #22: Ports 2, 3 & 6; Tank 1; and Eluent Samples

Notes: 1) Values are from samples analyzed undigested; 2) Samples preserved prior to analysis;  
3) Detection limit is 0.100 ppm; 4) Values below should be considered estimates.

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
22-TANK-1-1		1.298	<0.10	8.505	0.019	0.032	4.476	48.333
22-TANK-1-2		1.307	<0.10	8.497	0.019	0.031	4.472	48.435
22-TANK-1-3		1.309	<0.10	8.511	0.019	0.029	4.490	48.338
22-TANK-1-4		1.324	<0.10	8.554	0.020	0.031	4.547	49.211
4-PSNS-CRPT-1		1.322	<0.10	8.625	0.021	0.032	4.556	48.878
5-PSNS-CRPT-1		1.318	<0.10	8.618	0.020	0.029	4.517	48.489

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
22-2-5-0000	0	1.042	0.060	6.719	0.000	0.030	4.189	84.498
22-2-5-0330	330	1.078	0.131	6.931	0.005	0.032	4.337	88.048
22-2-5-0810	810	1.040	0.189	6.696	0.002	0.029	4.207	84.533
22-2-7-3000	3000	1.172	0.009	7.418	0.003	0.029	4.193	64.037
22-2-7-3652	3652	1.215	0.053	7.698	0.003	0.030	4.351	64.113
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0
<b>Avg. Feed (mg/l)</b>		1.109	0.088	7.092	0.003	0.030	4.255	77.046
<b>Avg. Feed (mmol/l)</b>		0.017	0.000	0.112	0.000	0.000	0.072	1.482
<b>Feed (mmol)</b>		234.48	5.90	1,543.89	0.33	3.69	1,002.07	20,480.58

Liters Processed: 13,823

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
22-3-5-0000	0	0.419	0.048	2.124	0.000	0.010	1.607	23.758
22-3-5-0210	210	1.076	0.135	6.787	0.000	0.023	4.335	55.512
22-3-5-0330	330	1.075	0.119	6.820	0.001	0.024	4.313	57.248
22-3-5-0570	570	1.086	0.132	6.870	0.006	0.030	4.360	61.027
22-3-5-0810	810	1.066	0.140	6.798	0.004	0.026	4.306	63.956
22-3-6-1970	1970	1.074	0.125	6.793	0.004	0.039	4.317	81.811
22-3-7-3000	3000	1.211	0.056	7.267	0.000	0.036	4.298	61.655
22-3-7-3652	3652	1.212	0.051	7.484	0.003	0.030	4.329	61.733
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0
<b>mmol</b>		240.46	6.28	1,535.96	0.32	4.14	1,016.53	17,991.74

**Continued Test Run #22**

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
22-3-5-0000	0	0.419	0.048	2.124	0.000	0.010	1.607	23.758
22-3-5-0210	210	1.076	0.135	6.787	0.000	0.023	4.335	55.512
22-3-5-0330	330	1.075	0.119	6.820	0.001	0.024	4.313	57.248
22-3-5-0570	570	1.086	0.132	6.870	0.006	0.030	4.360	61.027
22-3-5-0810	810	1.066	0.140	6.798	0.004	0.026	4.306	63.956
22-3-6-1970	1970	1.074	0.125	6.793	0.004	0.039	4.317	81.811
22-3-7-3000	3000	1.211	0.056	7.267	0.000	0.036	4.298	61.655
22-3-7-3652	3652	1.212	0.051	7.484	0.003	0.030	4.329	61.733
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0
<b>mmol</b>		240.46	6.28	1,535.96	0.32	4.14	1,016.53	17,991.74

Liters Processed: **13,823**

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr
22-6-5-0000	0	0.032	-0.001	0.045	-0.002	-0.001	0.010	18.181
22-6-5-0210	210	1.058	0.107	6.578	-0.001	0.024	4.253	51.649
22-6-5-0330	330	1.068	0.099	6.717	0.001	0.025	4.304	52.704
22-6-5-0570	570	1.084	0.146	6.746	0.004	0.025	4.304	53.775
22-6-5-0810	810	1.068	0.157	6.712	0.002	0.025	4.276	54.898
22-6-5-1970	1970	1.060	0.108	6.776	0.003	0.025	4.248	61.504
22-6-5-3000	3000	1.207	0.059	7.122	0.004	0.035	4.319	46.546
22-6-7-3652	3652	1.225	0.060	7.301	0.005	0.045	4.299	54.411
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0
<b>Avg. Effluent (mg/l)</b>		0.975	0.092	6.000	0.002	0.025	3.752	49.209
<b>Effluent (mmol/l)</b>		35.591	3.800	221.403	0.065	0.864	140.003	1,837.715
<b>Effluent (mmol)</b>		239.50	6.07	1,512.42	0.43	3.85	1,008.99	14,413.23

Identity	Gallons	K	Mg	Ca	Na
22-2-5-0015	15	212.69	12.72	25.59	1,015.22
22-2-5-0270	270	220.88	13.14	25.75	1,114.75
22-2-5-1230	1230	220.25	13.82	27.74	1,207.24
22-2-6-1790	1790	219.00	12.51	19.50	1,134.44
22-2-7-3540	3540	227.81	14.43	24.94	1,613.67

Identity	Gallons	K	Mg	Ca	Na
22-3-5-0030	30	202.71	14.75	20.76	1,257.96
22-3-5-0270	270	222.12	14.51	21.53	1,264.81
22-3-5-1230	1230	217.34	15.17	17.27	1,086.93
22-3-6-1790	1790	207.35	13.28	22.36	1,413.75
22-3-7-3540	3540	217.89	15.34	22.66	1,592.72

Identity	Gallons	K	Mg	Ca	Na
22-6-5-0015	15	202.50	13.98	22.22	1,395.45
22-6-5-0270	270	211.37	15.41	22.50	1,441.15
22-6-5-1230	1230	210.61	15.22	22.00	1,412.14
22-6-6-1790	1790	215.47	15.33	21.99	1,420.15
22-6-7-3540	3540	214.98	13.90	22.86	1,572.84

## Continued Test Run #22

### Column 3 Bed volumes

Identity	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Fe	Cr VI	Cr III
22-0BV-C3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	192.93	<0.50	208.00	-15.08
22-1BV-C3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	412.02	3.32	296.00	116.02
22-2BV-C3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	289.98	3.56	186.00	103.98
22-3BV-C3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	227.79	3.27	126.00	101.79
22-4BV-C3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	181.36	3.29	73.90	107.46
22-5BV-C3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	141.13	2.65	45.70	95.43
22-6BV-C3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	129.98	2.84	32.40	97.58
22-7BV-C3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	117.75	2.67	26.30	91.45
22-8BV-C3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	107.39	2.49	21.90	85.49
22-10BV-C3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	98.79	2.67	18.40	80.39
22-COMP-C3-30	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	202.30	3.39	114.00	88.30
22-COMP-C3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	201.70	3.38	114.00	87.70
<b>Composite (l)</b>	<b>M.W.</b>	65.4	207.2	63.5	107.9	112.4	58.7	55.8	52.0	52.0
<b>113.55</b>	<b>mg/l (avg)</b>	N/A	N/A	N/A	N/A	N/A	202.001	3.386	114.000	88.001
	<b>mmol</b>	N/A	N/A	N/A	N/A	N/A	441.10	6.89	248.94	192.16

