

US EPA ARCHIVE DOCUMENT



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

**Evaluation of USFilter Corporation's RETEC® Separated Cell
Purification of Chromic Acid Anodize Bath Solution**

Revision 1

September 13, 2000

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



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**Prepared by: ETV-MF Program and
USFilter Corporation**

**TITLE: EVALUATION OF USFILTER CORPORATION'S RETEC®
SEPARATED CELL PURIFICATION OF CHROMIC ACID
ANODIZE BATH SOLUTION**

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**Environmental Technology Verification for Metal Finishing Technologies Program
(ETV-MF) Test Plan for the Verification of USFilter Corporation's RETEC®
Separated Cell Purification of Chromic Acid Anodize Bath Solution.**

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US EPA ARCHIVE DOCUMENT

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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 at DV Industries (DVI), Inc., of Lynwood, California, during verification testing of a separated cell purification system for mineral acid bath maintenance manufactured by USFilter Corporation. USFilter's RETEC[®] Separated Cell Purification System is a pollution prevention technology applicable to any conventional metal finisher who uses mineral acids, including chromic acid, in one form or another. This test plan has been prepared to evaluate the performance of the technology in conjunction with the U.S. Environmental Protection Agency's (EPA's) Environmental Technology Verification for Metal Finishing (ETV-MF) Program. The objective of the ETV-MF program is to identify promising and innovative pollution prevention treatment technologies through EPA supported performance verifications and to provide objective performance data to providers, purchasers, and permittees of environmental technologies.

USFilter Corporation/Memtek Products, Billerica, Massachusetts, distributes the RETEC Separated Cell Purification (SCP) System, which purifies and reconditions spent chromic acid by circulating it through a specialized electrochemical cell. Ions of tramp metals in the process solution are converted to their respective hydroxides at the cathode; and trivalent chrome ions are converted back to useful hexavalent chrome ions. The metal hydroxides are separated from the treated process solution in a clarifier, and the process solution is then returned to the bath. The RETEC technology was developed by Memtek Products in 1992 and is now being distributed by USFilter in the US, Canada, Mexico, Argentina, and Brazil. Installed applications include chromic acid recovery and purification, and treatment of spent electroless nickel baths, chromate conversion coating, phosphoric acid recovery, and wastewater treatment.

DV Industries, Inc. (DVI), where the technology has been installed and operating for over four years, was selected by USFilter as the test site for this technology. DVI has one of the nation's largest anodizing departments. They serve a variety of customers, with the majority of their work dedicated to the aerospace, military, and commercial industries. They maintain three independent rack finishing lines and five separate paint booths, allowing for the acceptance of critical hardware across the full spectrum of size, quantity, tolerance requirements, and color. DVI finishes virtually almost all grades of aluminum.

This project will evaluate the ability of the USFilter RETEC separated cell purification system to extend the bath life of DVI's chromic acid anodizing operation. Evaluating and verifying the performance of USFilter's system will be accomplished by collecting operational data and in-process samples for analysis. The resultant test data will be used to prepare a material balance and determine the efficiency of chromic acid recovery/purification for a given set of operating conditions.

The test plan described in this document has been structured to allow the above objectives to be met using sound scientific principles. This document explains testing plans with respect to areas such as test methodology, procedures, parameters, and instrumentation. Section 5.0 describes Quality Assurance/Quality Control requirements of this task that will

ensure the accuracy of data. Also presented within this document are data interpretation procedures and hypothesized results. Worker health and safety considerations are covered in Section 8.0.

This test plan will be available at the test site, and verification testing will be conducted in accordance with the test plan requirements.

1.1 Background

The surface finishing and plating industries often use mineral acids to clean, prepare, treat, and finish metal parts. The RETEC system is designed to be used to recover mineral acids from dilute rinse waters and/or to purify concentrated acid baths, prolonging bath life and recovering bath constituents for reuse. This verification will focus on the use of a USFilter RETEC separated cell purification system installed on a chromic acid anodizing line at DVI in Lynnwood, California. Prolonging the chromic anodizing bath life is of great importance, not only from a financial standpoint, but a regulatory standpoint as well. Chromium is an RCRA listed characteristic waste (D007), and certain anodizing waste streams can be listed as non-specific source hazardous wastes (F006), by the EPA. Chromium has also been identified as a high priority for reduction and elimination in plating operations by the National Metal Finishing R&D Plan, and is addressed in the Strategic Goals Program regarding toxic metals reduction.

The tank life of the typical chromic anodizing bath is limited by the buildup of trivalent chromium; trace metals such as aluminum, magnesium, nickel, zinc, copper, iron, and other contaminants. Chromic acid is an important metal finishing chemical as well as an expensive chemical to purchase and waste-treat. USFilter's RETEC separated cell electro dialysis system is envisioned to be used to economically recover chromic acid from rinsewater, purify the chromic acid used in the anodizing process bath, and convert contaminant trivalent chromium into usable hexavalent chromium within the process bath. Anodizing process rinsewater can be simultaneously conditioned for recycling. Bath replacement is minimized resulting in lower costs for reclamation and waste disposal.

There are no adverse effects foreseen on plating quality when the RETEC unit is in operation. The unit has no effect on chlorides and sulfates. The purification process should extend the life of the anodizing bath and improves plating efficiency.

2.0 TECHNOLOGY DESCRIPTION

2.1 Theory of Operation

Typically, chrome plating and anodizing tanks become less efficient over time due to the build-up of trivalent chromium and tramp metal impurities. Unless treated to improve its quality, the plating bath presents the electroplater with an expensive disposal problem. The RETEC Chrome Purification (CP) system's functions include

oxidation of trivalent chromium and removal of metal impurities. *Figure 1* shows a simplified diagram of a typical application of a RETEC system for bath purification.

Anodizing solution is recirculated between the anolyte section of the RETEC CP cell and the anodizing tank. During this process, trivalent chromium in the solution is oxidized to hexavalent chromium, and metal cations in solution are transported through the cell separator to the catholyte section of the cell. The rate of trivalent chromium oxidation and the transfer rate of metal cations are related to the operating conditions. The oxidation rate of trivalent chromium will vary with cell current, and will be greater at high current and high trivalent chromium concentration. The anolyte to catholyte transfer rate of metal cations will depend on the species of cation present in solution, cation concentration, and the pH of the catholyte. Process operating conditions will vary and will depend upon the type and degree of contamination of the anodizing bath.

Catholyte solution is circulated between the catholyte section of the RETEC cell and the clarifier. The catholyte pH is controlled by the addition of anodizing solution or straight chromic acid to the clarifier. Hexavalent chromium in the catholyte is transferred through the cell separator to the anolyte side of the cell and then to the anodizing tank. Metal impurities in the chemicals added to the clarifier tank for pH control, and those impurities which are transferred from the anolyte through the cell separator into the catholyte, all accumulate in the catholyte solution.

