

# ESTCP Cost and Performance Report

(CP-0001)



## Removing Chromium(VI) from Wastewater by Anionic Liquid Ion Exchange (A-LIX)

January 2003



ENVIRONMENTAL SECURITY  
TECHNOLOGY CERTIFICATION PROGRAM

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# COST & PERFORMANCE REPORT

## ESTCP Project: CP-0001

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## LIST OF ACRONYMS

---

|   |   |
|---|---|
| A/E ratio   | Aqueous/Extractant (i.e., organic) ratio  |
| AFRL  | Air Force Research Laboratory   |
| Alamine <sup>®</sup> 336                              | Cognis trade name for their proprietary tri-octyl/decyl amine   |
| Alum, Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> | Aluminum sulfate used for particulate coagulation in Cr (VI) treatment  |
| ALC   | Air Logistics Center  |
| A-LIX   | Anionic Liquid Ion Exchange   |
| Conoco <sup>®</sup> 170 ES                            | Conoco trade name of kerosene-like organic diluent  |
| Cr  | Chromium  |
| Cr(OH) <sub>3</sub>                                   | Chromium hydroxide  |
| CTL   | Columbus Testing Laboratory   |
| E   | Extractor   |
| ECAM  | Environmental Cost Analysis Methodology   |
| ES  | Exempt solvent  |
| ESTCP   | Environmental Security Technology Certification Program   |
| Exxal <sup>®</sup> 10                                 | Exxon trade name for their iso decanol  |
| FeSO <sub>4</sub>                                     | Ferrous sulfate   |
| Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>       | Ferric sulfate  |
| 40CFR   | Section 40 of the Code of Federal Regulations   |
| gpd   | Gallons per day   |
| GOTW  | Government Owned Treatment Works  |
| H <sub>2</sub> SO <sub>4</sub>                        | Sulfuric acid   |
| HCrO <sub>4</sub> <sup>-</sup>                        | Bichromate anion  |
| IRR   | Internal rate of return   |
| IWTP  | Industrial Wastewater Treatment Plant   |
| Kgal/d  | Thousand gallons per day  |
| MP&M  | Metal Products and Machinery rule by US EPA to reduce allowable Cr and metal discharge levels from metal plating and machining facilities |
| Na <sub>2</sub> CrO <sub>4</sub>                      | Sodium chromate   |
| NFESC   | Navy Facilities Engineering Service Center  |
| NaOH  | Sodium hydroxide  |
| Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub>         | Sodium metabisulfite  |
| NPDES   | National Pollutant Discharge Elimination System   |
| NPV   | Net Present Value   |

## LIST OF ACRONYMS (continued)

---

|          |  |
|----------|--|
| O&M      | Operating and maintenance  |
| OSHA/EHS | Occupational Safety and Health Act/Environmental Health and Safety |
| OO-ALC   | Ogden Air Logistics Center   |
| POTW     | Publicly Owned Treatment Works                                     |
| ppm      | Parts per million (~ equal to mg/L)                                |
| S        | Stripper   |
| TSS      | Total suspended solids   |
| UTTR     | Utah Test and Training Range                                       |
| WAA      | Watervliet Army Arsenal  |
| WR       | Warner Robins  |
| WR-ALC   | Warner Robins Air Logistics Center                                 |

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Points of contact can be found in Appendix A.

*Technical material contained in this report has been approved for public release.*



## 1.0 EXECUTIVE SUMMARY

### 1.1 BACKGROUND

Chromium (Cr) is widely used within the Department of Defense (DoD) and industry for critical metal plating, corrosion control and surface finishing requirements. However, the hexavalent Cr (VI) anion is toxic and must be removed from wastewaters prior to discharge. The current technology to remove Cr (VI) involves Cr (VI) reduction to Cr (III) followed by precipitation. While the precipitation process is effective, it generates large amounts of hazardous sludge. These sludges represent the single largest type of hazardous waste at many DoD maintenance facilities. As noted in Table 1, based on Air Force Air Logistic Center (AF-ALC) data, it has been estimated that about 1,960 tons per year of hazardous sludges are generated by the DoD primarily as a result of efforts to curtail Cr(VI) discharges.<sup>(1)</sup>

**Table 1. Chromium Hydroxide Sludge Production.**

| Chromium Hydroxide Sludge Produced from DoD Installations, by Site (ton/year) |                   |                                      |
|---|-------------------|--------------------------------------|
| Robins AFB (WR-ALC)   | Hill AFB (OO-ALC) | DoD Wide (Based on 14 installations) |
| 186   | 100               | 1,960                                |

The high cost of handling Cr(VI) in wastewater and the associated sludge generation is the key reason behind the DoD need to eliminating Cr(VI) use<sup>(2)</sup>. While DoD and others are evaluating alternatives to Cr (VI), it is still the material of choice for corrosion resistance. Until all use of chromium is eliminated, there will be chromium discharges. Therefore, an improved Cr (VI) removal technology is needed. This project demonstrated the Anionic Liquid Ion Exchange (A-LIX) process for chromium separation and concentration for recycle. A-LIX technology continually extracts the chromate anion, from plating shop wastewater using a water immiscible tertiary amine extractant in a closed-loop process. The chromate anion is later stripped from the extractant phase of this process and the chromium is concentrated as sodium chromate. The A-LIX process produces a clean water stream that meets chromium discharge requirements, eliminates the need for the reducing agent, and drastically decreases plating shop wastes. A-LIX may also eventually lead to effective water recycle and reuse.

### 1.2 REGULATORY DRIVERS

The federal discharge limits are outlined in 40 CFR 433.10 (Metal Finishing). The most stringent limit is 1.71 mg/L (~1,710-ppb) total chrome. Federal environmental regulations, especially the proposed Metal Products and Machinery (MP&M) Rule to be applied to DoD facilities, may require much more stringent control of Cr (VI) and other metals<sup>(3,4)</sup>. Compliance with MP&M may require the use of a more effective Cr (VI) technology like the A-LIX technology. Local authorities can institute more stringent requirements. The target Cr (VI) discharge limit was 0.3 ppm, below the federal or local limits on chromium.

### 1.3 OBJECTIVES

The objectives of this project were met. They included the construction of a portable, demonstration-scale A-LIX unit device that was operated to validate previous test results and provide the data needed for a process economics evaluation based on full-scale conditions of the plating shops of the Watervliet Army Arsenal (WAA) located in Albany, NY and the Warner-Robins Air Logistics Center (WR-ALC) located at Robins AFB, GA. The discharge regulations for WAA and WR-ALC are noted in Table 2. The results provide the basis for implementation of the A-LIX process in plating shops and metal treatment operations throughout the DoD.

**Table 2. Discharge Regulations.**

| Location                           | NPDES Limit  |  |
|------------------------------------|--|--|
|                                    | Daily Weight Limit                                     | Daily Concentration Limit                                |
| Warner-Robins Air Logistics Center | 1.2 lb average total Cr and<br>1.7 lb maximum Cr total | 0.3 mg/L (ppm) average total Cr<br>and 0.45 mg/L maximum |
| Watervliet Army Arsenal            | 3 lb total Cr and 0.3 lb Cr(VI)                        | ~16 mg/L total Cr and ~1.6 mg/L<br>Cr(VI) <sup>(a)</sup> |

<sup>(a)</sup> Conversion to ppm based on 23,000 gallons per day of Cr contaminated effluent

### 1.4 DEMONSTRATION RESULTS

The demonstration was conducted in two parts. In March-April 2001, the portable A-LIX system was tested on diluted plating solution from WAA at the Battelle Memorial Institute, Columbus, OH, and then was tested at WR-ALC during May-June 2001 on plating shop wastewater.

The technical and economic goals and the program achievements are noted in Table 3 below.

**Table 3. Demonstration Results.**

| Goals   | Accomplishments  |
|---|--|
| Reduce the Cr(VI) concentration in the aqueous stream below a target level of 0.3 ppm | A Cr (VI) extraction efficiency of 99+% was demonstrated. This allowed the production of a < 0.3 ppm Cr (VI) level in the product water under normal operating conditions. However, at extremely high feed Cr(VI) conditions, while the extraction level remained near 99%, the 0.3 ppm Cr(VI) could not always be met.  |
| Produce a useful concentrate using the compact, automated A-LIX plant                 | This was achieved and the concentrate was successfully recycled at the Inmetco plant in Pennsylvania.  |
| Demonstrate significant cost savings and equipment payback in 3 years or less         | The capital costs for a full-scale A-LIX system were estimated at \$491K for WAA and \$449K for WR-ALC. The payback periods for the two sites evaluated were 3.9 and 2.4 years for WAA and WR-ALC, respectively. Treatment costs per 1,000 gallons wastewater were \$17.50 and \$19.90 for WAA and WR-ALC, respectively. |

Oil and grease levels are also regulated. The A-LIX demonstration at WR-ALC removed oil and grease levels to 5 ppm, which was well below the 15-ppm limit at WR-ALC.

## **1.5 STAKEHOLDERS/END USER ISSUES**

Stakeholders include the Air Force, Army, and Navy. They see this demonstration program as providing valuable data and operating experience needed before they can implement the process in their respective plating shops. Hill AFB has authorized funds for preparation of a site-specific design for a 50,000 - 70,000 gal/day A-LIX plant to be installed adjacent to the Hill AFB plating shop. Robins AFB has requested funds for a full-scale unit.