### Column 4 Bed volumes

Identity	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Fe	Cr VI	Cr III
22-0BV-C4	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	79.49	<0.50	79.40	0.09
22-1BV-C4	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	206.00	1.32	104.00	102.00
22-2BV-C4	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	170.28	1.27	57.50	112.78
22-3BV-C4	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	151.59	1.31	45.90	105.69
22-4BV-C4	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	135.43	1.19	32.10	103.33
22-5BV-C4	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	128.98	1.14	29.80	99.18
22-6BV-C4	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	116.22	1.12	24.60	91.62
22-7BV-C4	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	111.82	1.11	20.40	91.42
22-8BV-C4	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	111.84	1.23	15.60	96.24
22-10BV-C4	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	111.56	1.24	12.80	98.76
22-COMP-C4 30	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	141.67	1.29	40.80	100.87
22-COMP-C4	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	127.14	1.14	34.20	92.94
<b>Composite (l)</b>	<b>M.W.</b>	65.4	207.2	63.5	107.9	112.4	58.7	55.8	52.0	52.0
<b>113.55</b>	<b>mg/l (avg)</b>	N/A	N/A	N/A	N/A	N/A	134.402	1.216	37.500	96.902
	<b>mmol</b>	N/A	N/A	N/A	N/A	N/A	293.49	2.47	81.89	211.60

Identity	Cr VI
22-1-5-001	17.72
22-1-5-002	4.09

**Continue Test Run #22**

<b>Identity</b>	<b>Gallons</b>	<b>Cr VI</b>	<b>Cr</b>	<b>Cr III</b>
22-2-5-0000	0	11.03	84.498	73.46
22-2-5-0015	15	10.10		
22-2-5-0030	30	3.94		
22-2-5-0060	60	5.82		
22-2-5-0090	90	6.41		
22-2-5-0120	120	6.78		
22-2-5-0150	150	5.74		
22-2-5-0180	180	0.47		
22-2-5-0210	210	2.78		
22-2-5-0270	270	18.06		
22-2-5-0300	300	18.13		
22-2-5-0330	330	18.69	88.048	69.36
22-2-5-0360	360	17.51		
22-2-5-0390	390	5.89		
22-2-5-0450	450	11.89		
22-2-5-0570	570	8.73		
22-2-5-0690	690	5.89		
22-2-5-0810	810	1.69	84.533	82.84
22-2-5-0950	930	11.85		
22-2-5-1050	1050	19.07		
22-2-5-1230	1230	16.58		
22-2-6-1610	1610	16.58		
22-2-6-1790	1790	8.12		
22-2-6-1970	1970	5.89		
22-2-6-2622	2622	9.40		
22-2-6-3000	3000	11.98		
22-2-6-3180	3180	13.59		
22-2-6-3360	3360	13.96		
22-2-6-3540	3540	12.06		
22-2-6-3652	3652	13.34		
<b>M.W.</b>		52.0		
<b>Avg. Feed (mg/l)</b>		10.399		
<b>Avg. Feed (mmol/l)</b>		0.200		
<b>Feed (mmol)</b>		2,764.25		
<b>Liters Processed:</b>		<b>13,823</b>		

**Continue Test Run #22**

<b>Identity</b>	<b>Gallons</b>	<b>Cr VI</b>	<b>Cr</b>	<b>Cr III</b>
22-3-5-0240	240	0.05		
22-3-5-0270	270	0.00		
22-3-5-0330	330	0.46	57.248	56.78
22-3-5-0450	450	1.47		
22-3-5-0570	570	3.39	61.027	57.64
22-3-5-0810	810	4.28	63.956	59.67
22-3-5-1230	1230	7.89		
22-3-6-1450	1450	10.69		
22-3-6-1790	1790	12.05		
22-3-6-1970	1970	12.10	81.811	69.71
22-3-6-2586	2586	14.55		
22-3-6-2662	2662	12.14		
22-3-6-3000	3000	14.65		
22-3-6-3180	3180	15.59		
22-3-6-3360	3360	15.00		
22-3-6-3540	3540	15.34		
22-3-6-3652	3652	14.01		
<b>M.W.</b>		52.0		
<b>mmol</b>		0.00		

**Liters Processed: 13,823**

<b>Identity</b>	<b>Gallons</b>	<b>Cr VI</b>	<b>Cr</b>	<b>Cr III</b>
22-6-5-0570	570	0.22	53.775	53.56
22-6-5-0810	810	0.90	54.898	54.00
22-6-5-1050	1050	1.38		
22-6-5-1230	1230	2.14		
22-6-6-1970	1970	5.32	61.504	56.18
22-6-6-2586	2586	6.02		
22-6-6-2662	2662	6.56		
22-6-6-3000	3000	8.34		
22-6-6-3180	3180	9.10		
22-6-6-3360	3360	9.89		
22-6-6-3540	3540	12.18		
22-6-6-3652	3652	11.05		
<b>M.W.</b>		52.0		
<b>Avg. Effluent (mg/l)</b>		6.091		
<b>Effluent (mmol/l)</b>		71,398.054		
<b>Effluent (mmol)</b>		1,373.04		

**Liters Processed: 13,823**

### Continued Test Run #22

	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Cr VI
<b>Recovery (%)</b>	N/A	N/A	N/A	N/A	N/A	N/A	3.6	2.4
<b>C3 Mass Balance (%)</b>	N/A	N/A	N/A	N/A	N/A	N/A	72.53	11.73
<b>C3 &amp; C4 Mass Bal. (%)</b>	N/A	N/A	N/A	N/A	N/A	N/A	74.0	12.3

### Mass Balance, Run #22

	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Cr VI
<b>Feed (mmol)</b>	234.5	5.9	1,543.9	0.3	3.7	1,002.1	20,480.6	13,822.8
<b>Feed (mg/l)</b>	1.1	0.1	7.1	0.0	0.0	4.3	77.0	0.2
<b>Effluent (mmol)</b>	239.5	6.1	1,512.4	0.4	3.9	1,009.0	14,413.2	1,373.0
<b>Effluent (mmol/l)</b>	0.017	0.000	0.109	0.000	0.000	0.073	1.043	5.165
<b>Eluate (mmol)</b>	N/A	N/A	N/A	N/A	N/A	N/A	734.59	330.82
<b>Extraction (%)</b>	-2.1	-2.9	2.0	-28.6	-4.4	-0.7	29.6	90.1
<b>Mass Balance (%)</b>	<b>N/A</b>	<b>N/A</b>	<b>N/A</b>	<b>N/A</b>	<b>N/A</b>	<b>N/A</b>	<b>74.0</b>	<b>12.3</b>

**Note:** Effluent Concentrations calculated on metal present in 3,652 gal processed. Assumes ND=0.0.  
 Add PQL value for worst case. Extraction calculated from (Feed-Eff.)/Feed x100.

