U.S. Environmental Protection Agency
Environmental Technology Verification Program
For Metal Finishing Pollution Prevention Technologies
Verification Test Plan

for the

Evaluation of Lobo Liquids Rinse Water Recovery System

Revision 0

January 8, 2002

Concurrent Technologies Corporation is the Verification Partner for the EPA ETV Metal Finishing Pollution Prevention Technologies Center under EPA Cooperative Agreement No. CR826492-01-0.

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Environmental Technology Verification Program for Metal Finishing Pollution Prevention Technologies Verification Test Plan for the Evaluation of the Lobo Liquids Rinse Water Recovery System.

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Signature denotes acceptance of this test plan as written regarding experimental design, quality assurance, test and analysis methods, operational procedures, equipment configuration, project management and current system operating effectiveness prior to testing.

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ACRONYMS & ABBREVIATIONS

µS	Microsiemens
amp	Ampere
BPT	Best Practical Treatment
C	Celsius
cfm	Cubic Feet per Minute
COC	Chain of Custody
CTC	Concurrent Technologies Corporation
DAF	Dissolved Air Flotation
DC	Direct Current
DQO	Data Quality Objectives
EC	Electrocoagulation
EHS	Extremely Hazardous Substances
EPA	U.S. Environmental Protection Agency
ETV-MF	Environmental Technology Verification – Metal Finishing
ft	Feet
g	Gram
gal	Gallon
gpd	Gallon per Day
gpm	Gallon per Minute
HEM	Hexane Extractable Material
hp	Horsepower
hr	Hour
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
ID	Identification
IDL	Instrument Detection Limit
JTA	Job Training Analysis
kg	Kilogram
kW	Kilowatt
kWh	Kilowatt Hour
L	Liter
L/min	Liter per Minute
LM	Laboratory Manager
Lobo Liquids System	Lobo Liquids Rinse Water Recovery System
m^3	Cubic Meters
MDL	Method Detection Limit
mg/L	Milligram/Liter
min	Minute
mL	Milliliter
MP&M	Metal Products & Machinery
MRL	Method Reporting Limit
MSDS	Material Safety Data Sheet(s)
NA	Not Applicable
ND	Not Detected

ACRONYMS & ABBREVIATIONS (continued)

NR	Not Regulated
NRMRL	National Risk Management Research Laboratory
O&G	Oil and Grease
O&M	Operating & Maintenance
P	Percent Recovery
PARCCS	Precision, Accuracy, Representativeness, Comparability, Completeness and Sensitivity
pH	Value used to express acidity or alkalinity
PLC	Programmable Logic Controller
POC	Point of Contact
POTW	Publicly Owned Treatment Works
PPE	Personal Protective Equipment
ppm	Parts per Million
PQL	Practical Quantification Limit
PSES	Pretreatment Standards for Existing Sources
psi	Pounds per Square Inch
QA/QC	Quality Assurance/Quality Control
QMP	Quality Management Plan
R	Raw Wastewater Samples
RPD	Relative Percent Difference
Rx	Reactor
SGP	Strategic Goals Program
SOP	Standard Operating Procedure
SR	Sample Result
SSR	Spiked Sample Result
STL	Severn Trent Laboratories
T	Treated Wastewater Samples
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
TOP	Total Organic Parameter
TSA	Technical System Audit
TSS	Total Suspended Solids
U.S.	United States

1.0 INTRODUCTION

The purpose of this test plan is to document the objectives, procedures, equipment, and other aspects of testing that will be utilized during verification testing of the Lobo Liquids Rinse Water Recovery System (Lobo Liquids system). This test plan has been prepared in conjunction with the U.S. Environmental Protection Agency's (EPA's) Environmental Technology Verification Program for Metal Finishing Pollution Prevention Technologies (ETV-MF). The objective of this program is to identify promising and innovative pollution prevention technologies through EPA-supported performance verifications. The results of the verification test will be documented in a verification report that will provide objective performance data to metal finishers, environmental permitting agencies, and industry consultants. A verification statement, which is an executive summary of the verification report, will be prepared and signed by the EPA National Risk Management Research Laboratory (NRMRL) Director and the Concurrent Technologies Corporation ETV-MF Program Manager.

1.1 Background

The Lobo Liquids system is designed to process and recover for reuse wastewaters generated by metal finishing processes. This wastewater usually contains dissolved metals and other chemicals that are associated with the plating baths. At metal finishing facilities, rinsing operations generate the majority of wastewater. A typical metal finishing job shop discharges approximately 34,000 gallons per day (gpd) of wastewater, which contains a residual concentration of pollutants after treatment. The average metal finishing facility pays about $30,000/per year in water and sewer charges (based on industry average of $3.50/1,000 gal) [Ref. 1]. Recovering and reusing wastewater reduces pollutant loadings on publicly owned treatment works (POTWs) or receiving streams and reduces operating costs.

The focus of testing will be to determine the quality of the effluent produced by the Lobo Liquids system at a pre-set flow rate and the quantity and characteristics of by-products produced during processing. In terms of effluent water quality, of particular interest is the ability of the treatment system to meet existing effluent standards for the Metal Finishing point source category [Ref. 2] and proposed effluent standards for the Metal Products and Machinery (MP&M) point source category [Ref. 3]. The metal finishing regulations were promulgated in July 1983, and for most metal finishing companies are the applicable current standards. The proposed MP&M limitations were published on January 3, 2001. EPA must take final action on the proposed rule by December 2002. This final action does not have to be new limits, but can be a decision by the Agency not to impose any additional regulations on the metal finishing industry. If new limits are imposed, the MP&M limitations will replace the metal finishing limitations for most metal finishing companies.

Testing of the Lobo Liquids system will be conducted at Gull Industries, located in Houston, Texas. Gull Industries is a metal finishing job shop that performs decorative chromium electroplating (consists of cleaning and nickel and chromium electroplating), electroless nickel plating, and passivation of stainless steel. A Lobo Liquids system has been installed at Gull Industries since January 2001. That system has a design flow rate of 83 liters/minute (L/min) (22 gallons per minute (gpm)) and it can be operated in a batch or continuous mode. Also, it can be operated as a standalone treatment system or as a polishing technology. At Gull Industries, an electrocoagulation system is also installed, which is a 38-L/min commercial unit with two electrocoagulation reactors (Rxs) connected in series.

The verification test will evaluate the Lobo Liquids system in two modes:

- Ion exchange polishing system following an electrocoagulation process
- Ion exchange standalone treatment system

During the first test, wastewater from the Gull Industries electroplating line will be processed through the electrocoagulation system and subsequently processed through the Lobo Liquids system. Testing will consist of three runs, with each run treating approximately 3,400 L of wastewater. During testing, samples of raw and treated wastewater and sludge, and ion exchange regenerate, will be collected and analyzed.

During the second test, the Lobo Liquids system will treat the raw wastewater as a standalone technology. Prior to this test, the Lobo Liquids system will be regenerated. It will then be put into service and will process wastewater until regeneration is required. During testing, samples of raw and treated wastewater, and ion exchange regenerate, will be collected and analyzed.

This test plan has been structured based on a format developed for ETV-MF projects. This document describes the intended approach and explains testing plans with respect to areas such as test methodology, procedures, parameters, and instrumentation. Also included are quality assurance (QA)/quality control (QC) requirements of this task that will ensure the accuracy of data, data interpretation procedures, and worker health and safety considerations.

1.2 Data Quality Objectives (DQO)

The systematic planning elements of the data quality objectives process identified in "Guidance for the Data Quality Objectives Process" (EPA QA/G-4, August 2000) [Ref. 4] were specifically utilized during preparation of this verification test plan. A project team composed of representatives from *CTC,* the testing organization, the technology vendor, the host site, the analytical laboratory, and EPA assisted in preparing this test plan. The team jointly developed the test objectives; critical and non-critical measurements; test matrix; sample quantity, type, and frequency; analytical methods; and QA objectives to arrive at an optimized test designed to verify the performance of the technology.

2.0 TECHNOLOGY DESCRIPTION

2.1 Theory of Operation

The Lobo Liquids system consists of three skid-mounted, ion exchange pressure vessels, with interconnecting piping and control valves. It is also equipped with a personal computer based control system running under Windows.

Ion exchange is a chemical reaction wherein an ion from solution is exchanged for a similarly charged ion attached to an immobile solid particle (i.e., ion exchange resin). Ion exchange reactions are stoichiometric (i.e., predictable based on chemical relationships) and reversible. The strategy employed in using this technology is to exchange somewhat harmless ions (e.g., hydrogen and hydroxyl ions), located on the resin, for ions of interest in the solution (e.g., regulated metals). In the most basic sense, ion exchange materials are classified as either cationic or anionic. Cation resins exchange hydrogen ions for positively charged ions such as nickel, copper and sodium. Anion resins exchange hydroxyl ions for negatively charged ions such as chromates, sulfates and cyanide [Ref. 5].

Ion exchange resins are usually contained in vessels referred to as columns. The basic column consists of a resin bed, which is retained, in the column with inlet and outlet screens, and service and regeneration flow distributors. Piping and valves are required to direct flow, and instrumentation is required to monitor water quality and control regeneration timing. The systems are operated in cycles consisting of the following four steps:

1) Service (exhaustion) - Water solution containing ions is passed through the ion exchange column or bed until the exchange sites are exhausted.
2) Backwash - The bed is washed (generally with water) in the reverse direction of the service cycle in order to expand and resettle the resin bed.
3) Regeneration - The exchanger is regenerated by passing a concentrated solution of the ion originally associated with it (usually a strong mineral acid or base) through the resin bed.
4) Rinse - Excess regenerant is removed from the exchanger, usually by passing water through it.

2.2 Description of the Lobo Liquids System

The Lobo Liquids system consists of skid-mounted pressure vessels, with interconnecting piping and control valves. The process parameters and equipment status are constantly monitored and fed back to the control system and can be viewed on a display located in the control panel, which is also mounted on the skid.

A schematic diagram of the Lobo Liquids system is shown in **Figure 1.** The system operates by receiving influent from a tank, via a three-way valve and the suction side of a pump. The water is then discharged from the pump under pressure, and is monitored for pH, specific conductance, pressure, and flow. The resultant analogue signals are sent to the control system for subsequent processing and display. Each of the analogue signals has two high-level and two low-level alarms. The alarms cause the valve systems to either open or close, which cause a change of direction or stopping of flow. The water is allowed to enter the top of the first vessel containing a cation resin to remove the initial shock loading of heavy metals, whereupon it exits at the bottom of that vessel.

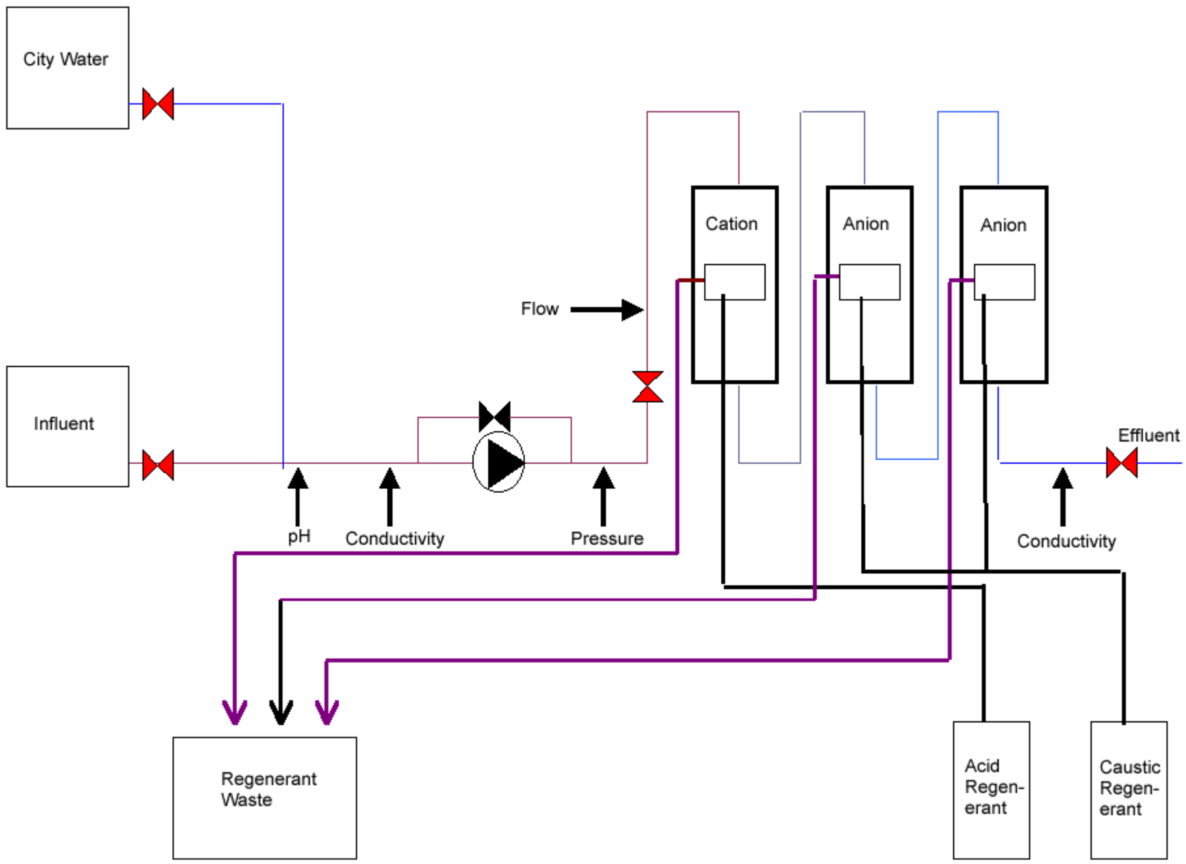

Figure 1. Diagram of Lobo Liquids System

The partially de-ionized water then enters the second and third vessels (anion columns) in the same manner as the first vessel, and there the remaining ionic loading is removed. The resultant discharge from the third vessel is again monitored for specific conductance and can then be reused in the metal finishing process.