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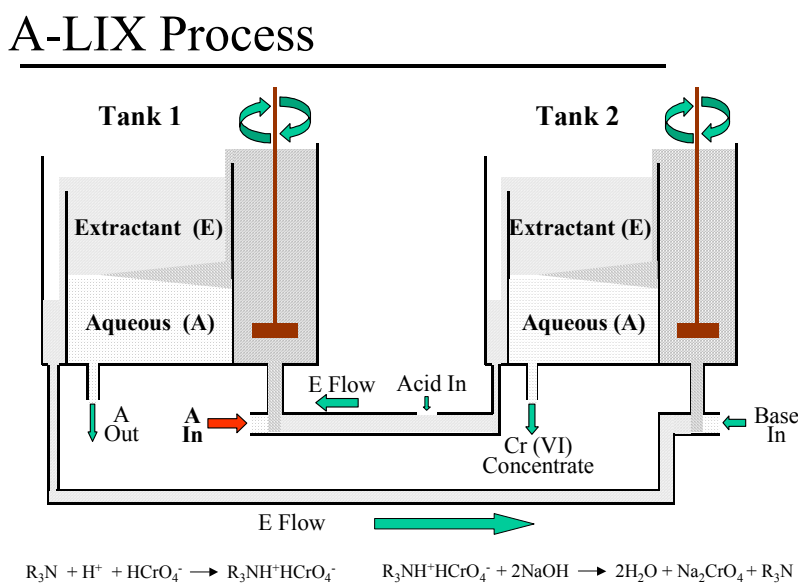
## 2.0 TECHNOLOGY DESCRIPTION

### 2.1 TECHNOLOGY DEVELOPMENT AND APPLICATION

The use of A-LIX is a novel extension of liquid-liquid extraction processes common to the chemical and metallurgical industries. Ion pairing is the primary extraction mechanism that selectively removes the Cr (VI) anion from dilute aqueous solution into an oil-soluble extractant. The extractant is a tertiary amine ( $R_3N$ ). In order for the amine to extract the bichromate anion ( $HCrO_4^-$ ), the extractant must first be protonated by the addition of an acid. In our process, this is accomplished by the addition of sulfuric acid ( $H_2SO_4$ ). After the Cr (VI) is extracted, it must be stripped. This is accomplished by contacting with base, i.e., sodium hydroxide ( $NaOH$ ). The reaction simultaneously regenerates the extractant and transfers the Cr to the aqueous phase for recovery. The addition of small amounts of water keeps the water “in and out” flows in balance and provides a way to purge the Cr concentrate from the system. The program demonstrated that a commercially available amine extractant, Alamine<sup>®</sup> 336, is an effective extractant when used as 5-percent solution in high flash-point organic<sup>(5)</sup>.

### 2.2 PROCESS DESCRIPTION

The A-LIX process is shown schematically in Figure 1. The aqueous rinse water from the plating shop contains many metals. Cr (VI) is present mostly as the ( $HCrO_4^-$ ). The A-LIX system employs a series of mixer/settlers to achieve a liquid-liquid extraction of the Cr. The Cr-contaminated feed water is introduced into the bottom of the first mixer where it is contacted with hydrogen ions ( $H^+$ ), in the form of sulfuric acid, and a tertiary amine ( $R_3N$ ) extractant. Ion pairing results in capture of the Cr (VI) as part of an oil soluble salt ( $R_3NH^+HCrO_4^-$ ) in the extractant phase.



**Figure 1. A-LIX Process Schematic.**

The high interfacial surface area provides fast kinetics even with feed Cr (VI) levels as low as 1 ppm. The extractant and cleaned water overflow the mixer into a settler. The Cr-extracted aqueous phase is drawn off the bottom of the settler. The low-density extractant separates from the aqueous phase in the settler and floats to the surface of the settler where it is drawn off and sent to the mixer in the stripper section. In the stripper, NaOH is added to react with the salt to regenerate the extractant and release the captured chromate as sodium chromate ( $\text{Na}_2\text{CrO}_4$ ). The stripped extractant and Cr concentrate overflow the mixer into the settler. The Cr (VI)-free extractant floats to the surface of the settler, is withdrawn, and is recycled back to the extraction mixer/settler. This continuous regeneration keeps working capital low. The aqueous Cr concentrate is withdrawn from the bottom of the stripper settling-chamber for recycle.

### **2.3 PREVIOUS TESTING OF THE TECHNOLOGY**

A-LIX tests were conducted at WR-ALC at Robins AFB, GA, with plating shop wastewater in 1997<sup>(5)</sup>. These tests provide valuable feasibility data for reducing the Cr (VI) concentration in the aqueous stream from 2 - 10 ppm to less than 0.05 ppm. Operating experience and limited parametric testing focused on pH control, stabilized operation and throughput that met the technical performance requirements and economic benefits. Cr (VI) concentration increased in the stripping solution to 1400-ppm over 20 days of cumulative testing. Eventually the concentration would rise sufficiently to make a valuable byproduct. To test the A-LIX operation under this condition, sodium chromate was added to the strip mixer-settler to raise the Cr (VI) concentration to 20,000 ppm. The Cr (VI) level in the clean water was consistently reduced to well below 0.05 ppm. Discussions with a chemical recycler indicated that a 20,000-ppm Cr concentrate would be a viable source for Cr recycling.

### **2.4 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY**

The demonstration involved treatment of simulated rinse water from the WAA plating shop and wastewater from the WR-ALC plating shop. The A-LIX process worked equally well in both applications. The A-LIX process could also be used for the treatment of general wastewater that is contaminated with Cr. It appears that as long as the Cr content is in the form of Cr (VI), reasonably high in Cr (at least a few ppm), and the wastewater can be acidified to the appropriate pH level (~ 3) the process should extract Cr. Excess particulates would eventually foul the system, so it will be necessary to screen the influent to remove solids. The presence of other metals should not have a detrimental affect. Oils in the water would likely be extracted into the extraction phase with little detrimental affect.

Alamine<sup>®</sup> must be protonated with an acid to extract metal anions. Even in a high pH environment with high levels of lead or aluminum, the wastewater could be acidified and successfully processed for the extraction of Cr. However, high cyanide (CN) containing streams should not be treated. The acidification of the CN wastewater could result in the release of toxic hydrogen cyanide gas. If the CN wastewater was oxidized for CN destruction, the effluent could then be treated by A-LIX for Cr extraction and recovery. However, the presence of residual oxidants might result in the degradation of the extractant and reduce extractant life.

Table 4 presents a comparison with other Cr (VI) control techniques.

**Table 4. Comparison of A-LIX and Alternative Treatment Technologies.**

| <b>Technology</b>  | <b>Pollution Prevention or Compliance</b> | <b>Hazardous Waste</b> | <b>Ionic Selectivity</b> | <b>Capital/ Operating Cost</b> | <b>Other Benefits/Issues</b>                                    |
|--|---|------------------------|--------------------------|--------------------------------|---|
| A-LIX  | Both                                      | No                     | Yes                      | Medium/Low                     | - Multiple applications.<br>- Non-proprietary commercial basis. |
| SO <sub>2</sub> /H <sub>2</sub> SO <sub>4</sub> /NaOH (current OO-ALC) | Compliance                                | Yes                    | No                       | Medium/High                    | - Discharge limit < 100 ppb.<br>- Need sludge conditioning.     |
| FeSO <sub>4</sub> /NaOH (Procedure used before June 2001 at WR-ALC)    | Compliance                                | Yes                    | No                       | Low/High                       | - Discharge limit < 50 ppb.<br>- Need sludge conditioning.      |
| Reverse osmosis (demonstrated)   | Both                                      | No                     | No                       | High/High                      | - Good for water reuse.   |
| Ion exchange   | Compliance                                | Yes                    | No                       | Medium/Unknown                 | - Not good for Cr(VI) recovery.                                 |
| Superlig CP-199805   | Both                                      | No                     | Yes                      | Medium/Unknown                 | - Proprietary.  |
| Diffusion dialysis PP-199705   | Both                                      | No                     | Limited                  | High/Medium                    | - Limited to conc. acid recovery.                               |
| Selective distillation PP-199501                                       | Both                                      | Yes                    | Limited                  | High/Medium                    | - Limited to conc. acid recovery.                               |

The related ongoing ESTCP program CP-199805 uses molecular recognition technology and is being tested by Dr. Katherine Ford of Naval Facilities Engineering Services Center.

A-LIX advantages include the following.

- No production of hazardous wastes.
- Selectivity for the specific ion of interest.
- Moderate capital and low operating costs.
- Suitable for multiple-site applications.
- Non-proprietary technology.
- Based on well-establish commercial liquid-liquid extraction technology.

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### 3.0 DEMONSTRATION DESIGN

#### 3.1 PERFORMANCE OBJECTIVES

The performance objectives are noted in Table 5. All were met, except that the 20,000 ppm Cr(VI) concentrate was only achieved during the demonstration with 0.8 ppm Cr(VI) in the raffinate.

**Table 5. Performance Objectives.**

| Type of Performance Objective | Primary Performance Criteria                                    | Expected Performance Metric  |
|-------------------------------|---|--|
| Quantitative                  | 1. Validate capacity  | 10,000 gpd   |
|                               | 2. Validate raffinate (i.e., clean water) Cr(VI) and oil levels | <0.3 ppm Cr(VI) and <50 ppm oil in product water<br>< 50 ppm Alamine® 336 in the product water |
|                               | 3. Increase Cr(VI) concentrate                                  | >20,000 ppm  |
|                               | 4. Validate process economics                                   | <3 year payback for full scale   |
| Qualitative                   | 1. Reliability  | Demonstrate robust operation with varying feed quality   |

#### 3.2 SELECTION OF TEST SITE/FACILITY

The Watervliet Army Arsenal (WAA) and Warner-Robins Air Logistics Center (WR-ALC) test sites were selected based on the following.