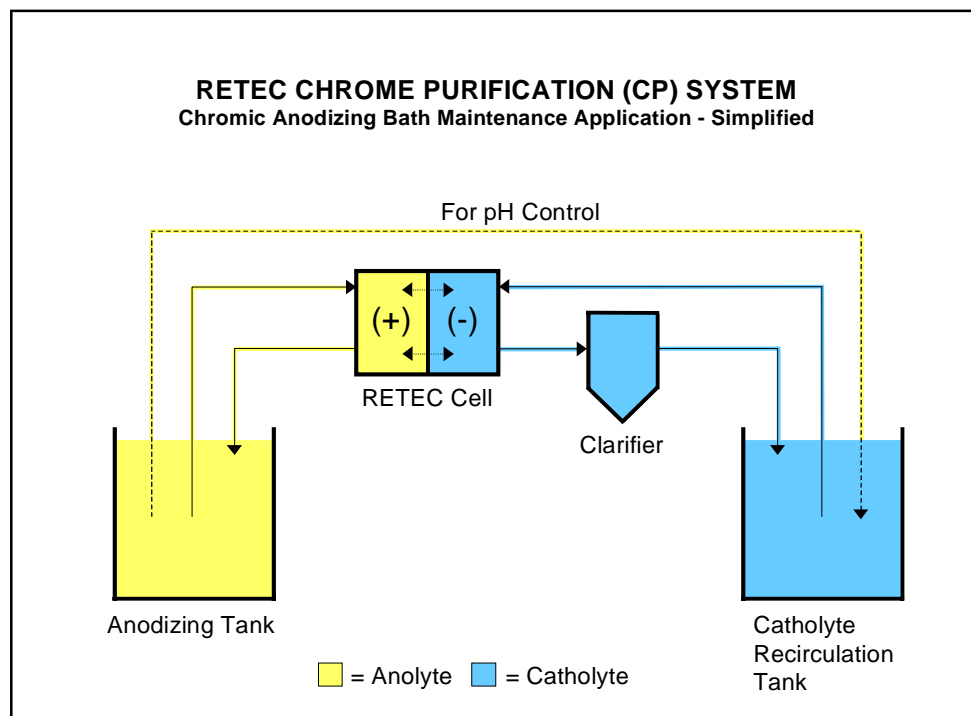


Figure 1: RETEC Chromic Anodizing Bath Maintenance Application – Simplified

The tramp metal impurities can be recovered from the catholyte solution by adjusting the cell operating conditions to precipitate the metal cations as their respective hydroxides, which are then separated from solution in the clarifier. Alternatively, and on a periodic basis, the catholyte solution can be chemically treated to remove the tramp metals from solution.

The diagram in **Figure 2** illustrates the reactions that typically take place in the RETEC cell. While this simplified diagram shows only one anode chamber, the remainder of the diagrams in this test plan will depict a RETEC Model 6, which contains up to six anode chambers. While USFilter makes systems with as many as 25 anode chambers, DVI uses a standard RETEC Model 6 installed on their 10,000 gallon anodizing line.

The RETEC chromic acid anodizing bath purification process offers a number of advantages - raw material usage is reduced through the oxidation of trivalent chromium to hexavalent chromium, tramp metal impurities are removed from the anodizing bath, and water quality of the anodizing bath is improved by the removal of common water contaminants such as calcium and magnesium. The purification process extends the life of the anodizing bath and improves anodizing efficiency.

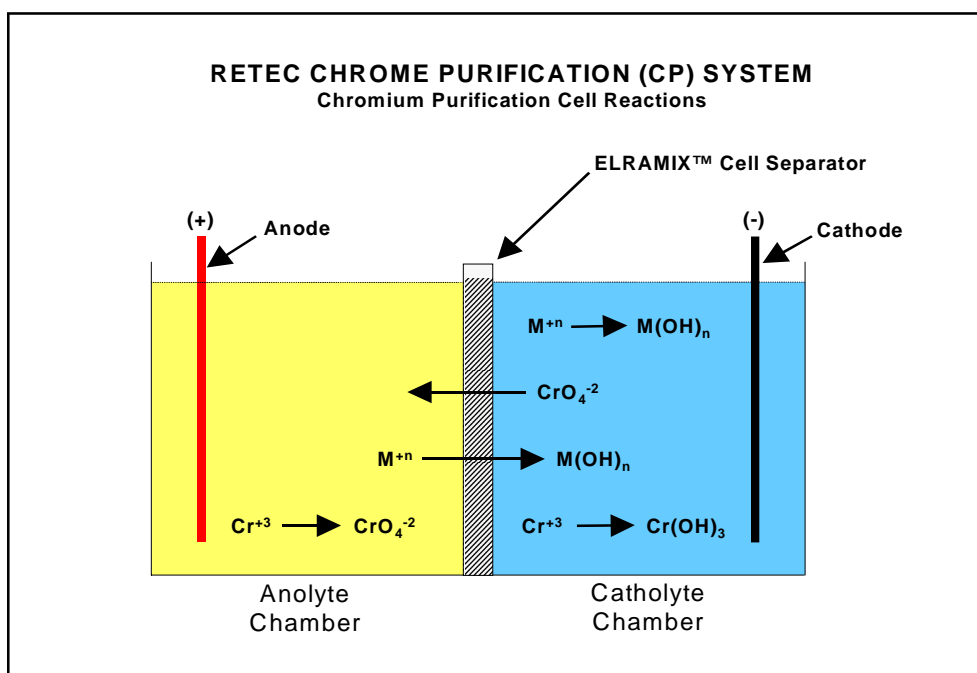


Figure 2: RETEC Chromium Purification Cell Reactions

2.2 Commercial Status

The USFilter RETEC system was introduced to the market in 1992. The target market was metal finishing, and in particular, chrome platers. This is the only market where this technology is being used at this time. There are two functional configurations, the RETEC Separated Cell Recovery (SCR) system, and the RETEC Separated Cell Purification (SCP) system. The RETEC SCR is used for chromic acid, sulfuric acid, and phosphoric acid recovery from dilute rinse waters or even from concentrated acid baths. The recovered acid is free of metal contaminants, and once concentrated, is suitable for reuse in the process. The metals are precipitated out of the rinse water as their hydroxides, and the purified water is suitable for more rinsing. The RETEC SCP system is basically the same design, but is designed for chromic acid, sulfuric acid, and phosphoric acid purification. Metals are precipitated out of the concentrated acid baths as their hydroxides, thus purifying the process bath, which extends its useful life. There are approximately 13 RETEC systems in operation in the U.S., and the system has been sold and installed in various other countries, including the Canada, Mexico, Argentina, and Brazil.

2.3 Pollution Prevention Classification

USFilter's RETEC CP system is a bath maintenance technology. Bath maintenance refers to a range of pollution prevention practices and technologies that preserve or restore the operating integrity of metal finishing process solutions, thereby extending their useful lives. Due to the rising costs of chemicals, energy, and treatment/disposal fees, and increasingly more stringent environmental requirements, bath maintenance has become a greater priority to metal finishing companies, and the methods and technologies they employ have increased in sophistication. Today, firms are willing to expend significant amounts of capital and operating funds for equipment and methods that primarily reduce the disposal frequency of their baths. In addition to extending bath lives, solution maintenance often improves the average operating efficiency and effectiveness of a process solution and therefore has a positive impact on production rates and finish quality.

2.2 Environmental Significance

The RETEC technology set up in an acid recovery mode may close-loop the rinsing from a sulfuric, phosphoric, or chromic acid bath. The water use reduction in these applications should approach 100%, and the recovered acid concentrated and reused in the plating or anodizing process. The fact that the system could electrolytically removed metals from acids or spent electroless nickel baths means less waste, and because it is an electrolytic reaction, additional waste-generating chemicals are not required in the RETEC process. In the case of the RETEC CP system, the additional environmental benefit of the recovery of hexavalent chromium for reuse in the plating or anodizing process is accomplished by the possible oxidation of trivalent chromium within the RETEC cell.