The contaminants from the influent (i.e., metal cations) and anions (hexavalent chromium and nonmetals) will remain in each of the three vessels bonded to each of the special purpose resins. The water is allowed to flow continuously through the system until such time that the resin is exhausted (i.e., its ability to remove cations and anions from the water is ended). This is determined by the specific conductance of the water exiting the system at the third vessel. At this point the system will go off line (usually outside production hours) and regenerate itself *in situ*.

The regeneration process (the process of removing cations and anions from the resin, which were captured during normal operation) is carried out automatically. Each vessel will regenerate itself in turn starting with the first vessel. Passing acids and/or bases over the resins, which will remove the captured cations and anions, carries out regeneration of the resin. City water is used as a rinse following regeneration. This regenerant will exit each of the vessels and be captured in the regenerant storage for subsequent processing and disposal. At this point the unit will then be ready to go back on line for the processing of influent.

2.3 Description of Electrocoagulation System

Various configurations of the electrocoagulation system are in use. A diagram of a typical system is shown in **Figure 2**. Wastewater initially flows into the electrocoagulation Rx. Systems can be configured with one or more Rxs in series. In the electrocoagulation Rx, a direct current (DC) (100 to 120 amperes (amp), 25 to 40 volts DC) is applied using an associated rectifier and sacrificial anode plates. The typical residence time in the reactor is 14 seconds. Reactions occur in the Rx, including the reduction of Cr^{+6} to Cr^{+3} and the generation of insoluble oxides and hydroxides. The wastewater flows from the Rx to a de-foam tank, which has a residence time of 30 min. Electrolysis gases are separated from the wastewater in the de-foam tank, which is agitated by a mechanical mixer. A polymer is sometimes added to improve floc formation as the wastewater exits the de-foam tank. The wastewater is then transferred through a sludge thickener to a conventional clarifier where solids separation takes place. The overflow from the clarifier is discharged to the intermediate storage tank. The underflow from the clarifier goes to a thickener. Thickened sludge is dewatered on a filter press and sent off-site for recovery or disposal.

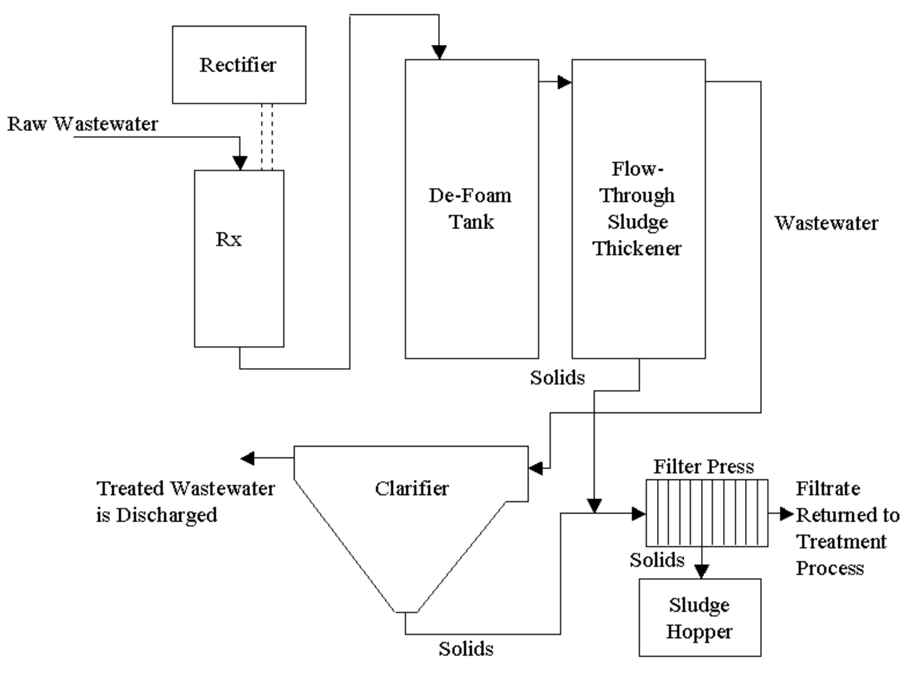

Figure 2. Diagram of Electrocoagulation System

2.4 Commercial Status

The Lobo Liquids system is a commercial product. The electrocoagulation system is a separate commercial product.

2.5 Environmental Significance

The Lobo Liquids system reportedly removes toxic metals to near or below detection limits and effectively deionizes wastewater. Resultant discharges of the wastewater contain little or no measurable concentrations of toxic metals. Also, the processed water may be sufficiently purified for reuse in the electroplating process, which may entirely eliminate the discharge of liquid to the sewer or receiving streams.

2.6 Local Installation

The Lobo Liquids and electrocoagulation systems will be tested at Gull Industries, located in Houston, Texas. Gull Industries is a metal finishing job shop that performs nickel and chromium electroplating, electroless nickel-plating, and passivation using nitric acid. The current discharge limits for Gull Industries are shown in **Table 1**.

A photograph of the decorative chromium plating line is shown in **Figure 3**.

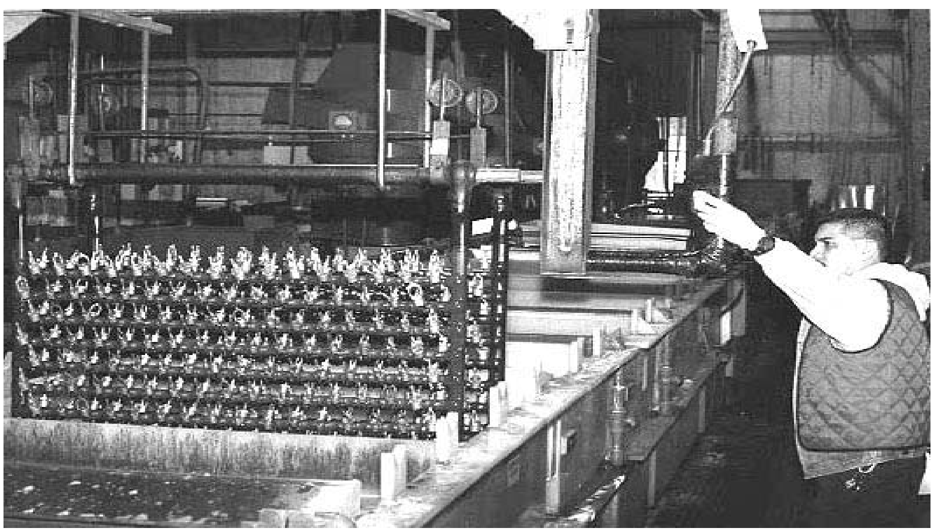

Figure 3. Photograph of Gull Industries' Decorative Chromium Plating Line

Gull Industries has established water-recycling specifications that must be met in order to reuse treated wastewater. These specifications are:

- Total Dissolved Solids (TDS) of 250 mg/L
- Specific conductance: maximum of 500 microsiemens (µS)
- pH: within the range of 5.0 to 9.0 standard units

The electrocoagulation system installed at Gull Industries is rated at 38 L/min and has dual electrocoagulation Rxs, piped in series. The Lobo Liquids system installed at Gull Industries is rated at 83 L/min. It has one cation column (1.02 cubic meters (m^3) of resin) and two anion columns (total of 1.13 m^3 of resin).

Parameter	Current Gull Industries Limitations		MP&M Pretreatment Standards for Existing Sources (PSES) Job Shop Subcategory	
	Daily Max., mg/L	4-Day Avg., mg/L	Daily Max., mg/L	Monthly Avg., mg/L
Cyanide T	NR	NR	0.21	0.13
Cyanide A	5.0	2.7	0.14	0.07
Cadmium	1.2	0.7	0.21	0.09
Chromium*	3.0	1.0	1.3	0.55
Copper*	3.0	2.0	1.3	0.57
Lead	0.6	0.4	0.12	0.09
Mercury*	0.02	0.01	NR	NR
Manganese	NR	NR	0.25	0.10
Molybdenum	NR	NR	0.79	0.49
Nickel*	3.0	2.0	1.5	0.64
Silver*	2.0	1.0	0.15	0.06
Tin	NR	NR	1.8	1.4
Zinc	2.61	1.48	0.35	0.17
O&G (local limit)	100	NR	NR	NR
O&G (as HEM)	NR	NR	52	26
TSS	NR	NR	NR	NR
TOC	NR	NR	78	59
TOP	NR	NR	9.0	4.3
Sulfide (as S)	NR	NR	31	13

NR = Not Regulated

*Local standard only (grab/composite instead of daily max./4-day average).

Current Gull Industries limitations are based on a combination of local and Federal standards (40 CFR 413 and 40 CFR 433).

O&G (as HEM) are not regulated under pretreatment standards for the Job Shop subcategory. However, it is regulated under the Best Practical Treatment (BPT) limitations for direct dischargers in the Job Shop subcategory (66 FR 423). The values shown are the BPT proposed limitations.

Table 1. Summary of Current and Proposed Regulations Applicable to Gull Industries

The majority of wastewater generated at Gull Industries is rinsewater and to a lesser extent spent cleaning baths. Approximately 7,500 L of wastewater is generated on a daily basis at Gull Industries. The concentration of regulated metals in the wastewater is typically above 300 mg/L (mostly nickel and chromium). Raw wastewater is stored in a 20,000-L equalization tank (**Figure 4**) prior to treatment.

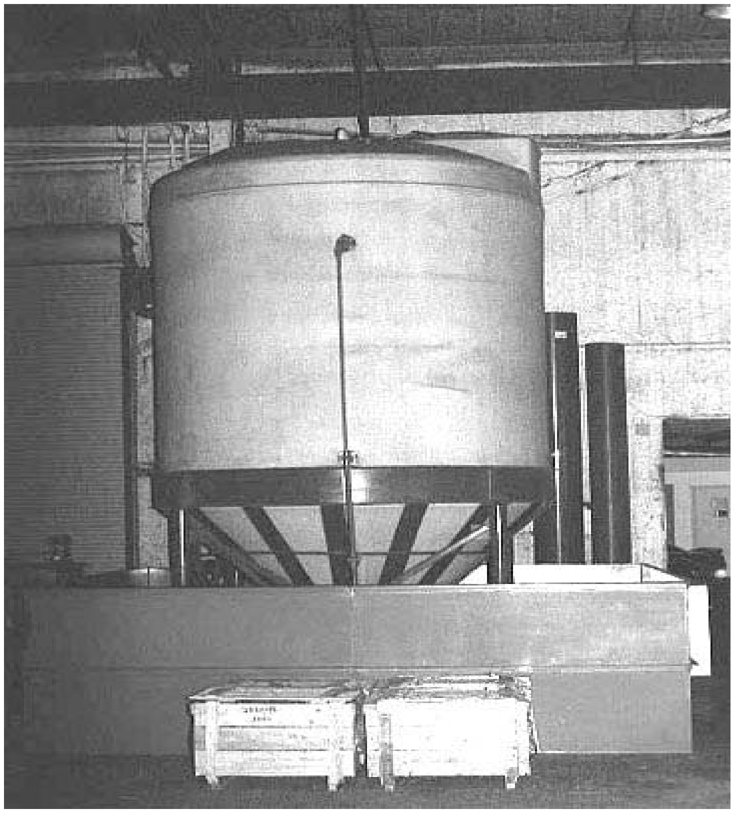

Figure 4. 20,000-Liter Equalization Tank

A diagram of the combined electrocoagulation/ion exchange system installed at Gull Industries is shown in **Figure 5**. It consists of electrocoagulation (two Rxs in series), de-foam tank, flow-through sludge thickener, clarifier, filter press, ion exchange system, storage tanks, and associated pumps, piping and controls. The treatment tanks (de-foam tank, flow-through sludge thickener, clarifier) have a total liquid capacity of approximately 3,400 L (900 gal).