1. Need to control Cr(VI) discharge from large plating shop operations.
2. Large wastewater production.
3. High concentrations of Cr(VI) wastewater.
4. Interest in exploring innovative solutions.
5. Space available to accommodate the portable system.

#### 3.3 TEST FACILITY HISTORY/CHARACTERISTICS

The two selected test sites operate large plating shop operations. Each is described in general terms in the following sections.

##### 3.3.1 Watervliet Army Arsenal

The WAA is a Munitions and Armaments Command Installation of Operations Support Command (Provisional) under the U. S. Army Materiel Command. Cannons remain the principle product of WAA. The guns manufactured at WAA provide the firepower for the Army's main battlefield tank, the M1A1 Abrams. The WAA contains one of the largest plating shops in the DoD. The plating/

surface coating facilities are available to handle small parts as well as long cylindrical parts up to 30 inches in diameter x 33-feet long.

The wastewater is divided into a soluble-oil contaminated and regular Cr-contaminated wastewater. Average data on the Cr-contaminated wastewater and the method of wastewater treatment is noted in Table 6 below.

**Table 6. WAA and WR-ALC Test Site Characteristics.**

| Parameter  | WAA                            | WR-ALC  |
|--|--------------------------------|---|
| <i>Common Values</i>                             |                                |   |
| Flow rate  | 58,000 gpd                     | 69,000 gpd  |
| Cr(VI) inlet, average                            | 25 ppm                         | 10 ppm  |
| Inlet wastewater pH                              | 7                              | ~6  |
| <i>Cr(VI) Reduction and Metals Precipitation</i> |                                |   |
| Acidifying agent                                 | H <sub>2</sub> SO <sub>4</sub> | None (recently switched to H <sub>2</sub> SO <sub>4</sub> )   |
| Cr(VI) reductant                                 | SO <sub>2</sub>                | Sodium meta bisulfite (recently switched to sodium bisulfite) |
| Metal hydroxide precipitation agent              | NaOH                           | NaOH  |
| Flocculating agent                               | Polymer                        | Polymer   |
| Coagulation                                      | Alum                           | Ferric sulfate (recently reduced)                             |
| <i>Sludge Handling</i>                           |                                |   |
| Metal sludge dewatering technique                | Drying beds                    | Plate and frame pressure filtration                           |

Prior to actual experimental testing, it was discovered that the activity level of the WAA plating shop had dropped dramatically. Rather than plating many gun barrels per month, demand had dropped to a small number. Therefore, there would not be sufficient plating shop rinse water to support the demonstration program. It was suggested by WAA that a drum of the actual plating solution be supplied and this could be diluted with water to simulate plating shop rinse water. Since the dilution could be done as well in Columbus as in Albany, NY, it was decided to do the WAA demonstration testing at Battelle in Columbus, OH. This change in the experimental testing site was approved by ESTCP prior to initiation of testing.

### 3.3.2 Battelle Memorial Institute

The Battelle Memorial Institute (Battelle) is located in Columbus, OH north of the downtown and adjacent to Ohio State University (OSU). The main Battelle campus consists of 19 buildings bordered on the west by the Olentangy River and OSU on the north. The A-LIX demonstration land/sea box was installed inside Building 9. This two-story building houses the Battelle machine shop and many industrial test and development areas.

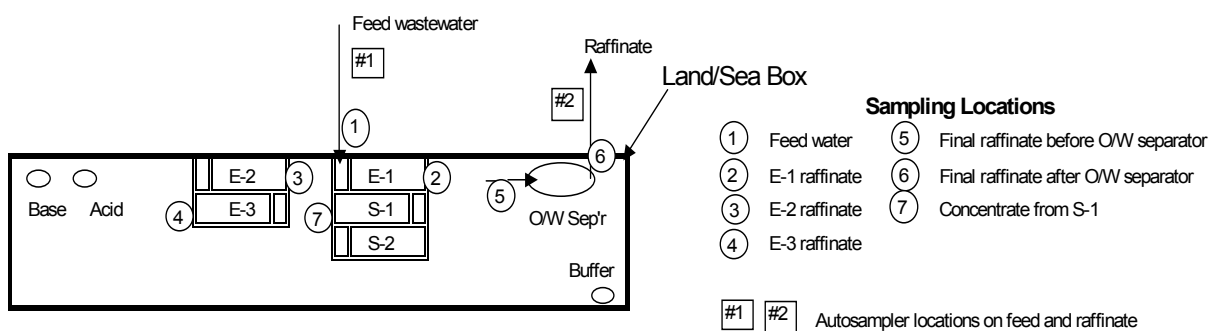
### 3.3.3 Warner-Robins Air Logistics Center

The WR-ALC is located in Warner Robins, GA at Robins AFB. Robins Air Force Base is the largest industrial complex in Georgia. The effluent from the plating shop, Building 142, is directed to the industrial wastewater treatment plant (IWTP) No. 2. The plant has been modified several times, but during the demonstration period was operated on a manual, batch-wise operation. The influent rates to and effluent rates from the IWTP are monitored on an infrequent basis. Average data were noted in Table 6 above. For many years, the base used ferrous sulfate ( $\text{FeSO}_4$ ) for Cr (VI) reduction. Within the last couple of years, they have switch to sodium metabisulfite ( $\text{Na}_2\text{S}_2\text{O}_5$ ) powder. After reduction, the pH of the suspension is raised by the addition of NaOH. Ferric sulfate,  $\text{Fe}_2(\text{SO}_4)_3$ , in acid is added as a floc conditioner and an organic polymer is added to promote solids removal. The suspension of precipitated solids is pumped to a clarifier. The underflow is pumped to a thickener, and the solids are ultimately dewatered in a filter. The solids are loaded in 1-ton containers and recycled for their iron content. In late June 2001, after completing the A-LIX tests, the bases switch to a sulfuric acid/liquid sodium bisulfite system for Cr (VI) reduction.

## 3.4 PHYSICAL SET-UP AND OPERATION

### 3.4.1 Demonstration Set-Up and Start-Up

The portable A-LIX plant was constructed off-site over a three-month period. Shakedown of the plant was conducted in January and February 2001 at Battelle in Columbus, Ohio. Testing with the WAA material was conducted in March and April 2001. The A-LIX plant was located in a 40-ft x 8-ft x 8-ft Land/Sea box. The equipment configuration is shown in Figure 2.



**Figure 2. Configuration of Equipment in the Land Sea Box.**

Tap water and Cr plating batch solution were mixed and pumped into a 1500-gal feed tank to make a consistent feed to the A-LIX unit. The product water (referred to as the raffinate) was sent to a 500-gal collection and then to the Battelle drain. Both feed and raffinate were monitored for pH and Cr (VI).

The Land/Sea box holding the A-LIX equipment was moved to the WR-ALC site in early May 2001. Testing was conducted in May and June 2001. Raffinate and concentrate tanks were located in an adjacent secondary containment tarp. Both were located along the fence line next to the IWTP. The base provided electrical power and tap water. The feed wastewater was withdrawn from a receiving basin in IWTP No. 2. It was pumped through a filter and into the Land/Sea box for

processing. The raffinate was discharged to the IWTP where the water was neutralized and all metals precipitated prior to thickening and filtration.

### **3.4.2 Period of Operation**

The operation test periods occurred on the following dates.

- Shakedown at Battelle: January – February 2001
- WAA material testing at Battelle: March – April 2001
- WR-ALC testing at Robins AFB: May – June 2001

### **3.4.3 Amount /Treatment Rate of Material Treated**

The plant was designed to continuously process ~ 7 gpm (10,000 gpd) of Cr (VI) rinse water. Testing at Battelle and WR-ALC were a mixture of 12 to 24 hr/day runs. Approximately 300,000 gal of simulated plating show rinse water was treated at Battelle and ~150,000 gal were treated at the WR-ALC test site.

### **3.4.4 Residuals Handling**

Four types of residual were produced during testing. The residuals were handled as noted below.

- Filters: an in-line cartridge filter was located on the process feed stream entering the A-LIX system. The filters were used to remove solids from the feed stream greater than approximately 10-microns. Because Cr (VI) is highly soluble, only the water entrained in the filter cartridge at the time of its disposal contained measurable amounts of Cr (VI). These cartridges were disposed of as hazardous wastes.
- Extractant: the extractant used for collecting the Cr (VI) was stripped at the conclusion of the demonstration test program at WR-ALC. It was transferred to 55-gal drums and is in a storage location at Battelle.
- Cr concentrate: at Battelle all the concentrated Cr (VI) water, containing ~20,000 ppm of sodium chromate ( $\text{Na}_2\text{CrO}_4$ ) was collected. About six 55-gal drums of concentrate were shipped to Inmetco for recycle evaluation. At WR-ALC, the majority was bled back into the IWTP for treatment; a small sample was saved for analysis.
- Rag layer: the waste rag layer containing water, extractant, Cr (VI) and Cr (III) in the form of green chromic hydroxide,  $\text{Cr}(\text{OH})_3$ , particles separated at the end of the series of runs was collected and sent to Inmetco for disposal.