## Test Run #23 Chromium Operational Parameters

MRT Chrome #23 Objective	Start Date/End Date	Work Order for Analy of Samples at PSNS	Superlig Material	Total Gall Processed	Flow Rate Processing	Flow Rate Regeneration	Change in Delta Pressure	pH Start/Fin	Regeneration Solution
Calculate Mass	10/27/2000	PS#00PS08429	Superlig 307	3652	2 gal/min	0.32-0.37 gal/m	no change	ph=5.32/4.5	1 M NaOH
Balance of Columns #3 and #4	10/29/2000	PS#00PS07653	Chrome VI only						
Slower Eluation Rate									

Conditioning of Columns for Processing	Conditioning of Column Start for Regeneration	Complications During Run
Column Stripped with 4M Sulfuric Acid to ensure all chrome stripped from column from Run #22 which was stripped with 1 M NaOH	Flushed with with approximately 20 gallons water	The totalizer stopped several times during the run. Correction: The time interval was multiplied by the flow rate.
Next column flushed with 40 gallons water		
Next column flushed with 0.2 M Sulfuric Acid		
Processing of wastewater began		

## Test Run #23 Chromium Analytical Data

Operation Test Run #23 Chromium  
Ports 2, 3 & 6; Tank 1; and Eluent Samples

Notes: 1) Values are from samples analyzed undigested; 2) Samples preserved prior to analysis;  
3) Detection limit is 0.100 ppm; 4) Values below should be considered estimates.

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Cr VI	Fe	Cr III
23-0-24-0001	1	4.29	<0.20	53.00	0.03	0.62	7.36	51.00	45.30	0.53	5.70
23-0-24-0002	2	4.28	<0.20	51.80	0.03	0.62	7.42	50.40	47.60	0.42	2.80

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Cr VI	Fe	Cr III
23-2-27-0010	10	3.18	<0.10	46.16	0.04	0.45	5.47	47.60	2.01	2.87	45.59
23-2-27-0020	20	3.97	<0.10	58.28	0.03	0.57	6.80	58.34	33.52	3.11	24.82
23-2-27-0040	40	3.95	<0.10	58.65	0.03	0.57	6.77	58.23	37.15	3.14	21.08
23-2-27-0080	80	3.94	<0.10	58.66	0.04	0.57	6.74	57.65	40.18	2.60	17.47
23-2-27-0120	120	3.99	<0.10	58.92	0.03	0.57	6.82	57.34	41.03	1.80	16.31
23-2-27-0160	160	4.01	<0.10	58.88	0.03	0.58	6.87	57.44	38.85	1.48	18.59
23-2-27-0200	200	4.01	<0.10	58.66	0.03	0.58	6.90	56.72	39.34	1.16	17.38
23-2-27-0240	240	4.00	<0.10	58.56	0.03	0.57	6.82	56.05	40.42	1.14	15.63
23-2-28-1080	1,080	3.95	<0.10	57.16	0.04	0.57	6.73	55.32	40.18	2.03	15.14
23-2-28-1560	1,560	3.89	<0.10	57.79	0.03	0.55	6.64	57.04	35.58	3.24	21.46
23-2-28-1920	1,920	3.98	<0.10	57.18	0.03	0.57	6.79	55.64	38.73	2.16	16.91
23-2-29-2040	2,040	3.68	<0.10	52.61	0.03	0.53	6.29	51.84	39.09	2.07	12.75
23-2-29-2160	2,160	3.95	<0.10	56.02	0.03	0.57	6.71	55.13	37.03	2.11	18.10
23-2-29-2400	2,400	4.01	<0.10	57.52	0.03	0.57	6.81	56.21	37.15	2.07	19.06
23-2-29-2640	2,640	4.00	<0.10	55.24	0.04	0.57	6.81	54.04	37.27	1.86	16.77
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0	52.0	55.8	52.0
<b>Avg. Feed (mg/l)</b>		3.900	N/A	56.686	0.034	0.560	6.664	55.639	35.835	2.187	19.804
<b>Avg. Feed (mmol/l)</b>		0.060	N/A	0.893	0.000	0.005	0.114	1.070	0.689	0.039	0.381
<b>Feed (mmol)</b>		595.93	N/A	8,920.20	3.12	49.80	1,134.38	10,691.65	6,886.17	391.63	3,805.48

Liters Processed: 9,992

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Cr VI	Fe	Cr III
23-3-27-0010	10	1.65	<0.10	1.36	0.02	0.25	2.44	5.31	0.01	0.26	5.30
23-3-27-0020	20	3.92	<0.10	28.73	0.04	0.54	6.76	8.73	0.01	0.94	8.72
23-3-27-0040	40	4.03	<0.10	49.65	0.05	0.57	6.92	9.94	0.02	1.15	9.92
23-3-27-0080	80	3.96	<0.10	51.86	0.04	0.57	6.78	9.57	0.02	0.66	9.55
23-3-27-0120	120	3.98	<0.10	52.84	0.03	0.56	6.77	9.82	0.03	0.51	9.79
23-3-27-0160	160	4.02	<0.10	53.09	0.04	0.59	6.87	10.72	0.02	0.54	10.70
23-3-27-0200	200	4.04	<0.10	53.77	0.03	0.58	6.93	12.20	0.02	0.54	12.18
23-3-27-0240	240	4.01	<0.10	53.60	0.04	0.58	6.90	13.62	0.03	0.53	13.59
23-3-27-0480	480	4.00	<0.10	53.67	0.04	0.58	6.87	22.09	6.50	0.41	15.59
23-3-27-0720	720	4.00	<0.10	53.95	0.04	0.57	6.81	31.53	16.43	0.39	15.10
23-3-28-1080	1,080	4.04	<0.10	53.76	0.04	0.58	6.89	44.68	30.61	0.39	14.07
23-3-28-1560	1,560	4.00	<0.10	53.89	0.04	0.57	6.87	52.69	39.50	0.44	13.19
23-3-28-1920	1,920	4.01	<0.10	53.25	0.04	0.57	6.83	55.11	40.67	0.40	14.44
23-3-29-2040	2,040	4.00	<0.10	44.22	0.04	0.58	6.83	53.94	41.88	0.36	12.06
23-3-29-2160	2,160	3.94	<0.10	50.63	0.04	0.57	6.71	52.77	43.70	0.35	9.07
23-3-29-2400	2,400	4.03	<0.10	53.08	0.04	0.59	6.89	52.95	41.03	0.51	11.92
23-3-29-2640	2,640	4.00	<0.10	52.64	0.04	0.57	6.82	52.31	40.55	0.54	11.76
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0	52.0	55.8	52.0
<b>mmol</b>		610.72	N/A	8,277.85	3.53	50.99	1,162.84	8,350.83	5,821.49	79.27	2,529.34