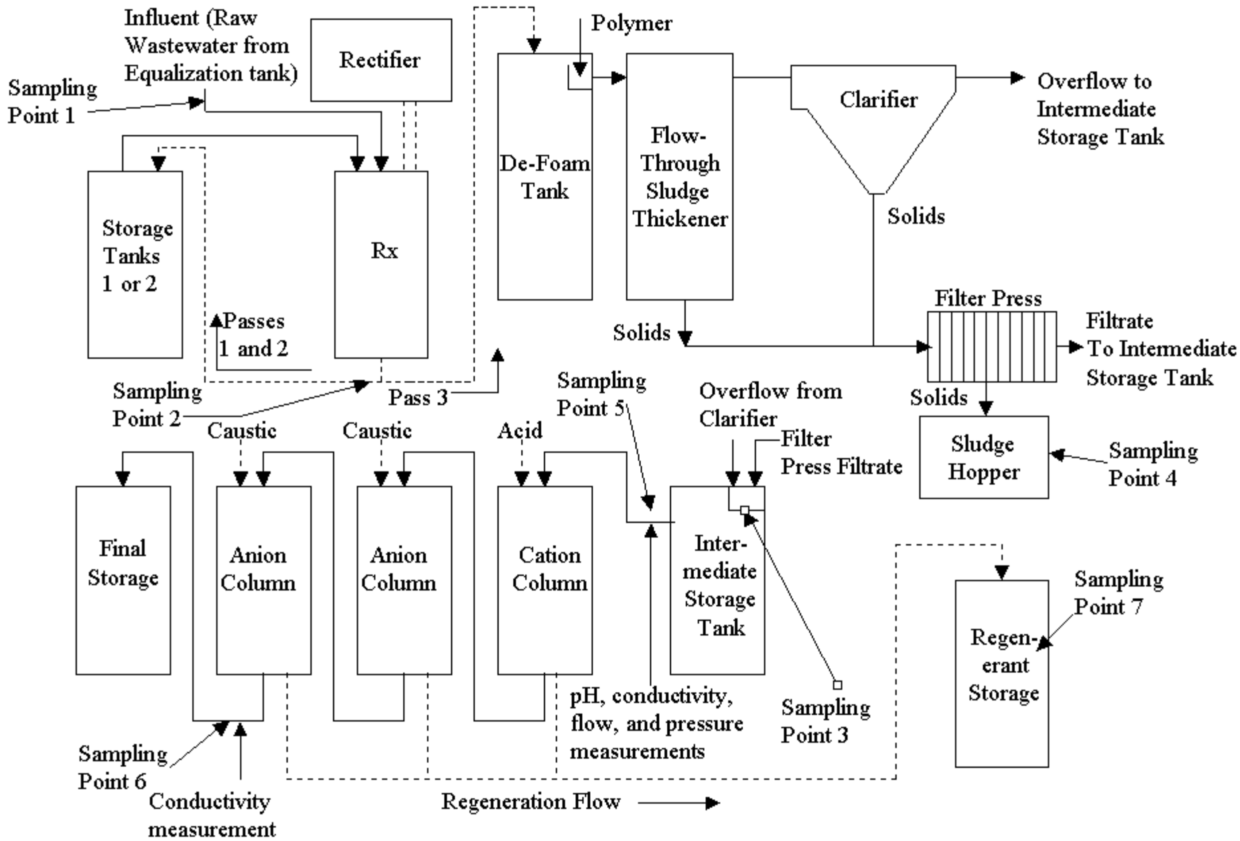

Figure 5. Diagram of the Combined Treatment System at Gull Industries

The following indicates the size and utility requirements of the Lobo Liquids system installed at Gull Industries:

- Flow Rate: 83 L/min (22 gpm)
- Electrical: 2.2 Kilowatts (kW)
- Air: 1 cubic foot per minute (cfm) @ 80 pounds per square inch (psi) intermittently
- City Water: Max flow 13.6 L/min (3.6 gpm), for regeneration only
- Dimensions: Length 15 feet (ft)
  Width 8 ft
  Height 10 ft

The Lobo Liquids system has the following parameters monitored and continuously logged to disk:

- Inlet specific conductance (µS)
- pH
- Pump discharge pressure (psi)
- Flow (gpm)
- Outlet specific conductance (µS)

All parameters are logged by software for subsequent analysis and archived for viewing at a later date. All events are date and time stamped. As part of the after sales back up, the system is connected to a modem, so that if a problem should arise the manufacturer is able to remotely interrogate the system for troubleshooting purposes. It also allows downloading of software updates.

The Gull Industries treatment system can be operated in two different modes, as described below.

2.6.1 Electrocoagulation/Ion Exchange Polishing Mode

In this mode, wastewater treatment is performed on a batch basis. Each batch consists of approximately 3,400 L (900 gal) and the processing rate is 38 L/min. Approximately one to two batches are processed each day. During treatment, wastewater is pumped from the 20,000-L equalization tank through the Rx. Wastewater exiting the Rx is diverted to one of two storage tanks (Tanks 1 and 2). Once the entire batch has been processed, the wastewater in that storage tank is tested using bench-top methods[1]. If the wastewater is insufficiently treated, it is reprocessed through the electrocoagulation system and diverted to the other storage tank, and retested using the bench-top methods. If the wastewater is determined to be sufficiently treated, i.e. the metals concentrations are below discharge limits as listed in **Table 1**, the wastewater is reprocessed through the electrocoagulation unit and the discharge is diverted to the de-foam tank. The wastewater then flows through the sludge thickener and the clarifier and is collected in the intermediate storage tank. From this point, the wastewater is processed through the Lobo Liquids system.

As discussed above, the electrocoagulation process is repeated as necessary until the bench-top methods indicate that the concentration of regulated parameters is sufficiently low. Typically, each 3,400 to 3,800-L batch at Gull Industries is processed through the Rx two times. During the first pass, most of the hexavalent chromium is reduced to trivalent chromium, and a portion of the dissolved metals is precipitated. During the second pass, the majority of the dissolved metals are precipitated. Analytical results of samples collected during a preliminary test of the technology reflect these expected results (see **Table 2**).

A photograph of the Lobo Liquids system is shown in **Figure 6.** The system operates by pumping wastewater (effluent from electrocoagulation system) from the intermediate storage tank to the ion exchange system. The wastewater passes continuously through one cation and two anion columns. The ion exchange polishing system removes any residual dissolved metals near or below detection levels and substantially lowers the TDS of the water. Analytical results of

[1] A sample of the wastewater from the storage tank in use is subjected to a simulated treatment process, performed in a beaker. A small amount of polymer is added to the beaker, which causes precipitated solids in the wastewater to form a dense floc and settle to the bottom of the beaker. The clarified wastewater or "supernatant" is then sampled and tested for nickel and chromium using bench-top analytical procedures.

samples collected during a preliminary test are shown in **Table 2**. The effluent of the ion exchange system is sufficiently purified that it is used as rinse water on the Gull Industries metal finishing line. The wastewater may also be discharged to the city sewer system. The pH and specific conductance of the wastewater are monitored at various points in the process. The system is capable of treating up to approximately 45,000 to 90,000 L of wastewater before the resin is exhausted (i.e., its ability to remove cations and anions from the wastewater is ended). This is determined by the specific conductance of the water exiting the system at the third vessel. Once the resin is exhausted, the system will go off line (usually outside production hours), and regenerate itself *in situ*.

Figure 6. Photograph of the Ion Exchange Polishing System Installed at Gull Industries

	Raw (mg/L)	Pass 1 (mg/L)	Pass 2 (mg/L)	IX
TDS	1320	876	655	127
Chromium (Hexavalent)	70.5	<0.015	<0.015	<0.015
Cadmium	0.008	ND	ND	ND
Chromium (T)	89.5	ND	0.287	ND
Copper	1.31	ND	0.011	ND
Iron	6.65	11.1	8.80	ND
Lead	0.252	0.013	ND	ND
Manganese	0.171	2.96	1.71	ND
Molybdenum	ND	ND	ND	ND
Nickel	202	45.6	1.29	1.98
Tin	0.057	ND	ND	ND
Zinc	3.09	0.734	ND	ND

ND = Not Detected

Table 2. Preliminary Analytical Results

2.6.2 Ion Exchange Primary Treatment Mode

A diagram of the Lobo Liquids system at Gull Industries operating as a standalone treatment system is shown in **Figure 7**. During operation, the raw wastewater is pumped from the equalization tank to the Lobo Liquids system, and the effluent is discharged to the final storage tank, from where it is available for reuse on the metal finishing line. In this mode, the Lobo Liquids system is operated continuously during production hours and is idled during non-production hours. The system is operated in this manner until the resin columns require regeneration, which occurs approximately every 10 to 20 business days. Regenerant is collected in the regenerant storage tank.

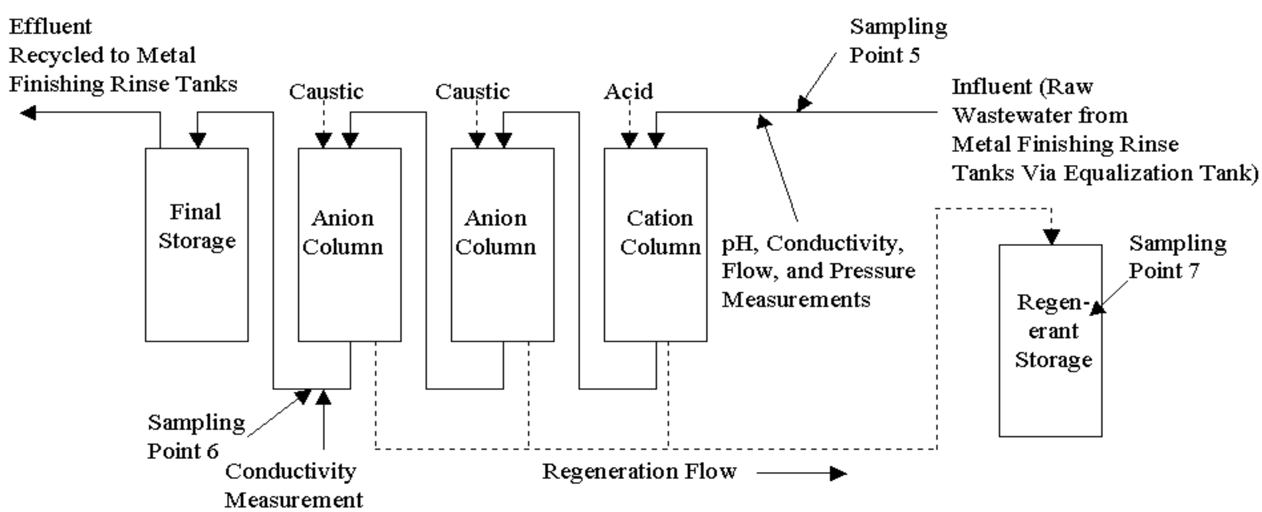

Figure 7. Diagram of Lobo Liquids System Used as Primary Treatment System

3.0 EXPERIMENTAL DESIGN

3.1 Test 1 - Combined Electrocoagulation/Lobo Liquids System Test

3.1.1 Test Goals and Objectives

The overall goals of Test 1 are: (1) evaluate the ability of the combined electrocoagulation system and Lobo Liquids ion exchange polishing system to remove pollutants from metal finishing job shop wastewaters with the metal finishing effluent guidelines used as target effluent concentrations, (2) determine the ability of the systems to recover water for reuse in the electroplating process, (3) evaluate the operating characteristics of the systems with respect to sludge and regenerant generation and operating costs, and (4) evaluate the environmental benefit by determining the reduction in metals discharged to the city sewer system.

The following is a summary of primary project objectives. For the installation at Gull Industries, verification testing is being conducted in order to:

- Determine the ability of the electrocoagulation and ion exchange polishing systems to remove specific contaminants from wastestreams and meet Gull Industries' target criteria for water reuse.
- Determine the quantity and chemical characteristics of the sludge generated by the electrocoagulation treatment process and the cost of sludge disposal.
- Determine the cost of operating the electrocoagulation and ion exchange polishing systems for the specific conditions encountered during testing:
  - Determine the amount and cost of operating and maintenance (O&M) labor.
  - Determine the quantity and cost of chemical reagents used and other materials (e.g., filters), including ion exchange regeneration.
  - Determine the quantity and cost of steel plates (anodes) consumed.
  - Determine the quantity and cost of energy consumed by operating the systems.
- Quantify the environmental benefit by determining the reduction in metals discharged to the sewer system beyond that required by the metal finishing standards.

3.1.2 Critical and Non-Critical Measurements for Test 1

Measurements that will be taken during testing are classified below as either critical or non-critical. Critical measurements are those that are necessary to achieve the primary project objectives. Non-critical measurements are those related to process control or general background readings.

<u>**Critical Measurements:**</u>

- volume of wastewater treated (L/run)
- quantity (kilogram (kg)/run) and costs ($/run) of chemical treatment reagents and other materials used in treatment
- mass of steel plates consumed (kg)/volume processed
- volume of filter press sludge generated (L/test)
- chemical characteristics of raw wastewater (specific conductance (µS)[2], mg/L of total suspended solids (TSS), O&G (as HEM), total organic carbon (TOC), cadmium, hexavalent chromium, chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, sulfide (as S), zinc, and TDS)
- chemical characteristics of treated effluent from the electrocoagulation system (specific conductance (µS), mg/L of TSS, O&G (as HEM), TOC, cadmium, hexavalent chromium, chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, sulfide (as S), zinc, and TDS)
- chemical characteristics of treated effluent from the Lobo Liquids system (specific conductance (µS), pH, mg/L of TSS, O&G (as HEM), TOC, cadmium, chromium (+6) chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, sulfide (as S), zinc, and TDS)
- chemical characteristics of filter press sludge from the electrocoagulation system (density, mg/L of solids, cadmium, chromium (T), copper, lead, manganese, molybdenum, nickel, tin, and zinc)
- O&M labor requirements (hours/run) and costs ($/run)
- energy use for components of the electrocoagulation and ion exchange polishing systems (e.g., rectifier, pumps) (kWh/run) and costs ($/run)
- Chemical use during ion exchange regeneration

<u>**Non-Critical Measurements:**</u>

- electrocoagulation rectifier DC output (amp-hours)
- pH of raw wastewater
- pH of treated wastewater following treatment by the electrocoagulation system
- flow, pH, and specific conductance of wastewater at various internal points within the Lobo Liquids system

3.1.3 Test Matrix for Test 1

The verification test will be conducted by processing batches (3,400-L each) of raw wastewater through the electrocoagulation system and subsequently through the Lobo Liquids system (each completely treated batch is referred to as a "run"). Each batch will be processed through the electrocoagulation reactors a minimum

[2] Specific conductance is a measure of the ability of a water solution to conduct an electrical current. It is commonly expressed in microsiemens. Specific conductance is related to the type and concentration of ions in solution and can be used for approximating the dissolved-solids content of the water.

of three times[3] (3 "passes") prior to clarification. Following clarification, the wastewater will be stored in an intermediate storage tank. It will then be processed through the Lobo Liquids system and collected in a final storage tank. For each run, samples will be collected of the initial raw wastewater, the discharge after each pass through the two electrochemical reactors, the treated wastewater following clarification, the influent to the Lobo Liquids system, and the effluent from the Lobo Liquids system. A single sludge sample will be collected after all the batches of wastewater are treated, and a single sample of the ion exchange regenerant will be collected after the Lobo Liquids system is regenerated. The operating conditions for the three runs are shown in **Table 3**.