### 3.4.5 Operating Parameters for the Technology

The key operating parameters for the A-LIX plant were the residence times in the mixers and settlers, the extraction A/E ratio (volume ratio of aqueous phase to extractant phase in the settlers), and the pH of the extraction and stripping stages. Automated pH controllers were used to monitor and automatically control the addition of acid and base to the plant.

### 3.4.6 Experimental Design

A series of eight experiments described in Table 7 were conducted using diluted WAA plating solution to address the critical technical issues including operability and reliability, control, extractant losses, concentrate value, and life cycle cost.

**Table 7. A-LIX Experimental Design Matrix.**

| Test Number | Conditions   |
|-------------|--|
| Run No. 1   | Center Point; mixer residence time = 2 min., settler residence time = 8 min., A/E= 6/1, extraction pH = 3.5, strip pH = 11, baseline impeller rpms                 |
| Run No. 2   | Mixer residence time = 2 min., A/E = 6/1, 110% of baseline impeller rpms   |
| Run No. 3   | Mixer residence time = 2 min., A/E = 6/1, Either 120% or 90% of baseline impeller rpms   |
| Runs No. 4  | Mixer residence time = 2 min., A/E= 8/1, new baseline impeller rpms  |
| Runs No. 5  | Mixer residence time = 2 min., A/E = 10/1, new baseline impeller rpms  |
| Runs No. 6  | Mixer residence time = 2 min., best A/E and impeller rpms from Run Nos. 1 through 5  |
| Runs No. 7  | Mixer residence time = 2 min., new baseline A/E and impeller rpms, Spike Strip No. 1 to ~20,000-ppm Cr(VI), withdraw concentrate to maintain Cr(VI) at ~20,000-ppm |
| Runs No. 8  | Mixer residence time = 1 min., A/E = 6/1, new baseline impeller rpms, withdraw concentrate to maintain Cr(VI) at ~20,000-ppm                                       |

Run No. 1 was the primary run for the system. The run was considered complete when steady operation was achieved and maintained for one hour. Such operation was defined by: constant feed, raffinate, and extractant flowrates; constant Cr (VI) concentrations in the feed and raffinate; and constant interface levels in the settlers. The liquid levels and pH of the extraction and stripping stages were pre-set based on startup tests conducted prior to the Run Nos. 1 through 8 program. Run Nos. 2 through 5 are modifications to Run No. 1 to help optimize system performance. Only one parameter was altered at a time. After three hours of operation, settings were changed to a new set of conditions.

Run No. 6 took place using the optimized conditions found from the previous runs. The duration of the run was ~1 week to demonstrate reliable system performance. During this run, the concentration of Cr (VI) in the first stripping unit increased to ~1,000-ppm. For Run No. 7, enough chromic acid (CrO<sub>3</sub>) and sodium hydroxide (NaOH) were added to the first stripping unit to increase the aqueous concentration to approximately 20,000 ppm while maintaining the pH near 13. During

continuous operation, water was pumped into stripper No. 2 which caused concentrate to flow to Stripper No. 1, which caused concentrated strip solution to overflow into a holding tank. The water addition rate was calculated to maintain the Cr (VI) concentration at this elevated level. This test confirmed that steady operation was possible while simultaneously producing the Cr (VI) concentrate and the <0.3-ppm Cr (VI) raffinate.

Run No. 7 was conducted over a period of 20 days. It demonstrated robustness and the ability to produce a consistent raffinate and Cr concentrate.

Run No. 8 was designed to evaluate the system at twice the throughput of the previous runs. The feed rate for this run was intended to be 20,000 gpd; however, due to plumbing and pumping limitations, a rate only slightly greater than the nominal 10,000-gpd rate could be achieved and the run was terminated after 1 day.

A similar set of runs was not needed for WR-ALC because the information needed to optimize the system was available from the WAA demonstration. The first WR-ALC run (designated R-1) corresponded to Run No. 6 in Table 7. And the second WR-ALC run (R-2) corresponded to Run No. 7 in Table 7.

### **3.5 SAMPLING/MONITORING PROCEDURES**

For the short-term experiments, Run Nos. 1 through 5 and Run No. 8 in Table 3-3, raffinate and concentrate samples were taken at 1-hour intervals to confirm steady-state operation. Sample ports with a discharge pipe and valve were installed where feasible. Grab samples were taken at the following locations (see Figure 2 presented earlier for configuration diagram).

1. Feed (by sample port).
2. Product water after each extraction stage (by syringe inserted into the overflow weir).
3. Raffinate, before the oil/water (O/W) separator (by sample port).
4. Raffinate (after the O/W separator) (by syringe inserted into the overflow sump).
5. Concentrate (by syringe inserted into the overflow weir).

Two auto samplers were used in experiments Nos. 6 and 7 (the long duration, steady-state tests). One was used to collect a 24-hour composite sample of the feed (No. 1 above) and the second auto sampler was used to collect a composite of the final raffinate (No. 4 above). Samples for oil and grease determination were collected in 1-L glass bottles with Teflon seals; the samples were acidified with sulfuric acid to a pH of <2, and stored in a refrigerator at 4 C until analyzed. Samples collected for total RCRA metals were acidified with 5 mL nitric acid/L of sample and stored in plastic bottles. The grab samples as well as samples from the auto-sampler were stored in a refrigerator until analyzed by Columbus Testing Laboratories (CTL).

### **3.6 ANALYTICAL PROCEDURES**

The following analytical procedures were used.

- pH: The pH of the solutions was determined using a hand held pH meter calibrated weekly with standard solutions.

- Cr (VI) in the raffinate: The Hach diphenylcarbazide colorimetric method was used for all Cr (VI) analyses [EPA 200.7, SW0846 6010A or APHA Standard Methods, 13th Ed., 156 (1071)]. This method allowed Cr (VI) concentrations from 0.01 to 0.50 ppm to be determined. The detection limit was 0.1 ppm. The method is US EPA accepted for analysis of wastewater.
- Metals in the raffinate and concentrate: Total metals analyses were determined by Atomic Absorption techniques [EPA 200.7 or SW0846 6010A].
- O/G: Oil and grease determinations were made following gravimetric methods (US EPA Method 413.1).
- Alamine<sup>®</sup> 336 in raffinate was determined by a colorimetric method suggested by Henkel for the “Colorimetric Determination of Alamine<sup>®</sup> 336 in Solvent Extraction Raffinates and Pulps”, as modified by Columbus Testing Laboratory.

Columbus Testing Laboratory was used for O/G, Alamine<sup>®</sup>, and total metals determinations for experiments conducted at Battelle. For tests at WR-ALC, the on-site base analytical laboratories were used for total metals analyses.

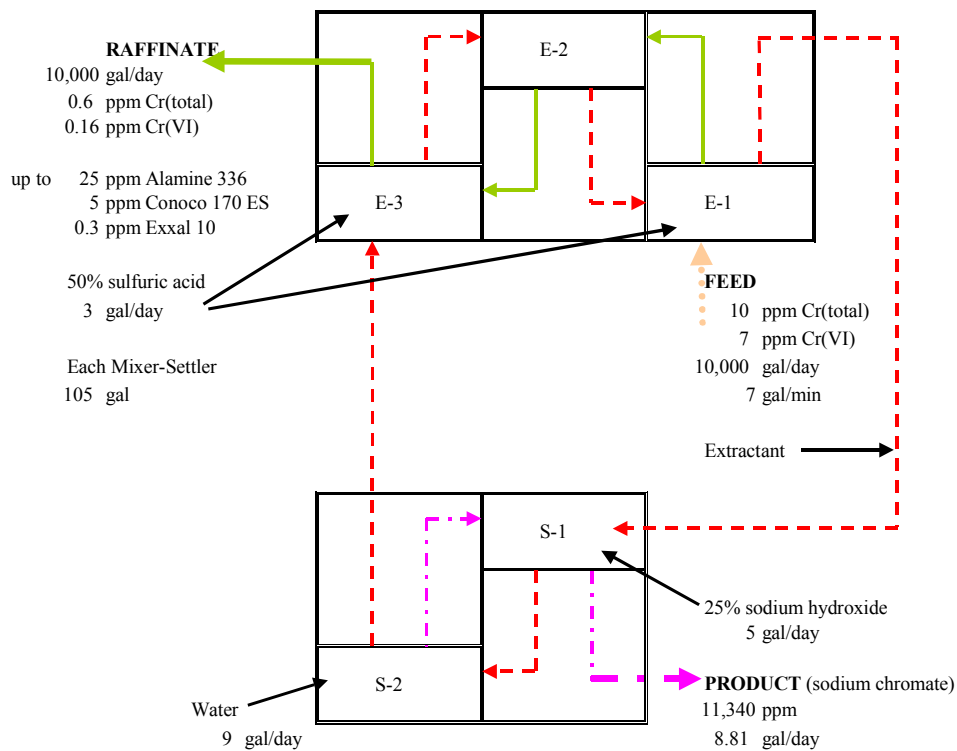
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## 4.0 PERFORMANCE ASSESSMENT

### 4.1 PERFORMANCE DATA

Figure 3 illustrates the test conditions and performance of the 5-stage mixer settler system tested at WR-ALC. A flow rate of 10,000 gal/day (gpd) of dilute Cr(VI) wastewater (from rinse tanks and other plating shop operations) was continuously contacted in countercurrent flow with the extractant to remove Cr(VI) from the aqueous effluent to produce effluent with discharge concentrations below 0.6 ppm composed of 0.2 ppm Cr(VI) and 0.4 ppm Cr(III). The Cr (VI) was then concentrated to 11,000-ppm (1.1 percent), representing a viable chromium source for chemical suppliers to reuse. Tests showed that other metals are not extracted to any significant extent in the A-LIX process, because the positively charged extractant only extracts anions; the other metals found in plating shop effluent are found in the cation (i.e., positively charged) form. A material balance on Cr (total) indicates 0.83 lb Cr/day entered with the feed. The raffinate and



**Figure 3. Demonstration Test Conditions and Results from WR-ALC Tests.**

Cr concentrate contain 0.05 and 0.83 lb Cr (total)/day, respectively. Thus, the A-LIX process was essentially in balance.