3.0 PROCESS DESCRIPTION

3.1 Equipment and Flow Diagram

The RETEC CP system consists of a rectifier, a clarifier to remove metal hydroxides that are formed in the catholyte as acids are purified and recovered, and an electrolytic cell. The electrolyzer box is fabricated of polyvinyl chloride and is supplied with inlet, outlet, and drain connections and valves. The cell, which is depicted in *Figure 3*, consists of a series of anodes and cathodes. The individual anodes are contained within separate anode chambers. The front and back sides of the anode chambers have diaphragms of ELRAMIX, a porous, polymeric membrane, separating the anolyte and catholyte compartments. ELRAMIX is manufactured by ELTECH, and was selected as the separator of choice after an extended evaluation of a wide variety of commercial materials having properties required for use as cell separators. Titanium mesh cathodes, which are easily removed from the cell, are placed between each anode chamber. The anodes and cathodes are connected to copper bus bars located on opposite sides of the cell box. The electrolyzer can operate with a full complement of anode chambers or any fewer numbers depending on capacity requirements. The cell is equipped with an air sparging system to prevent metals and metal hydroxides formed in the catholyte from settling in the cell. However, it has been determined that the formation of solids can be controlled by maintaining the pH of the catholyte <2 with additions of chromic acid, therefore the air sparging system is not used at DVI.

A thermo-sensor is included with the electrolyzer to shut off the rectifier if the temperature of the liquid being treated exceeds the pre-set limit. A thermo-controller resets the system. The RETEC CP electrolyzer is also supplied with a manifold located beneath the electrolyzer box that hydraulically connects each anode compartment to the anolyte reservoir. The anolyte feed manifold is located beneath the cathode bus bar. Solution from the anodizing tank is pumped into the anode frames through the anolyte feed manifold. The anodizing solution is returned by gravity to the anodizing tank from the anolyte reservoir. *Figure 4* shows an isometric of the six-compartment acid purification cell (RETEC CP-6) used at DVI.

The RETEC electrolyzer sits on a steel chassis, which mounts onto a stand to provide the proper height when installed at the anodizing line. The stand contains shelves for mounting the liquid feed pumps and the air compressors for the air sparger system.

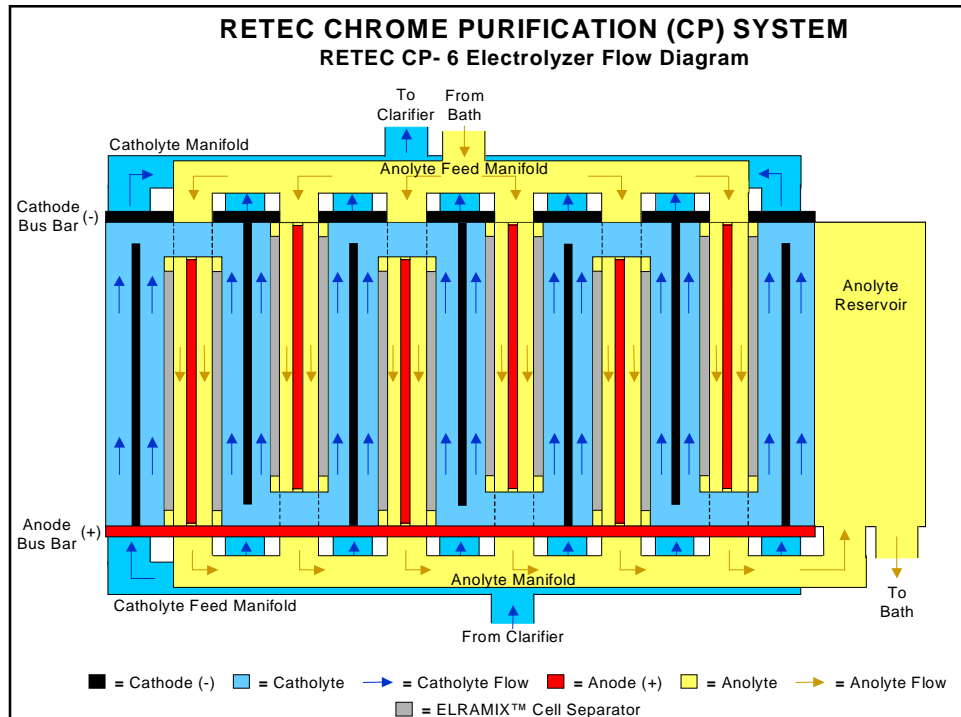
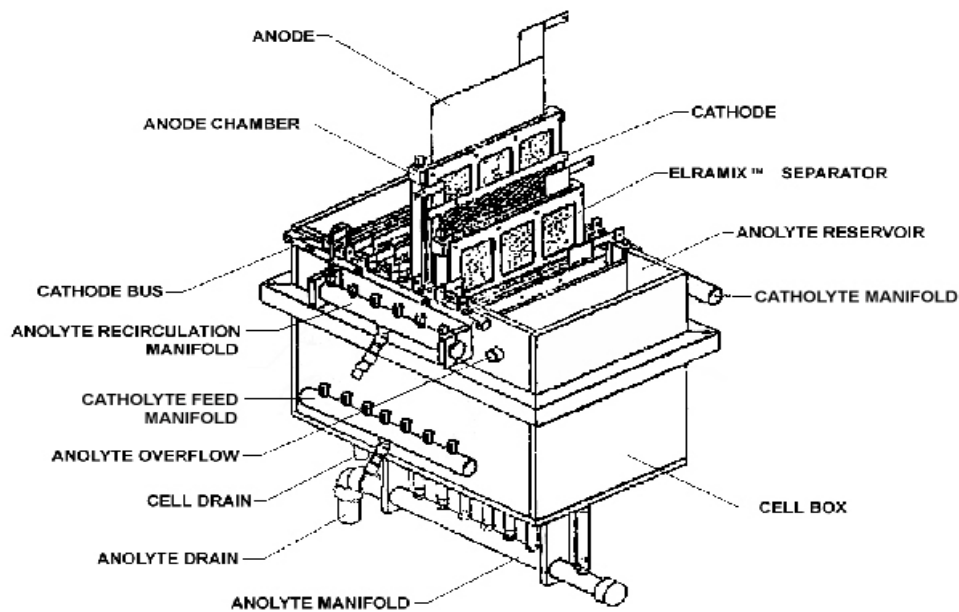


Figure 3: RETEC CP-6 Electrolyzer Flow Diagram



RETEC CP-6 Chrome Purification Cell

Figure 4: RETEC CP-6 Chrome Purification Cell

The system is installed online and operates continuously, but can also operate in a batch mode. A liquid transfer pump is used to deliver solution from the anodizing

tank to the anolyte feed manifold of the RETEC cell at a flow rate of about 7.5 L/min. From the anolyte outlet, the solution is returned to the anodizing tank. At DVI, anodizing solution is also used in the catholyte circulation loop. The pH of the catholyte is controlled by the periodic addition of chromic acid to the clarifier.

The purpose of the catholyte solution is to provide sufficient conductivity and conditions for the desired reactions occurring in the cell to proceed at a reasonable rate, and to maintain the pH in an acceptable range. Under normal operating conditions, metals in the catholyte solution would be precipitated as their hydroxides and removed in the clarifier. This installation, however, maintains the catholyte pH <2, so metal hydroxides are not formed. The clarifier is decanted when the concentration of tramp aluminum ions reaches the point of lowering RETEC efficiency. Controlling the pH of the catholyte is important and relatively easy to accomplish since chromic acid can be added to the catholyte as needed.

The catholyte solution is delivered to the RETEC cell by a liquid transfer pump. The solution then flows by gravity from the cell to the clarifier. When the catholyte is saturated with contaminants and requires disposal, it is drained into the facility waste storage tank.

Figure 5 below shows a detailed schematic of the RETEC CP chromic anodizing bath maintenance application complete with suggested sampling points.