Run	Wastewater Processed	Electrocoagulation System Conditions	Lobo Liquids System Conditions
1	3,400 L of raw wastewater	• 38 L/min • 3 EC cycles • 100 to 120 amps • 25 to 40 volts DC • Variable pH • 10-20 mg/L polymer addition	• 80 L/min • Variable pH
2	3,400 L of raw wastewater	• 38 L/min • 3 EC cycles • 100 to 120 amps • 25 to 40 volts DC • Variable pH • 10-20 mg/L polymer addition	• 80 L/min • Variable pH
3	3,400 L of raw wastewater	• 38 L/min • 3 EC cycles • 100 to 120 amps • 25 to 40 volts DC • Variable pH • 10-20 mg/L polymer addition	• 80 L/min • Variable pH

Table 3. Test Matrix for Test 1: Combined Electrocoagulation System and Ion Exchange Polishing System Test

Test objectives and measurements are summarized in **Table 4**. The analytical test parameters selected for this verification test are the parameters found in the Metal Finishing and proposed MP&M regulations, plus iron, which is contributed to the wastewater during the electrocoagulation process.

[3] As discussed in section 2.6.1, Gull Industries repeats the electrocoagulation process until the wastewater is sufficiently treated to meet local standards. Typically, the wastewater is processed for two times through the Rx; however, a greater number of cycles are possible to attain complete treatment.

Test	Test Objective	Test Measurement
Runs 1 to 3	Determine the ability of the electrocoagulation system to remove specific pollutants from wastestreams and meet the applicable Metal Finishing and proposed MP&M limitations and water recycling specifications of Gull industries.	- Volumes of raw wastewater processed. - Chemical characteristics of the influent and effluent.
Runs 1 to 3 (combined)	Determine the quantity and chemical characteristics of the sludge and regenerant generated by the treatment processes.	- Volumes of raw wastewater treated. - Quantity, density and chemical characteristics of the sludge. - Quantity and chemical characteristics of the regenerant.
Runs 1 to 3	Determine the cost of operating the treatment systems for the specific conditions encountered during testing.	- Volume of raw wastewater processed. - O&M labor requirements. - Energy use. - Input quantity and costs of chemical treatment reagents (pounds/test run) and other materials used in treatment. - Cost of sludge disposal. - Cost of steel plates consumed.
Runs 1 to 3 (combined)	Quantify the environmental benefit by determining the reduction in metals discharged to the Houston POTW beyond that required by the Metal Finishing regulations.	- Volume of raw wastewater processed. - Chemical characteristics of the effluent.

Table 4. Test Objectives and Related Test Measurements for Test 1: Combined Electrocoagulation and Lobo Liquids Systems

3.2 Test 2 - Lobo Liquids System Used as Standalone Treatment System

3.2.1 Test Goals and Objectives

The overall goals of Test 2 are: (1) evaluate the ability of the Lobo Liquids ion exchange system to remove pollutants from metal finishing job shop wastewaters with the metal finishing effluent guidelines used as target effluent concentrations, (2) determine the ability of the Lobo Liquids system to recover water for reuse in the electroplating process, (3) evaluate the operating characteristics of the ion exchange system with respect to regeneration and operating costs, and (4) evaluate the environmental benefit by determining the reduction in metals discharged to the city sewer system.

The following is a summary of primary project objectives. For the installation at Gull Industries, verification testing is being conducted in order to:

- Determine the ability of the Lobo Liquids system to remove specific contaminants from wastestreams and meet Gull Industries' target criteria for water reuse.

- Determine the cost of operating the Lobo Liquids system for the specific conditions encountered during testing:
- Determine the amount and cost of O&M labor.
- Determine the quantity and cost of chemical reagents used and other materials (e.g., filters), including ion exchange regeneration.
- Determine the quantity and cost of energy consumed by operating the system.
- Quantify the environmental benefit by determining the reduction in metals discharged to the sewer system beyond that required by the metal finishing standards.

3.2.2 Critical and Non-Critical Measurements

Measurements that will be taken during testing are classified below as either critical or non-critical. Critical measurements are those that are necessary to achieve the primary project objectives. Non-critical measurements are those related to process control or general background readings.

Critical Measurements:

- volume of wastewater treated (L/run)
- quantity (kg/run) and costs ($/run) of chemical treatment reagents and other materials used in treatment
- chemical characteristics of raw wastewater (specific conductance (µS), mg/L of TSS, O&G (as HEM), TOC, cadmium, hexavalent chromium, chromium (T), copper, lead, manganese, molybdenum, nickel, tin, sulfide (as S), zinc, and TDS)
- chemical characteristics of treated effluent from the Lobo Liquids system (specific conductance (µS), pH, mg/L of TSS, O&G (as HEM), TOC, cadmium, hexavalent chromium, chromium (T), copper, lead, manganese, molybdenum, nickel, tin, sulfide (as S), zinc, and TDS)
- chemical characteristics of the Lobo Liquids system regenerant (mg/L of solids, cadmium, chromium (T), copper, lead, manganese, molybdenum, nickel, tin, zinc)
- O&M labor requirements (hours/run) and costs ($/run)
- energy use for components of the Lobo Liquids system (e.g., pumps) (kWh/run) and costs ($/run)

Non-Critical Measurements:

- pH and specific conductance of wastewater at various internal points within the Lobo Liquids system

3.2.3 Test Matrix for Test 2

A single run will be performed during Test 2. The run will be conducted by processing 80 L/min of raw wastewater through the ion exchange system until the system requires regeneration. The Lobo Liquids system will be regenerated prior to the run and will be operated continuously during production hours at Gull Industries (approximately 8 hr/day) and idled during non-production hours. The treated wastewater will be reused on the Gull Industries metal finishing line. Samples will be collected of the initial raw wastewater and the effluent from the ion exchange system. A single sample of the ion exchange regenerant will be collected after the Lobo Liquids system is regenerated. The operating conditions for the run are shown in **Table 5**.

Wastewater Processed	Test Conditions
Raw wastewater generated from rinsing operations at Gull Industries.	• Regeneration prior to run • Operate system during production hours • 80 L/min flow rate • Stop run when system requires regeneration.

Table 5. Test Matrix for Test 2: Standalone Lobo Liquids System

Test objectives and measurements are summarized in **Table 6**. The analytical test parameters selected for this verification test are the parameters found in the Metal Finishing and proposed MP&M regulations, plus iron, which is present in the Gull Industries wastewater.

Test	Test Objective	Test Measurement
Run 1	Determine the ability of the Lobo Liquids system to remove specific pollutants from wastestreams and meet the water recycling criteria of Gull industries.	- Volumes of raw wastewater processed. - Chemical characteristics of the influent and effluent.
Run 1	Determine the quantity and chemical characteristics of the regenerant generated by the treatment processes.	- Volumes of raw wastewater treated. - Quantity and chemical characteristics of the regenerant.
Run 1	Determine the cost of operating the treatment system for the specific conditions encountered during testing.	- Volume of raw wastewater processed. - O&M labor requirements. - Energy use. - Input quantity and costs of chemical treatment reagents (pounds/run) and other materials used in treatment.
Run 1	Quantify the environmental benefit by determining the reduction in metals discharged to the Houston POTW beyond that required by the local regulations.	- Volume of raw wastewater processed. - Chemical characteristics of the effluent.

Table 6. Test Objectives and Related Test Measurements for Test 2: Standalone Lobo Liquids System

3.3 Testing and Operating Procedures

3.3.1 Set-Up and System Initialization Procedures

3.3.1.1 Test 1

The electrocoagulation and ion exchange systems are currently installed at Gull Industries, and no additional equipment set-up is required. The electrocoagulation system will be drained and cleaned and the ion exchange system will be regenerated prior to testing. The quantity of chemicals used for regeneration and the total volume of regenerant collected will be measured and recorded in accordance with procedures outlined in sections 3.3.4.3 and 3.3.4.4. A diagram of the system configuration for Test 1 is shown in **Figure 5**.

Prior to initiating each run, the entry of wastewater into the 20,000-L equalization tank (raw wastewater storage) will be stopped until the pumping of the raw wastewater through the electrocoagulation reactor is complete (approximately 90 min). This procedure will eliminate variability of raw wastewater characteristics during each batch and allow for grab sampling of the raw wastewater.

3.3.1.2 Test 2

The ion exchange system will be regenerated prior to testing, and the regenerant storage tank will be subsequently drained. A diagram of the system configuration for Test 2 is shown in **Figure 7**.

3.3.2 System Operation

During Tests 1 and 2, the systems will be operated by Gull Industries according to the standard procedures found on file at Gull Industries. These are the procedures used on a daily basis at Gull Industries for conducting wastewater treatment. Representatives of the electrocoagulation and the ion exchange system manufacturers will assist with operation of the systems. During Test 1, the source of raw wastewater during testing is the equalization tank that is part of the existing Gull Industries wastewater treatment system. The effluent from the Lobo Liquids system will be collected into the final storage tank and tested by Gull Industries and reused on their metal finishing lines or discharged to the city sewer system in accordance with their present discharge permit. During Test 2, the raw wastewater will be pumped from the equalization tank to the ion exchange system, and the effluent will discharge to a storage tank (to be installed), from where it will be pumped for reuse on the metal finishing line.

During Test 1, both the electrocoagulation system and the ion exchange system will be operated in batch modes, and only one system will be operated at a time.

Initially the electrocoagulation system will process a batch of raw wastewater, and the treated water will be collected in the intermediate storage tank. The electrocoagulation system will then be idled. The ion exchange system will then be initiated; it will process the batch of wastewater in the intermediate storage tank and discharge it to the final storage tank. This cycle will be repeated for each run.

As discussed in section 2.6, the treatment tanks installed at Gull Industries have a liquid capacity of approximately 3,400L. Prior to the start of the first run, the tanks will be drained and cleaned. The system will then process one batch (3,400 L) of wastewater using three passes through the Rx units before diverting the wastewater to the de-foam tank, thickener, and clarifier. Processing the first batch of wastewater through the electrocoagulation system will fill the treatment tanks (de-foam, thickener, and clarifier) with wastewater, but little or no discharge of treated wastewater to the intermediate storage tank will occur. When the second batch of wastewater is treated and passes through these tanks, this will cause an overflow of approximately 3,400L of wastewater from the clarifier to the intermediate storage tank. In effect, the raw wastewater volume from one batch treatment is discharged to the intermediate storage tank during the treatment of the subsequent batch. Some commingling of wastewater batches will occur, but this is not expected to have an effect on the results. Due to the "plug-flow" design of the system, samples of the raw wastewater from one run can be accurately paired with intermediate and final discharge samples of the subsequent run to determine the approximate pollutant removal efficiency of the systems. For example, when the Run 2 influent is being processed, samples from the intermediate storage tank, ion exchange influent, and final treated wastewater will be collected and the results paired with the Run 1 influent results.

During Test 2, the Lobo Liquids system will be set to automatically stop the service cycle and initiate the regeneration cycle when the specific conductance of the effluent is above 500 µS. This event is expected to occur approximately 10 to 20 operating days after initiation of Test 2.

The collection of samples from each batch treatment event during Test 1 and its relation to the runs is described in **Table 7**.

Batch	**Run**	**Raw Wastewater Sample Point 1**	**EC Reactor Discharge Sample Point 2**	**Intermediate Treated Wastewater Sample Point 3**	**Sludge Sample Point 4**	**IX Inf. Sample Point 5**	**Final Treated Wastewater Sample Point 6**	**Regeneran t Sample Point 7**
1	Run 1	R_1	EC_1	-	-	-	-	-
2	Run 1/2	R_2	EC_2	I_1	-	IX_1	F_1	-
3	Run 2/3	R_3	EC_3	I_2	-	IX_2	F_2	-
4	Run 3	R_4	-	I_3	$S_{1\text{-}3}$	IX_3	F_3	REG

Table 7. Sampling Sequence for Batch Treatment and Runs for Test 1

During Test 2, the ion exchange system will be operated continuously during production hours at Gull Industries, and the system will be idled during non-production hours. The ion exchange system will be operated in this manner until the system requires regeneration. The length of Test 2 is expected to be four to five business days.