Materials of construction were evaluated to assess their compatibility with the acid and base as well as the extractant components. The Alamine<sup>®</sup> 336 extract, Conoco<sup>®</sup> 170 ES carrier fluid, and Exxal<sup>®</sup> 10 (isodecanol) modifier were all found compatible with the polyvinyl chloride used in the mixer settlers, polyethylene used in auxiliary tank, and pumps. It was found to have poor compatibility with rubber (used in the seals of some of the pumps).

The extractant used in this process is Alamine<sup>®</sup> 336. It has been found to be a selective extractant for Cr (VI), even over sulfate ion. Exxal<sup>®</sup> 10 is added as a modifier to decrease phase separation time and increase the ion-pairing kinetics. To minimize costs and control performance, only a small portion of the extractant phase is actually the amine or the modifier. The majority is an organic diluent. The extractant phase used in these tests was composed of 5 vol. % Alamine<sup>®</sup> 336 (Cognis), 5 vol. % Exxal<sup>®</sup> 10 [isodecanol (Exxon Chemical, Corp.)], and 90 vol. % Conoco<sup>®</sup> 170 ES aliphatic diluent (Conoco, Inc.).

#### 4.1.1 Watervliet Army Arsenal

The commercial scale testing using the simulated rinse water processed about 300,000 gallons (4.2 million liters) of feed during the seven runs described in Table 7 with the following results.

**Runs Nos. W-1 - W-5:** This series of runs established the primary operating conditions: feed rate, A/E flow ratio, stirrer tip speeds, and pH control.

**Run No. W-6:** This run demonstrated operability and extraction performance over a 5-day 24 hr/day test.

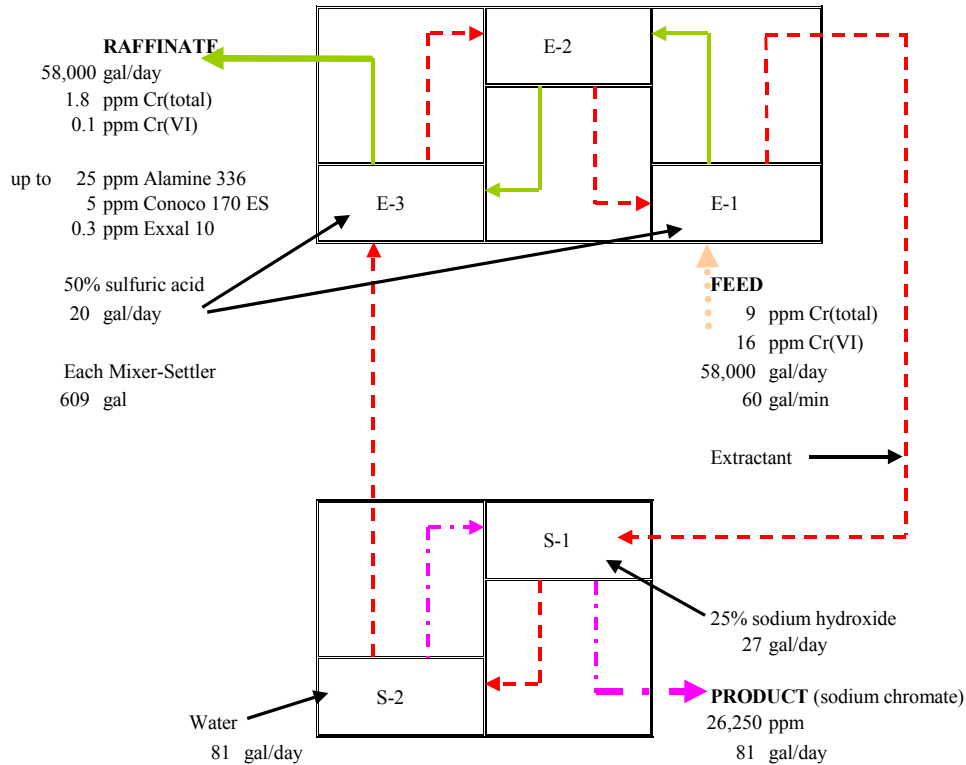
**Run No. W-7:** This 20 days test operated for 24 hr/day demonstrated the system robustness and consistent performance operation with the strippers at high (10,000 to 15,000 ppm) Cr (VI) concentrate levels, see Table 8. Steady operation over long periods is clearly illustrated. Various operational modifications were tested. The impact of these changes on raffinate Cr (VI) residual levels correlates well. Gradual extractant losses were made up at day 14 resulting in improved performance.

**Table 8. WAA Run Results.**

| Parameter   | Run No. W-6                          | Run No. W-7   |
|---|--------------------------------------|---|
| Feed conditions   | 6 gal/min; 12.8 ppm Cr(VI)           | 7 gal/min; 16 ppm Cr(VI)  |
| Aqueous/Extractant (A/E) ratio                                  | 6/1                                  | 6/1   |
| pH  | 2.7 E (Extractor); 12.9 S (Stripper) | 2.7 E, 13.4 S   |
| Cr(VI) in Extractor 1   | 0.86 ppm (93% extraction)            | 1.71 ppm (89% extraction)   |
| Cr(VI) in Extractor 2   | 0.16 ppm (84% extraction)            | 0.40 (66% extraction)   |
| Cr(VI) in Extractor 3   | 0.11 ppm (31% extraction)            | 0.19 ppm (53% extraction)   |
| Cr(VI) composite raffinate                                      | 0.04 ppm (99.7% overall extraction)  | 0.23 ppm (98.6% overall); 0.11 ppm (99.4%) over Days 14 - 18 after Alamine <sup>®</sup> 336 level fortified |
| Cr(VI) in Stripper 1  | 4,000 ppm (300 times concentration)  | 12,000 ppm (800 times concentration)  |
| Organics entrainment in raffinate as measured by Oil and Grease | 76 mg/L <sup>(a)</sup>               | 119 mg/L <sup>(a)</sup>   |

<sup>(a)</sup> High O/G figures obtained due to very low operating temperatures.

Figure 4 represents a projected process flow diagram for a commercial 3-extractor, 2-stripper mixer settler system for the nominal WAA conditions. The acid, base, raffinate, and Cr concentrate figures were based on test results.



**Figure 4. Projected Full-Scale A-LIX System Performance for WAA.**

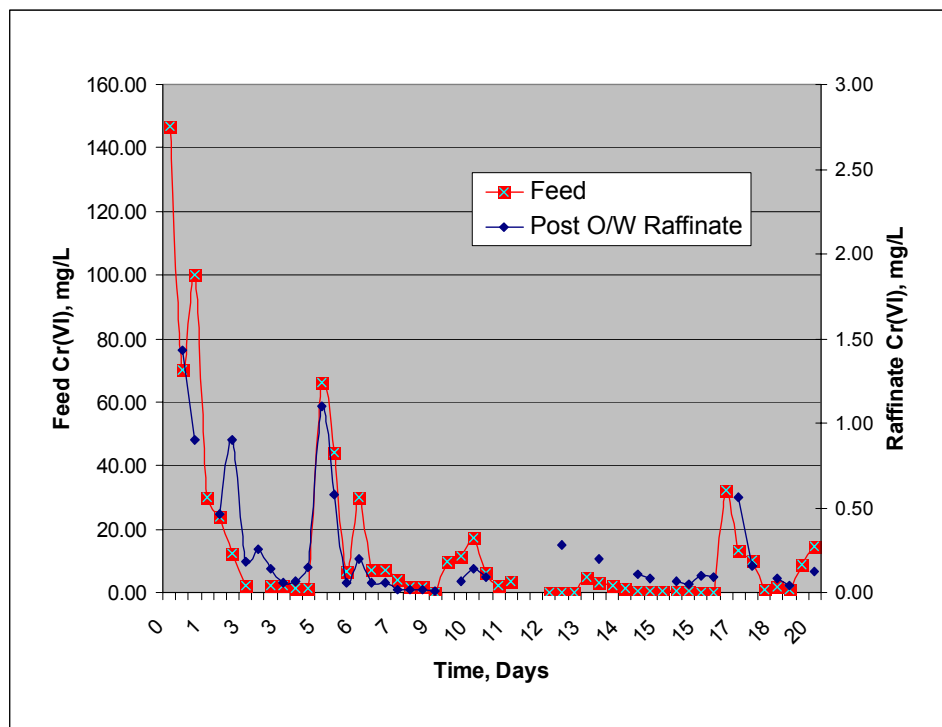
Over a seven-month period, data from the plating shop indicated an average flow rate of ~47,000 gal/day with a Cr (VI) concentration of 13 to 52 ppm<sup>(6)</sup>. In the demonstration tests, the simulated Cr (VI) rinse water was continuously contacted in countercurrent flow with the extractant to remove Cr (VI) from the aqueous effluent to produce clean water with discharge concentrations below 0.1 ppm. The Cr (VI) was concentrated to 13,000 ppm, representing a viable chromium source for recycle and reuse.