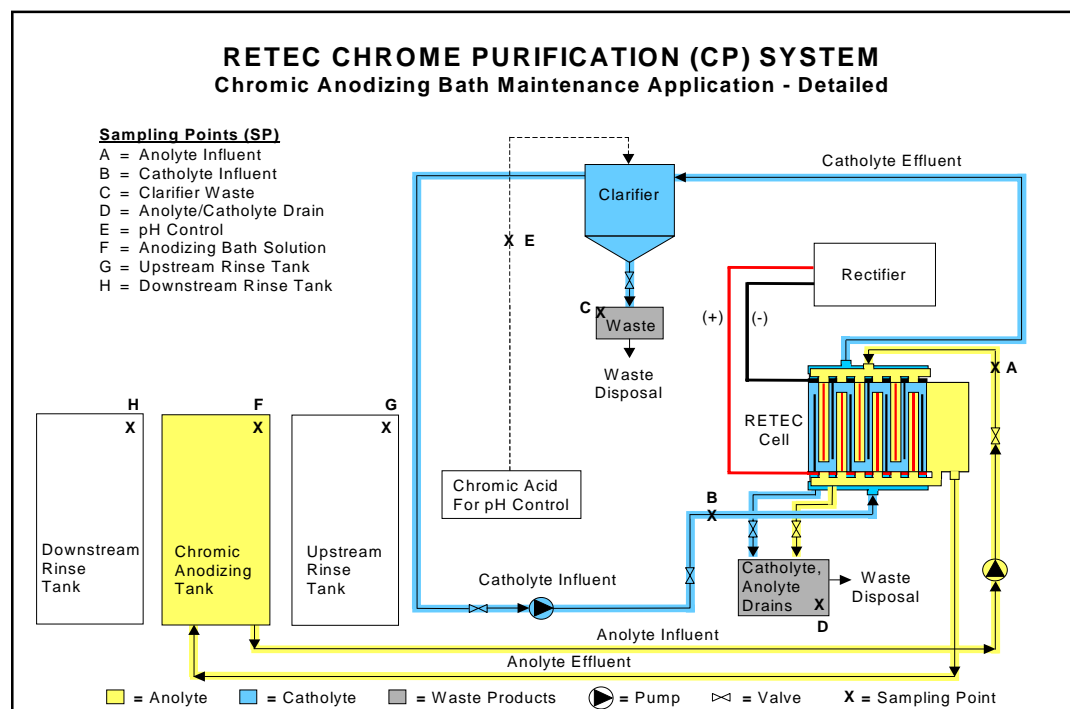


Figure 5: RETEC Chromic Anodizing Bath Maintenance Application – Detailed

3.2 Testing Site

The metal finisher selected for testing is DVI in Lynwood, California. Established in 1957, the 135,000 square foot facility has one of the nation's largest anodizing departments. They serve a variety of customers, with a large majority of the work dedicated to the aerospace, military, and commercial industries. They maintain three independent processing lines:

- 62 foot chromic and sulfuric anodize; chem-film (Alodine); pre-penetrant cleaning and etching.
- 27 foot chromic and sulfuric anodize; chem-film (Alodine); pre-penetrant cleaning and etching; passivation; and titanium pickle.
- 16 foot sulfuric anodize; chem-film (Alodine); pre-penetrant cleaning and etching; and titanium pickle.

Together, these three lines allow the acceptance of critical hardware across the full spectrum of size, quantity, tolerance requirements, and color. DVI also operates five paint booths ranging in size up to 62 feet in length and five paint curing systems within the 20,000 square foot painting department of the company. Masking, painting, curing, and polishing performed in this department allow for the application of virtually any coating to exact standards.

The Lynwood plant utilizes a USFilter RETEC Model CP-6 Separated Cell Purification system installed on the 27 foot, 10,000 gallon chromic acid anodizing (type I) line. The Model CP-6 has been operating successfully in purification mode since 1995. Anodizing bath solution is purified of tramp metals and trivalent chromium, and the catholyte, which is also anodizing bath solution, is circulated through the RETEC system, resulting in the recovery of hexavalent chromium for reuse in the anodizing bath. DVI has not experienced any degradation in plating quality since the installation of the RETEC unit. The DVI anodizing line uses a manually operated rack system. Materials anodized on the line consist of various grades of aluminum, primarily 7075, 2024, 2219, 6061, and 7050. Parts first go through an alkaline clean, then a caustic etch, then a deoxidizing tank. Subsequently, they go to the anodizing step, and finally the parts are sealed. Each process step is followed by a single-stage flowing rinse. See *Appendix A* for a pictorial representation of DVI's aluminum anodizing process.

The solution from the anodizing bath is re-circulated through the anolyte compartment of the cell at a rate of about 7.5 L/min. The cell applies approximately 400A @ 4 VDC for the electrolytic reaction to take place. Trivalent chrome is oxidized to hexavalent chrome at a rate of about 0.75 grams per amp-hour (g/Ahr), and at the same time, tramp metals are rejected through the ELRAMIX separator into the catholyte compartment. Anodizing solution also acts as the catholyte and is pumped continuously through the catholyte compartment. Hexavalent chrome in the catholyte passes through the ELRAMIX into the anolyte compartment - this gives a substantial recovery of the hex chrome from the catholyte at the same time. Effective

chrome oxidation and recovery for this operation increases the value of hex chrome pickup to about 1.25 g/Ahr.

A picture of the DV Industries RETEC CP-6 system is provided for reference in *Figure 6*.



Figure 6: DVI's RETEC CP-6 System

4.0 EXPERIMENTAL DESIGN

4.1 Test Goals and Objectives

The overall goal of this project is to establish the technical and economic performance parameters that will enable a potential user to determine if the USFilter RETEC Chrome Purification (CP) system is appropriate and feasible under their specific operating conditions. The objective of testing is to generate the analytical data and performance observations required to support these technology verification efforts.

The following are statements of specific project objectives:

- Evaluate, document, and verify the performance of the RETEC separated cell technology for the oxidation of trivalent chromium formed in the bath during the anodizing process.
- Evaluate, document, and verify the performance of the RETEC separated cell technology for the removal of aluminum and other tramp metals from the process bath that build up during the anodizing process.

- Evaluate, document, and verify the performance of the RETEC separated cell technology for the recovery of chromic acid from the catholyte solution.
- Quantify the energy required to operate the system. Primary energy users include the rectifier for the electrolyzer cell and liquid feed pumps. This information will be used to estimate operating costs for the RETEC CP system.
- Quantify environmental benefit by determining the reduction in bath disposal frequency.

4.2 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 project objectives. Non-critical measurements are those related to process control or general background readings.

Operational data will be collected on the RETEC CP system performance during the treatment of anodizing solution. The following operational data will be collected:

Critical Measurements

- Chemical additions: Quantity and frequency
 - Chromic acid - for pH control (mass (kg) of each addition, time of each addition)
 - Water - for bath makeup, pH control and chromic acid additions (volume (L) of each addition, time of each addition)
- Concentrations of trivalent chromium, hexavalent chromium, and trace metals (Al & Mg) in anolyte/catholyte (g/L)
- Amperage applied to cell & cell voltage (amps & VDC)
- pH of anodizing bath, and anolyte/catholyte (pH units)
- Elapsed time of cell operation (hrs)
- Production throughput rates (parts/hour, pounds/hour, surface area/unit of time)
- O & M labor requirements
- Solution processing rate (L/hr) & chemical characteristics of solutions (oxidation rate of tri-chrome to hex-chrome & removal of contaminants (g/Ahr))
- Waste volumes, characteristics, & costs

Non-Critical Measurements

- Bath and cell liquid temperatures (°C)
- Bath aeration rates (CFM)
- Bath volume (L)
- RETEC rectifier Amp hours (Ah)
- Hexavalent Chromium Air Emissions ($\mu\text{g}/\text{m}^3$)

This data will be used to determine the system material balance, bath stability and the chromic acid purification rate, operation and maintenance requirements, and cost effectiveness for a given set of operating conditions.

Historical Data

DVI's historical data on chromic acid bath disposal frequency prior to installation of the RETEC CP system will be collected and provided in the verification report to determine the environmental benefit.