3.3.3 Sample Collection and Handling

3.3.3.1 Test 1 Sample Collection and Handling

During Test 1, samples will be collected from the sampling points shown in **Figure 5**.

- **Raw wastewater (sample point 1).** A sampling port has been installed from which the raw wastewater samples will be collected. Grab samples of the raw wastewater will be collected 30 min (+/- 10 min) after initiation of each run (four) and placed into the appropriate sample containers. In order to generate a treated wastewater sample for the final run, it will be necessary to process a fourth batch of raw wastewater. A grab sample from this batch will also be collected, since some commingling of batches will occur. Although this sample will not be paired with a treated wastewater sample, the analytical results of this sample may be useful during an evaluation of data. The sampling sequence is described in **Table 7**.
- **Electrocoagulation reactor discharge (sample point 2).** A sampling port has been installed on the electrocoagulation reactor unit from which discharge samples will be collected. Grab samples of the discharge for hexavalent chromium and other metals analyses will be collected 30 min (+/- 10 min) after the first and second passes are initiated and placed into the appropriate sample containers. The electrocoagulation discharge contains both water and precipitated solids. These samples will be filtered at the analytical laboratory prior to analyses.
- **Intermediate treated wastewater (sample point 3).** Treated wastewater is discharged from the clarifier and filter press (separate pipes) to the intermediate storage tank. This tank cannot be fully drained due to its design. Therefore, to collect a representative sample, it is necessary to intercept the incoming flow before it commingles with the water in the intermediate storage tank. To accomplish this, a five-gal container will be hung inside the storage tank, above the water level, to intercept the two discharges. The discharges will enter the container and overflow into the intermediate storage tank. Grab samples for hexavalent chromium, other metals, pH, TDS, specific conductance, O&G, and sulfide will be collected 30 min (+/- 10 min) following initiation of the third pass. Samples will be collected using a glass ladle to draw treated wastewater from the

five-gal container and pour it into the appropriate sample bottles.

- **Wastewater treatment sludge (sample point 4).** After completion of the three runs, the filter press will be discharged to the sludge hopper. Grab samples of the sludge will be collected from the sludge hopper at five separate points using a clean spatula, after first completely mixing the material. The sludge sample will be placed into 1-L, wide mouth glass jars and mixed again.
- **Ion exchange system influent (sample point 5).** Grab samples of influent to the Lobo Liquids system will be collected from a sampling port for hexavalent chromium, other metals, pH, TDS, and specific conductance analyses. The samples will be collected 10 min (+/- 5 min) after initiation of the ion exchange treatment cycle.
- **Final treated wastewater (sample point 6).** Grab samples of treated wastewater from the Lobo Liquids system will be collected from a sampling port. Grab samples will be collected 20 min (+/- 5 min) after initiation of the ion exchange treatment process for hexavalent chromium, other metals, pH, TDS, specific conductance, O&G, and sulfide analyses.
- **Ion exchange system regenerant (sample point 7).** The Lobo Liquids system is regenerated approximately every 20 days when it is used in combination with the electrocoagulation system. The regenerant is collected in the regenerate storage tank. A Gull Industries employee, who will be trained by the ETV-MF Project Manager, will take grab samples of the regenerate from the regenerate storage tank for metals analyses.

3.3.3.2 Test 2 Sample Collection and Handling

During Test 2, samples will be collected from the sampling points shown in **Figure 7**. Samples will be collected from sample points 5 and 6 during the first three days of verification testing. Sampling will not be conducted during days 4 through 7 of verification. Sampling will resume on day 8 of testing and continue until the resin columns are exhausted. After day 4, only the samples collected on the day prior to regeneration after day 8 will be analyzed. Sampling procedures are described below.

- **Ion Exchange System Influent (sample point 5).** Grab samples of influent to the Lobo Liquids system will be collected from a sampling port for hexavalent chromium, other metals, pH, TDS, specific conductance analyses. Grab samples will be collected each day four hours (+/- one-hour) after daily start-up of the ion exchange treatment process.
- **Final treated wastewater (sample point 6).** Grab samples of treated wastewater will be collected on a daily basis from a sampling port. Grab samples will be collected each day four hours (+/- one-hour) after daily start-up of the ion exchange treatment process. The

samples will be analyzed for hexavalent chromium, other metals, pH, TDS, specific conductance, O&G, and sulfide.

- **Ion exchange system regenerant (sample point 7).** The Lobo Liquids system is regenerated approximately every 10 to 20 days when it is used as the primary treatment system. The regenerant is collected in the regenerate storage tank. A grab sample of the regenerate from the regenerate storage tank will be collected for metals analyses.

3.3.3.3 Additional Information on Sample Collection and Handling

Samples will be collected according to the schedule presented in **Tables 8** and **9**. All sampling events will be recorded on the forms shown in **Appendix A**.

At the time of sampling, each sample container will be labeled with the date, time, and sample identification (ID) number. Samples to be analyzed at an off-site laboratory will be accompanied by a chain of custody (COC) form. The COC form will provide the following information: project name, project address, sampler's name, sample numbers, date/time samples were collected, matrix, required analyses, and appropriate COC signatures. All samples will be transported in appropriate sample transport containers (e.g., coolers with packing and blue ice) directly to the lab. The transport containers will be secured with COC tape to ensure sample integrity during the delivery process to the analytical laboratory. The Project Manager (or trained designee) will perform sampling, and labeling, and ensure that samples are properly secured and shipped to the laboratory for analysis.

3.3.4 Process Measurements and Information Collection

Process measurements and information collection will be conduced to provide the following data: duration of treatment, volume of wastewater processed, reagent usage, steel plate (anode) consumption, sludge quantity, electricity use, and O&M activities. The methods that will be used for process measurements and information collection are discussed in this section.

3.3.4.1 Duration of Treatment and Wastewater Volume Processed

During both tests, the duration of each treatment cycle will be measured by recording the start and stop times for both the electrocoagulation and/or ion exchange polishing systems onto the data collection form found in **Appendix A**. During Test 1, the volume of wastewater processed during each batch will be measured after the first pass using the graduated scale found on storage tanks 1 and 2. During Test 2, the volume of water processed each day will be determined from the flow data recorded by the Lobo Liquids system. Prior to testing, the flow accuracy of the flow meter will be measured, and final flow measurements will be corrected if necessary.

Sample	Sample Location	Frequency/Type	Parameters
Raw wastewater	Sample point 1 (sample port)	One/run, plus a sample from the fourth batch. Grab sample collected after 30 min (+/- 10 min) of initiation of run.	TSS, TOC, cadmium, hex chromium, chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, zinc, TDS, O&G, sulfide, pH
Electrocoagulation reactor discharge	Sample point 2 (sample port)	One/run. Grab sample collected after 30 min (+/- 10 min) of initiation of run.	cadmium, hex chromium, chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, zinc, pH
Intermediate treated wastewater	Sample point 3 (container located in mouth of intermediate storage tank)	One/run. Grab sample collected after 30 min (+/- 10 min) of initiation of run.	TSS, TOC, cadmium, hex chromium, chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, zinc, TDS, O&G, sulfide, pH, specific conductance
Sludge	Sample point 4 (sludge hopper)	One/verification test. Representative grab sample collected after completion of the runs.	% solids, density, cadmium, chromium, copper, iron, lead, manganese, molybdenum, nickel, tin, zinc
Ion exchange system influent	Sample point 5 (sample port)	One/run. Grab sample collected after 15 min (+/- 5 min) of initiation of run.	TSS, TDS, TOC, cadmium, hex chromium, chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, zinc, TDS, O&G, sulfide, pH, specific conductance
Final treated wastewater	Sample point 6 (sample port)	One/run. Grab sample collected after 20 min (+/- 5 min) of initiation of run.	TSS, TDS, TOC, cadmium, hex chromium, chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, zinc, TDS, O&G, sulfide, pH, specific conductance
Ion exchange system regenerant	Sample point 7 (regenerant storage tank)	One/verification test. Representative grab sample collected after completion of the ion exchange system regeneration.	cadmium, hex chromium, chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, zinc

Table 8. Sampling Locations, Frequency and Parameters for Test 1

3.3.4.2 Polymer Usage Data

During Test 1, the quantity of polymer used by the electrocoagulation system will be measured and recorded after completion of the verification test. This will be accomplished by subtracting the quantity of polymer in the feed tank at the start and completion of the verification tests (four batches). Measuring the height of the polymer in the cylindrical feed tank and calculating the volume using the formula for a right circular cylinder will determine the quantity of polymer. The depth of polymer at the start and completion of the verification test will be entered into the forms in **Appendix A**.

Sample	Sample Location	Frequency/Type	Parameters
Ion exchange system influent	Sample point 5 (sample port)	One per day for the first three days plus one on the last day of test.	TSS, TDS, TOC, cadmium, hex chromium, chromium (T), copper, lead, manganese, iron, molybdenum, nickel, tin, zinc, TDS, O&G, sulfide, pH, specific conductance
Final treated wastewater	Sample point 6 (sample port)	One per day for the first three days plus one on the last day of test.	TSS, TDS, TOC, cadmium, hex chromium, chromium (T), copper, lead, manganese, iron, molybdenum, nickel, tin, zinc, TDS, O&G, sulfide, pH, specific conductance
Ion exchange system regenerant	Sample point 7 (regenerant storage tank)	Representative grab sample collected after completion of the ion exchange system regeneration. One/verification test.	cadmium, chromium (+6), chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, zinc

Table 9. Sampling Locations, Frequency and Parameters for Test 2

3.3.4.3 Volume of Ion Exchange Regenerant

The Lobo Liquids system will be regenerated prior to and following both verification tests. Volume measurement data from the regeneration process prior to Test 1 and following Test 2 will be used in order to determine the volume of regenerant. During regeneration, the columns are flushed with hydrochloric acid (cation column), sodium hydroxide (anion columns), and water. These solutions are combined into the regenerant storage tank. The storage tank will be emptied prior to regeneration. The depth of regenerant at the completion of the ion exchange cycle will be entered into the forms in **Appendix C**.

3.3.4.4 Ion Exchange System Regeneration Chemical Use

During Tests 1 and 2 the quantity of hydrochloric acid and sodium hydroxide used by the Lobo Liquids system for regeneration will be determined after completion of the system regeneration described in section 3.3.4.3. This will be accomplished by subtracting the quantities of hydrochloric acid and sodium hydroxide in their respective feed tanks at the start and completion of the ion exchange cycle. Measuring the height of the hydrochloric acid and sodium hydroxide in the cylindrical feed tanks and the circumferences of the tanks and calculating the volume using the formula for a right circular cylinder will determine the quantity of hydrochloric acid and sodium hydroxide. The depths of hydrochloric acid and sodium hydroxide at the start and completion of the ion exchange cycle will be entered into the forms in **Appendix A**.

3.3.4.5 pH

The pH provides a general indication of the acidity or alkalinity of a wastewater. It is also a regulated parameter for most dischargers. The pH of the influent and effluent samples will be determined by using a digital meter (electrometric). The digital meter will be calibrated using two buffers that bracket the expected measurement range. The calibration will be verified by measuring the pH of a buffer solution with a pH near the center of the calibration range. The ETV-MF Project Manager will record the manufacturer, lot number, and the expiration date of the buffer in the field notebook.

Sample Location/Parameters	Bottle Type	Batch 1	Batch 2	Batch 3	Batch 4	Total Sample
Raw Wastewater (Sample point 1)						
TOC	1000 mL plastic bottle	1	1	1	1	4
Cr^{+6}, TSS, TDS, pH, specific conductance	500 mL plastic bottle	1	1	1	1	4
Metals*	500 mL plastic bottle	1	1	1	1	4
O&G (HEM)	1,000 mL wide mouth glass jar (2 each)	1	1	1	1	4
Sulfide	250 mL plastic bottle	1	1	1	1	4
EC Reactor Discharge (Sample point 2)						
Cr^{+6}, pH	500 mL plastic bottle	3	6**	3	-	12
Metals*	500 mL plastic bottle	3	6**	3	-	12
Intermediate Treated Wastewater (Sample point 3)						
TOC	1000 mL plastic bottle	-	3**	1	1	5
Cr^{+6}, TSS, TDS, pH, specific conductance	500 mL plastic bottle	-	3**	1	1	3
Metals*	500 mL plastic bottle	-	3**	1	1	5
O&G (HEM)	1,000 mL wide mouth glass jar (2 each)	-	3**	1	1	5
Sulfide	250 mL plastic bottle	-	3**	1	1	5
Sludge (Sample point 4)						
Metals*, % solids, density	1,000 mL wide mouth glass jar				3**	3
Ion exchange Influent (Sample point 5)						
TOC	125 mL amber glass bottle (4 each)	-	1	1	1	4
Cr^{+6}, TSS, TDS, pH, specific conductance	500 mL plastic bottle	-	1	1	1	4
Metals*	500 mL plastic bottle	-	1	1	1	4
O&G (HEM)	1,000 mL wide mouth glass jar (2 each)	-	1	1	1	4
Sulfide	250 mL plastic bottle	-	1	1	1	4
Final Treated Wastewater (Sample point 6)						
TOC	1000 mL plastic bottle (4 each)	-	1	1	3**	5
Cr^{+6}, TSS, TDS, pH, specific conductance	500 mL plastic bottle	-	1	1	3**	5
Metals*	500 mL plastic bottle	-	1	1	3**	5
O&G (HEM)	1,000 mL wide mouth glass jar (2 each)	-	1	1	3**	5
Sulfide	250 mL plastic bottle	-	1	1	3**	5
Regenerant (Sample point 7)						
Cr^{+6}	500 mL plastic bottle	-	-	-	1	1
Metals*	500 mL plastic bottle	-	-	-	1	1