Liters Processed: 9,992

### Continue Test Run #23

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Cr VI
23-6-27-0010	10	0.02	<0.10	0.01	0.01	0.00	0.01	9.99	0.00
23-6-27-0020	20	2.05	<0.10	0.13	0.02	0.29	2.95	10.42	0.00
23-6-27-0040	40	4.13	<0.10	36.71	0.04	0.57	7.24	13.45	0.02
23-6-27-0080	80	3.96	<0.10	49.96	0.04	0.57	6.78	12.75	0.03
23-6-27-0120	120	4.01	<0.10	50.87	0.04	0.58	6.87	11.54	0.03
23-6-27-0160	160	4.01	<0.10	51.32	0.04	0.57	6.90	10.75	0.03
23-6-27-0200	200	4.04	<0.10	51.87	0.04	0.58	6.91	10.15	0.03
23-6-27-0240	240	4.00	<0.10	51.88	0.03	0.57	6.87	9.82	0.02
23-6-27-0480	480	4.05	<0.10	52.63	0.04	0.58	6.97	11.38	0.03
23-6-27-0720	720	3.97	<0.10	52.35	0.04	0.57	6.81	13.90	0.12
23-6-28-1080	1,080	3.95	<0.10	51.03	0.04	0.56	6.76	17.64	5.29
23-6-28-1560	1,560	3.98	<0.10	51.26	0.04	0.57	6.80	29.00	15.83
23-6-28-1920	1,920	3.96	<0.10	51.24	0.04	0.57	6.80	38.53	27.82
23-6-29-2040	2,040	3.98	<0.10	34.95	0.04	0.57	6.84	39.56	31.10
23-6-29-2160	2,160	3.99	<0.10	47.45	0.04	0.57	6.83	43.61	36.91
23-6-29-2400	2,400	4.02	<0.10	50.93	0.04	0.58	6.86	48.64	36.43
23-6-29-2640	2,640	4.00	<0.10	51.17	0.04	0.57	6.85	51.87	42.00
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0	52.0
<b>Avg. Effluent (mg/l)</b>		3.654	N/A	43.280	0.035	0.522	6.238	22.528	11.511
<b>Effluent (mmol/l)</b>		90.757	N/A	1,046.810	0.874	12.931	155.383	405.758	125.372
<b>Effluent (mmol)</b>		605.76	N/A	7,866.87	3.55	50.36	1,156.20	5,603.24	3,387.90

**Liters Processed: 9,992**

Identity	Gallons	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Cr VI
23-1-27-0000	0	4.50	<0.10	53.96	0.03	0.63	7.59	49.01	37.88
23-1-27-0480	480	4.48	<0.10	54.94	0.03	0.63	7.61	50.44	35.34
23-1-28-1440	1,440	4.49	<0.10	55.91	0.03	0.62	7.70	50.35	34.97
23-1-29-2400	2,400	4.43	<0.10	51.36	0.03	0.62	7.44	46.87	35.58
<b>M.W.</b>		65.4	207.2	63.5	107.9	112.4	58.7	52.0	52.0
<b>mg/l (avg)</b>		4.474	N/A	54.042	0.027	0.623	7.585	49.169	35.943
<b>mmol/l (avg)</b>		0.068	N/A	0.851	0.000	0.006	0.129	0.946	0.691

**Continued Test Run #23**

Identity (Eluents)	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Cr VI
23-BVOH-C3	0.27	0.39	41.75	0.04	0.03	0.42	371.91	244.31
23-BV0-C3	0.35	0.64	65.64	0.07	0.04	0.53	621.43	407.89
23-BV1-C3	0.02	<0.10	11.01	0.10	<0.02	<0.02	1,648.00	1,676.00
23-BV2-C3	0.02	<0.10	12.98	0.09	<0.02	<0.02	649.03	755.19
23-BV3-C3	0.02	<0.10	14.48	0.09	<0.02	<0.02	346.10	378.81
23-BV4-C3	0.02	<0.10	14.10	0.09	<0.02	<0.02	96.65	58.84
23-BV5-C3	0.02	<0.10	13.69	0.09	<0.02	<0.02	35.15	18.49
23-BV6-C3	0.03	<0.10	13.34	0.09	<0.02	<0.02	19.44	8.56
23-BV7-C3	0.02	<0.10	12.56	0.09	<0.02	<0.02	13.40	8.80
23-BV8-C3	0.02	<0.10	11.55	0.09	<0.02	<0.02	10.23	2.34
23-BV9-C3	0.02	<0.10	6.28	0.09	<0.02	<0.02	19.04	1.79
23-BV10-C3	0.02	<0.10	9.32	0.09	<0.02	<0.02	9.26	6.01
23-BV11-C3	0.02	<0.10	9.11	0.09	<0.02	<0.02	5.40	2.01
23-COMP-C3	0.01	<0.10	9.43	0.09	<0.02	<0.02	393.68	411.07
23-COMP-C3	0.02	<0.10	9.50	0.09	<0.02	<0.02	394.77	428.49
Composite (l)	M.W.	65.4	207.2	63.5	107.9	112.4	58.7	52.0
179.41	mg/l (avg)	0.013	N/A	9.463	0.091	N/A	N/A	394.220
	mmol	0.04	N/A	26.74	0.15	N/A	N/A	1,360.13

Identity (Eluents)	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Cr VI
23-BV0-C4	0.124	<0.10	19.22	0.04	<0.02	0.121	860.16	781.85
23-BV1-C4	<0.02	<0.10	6.25	0.07	<0.02	<0.02	1,424.86	1,431.32
23-BV2-C4	<0.02	<0.10	5.12	0.10	<0.02	<0.02	2,221.08	1,964.46
23-BV3-C4	<0.02	<0.10	3.16	0.10	<0.02	<0.02	420.88	481.35
23-BV4-C4	<0.02	<0.10	2.70	0.09	<0.02	<0.02	83.42	92.85
23-BV5-C4	<0.02	<0.10	2.67	0.10	<0.02	<0.02	29.67	20.92
23-BV6-C4	<0.02	<0.10	2.81	0.10	<0.02	<0.02	16.59	13.04
23-COMP-C4	<0.02	<0.10	2.80	0.09	<0.02	<0.02	601.16	693.85
23-COMP-C4	<0.02	<0.10	2.83	0.09	<0.02	<0.02	604.89	669.61
Composite (l)	M.W.	65.4	207.2	63.5	107.9	112.4	58.7	52.0
179.41	mg/l (avg)	0.000	N/A	2.816	0.089	N/A	N/A	603.022
	mmol	N/A	N/A	7.95	0.15	N/A	N/A	2,080.54

### Continued Test Run # 23

	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Cr VI
Recovery (%)	N/A	N/A	0.4	9.6	N/A	N/A	32.2	55.2
C3 Mass Balance (%)	N/A	N/A	88.5	118.7	N/A	N/A	65.1	70.2
C3 & C4 Mass Bal. (%)	N/A	N/A	88.6	123.4	N/A	N/A	84.6	104.4

### Mass Balance, Run #23

	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Cr VI
Feed (mmol)	595.9	N/A	8,920.2	3.1	49.8	1,134.4	10,691.7	6,886.2
Feed (mg/l)	3.9	N/A	56.7	0.0	0.6	6.7	55.6	35.8
Effluent (mmol)	605.8	N/A	7,866.9	3.5	50.4	1,156.2	5,603.2	3,387.9
Effluent (mmol/l)	0.009	N/A	0.105	0.000	0.001	0.016	0.041	0.013
Eluate (mmol)	N/A	N/A	34.69	0.30	N/A	N/A	3,440.68	3,800.41
Extraction (%)	-1.7	N/A	11.8	N/A	-1.1	-1.9	47.6	50.8
Mass Balance (%)	N/A	N/A	88.6	123.4	N/A	N/A	84.6	104.4

Note: Effluent Concentrations calculated on metal present in 2,640 gal processed. Assumes ND=0.0.  
Add PQL value for worst case. Extraction calculated from (Feed-Eff.)/Feed x100.