DVI's historical records regarding plating quality measurements, and chloride/sulfate levels in the anodizing bath will also be reviewed and summarized in the verification report in order to alleviate plating quality concerns regarding the implementation of the USFilter RETEC unit.

4.3 Test Matrix

The unit will be evaluated on its ability to efficiently purify and recover chromic acid. The specific tests planned are described below and listed in *Table 1*.

In this verification the anodizing bath solution will be circulated through the cell as the anolyte solution. Trivalent chromium in the anolyte will be oxidized back to hexavalent chromium. Aluminum in the anolyte will be transported across the cell separator to the catholyte compartment of the cell. Due to Boeing specified guidelines for rinsewater, DVI uses deionized water in their rinse tanks. Since deionized water lacks the required conductivity to facilitate the electrolytic reaction within the RETEC cell, it cannot be used as the catholyte in this instance. However, measuring rinse bath constituents will help determine the mass balance of the RETEC system. Anodizing bath solution, which possesses the proper pH and conductivity to facilitate the electrolytic reaction, will also act as the catholyte solution. Hexavalent chromium in the catholyte will migrate across the cell separator to the anolyte compartment of the cell and subsequently return to the anodizing tank. Aluminum, trivalent chromium, and other tramp metals in the catholyte will remain in solution in the catholyte until the saturation limit is reached, at which time the pH will become too high to maintain the proper electrolytic reactions. At this time, catholyte is drained from the system and sent to a waste storage tank. Water and chromic acid are added to replenish the catholyte to its operating level. In order to gain the valuable system performance information desired for the ETV-MF Program, sampling, testing, and documentation will take place in two different test matrixes:

- Baseline Mode - where contaminant buildup data is gathered with the RETEC CP system turned off, and
- Operational Mode - where the RETEC system is operated with the anodizing bath solution flowing through the catholyte compartment of the cell and is disposed of as it becomes too contaminated to facilitate the electrolytic reaction within the RETEC cell.

Initial anodizing bath samples will be taken with the RETEC CP system turned off in order to establish a baseline. Trivalent chrome, aluminum and magnesium contamination buildup rates will be determined for the anodizing bath at normal operating conditions without the RETEC CP system involved in the process. The RETEC CP system will then be turned on, and sampling will continue to determine the specified test measurements in the Operational Mode.

Periodically, the clarifier is partially drained to remove the spent catholyte. This waste consists of chromium ions, which have not yet migrated into the anolyte solution and the scavenged tramp metal cations. The clarifier is decanted, and the waste is sent to the facility waste storage tank for subsequent disposal. The frequency of draining and the volume of waste drained are contingent upon the amount of impurities in the anodizing bath, and the process operating conditions.

Verification Testing

Testing in each mode will be conducted at normal operating parameters recommended by USFilter and DV Industries. The key operating data, as discussed in Section 4.2, will be recorded in a bound laboratory notebook and transcribed onto the form shown in *Figure 7* in conjunction with weekly sampling. Analytical testing of samples will be conducted to evaluate the indicated parameters in *Table 1*.

4.4 Operating Procedures

DVI personnel will perform normal operation and maintenance activities during testing. These activities will be observed and noted by an ETV-MF representative.

The RETEC system will be operated 24 hrs/day for 5 to 6 days per week. The exact number of operating days will depend on the work load schedule at DVI. Each test mode will consist of five work weeks.

4.5 Sampling, Process Measurements, and Testing Procedures

4.5.1 Sampling Responsibilities & Procedures

Samples will be taken from the sampling points specified in *Figure 5*. Sampling will occur at the frequency listed in *Table 1* for each parameter. The appropriate sampling container will be used as outlined in *Table 3* for each test parameter. Each laboratory sample bottle will be labeled with the date, time, sample ID number, and test parameters required. Sampling will take place at least one hour after the system reaches normal operation stasis following any chemical or water additions or any system shut-down/start-up operations. Normal operating stasis is defined as the equilibrium of all operational parameters within the systems standard operating limits. Sample preparation methods are described in each individual analytical method.

Samples to be analyzed at off-site laboratories will be accompanied by a chain of custody form. The samples will be stored and transported in appropriate sample transport containers (e.g., coolers with packing and blue ice) by common carrier. The transport containers will be secured with tape to ensure sample integrity during the delivery process to the analytical laboratories. The Project Manager or designee will perform sampling and labeling, and ensure that samples are properly stored and secured for transport to the analytical laboratories. Columbia Analytical Services in Canoga Park, California will perform all aqueous analytical transportation and the testing of all rinse waters and total metals analysis for process bath samples. Anachem Labs of Los Angeles, California will perform all process bath titrations for hexavalent and trivalent chromium, and submit results to CAS for incorporation into all analytical reports presented to the ETV-MF Team. Total Air Analysis of Carson, California, will conduct the air sampling operations, and West Coast Analytical Services of Santa Fe Springs, California, will perform the air sample analytical testing.

4.5.2 Process Measurements

Monitoring of the tests will be accomplished by recording key operating data. Monitoring instrumentation will be provided and calibrated by the Project Manager or his designee according to manufacturer recommendations. See Section 4.5.4 for specific calibration procedures. Process measurements will be recorded at the same time that aqueous sampling occurs, that is to say, once a week, in accordance with *Table 1*. On-site measurements will be performed three times for each sampling day in order to ensure compliance with the QA objectives stated in *Table 2*.

Electricity use will be measured by determining the power requirements and cycle times of pumps and other powered devices. DVI will provide the cost of labor, electricity, and other data needed for cost analysis. Cumulative amp hours (Ah) will be measured by the installation of a Darrah Electric Company, A.H.M. 500 amp, 50 milli-volt ampere hour meter on the RETEC system rectifier. See *Table 2* for equipment performance details.

Process fluid flow rates for this demonstration will be measured by an Omega Engineering, Inc., Model FD-7000 liquid flowmeter, a multi-liquid ultrasonic flowmeter with non-penetrating transducers. See *Table 2* for equipment performance details.

Anodizing bath surface tension will be monitored using a Central Scientific Company, Inc., catalog number 70535, du Nouy tensiometer. See *Table 2* for equipment performance details.

Data will be recorded in a bound laboratory notebook and transcribed onto the form shown in *Figure 7*.

SAMPLE LOCATION	TEST MODE	NUMBER OF SAMPLES	FREQUENCY	TEST DURATION ¹	TEST PARAMETERS
SP-A Anolyte Influent	Operational	1	1/week	5 weeks	Hexavalent Chromium Trivalent Chromium Selected Metals (Cr, Al & Mg)
SP-B Catholyte Influent	Operational	1	1/week	5 weeks	Same as SP-A
SP-C Clarifier Waste	Operational	1	1/week	5 weeks	Same as SP-A
SP-D Anolyte/Catholyte Drain	Operational	1	1/week	5 weeks	Same as SP-A
SP-E pH Control	Baseline Operational	As Required	Each addition	5 weeks (for each test mode)	Mass of Chromic Acid (kg) Volume of Water (L)
SP-F Anodizing Bath Solution	Baseline Operational	1 (for each test mode)	1/week (for each test mode)	5 weeks (for each test mode)	Surface Tension Hexavalent Chromium Trivalent Chromium Selected Metals (Cr, Al & Mg)
SP-G Upstream Rinse Water	Baseline Operational	1 (for each test mode)	1/week	5 weeks (for each test mode)	Same as SP-A
SP-H Downstream Rinse Water	Baseline Operational	1 (for each test mode)	1/week	5 weeks (for each test mode)	Same as SP-A
Personal Air Sampling	Baseline Operational	3 (2 hour samples) (for each test mode)	Once in fourth week and once in ninth week of verification testing	1 day (for each test mode)	Airborne Hexavalent Chromium
Process Emissions Air Sampling	Baseline Operational	3 (2 hour samples) (for each test mode)	Once in fourth week and once in ninth week of verification testing	1 day (for each test mode)	Airborne Hexavalent Chromium

1. Each mode of operation will have a test duration of five weeks, however the Thanksgiving holiday falls in the middle of the Operational Phase, so there will be no testing done that week. The Operational Phase will therefore be extended one week in order to maintain five sampling weeks for each mode of operation.