*Cadmium, chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, and zinc
**Includes duplicate and matrix spike

Table 10. Test 1 Sample Quantities from Each Sampling Point

Sample Location/Parameters	Bottle Type	Day 1	Day 2	Day 3	Last Day	Total Sample
Ion Exchange Influent (Sample point 5)						
TOC	1000 mL plastic bottle (4 each)	1	1	1	1	4
Cr^{+6}, TSS, TDS, pH, specific conductance	500 mL plastic bottle	1	1	1	1	4
Metals*	500 mL plastic bottle	1	1	1	1	4
O&G (HEM)	1,000 mL wide mouth glass jar (2 each)	1	1	1	1	4
Sulfide	250 mL plastic bottle	1	1	1	1	4
Final Treated Wastewater (Sample point 6)						
TOC	1000 mL plastic bottle (4 each)	1	1	3**	1	6
Cr^{+6}, TSS, TDS, pH, specific conductance	500 mL plastic bottle	1	1	3**	1	6
Metals*	500 mL plastic bottle	1	1	3**	1	6
O&G (HEM)	1,000 mL wide mouth glass jar (2 each)	1	1	3**	1	6
Sulfide	250 mL plastic bottle	1	1	3**	1	6
Regenerant (Sample point 7)						
Cr^{+6}	500 mL plastic bottle	-	-	-	1	1
Metals*	500 mL plastic bottle	-	-	-	1	1

*Cadmium, chromium (T), copper, iron, lead, manganese, molybdenum, nickel, tin, and zinc
**Includes duplicate and matrix spike

Table 11. Test 2 Sample Quantities from Each Sampling Point

3.3.4.6 Quantity of Sludge Generated

The quantity of sludge generated will be measured at the end of the first verification test. This will be accomplished by transferring the sludge that is discharged from the filter press into an empty drum, measuring the height of the sludge and the circumference of the drum, and calculating the sludge volume using the formula for a right circular cylinder. The height of sludge in the drum will be recorded on the forms found in **Appendix A**. The analytical laboratory will determine the density of the sludge, and the mass of sludge will be calculated by multiplying the sludge volume by its density.

3.3.4.7 Electricity Usage Data

Electricity usage will be calculated by determining the input power requirements of pumps, the rectifier, and other powdered devices associated with the electrocoagulation system and Lobo Liquids system.

3.3.4.8 System Operation and Maintenance Data

System operation and maintenance activities will be observed during each run. Any non-routine operational or maintenance procedures performed will be documented. This includes changes to the flow rate or chemical feed rate, filter replacement, and similar activities. Labor requirements (hrs.) will also be recorded. The team leader will record notes pertaining to these activities on the data forms in **Appendix A**.

3.3.4.9 Cost Data

Gull Industries will provide the cost data for steel plates, electricity, labor, chemical reagents, and sludge disposal. Gull Industries will also provide one month of historical data for chemical reagent use and volumes of wastewater treated.

3.3.4.10 Steel Plate Consumption

Steel plate consumption is relatively slow and cannot be accurately measured during the short duration of the verification test. Therefore, six months of historical data will be collected from Gull Industries regarding steel plate consumption.

3.3.4.11 Ion Exchange System Operational Data

Data from the internal logging system of the ion exchange unit will be used to provide operational data (non-critical). The system has the following parameters monitored and continuously logged to disk:

- Inlet specific conductance (µS)
- pH
- Pump discharge pressure (psi)
- Flow (gpm)
- Outlet specific conductance (µS)

Prior to testing, the measurement devices will be calibrated according to each manufacturer's procedures. All events are date and time stamped. Gull Industries will provide these data to the ETV Project Manager at the completion of the test in a spreadsheet format.

3.4 Analytical Procedures

All analytical procedures that will be used during this verification test are EPA methods or other recognized methods. A summary of analytical tests is presented in **Table 12**.

Parameter	Test Method	Preservation/Handling	Hold Time
Metals (dissolved)	EPA 200.7	Cool storage (<4°C) pH <2 w/HNO_3	6 months
Metals (sludge)	SW-846 3050B/6010B	cool storage (<4°C)	6 months
Chromium (hexavalent)	SW-846 7196A	cool storage (<4°C)	24 hrs
O&G (as HEM)	EPA Method 1664	cool storage (<4°C) pH<2 w/HNO_3	28 days
pH	digital meter	NA	analyze immediately
sulfide (S)	EPA Method 376.2	cool storage (<4°C) zinc acetate + NaOH to pH >12	7 days
TDS	EPA Method 160.1	cool storage (<4°C)	7 days
TOC	EPA Method 415.1	cool storage (<4°C) acidify to pH <2 w/HNO3	28 days
TSS	EPA Method 160.2	cool storage (<4°C)	7 days
specific conductance	EPA Method 120.1	cool storage (<4°C)	28 days
sludge % water	SW-846 Draft Update IVA 9000	cool storage (<4°C)	28 days
sludge specific gravity	SM2710F	cool storage (<4°C)	28 days

Table 12. Summary of Analytical Tests and Requirements

4.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

Quality Assurance/Quality Control (QA/QC) activities will be performed according to the applicable section of the Environmental Technology Verification Program Metal Finishing Technologies Quality Management Plan (ETV-MF QMP) [Ref. 6].

4.1 Quality Assurance Objectives

The first QA objective is to ensure that the process operating conditions and test methods are maintained and documented throughout each test and laboratory analysis of samples. The second QA objective is to use standard test methods (where possible) for laboratory analyses. The test methods to be used are listed in **Table 12**.

4.2 Data Reduction, Validation, and Reporting

4.2.1 Internal Quality Control Checks

Raw Data Handling. Raw data are generated and collected by laboratory analysts at the bench and/or sampling site. These include original observations, printouts, and readouts from equipment for sample, standard, and reference QC analyses.

Data are collected both manually and electronically. At a minimum, the date, time, sample ID, raw signal or processed signal, and/or qualitative observations will be recorded. Comments to document unusual or non-standard observations also will be included on the forms, as necessary. The forms presented in **Appendix A** will be used for recording data on-site.

The on-site Project Team member will generate COC forms, and these forms will accompany samples when they are shipped off-site.

Raw data will be processed manually by the analyst, automatically by an electronic program, or electronically after being entered into a computer. The analyst will be responsible for scrutinizing the data according to laboratory precision, accuracy, and completeness policies. Raw data bench sheets and calculation or data summary sheets will be kept together for each sample batch. From the standard operating procedure and the raw data bench files, the steps leading to a final result may be traced. The ETV-MF Project Manager will maintain process-operating data for use in verification report preparation.

<u>Data Package Validation.</u> The generating analyst will assemble a preliminary data package, which shall be initialed and dated. This package shall contain all QC and raw data results, calculations, electronic printouts, conclusions, and laboratory sample tracking information.

A second analyst will review the entire package and check sample and storage logs, standard logs, calibration logs, and other files, as necessary, to ensure that all tracking, sample treatments, and calculations are correct. After the package is reviewed in this manner, a preliminary data report will be prepared, initialed, and dated. The entire package and final report will be submitted to the Laboratory Manager (LM).

The LM shall be ultimately responsible for all final data released from the laboratory. The LM or designee will review the final results for adequacy to task QA objectives. If the LM or designee suspects an anomaly or non-concurrence with expected or historical performance values, or with task objectives for test specimen performance, the raw data will be reviewed, and the generating and reviewing analysts queried. If suspicion about data validity still exists after internal review of laboratory records, the LM will authorize a re-test. If sufficient sample is not available for re-testing, a re-sampling shall occur. If the sampling window has passed, or re-sampling is not possible, the LM will flag the data as suspect. The LM signs and dates the final data package.

<u>Data Reporting.</u> A report signed and dated by the LM will be submitted to the ETV-MF Project Manager. The ETV-MF Project Manager will decide the appropriateness of the data for the particular application. The final report contains the laboratory sample ID, date reported, date analyzed, the analyst, the standard operating procedure (SOP) used for each parameter, the process or

sampling point identification, the final result, units, and all QC data generated. The ETV-MF Project Manager shall retain the data packages as required by the ETV-MF QMP [Ref. 6].

4.2.2 Calculation of Data Quality Indicators

Analytical performance requirements are expressed in terms of precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS). Summarized below are definitions and QA objectives for each PARCCS parameter.

One influent sample from each run shall be submitted with a field duplicate. All analytes from this sample will have matrix spike and matrix spike duplicate analyses performed.

4.2.2.1 Precision

Precision is a measure of the agreement or repeatability of a set of replicate results obtained from duplicate analyses made under identical conditions. Precision is estimated from analytical data and cannot be measured directly. The precision of a duplicate determination can be expressed as the relative percent difference (RPD), and calculated as:

$$RPD = \left\{ \frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}} \right\} x100\,\%$$

where:

X_1 = larger of the two observed values
X_2 = smaller of the two observed values

Multiple determinations will be performed for each test on the same test specimen. The replicate analyses must agree within the relative percent deviation limits provided in **Table 13.**

4.2.2.2 Accuracy

Accuracy is a measure of the agreement between an experimental determination and the true value of the parameter being measured. Accuracy is estimated through the use of known reference materials or matrix spikes. It is calculated from analytical data and is not measured directly. Spiking of reference materials into a sample matrix is the preferred technique because it provides a measure of the matrix effects on analytical accuracy. Accuracy, defined as percent recovery (P), is calculated as:

$$P = \left[\frac{(SSR - SR)}{SA}\right] x\ 100\%$$

where:

SSR	=	spiked sample result
SR	=	Sample result (native)
SA	=	the concentration added to the spiked sample

Analyses will be performed with periodic calibration checks with traceable standards to verify instrumental accuracy. These checks will be performed according to established procedures in the contracted laboratory(s) that have been acquired for this verification test. Analysis with spiked samples will be performed to determine percent recoveries as a means of checking method accuracy. QA objectives will be satisfied if the *average* recovery is within the goals described in **Table 13**.

4.2.2.3 Completeness

Completeness is defined as the percentage of measurements judged to be valid compared to the total number of measurements made for a specific sample matrix and analysis. Completeness is calculated using the following formula:

$$\text{Completeness} = \frac{\text{Valid Measurements}}{\text{Total Measurements}} \times 100\%$$

Experience on similar projects has shown that laboratories typically achieve about 90 percent completeness. QA objectives will be satisfied if the percent completeness is 90 percent or greater as specified in **Table 13**.

Critical Measurements	Matrix	EPA Test Method	Reporting Units	Method of Determination	MDL	Precision (RPD)	Accuracy (% Recovery)	Completeness
cadmium	Water/ sludge	200.7 SW3050B SW6010B	mg/L	ICP-AES	.005	<30	80 – 120	90
chromium (T)	water/ sludge	200.7 SW3050B SW6010B	mg/L	ICP-AES	.010	<30	80 – 120	90
copper	water/ sludge	200.7 SW3050B SW6010B	mg/L	ICP-AES	.010	<30	80 – 120	90
iron	water/ sludge	200.7 SW3050B SW6010B	mg/L	ICP-AES	.400	<30	80 – 120	90
lead	water/ sludge	200.7 SW3050B SW6010B	mg/L	ICP-AES	.010	<30	80 – 120	90
manganese	water/ sludge	200.7 SW3050B SW6010B	mg/L	ICP-AES	.030	<30	80 – 120	90
molybdenum	water/ sludge	200.7 SW3050B SW6010B	mg/L	ICP-AES	.020	<30	80 – 120	90
nickel	water/ sludge	200.7 SW3050B SW6010B	mg/L	ICP-AES	.020	<30	50 – 150	90
O&G (as HEM)	water	1664	mg/L	gravimetric	5.0	<30	50 – 150	90
pH	water	150.1	std. units	electrometric	0.1	<30	+/- .2 pH units	90
sulfide (S)	water	376.2	mg/L	colormetric	5.0	<30	80 – 120	90
total solids	sludge	160.3		gravimetric	1.0	<30	-	90
TDS	water	160.1	mg/L	gravimetric	10	<30	-	90
TOC	water	415.1	mg/L	combustion/ oxidation	1.0	<30	-	90
tin	water/ sludge	200.7 SW3050B SW6010B	mg/L	ICP-AES	.020	<30	80 – 120	90
TSS	water	160.2	mg/L	gravimetric	1.0	<30	-	90
zinc	water/ sludge	200.7 SW3050B SW6010B	mg/L	ICP-AES	.030	<30	80 – 120	90
chromium (hexavalent)	water/ sludge	SW 846 3050B/7196A	mg/L	colorimetric	.015	<30	80 – 120	90
silver	water/ sludge	200.7 SW3050B SW6010B	mg/L	ICP-AES	.010	<30	80 – 120	90
specific conduct.	water	120.1	µS	Wheatstone bridge	.01	<30	-	90

Table 13. QA Objectives

4.2.2.4 Comparability

Comparability is another qualitative measure designed to express the confidence with which one data set may be compared to another. Sample collection and handling techniques, sample matrix type, and analytical method all affect comparability. Comparability is limited by the other PARCCS parameters because data sets can be compared with confidence only when precision and accuracy are known. Comparability will be achieved in this technology verification by the use of consistent methods during sampling and analysis and by traceability of standards to a reliable source.