#### 4.1.2 Warner-Robins Air Logistic Center

The WR-ALC testing using the Cr-contaminated wastewater from the Building 142 plating shop processed about 100,000 gallons (0.4 million liters) of feed during the two runs with the following results.

**Runs No. R-1:** This start up run established the primary operating conditions, i.e., feed rate, A/E flow ratio, stirrer tip speeds, and pH control, were also suitable for WR-ALC operation. After this testing, sodium chromate was added to the strippers to increase the Cr concentration to typical steady state level of 10,000 ppm of Cr (VI).

**Run No. R-2:** This extended duration run demonstrated operability, extraction performance, robustness, and consistent performance over 20 days at 12- to 24-hr/day operations with the strippers containing 4,000 to 22,000 ppm Cr (VI) concentrate levels. Results are summarized in Figure 5 and Table 9.



**Figure 5. Cr (VI) Versus Time for Run No. R-2.**  
(Excludes data for day 9 where pH of feed was ~ 1)

**Table 9. WR-ALC Run Results.**

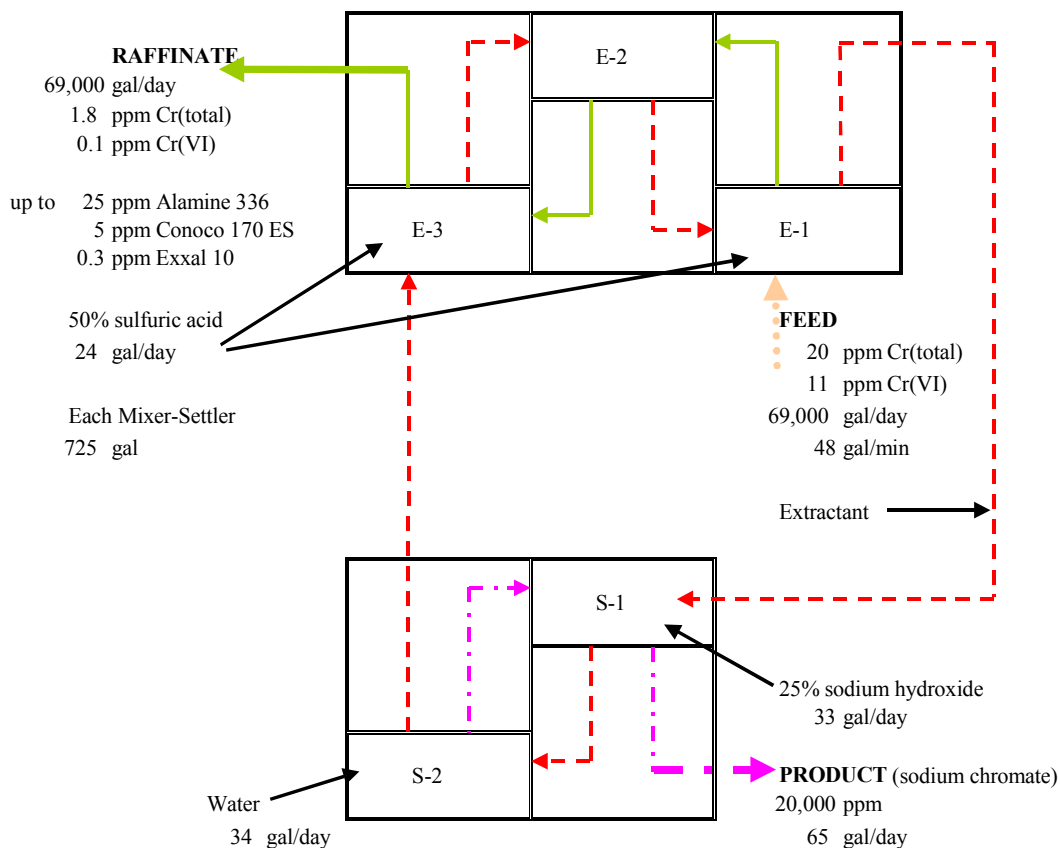
| Parameter   | Run No. R-1                            | Run No. R-2                            |
|---|--|--|
| Feed conditions   | 6.8 gal/min; 55.0 ppm Cr(VI)           | 6.9 gal/min; 7.6 ppm Cr(VI)            |
| Aqueous/Extractant (A/E) ratio                                  | 8/1                                    | 7/1                                    |
| pH  | 2.7 E (Extractor); 13.4 S (Stripper)   | 2.7 E, 13.3 S                          |
| Cr(VI) in Extractor 1   | 6.8 ppm (88% extraction)               | 2.0 ppm (74% extraction)               |
| Cr(VI) in Extractor 2   | 1.8 ppm (73% extraction)               | 0.7 (63% extraction)                   |
| Cr(VI) in Extractor 3   | 0.8 ppm (57% extraction)               | 0.2 ppm (73% extraction)               |
| Cr(VI) composite raffinate                                      | 0.8 (99.2% overall extraction)         | 0.17 ppm (98.9% overall)               |
| Cr(VI) in Stripper 1  | 21,000 ppm (3,200 times concentration) | 11,000 ppm (1,900 times concentration) |
| Organics entrainment in raffinate as measured by Oil and Grease | 27 mg/L                                | 5 mg/L                                 |

Steady operation over the entire period was clearly illustrated. Gradual extractant losses were made up after day 13. Oil entrainment in the raffinate, as measured by oil and grease were significantly

lower than in the WAA testing (119 vs. 5 ppm). The difference was attributed to the higher wastewater temperatures (~ 100 to 110 F).

Full metals analyses for the feed and raffinate (see Final Report, Ref. 7) show that the A-LIX process did not extract most positively charged metal cations. However, like in the WAA test series, the process did remove a significant portion of the Cr (III) and nearly all the Cr (VI) from the feed water.

Figure 6 illustrates a process flow diagram for a commercial 3-extractor, 2-stripper mixer-settler system based on the test performance and typical WR-ALC flow and feed concentration figures at the IWTP servicing the WR-ALC plating shop. Over the period October 1999 through January 2000, flow ranged from 37,000 to 72,000 gal/day (69,000 gal/day average) and Cr(VI) feed concentration ranged from 1.8 to several hundred ppm (15 ppm average).<sup>(8)</sup>



**Figure 6. Projected Full-Scale A-LIX System Performance for WR-ALC.**

## 4.2 DATA ASSESSMENT

The effectiveness of the A-LIX system was evaluated against the performance parameters presented in Table 10. As noted, the operations met all performance criterion elements except Alamine<sup>®</sup> content in the raffinate. There was some uncertainty in the accuracy of this parameter. Overall, the unit performance was considered a success.

**Table 10. Expected and Actual Performance.**

| <b>Performance Criteria</b>                                 | <b>Performance Confirmation Method</b>         | <b>Expected Performance</b>                              | <b>Actual Performance</b>  |
|---|--|--|--|
| Effluent stream Contaminant concentration (after treatment) | Hach diphenyl-carbazide colorimetric method    | < 0.3 ppm Cr(VI)   | ≤0.3 ppm whenever feed Cr(VI) levels were below 30 ppm                             |
| Concentrate produced  | Hach diphenyl-carbazide colorimetric method    | > 20,000-ppm Cr(VI)                                      | 15,000 to 20,000 ppm Cr(VI) as sodium chromate                                     |
| Total Cr  | Atomic absorption                              | < 0.3 ppm Cr if no Cr(III) in feed                       | All test found that there was significant Cr(III) in the feed and in the raffinate |
| Residual oil  | Oil and grease                                 | < 50 ppm   | ~ 5 ppm when the feed water temperature was elevated                               |
| Alamine® 336  | Modified Henkel Extraction test <sup>(a)</sup> | < 5 ppm  | <1 ppm based on oil and grease figures; or 25 ppm based on Henkel test             |
| Cost  | Cost calculation                               | < 3 year payback   | 2.4 to 3.9 years   |
| Reliability   | Record keeping                                 | Achieve multi-day uninterrupted operation                | Achieved   |
| Ease of use   | Experience from demonstration operations       | No excessive maintenance or operating labor requirements | Achieved   |
| Versatility of mobile system                                | Experience from demonstration operation        | Ease of shutdown, transport, and startup                 | Shutdown and moved in 5 days   |

<sup>(a)</sup> Colorimetric method developed by Henkel for the “Colorimetric Determination of Alamine® 336 in Solvent Extraction Raffinates and Pulps” as modified by CTL.

### 4.3 TECHNOLOGY COMPARISON

There are four primary methods for conventional treatment of Cr (VI) contaminated plating-shop wastewater. All involve Cr (VI) reduction followed by precipitation of the metal hydroxides using NaOH. The four methods vary in the pH required for treatment, the choice of reductant, and amount of sludge produced. Use of the A-LIX process eliminates the need for reductant chemicals, eliminates the production of Cr(VI)-metal hydroxide sludge, avoids the need to handle highly toxic chemicals, avoids long-term liability problems (related to the production of sludge) and provides a means to recycle the Cr back into a useful product.

## 5.0 COST ASSESSMENT

### 5.1 COST REPORTING

Cost issues are critical to the evaluation of the A-LIX process. Battelle estimated capital and operating costs of a full-scale commercial A-LIX facility. Battelle completed a pseudo level II cost assessment of both the existing Cr treatment technology and the A-LIX process using a modified version of the Environmental Cost Analysis Methodology (ECAM)(9). A cost estimating procedure was followed to identify, quantify, and assign environmental costs to the baseline and A-LIX process for WAA and WR-ALC. Further detail is available in the ESTCP Final Report<sup>(7)</sup>.