Table 1: Test Matrix

4.5.3 Testing Parameters

4.5.3.1 Flowrate

Liquid transfer pumps deliver anodizing process solutions to various parts of the RETEC CP system during the purification process. The rate at which

these liquids are transferred is of major importance in the RETEC CP system. Liquid flowrates will be determined with a non-penetrating ultrasonic flowmeter for the anolyte influent (SP-A), the catholyte influent (SP-B), the clarifier waste (SP-C), the anolyte/catholyte drain (SP-D), and the pre- and post-anodizing rinse tanks (SP-G & SP-H).

4.5.3.2 Temperature

While temperature is not a critical parameter in the RETEC process, it is very critical in the chromic acid anodizing process, and therefore will be monitored at the liquid sampling points SP-A through SP-D, and SP-F through SP-H. (EPA/600/4-79/020 Method 170.1) Temperature will be measured with a Davis Instruments Model # 9214 microprocessor controlled, Automatic Temperature Compensated (ATC) pH meter with built-in temperature sensor. An immersion heater typically maintains the bath temperature between 91°F and 99°F, with an average operating temperature of 95°F.

4.5.3.3 pH Control

The catholyte pH is maintained by the addition of chromic acid to the catholyte re-circulation loop at the rate required to maintain the catholyte solution within the pH range conducive to the trivalent chromium oxidation process (< pH 2). pH is therefore a critical parameter in the RETEC process. pH will be measured with a Davis Instruments Model # 9214 microprocessor controlled, Automatic Temperature Compensated (ATC) pH meter with built-in temperature sensor. pH will be measured at all liquid sampling points SP-A through SP-D, and SP-F through SP-H. (EPA/600/4-79/020 Method 150.1)

4.5.3.4 Hexavalent Chromium Concentration

Over time, a certain amount of hexavalent chromium will reduce to trivalent chromium in the typical anodizing bath. Hexavalent chromium in the anolyte, catholyte and clarifier waste can be determined by Sodium Thiosulfate titration (Iodide method). This is a standard industry method for determining high concentrations of hexavalent chromium in anodizing baths, and is described in detail in the 1999 Metal Finishing Guidebook in Table VIII on page 538. Hexavalent chromium in the rinse tanks is more dilute, and will therefore require EPA SM 3500 Cr D, a more precise colorimetric method for measuring dilute hexavalent chromium levels in rinsewater/wastewaters. Hexavalent chromium levels will be measured at all liquid sampling points SP-A through SP-D, and SP-F through SP-H (1999 Metal Finishing Guidebook, Table VIII method for CrO₃ (Cr⁶⁺) & EPA SM 3500 Cr D)

4.5.3.5 Trivalent Chromium Concentration

Trivalent chromium in the anodizing bath adversely effects anodizing quality, and therefore must be removed from the process. The RETEC CP system is designed to oxidize trivalent chromium in the anodizing solution back to hexavalent chromium, thus extending the life of the bath. The operating conditions set for the RETEC CP system are directly related to the rate of trivalent chromium buildup in the anodizing bath. Trivalent chromium in the anolyte, catholyte and clarifier waste can also be determined by the Sodium Thiosulfate titration (Iodide method). Determine the total hexavalent chromium on a sample of solution, and again after the trivalent chrome has been oxidized to the hexavalent state. The difference represents the concentration of trivalent chromium in the original sample. The method is described in detail in the 1999 Metal Finishing Guidebook in Table VIII on page 538. Trivalent chromium in the rinse tanks is more dilute, and will therefore also require EPA SM 3500 Cr D, a more precise colorimetric method for measuring dilute hexavalent chromium levels in rinsewater/wastewaters. The same double titration method that is described above is used to determine the trivalent chromium in the water. Trivalent chromium levels will be calculated at all liquid sampling points SP-A through SP-D, and SP-F through SP-H. (1999 Metal Finishing Guidebook, Table VIII method for Cr³⁺ & EPA SM 3500 Cr D)

4.5.3.6 Other Selected Metals (Cr, Al & Mg)

Based on the type of substrate being anodized, certain metals will accumulate in the process bath. As the concentration of these metals increases, anodizing efficiency decreases, resulting in the need to dump the anodizing bath. Bath life is extended by the RETEC system when tramp metals are transferred across the cell separator, precipitated as their respective hydroxides and separated from the catholyte within the clarifier. Acid digestion followed by ICP-AES analysis of the anolyte, catholyte, clarifier waste, and rinse tanks can provide a metals scan of the three most prevalent metals in the DVI RETEC system. Total chromium levels will be monitored as a check to the titrimetric and colorimetric methods described above. Periodic sampling and analysis at SP-A through SP-D and SP-F through SP-H will enable the ETV-MF team to determine the removal rate of aluminum and magnesium from the anodizing bath. (EPA SW-846 Method 3010A & 6010B)

4.5.3.7 Surface Tension

Surface tension is a critical parameter in the anodizing process, and will be maintained in the optimum process range (26-40 dynes/cm). Surface tension will be measured at the anodizing process bath (SP-F) with a du Nouy tensiometer. (ASTM Method D-1331-89)

4.5.3.8 Bath Volume

The anodizing and rinse bath volumes should be maintained at relatively stable levels. The recirculation of process bath solutions and the pH adjustment of the catholyte should not adversely affect bath volumes. Rinse tanks operate on a continuous overflow basis, so bath volumes remain consistent. Bath volume measurements will be used in calculating overall purification, recovery efficiencies, and mass balance. Bath volumes will be calculated for anodizing and rinse baths located at sampling points SP-F through SP-H.

4.5.3.9 Amperage Provided to Cell

Cell amperage is the main factor that drives the RETEC electrolytic reaction. Trivalent chromium is oxidized to hexavalent chromium, and tramp metals diffuse over the cell separator to the catholyte solution for subsequent removal based on the current density, and will be greater at high current and high trivalent chromium concentrations. Process operating conditions will be monitored and adjusted based on the type and degree of contamination of the anodizing bath. Cell current will be monitored and recorded directly from the rectifier.

4.5.3.10 RETEC Rectifier Amp Hours

Cumulative amp hours (Ah) applied to the RETEC electrolytic cell will be measured by the installation of a Darrah Electric Company, A.H.M. 500 amp, 50 milli-volt ampere hour meter on the RETEC system rectifier.

4.5.3.11 Time

Time is important in any testing procedure where rates are concerned. The testing parameters listed above, coupled with accurate time measurements, will provide the means required to determine valuable process rates such as oxidation rate of trivalent chromium to hexavalent chromium, transportation rate of tramp metals in the bath from the anolyte to the catholyte compartment, and the recovery rate of chromic acid from the catholyte.

4.5.3.12 Clarifier Waste Composition

According to USFilter, waste liquid drained off of the clarifier primarily contains chromium, aluminum and small amounts of magnesium. It is important to know how much waste the system will generate, since it must be disposed of as hazardous waste. The quantity and composition of this waste will enable the ETV-MF team to determine how the waste must be handled, quantify waste generation, and estimate waste disposal costs. Acid digestion and ICP-AES analysis can quantify most metals in the waste. The titrimetric

test method will be used for the quantification of hexavalent and trivalent chromium in the waste. Clarifier waste samples will be taken at sampling points SP-C & SP-D. (EPA SW-846 Method 3010A/6010B & 1999 Metal Finishing Guidebook Titrations).