4.2.2.5 Representativeness

Representativeness refers to the degree to which the sample represents the properties of the particular wastestream being sampled. For the purposes of this demonstration, representativeness will be determined by submitting identical samples (field duplicates) to the laboratory for analysis. The samples will be representative if the relative percent difference between the sample and the field duplicate is similar to or less than the precision (laboratory duplicates) calculation of the sample. Three identical samples (one each of influent, effluent, and sludge) will be collected during the course of the verification test. Representativeness will be satisfied if the analytical results for each parameter are within 25 percent of the results for the associated duplicate sample.

4.2.2.6 Sensitivity

Sensitivity is the measure of the concentration at which an analytical method can positively identify and report analytical results. The sensitivity of a given method is commonly referred to as the detection limit. Although there is no single definition of this term, the following terms and definitions of detection will be used for this program.

Instrument Detection Limit (IDL) is the minimum concentration that can be measured from instrument background noise.

Method Detection Limit (MDL) is a statistically determined concentration. It is the minimum concentration of an analyte that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero as determined in the same or a similar matrix. (Because of the lack of information on analytical precision at this level, sample results greater than the MDL but less than the practical quantification limit (PQL) will be laboratory qualified as "estimated.")

MDL is defined as follows for all measurements:

$$MDL = t_{(n-1, 1-\alpha = 0.99)} \times s$$

where: MDL = method detection limit

$t_{(n-1,1-\alpha = 0.99)}$ = students t-value for a one-sided 99 percent confidence level and a standard deviation estimate with n-1 degrees of freedom

s = standard deviation of the replicate analyses

Method Reporting Limit (MRL) is the concentration of the target analyte that the laboratory has demonstrated the ability to measure within specified limits of precision and accuracy during routine laboratory operating conditions. (This value is variable and highly matrix dependent. It is the minimum concentration that will be reported without qualifications by the laboratory.)

4.3 Additional Data Calculations

4.3.1 Ability to Meet Metal Finishing and Proposed MP&M Limitations

The results of each test cycle will be compared to the applicable metal finishing limitations (**Table 14**) and Proposed MP&M limitations (**Table 15**). To meet a metal finishing or MP&M limit, the analytical result must be equal to or below the corresponding daily maximum limit.[4] The comparison will be made on a parameter-by-parameter basis for each cycle. The applicable limitations are the pretreatment standards for existing sources for the metal finishing category (40 CFR 433.15) and proposed pretreatment standards for existing sources for the MP&M Job Shop subcategory (66 FR 424).

Parameter	Metal Finishing Category (40 CFR 433.15)	
	Daily Max., mg/L	Monthly Avg., mg/L
Cadmium	0.69	0.26
Chromium	2.77	1.71
Copper	3.38	2.07
Lead	0.69	0.43
Nickel	3.98	2.36
Zinc	2.61	1.48

Table 14. Applicable Pretreatment Standards for Existing Sources for the Metal Finishing Category (40 CFR 433.15)

[4] It is anticipated that for certain parameters the influent concentration will be below the discharge limit. These instances will be identified during data reduction and reported as such in the verification report.

Parameter	MP&M Job Shop Subcategory (66 FR 424)	
	Daily Max., mg/L	Monthly Avg., mg/L
Cadmium	0.21	0.09
Chromium	1.3	0.55
Copper	1.3	0.57
Lead	0.12	0.09
Manganese	0.25	0.10
Molybdenum	0.79	0.49
Nickel	1.5	0.64
Tin	1.8	1.4
Zinc	0.35	0.17
O&G (as HEM)	52	26
TOC	78	59
Sulfide (as S)	31	13

O&G (as HEM) are not regulated under pretreatment standards for the Job Shop subcategory. However, it is regulated under the BPT limitations for direct dischargers in the Job Shop subcategory (66 FR 424). The values shown are the BPT proposed limitations.

Table 15. Applicable Proposed Pretreatment Standards for Existing Sources for the MP&M Job Shop Subcategory (66 FR 424)

4.3.2 Mass Balance

Mass balance calculations will be performed for chromium (T) and nickel, which are the two metal parameters of greatest significance at Gull Industries. During Test 1, the mass balance will be performed for the electrocoagulation system only since the ion exchange system will not be tested over a full cycle. The mass balance results will be used as an indicator of the accuracy of the verification test. The mass balance criterion will be satisfied when the mass balance is within the range of 75 percent to 125 percent.

The equation for the chromium mass balance for Test 1 is shown below. The nickel mass balance equation will be similar.

$$\text{mass bal. (\%)} = [((C_E \times V_E) + (C_S \times V_S)) / (C_I \times V_I)] \times 100\%$$

where:

- C_E = intermediate treated wastewater chromium concentration (mg/L)
- V_E = intermediate treated wastewater volume processed during the test period (L)
- C_S = filter press sludge chromium concentration (mg/L)
- V_S = filter press sludge volume generated during the test period (L)
- C_I = raw wastewater chromium concentration (mg/L)
- V_I = raw wastewater volume processed during the test period (L)

The equation for the chromium mass balance for Test 2 is shown below. The nickel mass balance equation will be similar.

$$\text{mass bal. (\%)} = [((C_F \times V_F) + (C_R \times V_R)) / (C_I \times V_I)] \times 100\%$$

where:

C_F	=	final treated wastewater chromium concentration (mg/L)
V_F	=	final treated wastewater volume processed during the test period (L)
C_R	=	regenerant chromium concentration (mg/L)
V_R	=	regenerant volume generated at end of the Test 2 (L)
C_I	=	raw wastewater chromium concentration (mg/L)
V_I	=	raw wastewater volume processed during the test period (L)

4.3.3 Pollutant Removal Efficiency

The pollutant removal efficiency is calculated based on a comparison of raw wastewater and treated wastewater concentrations for each pollutant parameter and each run. The equation for zinc removal is shown below. The removal efficiency rate for each pollutant parameter will be separately calculated. These include: O&G (as HEM), TOC, cadmium, hexavalent chromium, chromium (T), copper, lead, manganese, molybdenum, nickel, sulfide (as S), tin, and zinc.

$$Z_{remove}\text{(\%)} = [((Z_I \times V_I) - (Z_E \times V_E)) / (Z_I \times V_I)] \times 100\%$$

where:

.Z_{remove}	=	zinc recovery efficiency
Z_I	=	raw wastewater zinc concentration (mg/L)
V_I	=	raw wastewater volume processed during the test cycle (L)
Z_E	=	treated wastewater zinc concentration (mg/L)
V_E	=	treated wastewater volume processed during the test cycle (L)

As a result of its design, the electrocoagulation system retains a volume of wastewater in the treatment system tanks approximately equal to the volume of one batch. The tanks operate as a plug flow reactor; as a batch of wastewater is processed through the tanks, the existing wastewater in the tanks is pushed through the system and is discharged to the final storage tank. To account for this design, during Test 1, the treated wastewater samples (E) will be paired with the raw wastewater samples (R) from the previous batch as shown in **Table 7**.

4.3.4 Reusability of Treated Wastewater

The reusability of the treated wastewater as process water will be determined by comparing the results of the pH and specific conductance analytical tests of the final treated water to standards used by Gull Industries for water reuse. Treated water meeting both of these standards will be deemed reusable. The Gull Industries standards are:

- Specific conductance: maximum of 500 µS
- pH: within the range of 5.0 to 9.0 standard units

4.3.5 Energy Usage

Separate energy usage analyses will be performed for Tests 1 and 2. Energy requirements for the electrocoagulation and Lobo Liquids systems will be calculated by summing the total quantity of horsepower (hp) hours for each system and dividing by 1.341 hp-hr/kWh to arrive at electricity needs. The energy requirements will be calculated separately for each system.

4.3.6 Cost Analysis

Separate cost analyses will be performed for Tests 1 and 2. These analyses will determine the operating costs of the electrocoagulation and Lobo Liquids systems considering the following cost parameters: chemical reagents, steel plates, other materials (e.g., filters), electricity, labor, and sludge management. Costs will be calculated and expressed in dollars per thousand liters processed ($/1000 L) by dividing the cost by the total volume of wastewater processed during the verification test. Total costs will be calculated separately for each system by summing the individual cost elements. The calculation of treatment cost is shown below.

$$C_{treat\ cost} = (R + A + M + E + L + S) / V$$

where:

$.C_{treat\ cost}$ = cost of treatment ($/1000 L)
R = cost of chemical reagents used ($)
A = cost of steel plates consumed ($)*
M = cost of materials used ($)
E = cost of electricity used ($)
L = cost of labor ($)
S = cost of sludge management ($)*
V = volume of wastewater processed during the verification test (1000 L)
*Test 1 only

4.3.7 Sludge Generation Analysis

During Test 1, the quantity of sludge generated will be measured at the end of the verification test as described in section 3.3.3.1. The quantity will be expressed as volume of sludge generated (wet basis) and weight of sludge generated (wet and dry basis). The weight of the sludge on a dry-weight basis will be calculated as follows:

$$S_{dry} = (S_{wet} \text{ x } \% \text{ solids}) / 100\%$$

where:
- S_{dry} = dry weight of sludge
- S_{wet} = wet weight of sludge as measured during verification test
- % solids = percent solids from lab analysis of sludge

4.3.8 Environmental Benefit

Separate environmental benefit analyses will be performed for Tests 1 and 2. This analysis will quantify the environmental benefit of the electrocoagulation/ion exchange polishing technologies installed at Gull Industries by determining the quantity of regulated pollutants removed beyond the level required by the metal finishing regulations (40 CFR 433).

$$P_B = P_V - P_H$$

where:
- P_B = quantity of regulated pollutants removed beyond the level required (gram (g)/1000 L)
- P_V = sum of allowable pollutant discharged (g/1000 L) (calculated by multiplying the daily maximum limit times the volume of wastewater processed and summing over all regulated parameters)
- P_H = sum of actual pollutant mass discharged during verification test (g/1000 L) (calculated by multiplying the average final concentration for the three runs times the volume of wastewater processed and summing over all regulated parameters)

4.4 Test Plan Modifications

In the course of verification testing, it may become necessary to modify the test plan due to unforeseen events. These modifications will be documented using a Test Plan Modification Request (**Appendix B**), which must be submitted to the *CTC* Project Manager for approval. Upon approval, the modification request will be assigned a number, logged, and transmitted to the requestor for implementation.

4.5 Quality Audits

Technical System Audits. An audit will not be performed during verification testing by the *CTC* QA Manager. The EPA QA Manager may also conduct an audit to assess the quality of the verification test.

Internal Audits. In addition to the internal laboratory QC checks, internal quality audits will be conducted to ensure compliance with written procedures and standard protocols.

Corrective Action. Corrective action for any deviations to established QA and QC procedures during verification testing will be performed according to section 2.10 Quality Improvement of the ETV-MF QMP [Ref. 6].

Laboratory Corrective Action. Examples of non-conformances include invalid calibration data, inadvertent failure to perform method-specific QA, process control data outside specified control limits, failed precision and/or accuracy indicators, etc. Such non-conformances will be documented on a standard laboratory form. Corrective action will involve taking all necessary steps to restore a measuring system to proper working order and summarizing the corrective action and results of subsequent system verifications on a standard laboratory form. Some non-conformances are detected while analysis or sample processing is in progress and can be rectified in real time at the bench level. Others may be detected only after a processing trial and/or sample analyses are completed. Typically, the LM detects these types of non-conformances. In all cases of non-conformance, the LM will consider sample re-analysis as one source of corrective action. If insufficient sample is available or the holding time has been exceeded, complete re-processing may be ordered to generate new samples if a determination is made by the ETV-MF Project Manager that the non-conformance jeopardizes the integrity of the conclusions to be drawn from the data. In all cases, a non-conformance will be rectified before sample processing and analysis continues.

5.0 PROJECT MANAGEMENT

5.1 Organization/Personnel Responsibilities

The ETV-MF Project Team that is headed by *CTC* will conduct the evaluation of the electrocoagulation and ion exchange polishing systems. The *CTC* ETV-MF Program Manager, Donn Brown, will have ultimate responsibility for all aspects of the technology evaluation. The ETV-MF Project Manager assigned to this evaluation is George Cushnie. Mr. Cushnie and/or his staff member will be on-site throughout the series of treatment runs and will conduct or supervise all sampling and related measurements, with one exception.[5] During Tests 1 and 2, the operating cycle of the Lobo Liquids system will extend beyond the time period the ETV-MF team will be on-site. During Test 1, at the end of the operating cycle, a Gull Industries employee will collect a sample of the ion exchange regenerate from the regenerant storage tank (sample point 7) and ship the

[5] The *CTC* ETV-MF Program Manager, Donn Brown, will make a determination as to the qualifications of any staff member assigned to the project. This will occur prior to testing.

sample to the laboratory. During Test 2, a Gull Industries employee will collect influent, effluent, and regenerant samples from the ion exchange system. The ETV-MF Project Manager will train the Gull Industries employee with regard to sampling protocol, sample preservation, and COC.

Ian Tunnicliffe will head the ion exchange staff. He will be on call throughout the entire test period to answer questions concerning operation of the system.

Gull Industries personnel will be responsible for operating the electrocoagulation and ion exchange polishing systems and providing historical wastewater volume and cost information. Gull Industries will also be responsible for the disposal of all residuals generated during the verification test.