Identity	Gallons	K	Mg	Ca	Na
23-2-27-0020	20	34.54	13.71	26.31	1,563
23-2-28-1920	1920	36.20	12.82	25.42	1,486
23-2-29-2400	2400	35.51	12.61	25.94	1,598

Identity	Gallons	K	Mg	Ca	Na
23-3-27-0020	20	36.83	13.02	16.49	979
23-3-27-0720	720	36.20	12.92	26.04	1,590
23-3-28-1920	1920	38.08	12.80	25.37	1,508
23-3-29-2400	2400	40.29	13.87	26.45	1,564

Identity	Gallons	K	Mg	Ca	Na
23-6-27-0020	20	32.11	9.96	18.07	1,184
23-6-27-0720	720	36.90	15.49	22.17	1,308
23-6-28-1920	1920	35.16	13.14	25.39	1,557
23-6-29-2400	2400	35.20	12.84	26.03	1,551

Identity (Eluents)	K	Mg	Ca	Na
23-BV0-C3	47.72	0.66	0.26	26,948
23-BV1-C3	41.96	0.65	0.40	25,052
23-COMP-C3	45.70	0.74	0.32	25,013
23-BV2-C4	42.31	0.70	0.28	25,962

**Continued Test Run #23**

<b>Identity</b>	<b>Gallons</b>	<b>Hex Cr</b>
<b>23-3-27-0360</b>	360	0.034
<b>23-3-27-0600</b>	600	4.30
<b>23-3-27-0854</b>	854	10.73
<b>23-3-29-2280</b>	2,280	29.37
<b>23-6-27-0600</b>	600	0.03
<b>23-6-27-0857</b>	857	0.04
<b>23-6-28-0966</b>	966	2.99
<b>23-6-29-2280</b>	2,280	27.40
<b>23-6-29-2520</b>	2,520	31.97

## Special Tests

### Special Samples During MRT Pilot Demonstration

Test	Identification No	General Conclusions
Tests Metal Contaminants in NaOH and Sulfuric Acid Tanks	99PS02655	High content of nickel and chromium found in IWPF supply tanks for NaOH and sulfuric acid
Sludge Prior to Dryer	00PS06464	Sludge samples at different times showed consistent results
Shipyards Water	99PS02618	Tests did not show water high in iron as expected.
Fluoride in shipyard Water	99PS02892	Shipyards water was < 1 ppm in fluoride content
Carbon Analysis of MRT Column Water to Determine If Organics from Superlig Leaching	00PS05913	Tests shows that there was no breakdown of the Superlig material

### Table Tests for Metals Content in Sulfuric and NaOH PSNS Report Number 99PPS02655

sample	Trace #	Description	Zn	Pb	Cu	Ag	Cd	Ni	Cr	Fe
2655-1	IF-NAOH-Tank-98-1026	NaOH Supply Tank	ND	ND	ND	ND	ND	ND	ND	ND
2655-2	IF-H2S04-Tank 98	Sulfuric Acid Tank	ND	ND	1.09	ND	ND	16.8	22	218
2618-6	2-S2-97-1320-M	Shipyards Water	0.065	0.032	0.124	ND	ND	ND	ND	0.1

**Shipyard Potable Water  
PSNS Test No. 99PS02618**

<b>Metal</b>	<b>Concentration mg/l</b>
<b>Zinc</b>	<b>0.065</b>
<b>Lead</b>	<b>0.0032</b>
<b>Copper</b>	<b>0.12</b>
<b>Silver</b>	<b>ND</b>
<b>Cadmium</b>	<b>ND</b>
<b>Nickel</b>	<b>ND</b>
<b>Chromium</b>	<b>ND</b>
<b>Iron</b>	<b>0.13</b>
<b>Calcium</b>	<b>7.24</b>
<b>Magnesium</b>	<b>1.84</b>

**Fluoride Test: 99PS02892**

A sample of treated effluent was analyzed to determine the fluoride content. The instrumental analysis of the sample indicated less than one PPM of fluoride present.

**Port Water Test of MRT: 00PS05913**

Two different samples were taken to test for organic content. If the organic content increased in the column, then there was breakdown of the macrocyclic polyethers. The analysis results showed for sample 001, 18 mg/l and sample 002 , 14 mg/l.

**Table of Sludge Analysis Prior to IWPF Sludge Dryer  
PSNS Test 00PS06464**

<b>Metal</b>	<b>Sample 8-30-2000 mg/l</b>	<b>Sample 9-7-2000 mg/l</b>
Zinc	18800	19200
Lead	759	767
Copper	27600	24700
Silver	184	190
Cadmium	1510	1590
Nickel	3870	3970
Chromium	12600	12700
Phosphorus	3800	3910



# **APPENDIX C**

## **Molecular Recognition Technology Skid Design & Operation**



# Molecular Recognition Technology Skid Design & Operation Manual

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## I. INTRODUCTION

This manual has been developed to help maximize and maintain the system's operating and process efficiency.

A general flow diagram and P&ID (process and instrumentation) of the system is included in the design drawings. This drawing will be referenced in the discussion that follows.

## II. MRT SYSTEM OPERATING INSTRUCTIONS

The following sections will describe operation of the US Navy MRT Pilot/Production Plant. In addition to this description, the manual includes four component manuals provided by manufacturers of major portions of the control system. These are the Cole-Parmer Digital Panel Meter, Seametrics Flow Sensor, Baldor Adjustable Speed DC Motor Controller and the ECO Gearchem Pulsafeeder pump. This operating instruction will refer to the manufacturers' manuals when appropriate.

### A. Plant Piping Design

The plant is designed for manual operation with semi-automatic control of the batch processes associated with pre and post wash, feed, and elution. Four separate intake lines are provided on the intake header array. One additional independent feed is installed on the header for future use if needed. These intake lines are intended to be piped directly to individual supply tanks. The source liquids are then controlled by the operator using the valves on the intake header.

**Important: All air must be purged from the supply piping to the pump. The pump must not be allowed to dry prime from a new source of liquid. It is strongly recommended that all supply tanks to the plant provide a net positive suction head at the pump intake and that all intake lines are purged and maintained air tight and liquid filled during operation of the test plant.**

The pump is a progressive cavity metering pump driven by a variable speed DC motor. The pump curve for this pump is noted on page 44 of the Pump Manual, G8. All pump curves noted for the G8 pump are available for this variable speed design. The character of these types of pumps provides for stable operation, on a specific pump curve, when the backpressure is maintained constant. The valve array down stream from the pump is designed to provide a steady backpressure during operation that is above the potentially variable backpressure of the process. For example, if the process is running at a flow of 5 gpm and a backpressure (delta-P) of between 3 and 6 psi, the backpressure valve is set for approximately 10 psi at the specified flow. The pump then operates in a very stable manner at 5 gpm and any backpressure up to 10 psi. The back pressure setting will be set by IBC start-up personnel for the pilot phase of this operation and will not require adjustment until full scale operation at 21 gpm is required. The backpressure, and thus the desired pump curve, is easily adjusted by varying the spring setting on the backpressure valve.