The following assumptions were included in this analysis.

- Capital costs for larger-scale A-LIX facilities can be estimated from the \$140,000 cost for the 7 gpm unit by assuming a scale up factor of 0.46; i.e.,  $\$140,000 * (\text{new capacity}/7 \text{ gpm})$  raised to the 0.46 power. To this cost is added \$50,000 for installation and 12% of the capital plus installation for utility connections.
- The feed is assumed to be 2/3 Cr (VI) and the balance is Cr (III).
- Extraction efficiency is 99.3% for Cr(VI) and 80% for Cr(III).
- Extractant ratio is 90/5/5 for Conoco<sup>®</sup> 170 ES, Alamine 336<sup>®</sup>, and Exxal<sup>®</sup> 10.
- Costs are \$2.65/gal of Conoco<sup>®</sup> 170 ES, \$19.96/gal of Alamine<sup>®</sup> 336, and \$5.67/gal of Exxal<sup>®</sup> 10.
- The desired A-LIX feed water pH was 3. The experimental H<sub>2</sub>SO<sub>4</sub> utilization rate was 1.7 lb of 50% H<sub>2</sub>SO<sub>4</sub>/10,000 gal of feed water (0.35 gal of 50% H<sub>2</sub>SO<sub>4</sub>/Kgal). This rate was used for costing purposes. A feed pH of 4 can be utilized if the Alamine<sup>®</sup> 336 is increased to 20% (from the normal 5% level) in the extractant; this could reduce the H<sub>2</sub>SO<sub>4</sub> utilization rate by approximately 20%.
- The desired A-LIX stripper pH level was 13. The experimental NaOH utilization rate was 2.1 lb of 25% NaOH/10,000 gal of feed water (0.475 gal 25% NaOH/Kgal). This rate was used for costing purposes. When a feed pH of 4 is utilized, the required quantity of NaOH can be decreased by about 20%.
- Cost for H<sub>2</sub>SO<sub>4</sub>, NaOH, coagulant/floc aids and polymers were based on WAA and WR-ALC specific rates.
- Labor rates were based on WAA and WR-ALC specific rates. Savings in labor were based both on IWTP labor savings and sludge handling/dewatering labor savings.
- An overhead rate of 80% was added to all the labor charges.
- Training, compliance audits, testing of liquids and solids, medical exams and loss of productive time, etc. were based on WAA and WR-ALC specific rates where possible.
- Losses of extractant are based on a 5 ppm overall loss rate (based on the 5 ppm O/G figure determined at WR-ALC); losses of Conoco<sup>®</sup> 170 ES, and Exxal<sup>®</sup> 10, 4, and 0.25 ppm, respectively. The loss of Alamine<sup>®</sup> 336 was estimated at from 0.25% to up to 25 ppm (based on chemical analysis); the 25-ppm loss figure was used for cost estimation.
- The raffinate produced by the A-LIX process has a pH of between 3 and 4; the extra NaOH required to increase the pH to the 3 to 6 level normally experienced at the base IWTPs represents only a few thousand dollars/year and was ignored.
- The reduction in the sludge production rate was directly proportional to the reduction in the total Cr discharged by the A-LIX system, i.e., the 92% decrease to total Cr resulted in a

- reduction in sludge production by 92%. Any extra NaOH required to increase the raffinate pH to the normal feed level would not result in any increase in sludge production.
- Particulate matter in the wastewater removed upstream from the A-LIX unit would be transferred to the IWTP hazardous waste disposal area with no net charge to the A-LIX system.
  - Other wastes such as rag layers are only generated during shutdown and movement of the equipment; there is no charge assigned as shutdown and movement would not be a normal operational activity.
  - The Cr concentrate was shipped to Inmetco for recycle. A recycle charge of \$1/gal and a transportation of \$1/ton-mile were applied. No other recyclers were identified.
  - The costs for sludge disposal were based on WAA and WR-ALC practices; a transportation of \$1/ton-mile was applied.

## **5.2 COST ANALYSIS**

A summary of the projected A-LIX capital and operating costs are provided in Table 11 for WAA and WR-ALC. Details are provided in Appendices C and D of the Final Report<sup>(7)</sup>. The startup, operating and maintenance, indirect environmental and other associated costs for A-LIX facilities at WAA and WR-ALC are detailed in Table 12. Treatment costs per 1,000 gallons of wastewater were \$17.50 and \$19.90 for WAA and WR-ALC, respectively.

### **5.2.1 Cost Drivers**

The cost drivers included (1) capital cost, (2) operating labor requirements, (3) chemical costs, (4) laboratory requirements, and (5) disposition cost of the Cr (VI) concentrate.

### **5.2.2 Life Cycle Cost Comparison**

The life-cycle costs of the Cr (VI) treatment processes were calculated based on the following considerations: (1) facility capital cost; (2) startup, operations and maintenance, and demobilization costs; (3) equipment replacement costs; and (4) environmental compliance costs. A 10-year time period was used for the life-cycle period for cost comparison. The base case IWTP operating costs for conventional Cr (VI) treatment as practiced by WAA and WR-ALC were estimated at \$426K/year and \$691K/year, respectively. Details of the conventional IWTP costs are provided in Table 13. A comparison of the projected financial performance is noted in Table 14. Financial indicators included payback period, net present value (NPV), and internal rate of return (IRR).

In general a payback period of less than 3 years, a positive NPV at the noted discount rate, and an IRR of >10 % indicates a very good investment. For both WAA and WR-ALC, the analysis indicates that installation of an A-LIX system would provide very good economic payback.

The WAA operations are not as favorable as WR-ALC, based on payback period and IRR. The lower performance indicators were due to lower labor and sludge handling/disposal costs in the conventional WAA treatment system compared to WR-ALC. Therefore, the improvements allowed by use of an A-LIX system are not as dramatic in the WAA case. The economic performance parameters are still very good, and an A-LIX application at WAA is still economically justified.



**Table 11. A-LIX Cost Summary.**

| Parameters                | A-LIX Capital Costs, \$K |        |
|---------------------------|--------------------------|--------|
|                           | WAA                      | WR-ALC |
| Total capital             | 491                      | 449    |
|                           | Annual Costs, \$K/year   |        |
| Operating and maintenance | 200                      | 300    |
| Indirect environmental    | 28                       | 58     |
| Other                     | 72                       | 142    |
| Total annual costs        | 300                      | 501    |

**Table 12. A-LIX Capital and Operating Costs.**

| Start-Up                           |                |     | Operating and Maintenance       |                |     | Indirect Environmental Activity Costs             |                |    | Other Costs                           |                |     |
|------------------------------------|----------------|-----|---------------------------------|----------------|-----|---|----------------|----|---------------------------------------|----------------|-----|
| Activity                           | Cost, \$K/year |     | Activity                        | Cost, \$K/year |     | Activity  | Cost, \$K/year |    | Activity                              | Cost, \$K/year |     |
|                                    | WAA            | WR  |                                 | WAA            | WR  |   | WAA            | WR |                                       | WAA            | WR  |
| Facility preparation, mobilization | 53             | 48  | Labor to operate equipment      | 64             | 138 | Compliance audits                                 |                |    | Overhead assoc. with Process          |                |     |
| Equipment design                   |                |     | Labor to manage hazardous waste | 26             | 26  | Document maintenance                              |                |    | Productivity /cycle time              |                |     |
| Equipment purchase                 | 388            | 351 | Utilities                       | 5              | 7   | Envir Mgmt Plan Dev & maintenance                 |                |    | Worker injury claims and health costs | 72             | 142 |
| Installation                       |                |     | Mgmt/ Treatment of by-products  | (a)            | 14  | Reporting requirements                            | 8              | 20 |                                       |                |     |
| Training of operators              | 50             | 50  | Hazardous waste disposal fee    | 15             | 14  | Test/analyze waste streams                        | 3              | 18 |                                       |                |     |
|                                    |                |     | Process chemicals               | 29             | 37  | Medical exams (including loss of productive time) | 1              | 1  |                                       |                |     |
|                                    |                |     | Consumable and supplies         | 8              | 8   | Waste transportation (on and off site)            | 16             | 16 |                                       |                |     |
|                                    |                |     | Equipment maintenance           | 53             | 51  | OSHA/EHS training                                 | 1              | 4  |                                       |                |     |
|                                    |                |     | Training of operators           | 1              | 5   |   |                |    |                                       |                |     |
|                                    |                |     | Subtotal                        | 200            | 300 | Subtotal  | 28             | 58 | Subtotal                              | 72             | 142 |
| Total Capital                      | 491            | 449 |                                 |                |     | Total Operating and Environmental                 |                |    |                                       | 300            | 301 |

(a) Included with waste sludge handling cost.