Severn Trent Laboratories (STL) in Houston, Texas, is responsible for analyzing verification test samples. The LM, Jodi Romine, will be point of contact (POC). STL is approved by the State of Texas for the analyses identified in this test plan.

The ETV-MF Project Manager and Gull Industries (host facility) have the authority to stop work when unsafe or unacceptable quality conditions arise. The *CTC* ETV-MF Program Manager will provide periodic assessments of verification testing to the EPA ETV Program Manager.

6.0 EQUIPMENT AND UTILITY REQUIREMENTS

The electrocoagulation system and Lobo Liquids system are permanently installed at Gull Industries. The only utility requirements for operating the systems are electricity, compressed air, and city water.

7.0 HEALTH AND SAFETY PLAN

This Health and Safety Plan provides guidelines for recognizing, evaluating, and controlling health and physical hazards throughout the workplace. More specifically, the Plan specifies for assigned personnel the training, materials, and equipment necessary to protect themselves from hazards created by acids and any waste generated by the process.

7.1 Hazard Communication

All personnel assigned to the project will be provided with the potential hazards, signs and symptoms of exposure, methods or materials to prevent exposures, and procedures to follow if there is contact with a particular substance. The Gull Industries (host facility) Hazard Communication Program will be reviewed during training and will be reinforced throughout the test period. All appropriate Material Safety Data Sheet(s) (MSDS) forms will be available for chemical solutions used during testing.

7.2 Emergency Response Plan

Gull Industries has a contingency plan to protect employees, assigned project personnel, and visitors in the event of an emergency at the facility. This plan will be used throughout the project. All assigned personnel will be provided with information about the plan during training.

7.3 Hazard Controls Including Personal Protective Equipment

All assigned project personnel will be provided with appropriate personal protective equipment (PPE) and any training needed for its proper use, considering their assigned tasks. The use of PPE will be covered during training as indicated in section 9.0.

The following PPE will be required and must be worn at all times while in the Gull Industries facility: eyeglasses with side splashguards.

7.4 Lockout/Tagout Program

The electrocoagulation and Lobo Liquids systems are fully installed. There is no need for implementation of a lockout/tagout program.

7.5 Material Storage

Any materials used during the project will be kept in proper containers and labeled according to state and Federal laws. Proper storage of the materials will be maintained based on associated hazards. Spill trays or similar devices will be used as needed to prevent material loss to the surrounding area.

7.6 Safe Handling Procedures

All chemicals and wastes or samples will be transported on-site in non-breakable containers used to prevent spills. Spill kits will be strategically located in the project area. These kits contain various sizes and types of sorbents for emergency spill clean up. Emergency spill clean up will be performed according to the Emergency Response Plan.

8.0 WASTE MANAGEMENT

The electrocoagulation and Lobo Liquids systems will process wastewater generated by manufacturing operations at Gull Industries. After processing, the effluent from treatment will be transferred to the existing Gull Industries storage tanks and subsequently reused as process water and/or discharged to the Houston POTW. Any residuals generated by the electrocoagulation or Lobo Liquids systems will be managed by Gull Industries in accordance with Gull Industries requirements.

9.0 TRAINING

It is important that the verification activities performed by the ETV-MF Program be conducted with high quality and with regard to the health and safety of the workers and the environment. By identifying the quality requirements, worker safety and health, and environmental issues associated with each verification test, the qualifications or training required for personnel involved can be identified. Training requirements will be identified using the Job Training Analysis (JTA) Plan [Ref. 8].

The purpose of this JTA Plan is to outline the overall procedures for identifying the hazards and quality issues and training needs for each verification test project. This JTA Plan establishes guidelines for creating a work atmosphere that meets the quality, environmental, and safety objectives of the ETV-MF Center. The JTA Plan describes the method for studying ETV-MF project activity and identifying training needs. The ETV-MF Operation Planning Checklist (**Appendix C**) will be used as a guideline for identifying potential hazards, and the JTA Form (**Appendix D**) will be used to identify training requirements. After completion of the form, applicable training will be performed. Training will be documented on the ETV-MF Project Training Attendance Form (**Appendix E**). Health and safety training will be coordinated with Gull Industries personnel.

10.0 REFERENCES

1) Data collected by the Metal Finishing Strategic Goals Program (SGP) (see www.strategicgoals.org).
2) EPA, Effluent Limitations Guidelines, Pretreatment Standards, and New Source Performance Standards for the Metal Finishing Point Source Category (40 CFR 433).
3) EPA, Effluent Limitations Guidelines, Pretreatment Standards, and New Source Performance Standards for the Metal Products and Machinery Point Source Category; Proposed Rule (66 FR 424, January 3, 2001).
4) U.S. EPA Office of Environmental Information, "*Guidance for the Data Quality Objectives Process, EPA QA/G-4,"* EPA/600/R-96/055, August 2000.
5) Cushnie, George C., *Pollution Prevention and Control for Plating Operations*, National Center for Manufacturing Sciences, Ann Arbor, MI, 1994.
6) Concurrent Technologies Corporation, "*Environmental Technology Verification Program Metal Finishing Technologies (ETV-MF) Quality Management Plan, Revision 1,*" March 26, 2001.
7) U.S. EPA Office of Research and Development, "*Preparation Aids for the Development of Category IV Quality Assurance Project Plans*," EPA/600/8-91/006, February 1991.
8) Concurrent Technologies Corporation, "*Environmental Technology Verification Program Metal Finishing Technologies (ETV-MF) Pollution Prevention Technologies Pilot Job Training Analysis Plan*," May 10, 1999.

11.0 DISTRIBUTION

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Jerry Givens, Gull Industries

Ian Tunnicliffe, Lobo Liquids

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APPENDIX A

Data Collection Forms for Electrocoagulation and Lobo Liquids Systems

Data Collection Form for Electrocoagulation and Lobo Liquids Systems
General Test Data

Date: ______________________
ETV-MF Project Manager: ______________________
Gull Industries Operator: ______________________

Parameter	Reading	Observations/Comments
Polymer Tank Dimensions		
IX Acid Tank Dimensions		
IX Caustic Tank Dimensions		
Sludge Generation Volume:		
Electricity Cost:		
Labor Cost:		
Acid Cost:		
Caustic Cost:		
Steel Plate Cost:		
Steel Plate Usage:		

Notes:

Data Collection Form for Electrocoagulation System
Cycle-Specific Data

Date: ____________________
ETV-MF Project Manager: ____________________
Gull Industries Operator: ____________________

Parameter/Time	**Reading or Sample #**	**Observations/Comments**
Batch ___/Pass___		
Electrocoagulation Start Time:		
Amp-hour reading A at start:		
Amp-hour reading B at start:		
Electrocoagulation Stop Time:		
Amp-hour reading A at stop:		
Amp-hour reading B at stop:		
Volume Treated (L):		
Polymer Height:		
Sample Point 1:		
Sample Point 2:		
Sample Point 3:		
Sample Point 4:		
Batch ___/Pass___		
Electrocoagulation Start Time:		
Amp-hour reading A at start:		
Amp-hour reading B at start:		
Electrocoagulation Stop Time:		
Amp-hour reading A at stop:		
Amp-hour reading B at stop:		
Volume Treated (L):		
Polymer Height:		
Sample Point 1:		
Sample Point 2:		
Sample Point 3:		
Sample Point 4:		

Notes:

Data Collection Form for Lobo Liquids System
Cycle-Specific Data

Date: ______________________
ETV-MF Project Manager: ______________________
Gull Industries Operator: ______________________

Regeneration

Parameter/Time	**Reading or Sample #**	**Observations/Comments**
Acid Tank Height at Start:		
Caustic Tank Height at Start:		
Ion Exchange Regen. Start Time:		
Ion Exchange Regen. Stop Time:		
Acid Tank Height at Completion:		
Caustic Tank Height at Completion:		
Sample Point 7:		
Volume of Regenerant:		

System Operation

Parameter/Time	**Reading or Sample #**	**Observations/Comments**
Batch ___		
Ion Exchange Start Time:		
Ion Exchange Stop Time:		
Sample Point 5:		
Sample Point 6:		

Notes:

APPENDIX B

Test Plan Modification Request

TEST PLAN MODIFICATION REQUEST

In the course of verification testing, it may become necessary to modify the test plan due to unforeseen events. The purpose of this procedure is to provide a vehicle whereby the necessary modifications are documented and approved.

The Test Plan Modification Request form is the document to be used for recording these changes. The following paragraphs provide guidance for filling out the form to ensure a complete record of the changes made to the original test plan.

The person requesting the change should record the date and project name in the form's heading. Program management will provide the request number.

Under Original Test Plan Requirement, reference the appropriate sections of the original test plan, and insert the proposed modifications in the section titled Proposed Modification. In the Reason section, document why the modification is necessary; this is where the change is justified. Under Impact, give the impact of not making the change, as well as the consequences of making the proposed modification. Among other things, the impact should address any changes to cost estimates and project schedules.

The requestor should then sign the form and obtain the signature of the project manager. The form should then be transmitted to the *CTC* ETV-MF Program Manager, who will either approve the modification or request clarification. Upon approval, the modification request will be assigned a number, logged, and transmitted to the requestor for implementation.

TEST PLAN MODIFICATION REQUEST

Date:__________ Number:__________ Project:________________________

Original Test Plan Requirement:____
____
____
____
____

Proposed Modification:____
____
____
____
____

Reason: ____
____
____
____
____

Impact:____
____
____
____
____

Approvals:

Requestor:___________________________

Project Manager:______________________

Program Manager:_____________________

APPENDIX C

ETV-MF Operation Planning Checklist

ETV-MF Operation Planning Checklist

The ETV-MF Project Manager prior to initiation of verification testing must complete this form. If a "yes" is checked for any items below, an action must be specified to resolve the concern on the Job Training Analysis Form.

Project Name: ______________________________ Expected Start Date:

ETV-MF Project Manager: ______________________________ ______________________

Will the operation or activity involve the following:	Yes	No	Initials & Date Completed
Equipment requiring specific, multiple steps for controlled shutdown? (e.g. in case of emergency, does equipment require more than simply pressing a "Stop" button to shut off power?) *Special Procedures for emergency shut-down must be documented in Test Plan.*			
Equipment requiring special fire prevention precautions? (e.g. Class D fire extinguishers)			
Modifications to or impairment of building fire alarms, smoke detectors, sprinklers or other fire protection or suppression systems?			
Equipment lockout/tagout or potential for dangerous energy release? *Lockout/tagout requirements must be documented in Test Plan.*			
Working in or near confined spaces (e.g., tanks, floor pits) or in cramped quarters?			
Personal protection from heat, cold, chemical splashes, abrasions, etc.? *Use Personal Protective Equipment Program specified in Test Plan.*			
Airborne dusts, mists, vapors and/or fumes? *Air monitoring, respiratory protection, and /or medical surveillance may be needed.*			
Noise levels greater than 80 decibels? *Noise surveys are required. Hearing protection and associated medical surveillance may be necessary.*			
X-rays or radiation sources? *Notification to the state and exposure monitoring may be necessary.*			
Welding, arc/torch cutting, or other operations that generate flames and/or sparks outside of designated weld areas? *Follow Hot Work Permit Procedures identified in Test Plan.*			
The use of hazardous chemicals? *Follow Hazard Communication Program, MSDS Review for Products Containing Hazardous Chemicals. Special training on handling hazardous chemicals and spill clean-up may be needed. Spill containment or local ventilation may be necessary.*			
Working at a height of six feet or greater?			

ETV-MF OPERATION PLANNING CHECKLIST

The ETV-MF Project Manager prior to initiation of verification testing must complete this form. If a "yes" is checked for any items below, an action must be specified to resolve the concern on the Job Training Analysis Form.

Project Name: ______________________________

ETV-MF Project Manager: ______________________________

Will the operation or activity involve the following:	**Yes**	**No**	**Initials & Date Completed**
Processing or recycling of hazardous wastes? *Special permitting may be required.*			
Generation or handling of waste?			
Work to be conducted before 7:00 a.m., after 6:00 p.m. and/or on weekends? *Two people must always be in the work area together.*			
Contractors working in *CTC* facilities? *Follow Hazard Communication Program.*			
Potential discharge of wastewater pollutants?			
EHS aspects/impacts and legal and other requirements identified?			
Contaminants exhausted either to the environment or into buildings? *Special permitting or air pollution control devices may be necessary.*			
Any other hazards not identified above? (e.g. lasers, robots, syringes) *Please indicate with an attached list.*			

The undersigned responsible party certifies that all applicable concerns have been indicated in the "yes" column, necessary procedures will be developed, and applicable personnel will receive required training. As each concern is addressed, the ETV-MF Project Manager will initial and date the "initials & date completed" column above.

ETV-MF Project Manager: ____________________ ____________________ ____________

(Name) (Signature) (Date)

APPENDIX D

Job Training Analysis Form

Job Training Analysis Form

ETV-MF Project Name: ____

Basic Job Step	Potential EHS Issues	Potential Quality Issues	Training

ETV-MF Project Manager:____

Name **Signature**

____

Date

APPENDIX E

ETV-MF Project Training Attendance Form

ETV-MF Project Training Attendance Form

ETV-MF Center Project: ____

Date Training Completed	Employee Name Last First	Training Topic	Test Score (If applic.)

ETV-MF Project Manager: ____