Each time the pump is started into a new valve arrangement, it is recommended that flow be verified by observing rotation of the turbine in the flow sensor. In the event that the valves have

not been properly set and a flow path does not exist, the backpressure in the system will rise and there will be no flow at the flow sensor.

Valve arrangements will correspond to Column 1 lead - Column 2 trail and vice versa for feed, wash, and elution. For pilot plant testing, the same approach applies to Columns 3 and 4. The valves that are operated to set up each of these modes of operation are labeled V11, V12, V13, V21, V22, and V23 for Columns 1 and 2. For Columns 3 and 4, the valves are V31, V32, V32, V42, V33 and V43. Valves V11, V21, V31, and V41 are operated from the Motor Valve Control Center. The other valves on the plant are for isolation purposes or non-standard operations and should not be operated during normal testing. In general, the valves that will be regularly operated are within easy reach or are motor operated.

The effluent from the plant, during normal operation at 2 to 21 gpm, flows through a second backpressure array. This design feature provides a method of maintaining a system pressure at the column tops that is above ambient. This will prevent the columns from draining or creating a siphon when the feed pump is shut down.

Operation of plant in pilot mode, at a reduced flow of 2 to 5 gpm, involves the use of Columns 3 and 4. The pilot phase is intended to be performed under the direct supervision of IBC personnel and care must be observed to prevent operation of Columns 3 and 4 in excess of 5 gpm.

## **B. Flow Indicator and Batch Controller**

When the control panel is energized by closing the Main Breaker, the panel flow meter will activate and perform self tests. This meter was programmed during testing at IBC and the flow totalizer was calibrated for water. The operation and programming of this meter is described in detail in the enclosed Cole-Parmer operating manual. Several key operations will be described here that consist of the variables that are adjusted during normal testing.

**K Factor Scaling.** The K factor for the flow sensor is noted on the name plate for the sensor. The factor was converted to gal/min and the value entered into the meter for initial calibration testing. The value was then adjusted in a calibration procedure using water. This calibration can be checked at any time using a stop watch and container graduated in gallons. The meter responds to "pulses per gallon" so an indicated volume measurement that is short results from too many pulses/unit in comparison to the amount of liquid actually measured. The meter can be adjusted by reducing proportionally the pulses per gallon. During the following test, the same time and volume measurement is run and the same number of pulses will be indicated as more volume units, more closely matching the actual volume measurement. Conversely, if the indicated volume is more than actually measured, the number of pulses per unit should be increased proportionally.

Totalizer set points one and two can be reset within the programming procedure, as noted in the manufacturers manual, or can be easily reset by pressing and holding Enter key for 3 seconds.

The flow indicator and totalizer can show rate or total alternately by pressing Enter. The display will shift to rate or total each time the Enter key is pressed.

Reset of the Totalizer set points 1 and 2 can be accomplished by pressing external reset button.

### **C. Variable Speed Control**

The DC variable speed control was tested at our factory and preset for speed control. The controller should not require adjustment during testing operations. In the event that adjustment or changes are required, please refer to the enclosed Baldor manual. A 10-amp fuse is necessary in the armature circuit. This has been installed in the factory.

### **D. Control Panel Operations**

The sequence of operation of the control panel and plant are described in the following points. Please refer to the attached circuit diagram for details on the control logic. The four valves that function as inlet controls for the columns are motorized and controlled from the Motor Valve Control Center. These actuators were specifically designed such that the valves will remain in the last position on loss of power. Therefore, the valves for the two columns not in use may be energized, set to the closed position, and then de-energized by moving the main switch to the OFF position. These valves will remain closed until power is restored and the selector switches are moved to a new position. The bank of valves for the columns that are in use may remain independently energized, indicated by the green panel light, during operation.

Energize the panel and the proper bank of inlet valves and allow for a 30 minute warm up period prior to start of testing. The totalizer will energize and self test on application of power (220 VAC, 50-60 Hz).

Set the plant manual valves for a specific mode of operation. Set the motor valves for the columns in use, making sure that the valves for the pair of columns not in use are in the Closed position.

Verify that the intended flow path is open by visually following the flow from the intake array to the discharge.

Set the approximate speed level at the variable speed selector based on previous operations or verify the selected speed is correct. If the required speed (flow rate) is not known set to a lower level and adjust appropriately. Please note that the pilot columns, Numbers 3 and 4, should not exceed 5 gpm of flow rate.

Set operation for Manual or Automatic using the Off-Manual-Auto switch.

If manual operation is selected, closing the Pump Start switch on the DC controller will start the pump at a preset flow rate. The pump will operate until disabled by the operator.

If automatic batch operation is selected, check set points 1 and 2 using the Enter key on the batch controller.

When the set points are verified, start the pump by closing the Pump Start Switch. There will be a brief delay between controller activation and motor start.

Verify that the pump is turning and the flow is passing the visual flow sensor.

Verify that the flow rate is correct. If not, adjust by changing the motor speed. Verify that the totalizer is operating by pressing the Enter button once to toggle to totalizer. Verify that the Pump Back Pressure Array is maintaining the desired back pressure on the pump (approximately 10 to 15 psi). Verify that the Column Back Pressure Array is maintaining approximately 5 to 8 psi of pressure in the system. Please note that once these back pressures are set for a specific test series, it is unlikely that any adjustment will be necessary.

When the preset volume has been reached, the batch controller will stop the pump. The Pump Start switch should be moved to Stop and the mode select switch to OFF.

The operator may then proceed to the next step in the process, changing the manual valves appropriately. The operator may reset set point one for the next batch or use the preset set point two. Note that moving the selector to Set Point 2, or resetting Set Point 1, will reactivate the pump unless the operator has disabled the pump using the Pump Start/Stop Switch or the OFF setting on the mode switch.

## **E. Manuals**

**Cole-Parmer Digital Panel Meter 94788-20**

**Seametrics SPX Low Flow Meter**

**Eco-Gearchem Pulsafeeder G2 and Baldor 2 HP BEC805 DC Motor**

**Baldor Motors and Drives**

**Bettis TorqPlus EM-Series Actuators**

August 17, 1999

Dr. Katherine Ford, Ph. D.  
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1100 23<sup>rd</sup> Ave.  
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**Re: Supplemental Questions on IBC MRT Process Skid**

Dear Dr. Ford:

You have requested that we address several questions regarding the upgraded MRT Skid. We will restate the questions as we understand them and provide the information you need to better understand the operation and design issues.

Columns 1 and 2, 30 inches in diameter and 60 inches tall, were designed for full operation based on 30,000 gallons in 24 hours or about 21 gpm. The columns are constructed from SDR 17 (high density polyethylene). Piping is CPVC (Chlorinated Polyvinyl Chloride). These materials were selected to be compatible with the concentrations of H<sub>2</sub>SO<sub>4</sub> used on this skid. The HDPE and CPVC materials are also compatible with temperatures up to about 160F, though this application will not involve elevated temperatures. Valves and fittings are CPVC. Pressure control valves and the flow sensor are made of PP (polypropylene) with Teflon diaphragms and Viton seals. The metering pump is a Pulsafeeder Chemical Pump (G8) compatible with the fluids in this system.