**Table 13. IWTP Operating Costs.**

| Operating and Maintenance       |                |     | Indirect Environmental Activity Costs             |                |    | Other Costs                           |                |     |
|---------------------------------|----------------|-----|---|----------------|----|---------------------------------------|----------------|-----|
| Activity                        | Cost, \$K/year |     | Activity  | Cost, \$K/year |    | Activity                              | Cost, \$K/year |     |
|                                 | WAA            | WR  |   | WAA            | WR |                                       | WAA            | WR  |
| Labor to operate equipment      | 127            | 191 | Compliance audits                                 |                |    | Overhead assoc. with Process          |                |     |
| Labor to manage hazardous waste | 26             | 26  | Document maintenance                              |                |    | Productivity/cycle time               |                |     |
| Utilities                       | 6              | 9   | Envir Mgmt Plan Dev & maintenance                 |                |    | Worker injury claims and health costs | 123            | 203 |
| Mgmt/Treatment of by-products   | --             | 36  | Reporting requirements                            | 9              | 23 |                                       |                |     |
| Hazardous waste disposal fee    | 44             | 40  | Test/analyze waste streams                        | 3              | 35 |                                       |                |     |
| Process chemicals               | 17             | 42  | Medical exams (including loss of productive time) | 0              | 1  |                                       |                |     |
| Consumable and supplies         | 9              | 9   | Waste transportation (on and off site)            | --             | 9  |                                       |                |     |
| Equipment maintenance           | 60             | 60  | OSHA/EHS training                                 | 1              | 4  |                                       |                |     |
| Training of operators           | 1              | 4   |   |                |    |                                       |                |     |
| Subtotal                        | 290            | 416 | Subtotal  | 13             | 72 | Subtotal                              | 123            | 203 |
|                                 |                |     |   |                |    | Total                                 | 426            | 691 |

**Table 14. Cost Comparison: Conventional Treatment Versus A-LIX.**

| Parameter   | WAA | WR-ALC |
|---|-----|--------|
| <i>Conventional Performance</i>   |     |        |
| Annual costs, \$/year   | 426 | 692    |
| <i>A-LIX</i>  |     |        |
| Annual costs, \$/year   | 300 | 501    |
| Projected savings, \$/year  | 126 | 191    |
| A-LIX startup capital and training costs, \$K                           | 491 | 449    |
| <i>A-LIX Performance</i>  |     |        |
| Payback period, years   | 3.9 | 2.4    |
| Net present value, \$K, at 8% annual discount rate and 10 year life, \$ | 355 | 830    |
| Internal rate of return, 10 year, %                                     | 22% | 41%    |

## **6.0 IMPLEMENTATION ISSUES**

### **6.1 COST OBSERVATIONS**

The cost analysis indicated the following factors influenced the cost advantage of the A-LIX process over alternative conventional Cr (VI) treatment technologies.

- Method of conventional Cr(VI) treatment.
- Concentration of Cr(VI) and Cr(III) in the wastewater.
- Cost for sludge disposal generated by conventional Cr(VI) treatment.
- pH required for conventional Cr(VI) treatment and for the A-LIX extraction step.
- Required A-LIX mixer and settler residence times.
- Extractant composition.
- Wastewater temperature.
- Extractant, especially Alamine<sup>®</sup> 336, loss rate.

### **6.2 PERFORMANCE OBSERVATIONS**

Based on 400,000 gal of wastewater treated by the A-LIX process, the following performance observations were made.

- Cr (VI) extraction was ~ 99% or higher.
- The extraction rate was nearly independent of inlet Cr(VI) concentration.
- Extractant losses, as measured by oil and grease determinations, were significantly lower at higher feed water temperatures.
- Alamine<sup>®</sup> 336 losses were difficult to quantify, but may be as high as 25 ppm.

### **6.3 SCALE UP**

The demonstration unit operated at 10,000 gpd. Full-scale applications at WAA and WR-ALC are expected to operate in the 50,000- to 70,000-gpd range. The 5-to-7:1 scale-up factor is small enough to not provide a significant scale-up problem.

### **6.4 END USER ISSUES**

End users include both the plating shops and the Environmental Management staff (or Civil Engineering staff) at WAA and WR-ALC. Their concerns include the following.

- Effluent Cr(VI) and total Cr levels.
- Residual oil levels.
- Ease of use (specifically the requirements of operating and maintenance labor).
- Cost savings.
- Capital requirements.
- Payback period.
- Ease of scale up.

The customized demonstration plant constructed and tested generated the needed operational and cost data to meet these concerns. Because it was designed to operate at a near full-scale level, using

standard commercial-off-the-shelf (COTS) components integrated into a customized design, the plant was designed to address the scale-up and commercialization issues. Operation of the demonstration plant also provided an opportunity for plant operators to observe the A-LIX plant in operation and to see the labor needed as well as a means to assess the ease of operation.

Battelle will continue to disseminate information about the technology and the demonstration results. Work is underway to commercialize the technology and install a system at Hill AFB. In addition, other DoD sites will be contacted for a demonstration run.

Efforts will also be expended to demonstrate the technology at a commercial plating shop. The data gathered during the WAA and WR-ALC demonstration program will be critical to interesting non-DoD shops to try the A-LIX technology. The availability of a mobile demonstration plant that can be quickly moved onto a user's site and tested will accelerate acceptance of this technology.

## **6.5 APPROACHES TO REGULATORY COMPLIANCE AND ACCEPTANCE**

### **6.5.1 Environmental Checklist**

Because of Battelle's role in research and development, short-term testing US EPA permits were not required. However, a Battelle "discharge to drain" permit as described in Battelle's EN-PC-02.0 Discharge to Drain Procedure were followed for testing at Battelle. The City of Columbus allows a discharge level of 1.9-ppm total Cr [Cr (III) +Cr (VI)] maximum composite sample concentration. Since we achieved a < 1 ppm discharge level, this was not a problem. The City also states that the hydrocarbon/fat/oil/grease discharge cannot exceed 200 ppm. Again since we achieved a 50 to 150 ppm level, this was not a problem.

The demonstration plant at WR-ALC did not significantly change the amount of Cr entering and leaving the IWTP. Therefore, no new permits were required.

At WR-ALC, an AF Form 813 was required. This form served to notify impacted individuals and organizations of changes in the process treatment procedure.

### **6.5.2 Other Regulatory Issues**

Battelle obtained the assistance of Mr. Dave Ferguson of the US EPA (Cincinnati office). Mr. Ferguson specializes in new controls/innovations affecting the plating shop industry. Battelle sought his guidance regarding new regulations on the discharge of Cr contaminated wastewater generated in Cr plating shops during two briefing meetings over the course of the program.

Currently the federal regulations, 40CFR 433.10 (Metal Finishing), only limits the total Cr discharge rate. The effluent concentration limit is <1.71 mg/L total chrome. Local regulations can be much more restrictive. Requirements for WAA were:

- 3.0 lb total Cr/day and 0.3 lb Cr(VI)/day and
- 6 mg/L (ppm) daily average total Cr and 0.6 mg/L Cr (VI) based on 58,000 gpd of Cr contaminated wastewater.

For WR-ALC, the State of Georgia, Department of Natural Resources Authorization to Discharge under the NPDES restricts the discharge at IWTP No. 2 to:

- 1.2-lb total Cr/day daily average [1.7-lb total Cr/day daily maximum] as total Cr and
- 0.30-mg/L daily average [0.45-mg/L daily maximum] as total Cr.

There are no specific Cr (VI) regulations at WR-ALC.

The raffinate produced by the A-LIX process has a pH of between 3 and 4. The low pH of the raffinate is not expected to detrimentally affect IWTP operations. The extra NaOH required to increase the pH to the 3 to 6 level normally experienced at the base IWTPs represents only a few thousand dollars/year and was ignored in the cost analysis in Section 5.2.

For a 50 ppm O/G discharge, the Alamine<sup>®</sup> 336 might constitute 5% to 10% of O/G or up to 5 ppm. Alamine<sup>®</sup> at high concentrations is toxic to aquatic wildlife and could present a problem for downstream biological sewage treatment. This was not a problem at Battelle because the 10,000 gpd discharge of Cr raffinate from the A-LIX process was diluted with 200,000 gpd of wastewater from other uses. Thus it was reduced to well below 1 ppm prior to leaving the Battelle property.

It was also not a problem at WR-ALC where the A-LIX discharge O/G level was ~ 5 ppm. The 10,000 gpd discharge of Cr raffinate from the A-LIX process was diluted with 50,000 to 70,000 gpd of acid/alkali wastewater then subjected to neutralization with NaOH, flocculated, thickened, and the treated water discharge to the river. There it is diluted with over a million gpd of clean water produced from WR-ALC combined sanitary and industrial wastewater treatment plants. Therefore, it is not anticipated that there would be any required changes to the WR-ALC NPDES permit.

The discharge of the Conoco<sup>®</sup> 170 ES should be treated like any other O/G constituent of the wastewater. No adverse impact to the IWTP operation was experienced.

## 6.6 LESSONS LEARNED

1. Good working relationships between the Battelle technology development staff and AFRL, ESTCP, and the WAA and WR-ALC test site staff were critical to the successful development of A-LIX technology.
2. Good communications between the A-LIX staff and the IWTP staff was critical in overcoming scheduling, analytical, and operational problems.
3. Frequent meetings and consultation with the ultimate process owner (be it the Plating Shop or the Civil Engineering directorate) is critical to get “buy in” prior to technology implementation.
4. The attractiveness of the A-LIX technology must be analyzed on a case-by-case basis; local factors such as discharge limitations, labor utilization, chemical costs, and waste disposal practices can affect plant savings and thus the estimated payback period.

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## APPENDIX A

### POINTS OF CONTACT

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| Nick Conkle                        | Battelle                                     | 614-424-5616<br>conkle@battelle.org           | Principle<br>Investigator  |
| Dave Bury                          | WR-ALC/EM                                    | 912-926-1197, x140<br>dave.bury@robins.af.mil | Test Site<br>Coordinator   |
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