Critical Measurements	Matrix	Sample/Test Responsibility	Sample Preparation Method	Method	Reporting Units	Method of Determination	MDL	Precision (RPD)	Accuracy (% Recovery)	Completeness
Hexavalent Chrome	Aqueous (Bath/RETEC)	MMTC/Anachem	-	See Note 1	g/L	Titration	200 µg/L	≤ 2	(95 – 105)	95
Trivalent Chrome	Aqueous (Bath/RETEC)	MMTC/Anachem	-	See Note 1	g/L	Titration	200 µg/L	≤ 2	(95 – 105)	95
Hexavalent Chrome	Aqueous (Rinsewater)	MMTC/CAS	-	EPA SM 3500 Cr D	µg/L	Colorimetric	10 µg/L	≤ 30	(75 – 125)	95
Trivalent Chrome	Aqueous (Rinsewater)	MMTC/CAS	-	EPA SM 3500 Cr D	µg/L	Colorimetric	10 µg/L	≤ 30	(75 – 125)	95
Selected Metals (Cr, Al & Mg)	Aqueous	MMTC/CAS	EPA 3010A (acid digestion)	EPA 6010B	µg/L	ICP-AES	2 – 100 µg/L	≤ 30	(75-125)	95
Hexavalent Chromium (Personal Exposure & Process Emissions)	Air	Total Air Analysis, Inc.	-	CARB 425	µg/m ³	IC-Colorimetric	0.025 µg/m ³	≤ 5.0	(90-110)	95
Liquid Flow (Catholyte, Anolyte, pH Control, rinse Water, Clarifier Waste)	Aqueous	MMTC	-	Flow Meter	L/hr	Ultrasonic	0.3-3.6 L/hr	≤ 1	+/- 2%	90
Surface Tension	Aqueous	MMTC	-	ASTM D-1331-89	dynes/cm	Tensiometer	0.1 Dynes/cm	≤ 1	+/- 0.6%	90
Cell Current	Aqueous	MMTC	-	Amp-hour Meter	Amps/VDC	Ammeter	0.5 Amps	≤ 0.5	+/- 2%	90
pH	Aqueous	MMTC	-	pH Meter (EPA 150.1)	pH Units	Electrometric	0.01 pH	≤ 1.0	+/- 0.02 %	90
Temperature	Aqueous	MMTC	-	Thermometer (EPA 170.1)	°F (°C)	Thermometric	0.1 °C	≤ 1	+/- 0.5 %	90

Note 1: Standard sodium thiosulfate titration will be used to determine the hexavalent and trivalent chromium concentration. These procedure were taken directly from the 1999 Metal Finishing Guidebook, Vol. 97, No. 1, Control, Analysis, and Testing Section – Chemical Analysis of Plating Solutions, Charles Rosenstein and Stanley Hirsch, Table VIII – Test Methods for Electroplating Solutions, page 538.

Table 2: QA Objectives For Precision, Accuracy, And Detection Limits

ANALYTE	SAMPLE PREPARATION METHOD	METH	SAMPLE BOTTLE	PRESERVATION METHOD	HOLDING TIME
Hexavalent Chromium	None	1999 Metal Finishing Guidebook Titration & EPA SM 3500 Cr D	500 ml High Density Polyethylene (HDPE)	Cool to 4° C ($\pm 2^\circ$ C)	24 hours
Trivalent Chromium	None	1999 Metal Finishing Guidebook Titration & EPA SM 3500 Cr D	500 ml HDPE	Cool to 4° C ($\pm 2^\circ$ C)	24 hours
Other Selected Metals (Cr, Al & Mg)	EPA SW-846 Method 3010A	EPA SW-846 Method 6010B	500 ml HDPE	HNO ₃ to pH<2 (if necessary)	6 months
Surface Tension	None	ASTM D-1331-89	50 ml glass	None	24 hours

Table 3: Aqueous Samples

PARAMETER	SAMPLE LOCATION	TEST DURATION	TEST RUNS	SAMPLING METHOD	ANALYSIS METHOD
Hexavalent Chromium	Personal Exposure	2 hours	3/Test Condition	NIOSH Method 7604	CARB Method 425
Hexavalent Chromium	Process Emissions	2 hours	3/Test Condition	Modified CARB Method 5 (Modified EPA 5)	CARB Method 425

Table 4: Air Samples

Test Data Collection Form

DATE: _____
 BATH VOLUME _____

OPERATION: Chromic Acid Bath Solution Purification
 BATH TYPE (CHECK ONE): Chromic Acid Anodizing Upstream Rinse Downstream Rinse

SAMPLE DATA				ANODIZING PROCESS PARAMETERS												
Sample #	Time	Sample Location	Initials	Temp. (°F)	pH	Flow Rate (L/hr)	Surface Tension (dynes/cm)	Temp. (°F)	pH	Flow Rate (L/hr)	Cell Amperage (amps)	Water Added (L)	Chromic Acid Added (kg)	Cell Voltage (VDC)	Amp-hours (Ah)	Waste Volume (L)

Figure 7: Test Data Collection Form

4.5.3.13 Air Sampling

The presence of airborne hexavalent chromium emissions may affect product quality as well as worker health and safety. As well as being a carcinogen, hexavalent chromium is an allergen, irritant, and sensitizer. Overexposure can lead to ulcers (chrome holes) on skin, inflammation and perforation of nasal septum, and dermatitis. The purpose of this air sampling will be to determine whether the RETEC system contributes to an increase in airborne hexavalent chromium emissions. The determination of hexavalent chrome emission fluctuation during the implementation of the RETEC system provides valuable information to project managers, technicians, operators, and risk assessors regarding the process variables and worker health and safety.

An area air sample collector will be placed in the vicinity of the anodizing process tank and the RETEC system. Samples will be collected before and after the RETEC unit is placed in operation during the fourth and ninth week of verification testing. The RETEC system technician will also be equipped with a personal air sampling device that will measure the hexavalent chromium exposure at the operator's breathing zone. Again, samples will be collected before and after the RETEC unit is placed in operation during the fourth and ninth week of verification testing. Total Air Analysis of Carson, California, will conduct the air sampling operations at DVI, and West Coast Analytical Services of Santa Fe Springs, California, will perform the air sample analysis. See *Table 4* for a summary of air sampling and analysis methods and frequencies.

4.5.3.13.1 Personal Exposure

Two hour samples will be collected. A total of three runs (samples) will be collected each sampling day. There will be two sampling days, one before and one after the RETEC unit is placed in operation during the fourth and ninth week of verification testing. The sampling will be conducted according to National Institute of Occupational Safety and Health (NIOSH) method 7604. The sampling train will consist of a SKC, Inc., personal pump with constant flow, and a cassette filter holder to house the filter. A PVC membrane from Pall Gleman Laboratory will be used to collect the hexavalent chromium according to NIOSH method 7604. The cassette filter holder and pump will be connected with Tygon tube. The pump will be attached to the waist of the worker, while the cassette filter holder will be attached to the collar of the worker.

4.5.3.13.2 Process Emissions

Two hour samples will be collected near the anodizing bath/RETEC unit. A total of three runs (samples) will be collected each sampling day. There will be two sampling days, one before and one after the

RETEC unit is placed in operation during the fourth and fifth week of verification testing. The sampling will be conducted according to a modified California Air Resources Board (CARB) method 5, which is equivalent to a modified EPA method 5 for the collection of hexavalent chromium. The modifications to method 5 include using a glass nozzle, a glass lined stainless steel probe, 0.1 N NaOH in the first two impingers, and a Teflon-coated glass fiber filter. Sample flow is through the first probe, then the impingers, and then the filter. The sampling train will consist of a control box, sampling pump, impingers, and a filter holder from Apex Instruments, Inc.