Columns 3 and 4 are intended for pilot operation. These are 10 inches in diameter and 24 inches tall. These columns are made from CPVC. CPVC material is practical up to a diameter of 12 inches. From 12 inches up to 30 inches, HDPE is recommended.

Design operating pressure in the MRT systems is based on the expected delta-P through the two columns in series (lead-trail) configuration. This delta-P should not exceed 12 psi or approximately 6 psi per column in either production or pilot mode. In pilot mode, for example, the delta-P in the system without *Superlig*<sup>®</sup> bed losses is approximately 6 psi at 5 gpm. The required system back pressure to prevent vacuum conditions at the column tops is 5 to 8 psi. The column inlet pressure is thus designed to be a maximum of 12 psi plus 8 psi or 20 psi. Most test or production conditions will allow for 10 to 15 psi of inlet pressure, as noted on our drawing. Using the maximum value, the system pressure relief valve is set to 40 psi and the columns have been designed and tested for 60 psi.

The large production columns and the small pilot columns were both designed using standard ASME 150 psi flanges. The system pressures used in this process are less than this value by

approximately a factor of 10. However, custom flanges designed specifically for 15 psi would be quite expensive and were not considered due to costs. The heavy bolts and flanges for both columns are, therefore, designed for pressures 10 times greater than the actual pressures in this process. It was not considered cost effective to provide column or flange specifications that would require custom design and fabrication of major components.

In terms of operator safety, the design pressure of the process in both production and pilot modes is 33% of the design test pressure of the columns (20 psi vs. 60 psi) providing a safety factor of 3. In reality, the columns and flanges are much heavier than this test value and the actual safety factor in the system is greater than 3. All piping in the skid design is CPVC schedule 80, rated for pressures in excess of 300 psi.

Columns 3 and 4 were designed for pilot mode operation. Materials of construction are described above. Inlet and outlet diffusers are vertically oriented cylinders due to the relatively small internal diameter of the shell. The outlet assembly is flush with the base of the vessel, thus draining 100% of the liquid retained in the glass bead bed if so desired.

The skid is designed for site-specific conditions at PSNS. Most of these features would fall under the general category of maximizing flexibility. Therefore, the metering pump and flow totalizing/indicating system has demonstrated accuracy over a wide range of flows from 2 gpm to 21 gpm. Columns are in two sizes, from full production to pilot mode. Flows can be arranged for lead trail or bulk removal, and lead trail can be easily re-configured. The motor operated valves were designed to maintain the last position when de-energized, thus allowing the columns not in use to be isolated and then switched off entirely. The skid was originally intended for full manual operation due to the long load times and relatively short cycle times for elution and wash (regeneration). Manual operation was thus recommended for the production mode due to simplicity and cost. For the pilot mode, manual operation was justified since pilot testing is normally monitored closely by operating personnel. In the case of both production and pilot modes, this skid is actually semi-automatic in terms of totalizing of a specific cycle flow (please refer to the MRT Skid operating manual for details on semi-automatic operation). The MRT skid was designed for specific PSNS conditions and for maximum flexibility. All components in the design, however, were chosen from readily available commercial sources. The MRT skid required one week of detailed engineering design and 6 weeks of procurement, fabrication and testing in our factory prior to shipment.

We would not recommend that any components in the current Production and Pilot MRT design be minimized or reduced for “minimal function” of the MRT skid. In the event that a specific US Navy application was defined in sufficient detail regarding target metals, pressures, temperatures or fluids, some savings might be realized in the materials of construction. However, in the case of a specific application, this would apply only if the semi-automatic control feature could be retained. Fully automatic operation would impact costs substantially. For different waste streams, the primary sensitive variable is the potentially corrosive nature of the elution and/or wash fluids and the temperature of the elution or feed streams. For systems involving acids, higher temperatures have a large impact on materials of construction. Pressure

is not a necessary factor in the MRT process and is present as a variable only in terms of the developed delta-P across the system. Pressure can become critical in polishing configurations involving 4 or more columns in series or highly viscose feed streams. Each general category of waste stream will likely require a specific design approach. In all cases, the most sensitive factor is the method of control, semi-automatic requiring the attention of an operator, or fully automatic involving a PLC and extensive programming.

The 100 mesh strainers that are installed in the inlet side of both columns are not made of glass. These are constructed of high impact plastic specifically intended for this service and do not, therefore, represent a hazard or weakness in the piping design. These strainers are in the MRT Skid piping design in order to provide a final line of defense against particulates in the feed stream. We intentionally used visible bowl strainers in order to provide an obvious and instant operator feed back on the quality of the feed stream. It is important to emphasize that the 100 mesh size is greater than the required quality of the feed stream (5 microns) but that system malfunctions or feed stream filter failure might introduce particulates that are difficult to remove from the columns. The manual valve array associated with the strainers is intended to allow servicing without shutting down the process. The valve array also allows operators to valve out the strainers entirely. We noted that this option was preferred by operators of the original full production skid used in pilot mode. We do not recommend that these strainers be removed. We also recommend that the strainers be valved back into the process flow and that operators frequently check the clear plastic bowls for early indications of potential problems with the feed stream.

We have addressed the issues you have raised and that also fall into our area of expertise. Please let us know if we can provide any additional assistance.

Sincerely,

L. J. Mott, PE  
Consulting Engineer

cc: Neil Izatt, IBC



# **APPENDIX D**

## **IWPF Volumes, Metal Concentrations, and Potential Contaminants**



## Volumes and Typical Concentrations of Metals in Metal Finishing Wastewaters

The purpose of this document is to describe volumes and typical concentrations of metals in metal finishing wastewaters received and treated at Puget Sound Naval Shipyard's Industrial Wastewater Pretreatment Facility at Bldg. 871. Though many specific waste streams exist, they are grouped into three generic profiles, or broad waste streams as follows: PMC001 is miscellaneous metal finishing wastewater (acidic/alkaline with metals), pH 0-14; PCR001 is metal finishing wastewater with hexavalent chromium, pH 0-9; PCN001 is metal finishing wastewater with cyanide, pH 7-13. Table 1, below, relates volumetric data for the three waste streams. Daily averages are based on 20 workdays per month.

**Table 1 – Wastewater Volumes Received from January 1997 through December 2000**

	Gallons of Acid/Alk.	Gallons Of Chromium	Gallons Of Cyanide	Total Gallons Combined
CY1997 TOTALS	662,290	487,900	136,300	1,286,490
CY1997 MONTHLY AVERAGE	55,191	40,658	11,358	107,208
CY1997 DAILY AVERAGE*	2,760	2,033	568	5,360
CY1997 1-DAY MAXIMUM	21,945	12,188	6,371	N/A
CY1998 TOTALS	774,272	490,300	157,177	1,421,749
CY1998 MONTHLY AVERAGE	64,523	40,858	13,098	118,479
CY1998 DAILY AVERAGE*	3,226	2,043	655	5,924
CY1998 1-DAY MAXIMUM	25,935	20,775	17,174	N/A
CY1999 TOTALS	293,297	213,800	31,700	538,797
CY1999 MONTHLY AVERAGE	24,441	17,817	2,642	44,900
CY1999 DAILY AVERAGE*	1,222	891	132	2,245
CY1999 1-DAY MAXIMUM	19,950	16,620	3,878	N/A
CY2000 TOTALS	401,768	137,700	20,200	559,668
CY2000 MONTHLY AVERAGE	33,481	11,475	1,683	46,639
CY2000 DAILY AVERAGE*	1,674	574	84	2,332
CY2000 1-DAY MAXIMUM	25,935	21,606	2,770	N/A
4 YEAR TOTAL (GALLONS)	2,131,627	1,329,700	345,377	3,806,704
YEARLY AVERAGE	532,907	332,425	86,344	951,676
MONTHLY AVERAGE	44,409	27,702	7,195	79,306
DAILY AVERAGE*	2,220	1,385	360	3,965

As is typical in pretreatment of electroplating wastes, hexavalent chromium wastewater is segregated until the hexavalent chromium is reduced to trivalent chromium, cyanide bearing wastewater is segregated until the cyanide is oxidized, then combined with the miscellaneous acid/alkali wastewater prior to precipitating metals. At this facility, hence in this document, after the acid/alkali waste is combined with treated cyanide waste and treated chromium waste, it is generally referred to as miscellaneous waste.

While the waste streams include other chemicals typical of electroplating wastes, the principal constituents of concern are the metals. Table 2, below, relates data regarding typical concentrations of metals (mg/l) in batches of untreated miscellaneous waste (confluent of all three waste streams following reduction of hexavalent chromium and destruction of cyanide). For a complete list of constituents of all three waste streams, including incidental contaminants, see Table 5.

**Table 2 – Metal Concentrations (mg/l) in Untreated Miscellaneous Waste**

	<b>Zn</b>	<b>Pb</b>	<b>Cu</b>	<b>Ag</b>	<b>Cd</b>	<b>Ni</b>	<b>Cr</b>
<b>Minimum</b>	0.47	<0.2	0.64	<0.1	<0.05	0.39	2.20
<b>Maximum</b>	224	6.28	263	1.60	19.1	35.6	651
<b>Average</b>	23.0	1.30	31.0	0.44	2.61	6.61	60.0

Symbols: Zn - Zinc, Pb - Lead, Cu - Copper, Ag - Silver, Cd - Cadmium, Ni - Nickel, and Cr - Chromium. All units are mg/l.

Table 3, below, relates data regarding typical concentrations of hexavalent chromium in batches of hexavalent chromium wastewater prior to reduction to trivalent chromium.

**Table 3 – Hexavalent Chromium Concentrations Prior to Reduction**

Minimum	<0.1 mg/l
Maximum	1010 mg/l
Average	127 mg/l
Maximum excluding anomalous maximum	540 mg/l
Average excluding anomalous maximum	98.5 mg/l

Note: The absolute maximum concentration is an anomaly attributable to a batch of chromate bearing ballast water from an inactivated ship. This is an infrequent waste, typically occurring every three to four years, in quantities of approximately 100,000 to 200,000 gallons.

Table 4, below, relates data regarding typical concentrations of cyanide found in samples of cyanide bearing wastewater prior to oxidizing the cyanide.

**Table 4 – Cyanide Concentrations Prior to Oxidation**

Minimum	0.01 mg/l
Maximum	1,100 mg/l
Average	93.4 mg/l
Maximum excluding anomalous maximum	100 mg/l
Average excluding anomalous maximum	17.3 mg/l

Note: The absolute maximum concentration is an anomaly that resulted from washing the old cyanide scrubber tunnels during refurbishment of the Metal Finishing Facility.

Numerous incidental waste constituents may be found in each waste stream in addition to the elemental metals listed in Table 2. Table 5 provides a complete list of constituents for each waste stream. These constituents vary with each batch of waste, and not all will be found in any single batch of waste. This information is provided merely to acknowledge that the wastewaters contain contaminants other than just elemental metals.

**Table 5 – Waste Stream Constituents, Including Incidental Contaminants  
PMC001-Miscellaneous Metal Finishing Wastewater**

diiron trisulfate; ferric sulfate; iron persulfate; iron tersulfate; iron(iii) sulfate; boric acid; calcium chloride; potassium peroxymonosulfate; periodic acid; 1,3-diethylthiourea; propargyl alcohol; phenol; morpholine; sodium acetate; sodium tetraborate decahydrate; ferric oxide; ferric subsulfate; potassium hydroxide; sodium hydroxide; ammonium hydroxide; ammonium bifluoride; oxalic acid; nickel diacetate; red squill; blue dye #1; sodium carbonate; formaldehyde; sodium-1,2-naphthoquinone-4-sulfonate; sulfamic acid; glycerin; thiourea; acetic acid; benzoic acid; isopropanol; acetone; disodium metasilicate; triazine; aluminum; lead; nickel; silver; arsenic; barium; cadmium; colloidal cadmium; chromium; copper; zinc; lithium bromide; iodine; trisodium phosphate; sodium molybdate; sodium nitrate; sodium nitrite; hydrochloric acid; phosphoric acid; hydrofluoric acid; sulfuric acid; sodium fluoride; sodium hypochlorite; nitric acid; nickel chloride; water; chromic acid; disodium sulfite; copper (ii) sulfate ; silver nitrate; monopotassium phosphate; selenium; ammonium thiosulfate; manganese ii sulfate (1:1); magnesium chloride; nickel sulfate; ammonium dichromate; oil; pyrogalllic acid; 1-amino-2-methylbenzene; 2-methylaniline; o-toluidine; concrete, mortars & plasters; detergents, soaps, dispersing agents; dirt; gravel & soil; soil; metallic waste, non rcra; paint chips & paint sludge; grease; lithium grease; sediment; sludges, muds w/debris; dye, non hazardous; surfactant; urea prills; phosphate
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<b>PCR001-Metal Finishing Wastewater with Hexavalent Chromium</b>
sodium dichromate; ammonium molybdate; chromic acid; propionic acid, sodium salt; nickel acetate tetrahydrate; lead; nickel; silver; arsenic; barium; cadmium; colloidal cadmium; chromium; copper; zinc; sodium phosphate, dibasic; sodium nitrate; sodium nitrite; orthophosphoric acid; phosphoric acid; sulfuric acid; sodium metabisulphite; nitric acid; water; chromic acid; sodium sulfite; silver nitrate; sodium chromate; selenium; tartaric acid; dye, non hazardous
<b>PCN001-Metal Finishing Wastewater with Cyanide</b>
ethylenediamine; sodium hydroxide; ammonium hydroxide; potassium dicyanoaurate; sodium cyanide, solid and solution ; sodium carbonate; formaldehyde; potassium silver cyanide; cyanide; cyanide anion; potassium carbonate; lead; nickel; nickel sponge; silver; cadmium; colloidal cadmium; chromium; copper; zinc; water

This document presents information based on current wastes. It is anticipated that future wastes will include chelating agents that must be treated. Some additional considerations that should be addressed are (1) that all three waste streams normally contain a significant amount of particulate matter that may require pre-filtration prior to introduction to a treatment unit; (2) that the pH of the treated effluent should be between 6 and 9; and (3) that sludge from the treatment process is hazardous waste, and water should be removed so that remaining water is less than 30% by weight prior to disposal.