4.5.3.13.3 Sample Analysis

Both the personal and process emissions samples will be analyzed according to CARB method 425. The samples will be analyzed using Dionex Ion Chromatography (IC) System 4000i (or equivalent). The IC method has a lower detection limit than the manual colorimetric method for air sample analysis.

4.5.4 Calibration Procedures and Frequency

The following procedures will be used to calibrate the instruments/equipment that will be used to collect critical measurements:

1. Instruments used to perform analytical methods will be calibrated according to the equipment manufacturer's specifications and quality assurance plan by.
2. Instruments used to conduct air sampling and analysis will be calibrated according to equipment manufacturer's specifications and Total Air Analysis's quality assurance plan by Total Air Analysis.
3. A certified amp hour meter will be installed on the RETEC system rectifier, and will be calibrated by the equipment manufacturer, Darrah Electric Company before the start of verification testing.
4. The ultrasonic liquid flow meter, used to measure the flow rate of liquids within the anodizing/RETEC process, will be calibrated at the start of each sampling day, and in accordance with the equipment manufacturer's calibration instructions by the on-site MMTC representative.
5. The du Nouy tensiometer used to measure the anodizing bath surface tension will be calibrated at the start of each sampling day, and in accordance with the calibration instructions found in method ASTM 1331-89 by the on-site MMTC representative.
6. Temperature is automatically controlled by the anodizing tank process controls, and pH is manually controlled by the monitoring and addition of chromic acid to the anodizing bath/RETEC process. These measurements will be taken each time a sample is drawn from its

respective sampling port. The digital pH reader/ temperature probe will be calibrated at the start of each sampling day by the on-site MMTC representative.

5.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

Quality Assurance/Quality Control 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 3].

5.1 Quality Assurance Objectives

One 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. Another QA objective is to use standard test methods for laboratory analyses. The test methods to be used are shown in *Tables 2, 3 & 4*. The analytical methods that will be used for analyzing the baths, waste samples, and air samples are standard metal finishing methods, ASTM, EPA, or EPA-equivalent CARB methods.

5.2 Data Reduction, Validation, and Reporting

5.2.1 Calculation of Results

The conservation of mass/energy in any isolated system is one of the most fundamental laws in science and engineering. The mass/energy balance is a tool that was developed to account for the inputs, outputs, consumption, and accumulation in a system. To determine system efficiency, measuring or quantifying all of the elements for a mass balance in an industrial setting is very difficult. The greatest challenge is generally defining the system boundaries and what degree of accuracy is required. Sampling, measurement, and analytical errors preclude absolute precision; however, the mass/energy balance provides us with a fundamental tool for evaluating the performance of environmental technologies where we are generally evaluating some form of efficiency.

Figure 8 illustrates the most fundamental form of the material balance equation. Batch systems and continuous systems can both be modeled using this general form.

Figure 9 illustrates the material flow into and out of DVI's RETEC system.

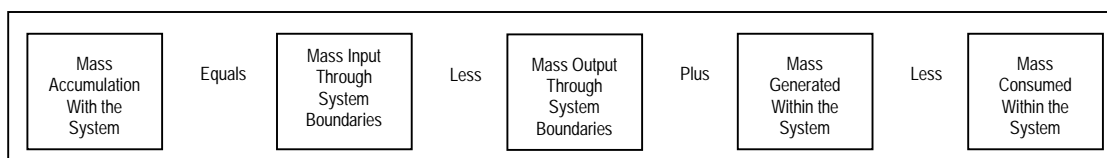


Figure 8: Fundamental Material Balance Equation

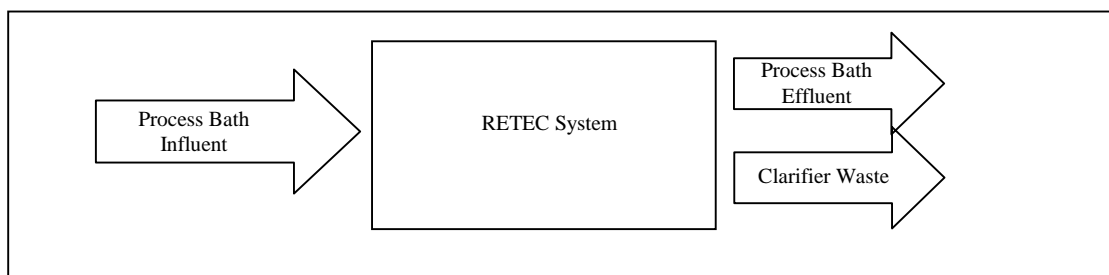


Figure 9: Material Balance Equation For DVI'S RETEC System

The goal of the USFilter verification project is to determine performance, and this can generally be measured in terms of efficiency. For solution maintenance technologies, percent contaminant removal and percent solution recovery are measures of system efficiency.

Sampling will occur at the frequency and duration specified in *Table 1*. Historical operating records at DVI indicate that when the RETEC unit is shut off, we will see an aluminum contamination increase of approximately 0.25 g/L per week in the anodizing bath. There will also be an increase of trivalent chromium in the anodizing bath as a byproduct of the hexavalent chromium deposition reaction. A five-week sampling period for each test mode was selected in order to show an appreciable increase in tramp metals as well as trivalent chromium. This is the minimum time frame in which the required analytical methods can accurately differentiate a measurable change in the testing parameter levels.

To determine the tramp metal removal efficiency, the material balance equation for the DVI RETEC system can be simplified as:

$$X_i = X_s + X_e$$

where: X_i = Mass in influent

X_s = Mass in waste

X_e = Mass in effluent

Removal efficiency can be determined by:

$$\text{Efficiency \%} = \frac{X_{i_a} - X_{e_a}}{X_{i_a}} \times 100$$

where: a = any contamination constituent 1 through n

Since the RETEC system is not a single-pass unit, but rather re-circulates the anodizing solution through the electrolyzer, there will not be a detectable difference in contamination concentrations between the influent and the effluent at any given point in time. Samples will be collected once each week for each mode of operation,

and cumulative and overall removal efficiency calculations will be determined for each time period.

For determining the recovery efficiency of converting trivalent chromium to hexavalent chromium within the DV Industries RETEC system, the material balance equation becomes a little more complex:

$$Xe = Xi_h - Xs + Xc + Xa$$

where: Xe = Mass of hexavalent chrome in effluent

Xi_h = Mass of hexavalent chrome in influent

Xs = Mass of hexavalent chrome in waste

Xc = Mass of hexavalent chrome converted from trivalent chrome
(oxidation)

Xa = Mass of hexavalent chrome accumulated (from catholyte solution)

Recovery efficiency can be determined by:

$$\text{Efficiency \%} = \frac{Xe - Xa}{Xi_h + Xi_t} \times 100$$

where: Xi_t = Mass of trivalent chrome in influent

As with the contaminant concentration, there will not be a detectable difference in chromium oxidation at any given point in time. Samples will again be collected once each week for each mode of operation, and cumulative and overall recovery efficiency calculations will be determined for each time period. This data will be presented graphically with error bars to indicate uncertainty in the verification test report.

5.2.2 Internal Quality Control Checks

Raw Data Handling. Raw data is 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 is collected both manually and electronically. At a minimum, the date, time, sample ID, instrument ID, analyst 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. Data collected on-site will be recorded in a bound laboratory notebook and transcribed onto the form presented in *Figure 7*. The on-site Project Team member will generate chain of custody forms that will accompany samples during shipment to the respective labs. 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 Program Manager will maintain process-operating data for use in report preparation.

Data Package Validation. 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.

The Laboratory Manager shall be ultimately responsible for all final data released from the laboratory. The Laboratory Manager or designee will review the final results for adequacy to task QA objectives. If the manager 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 manager 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 manager will flag the data as suspect. The Laboratory Manager signs and dates the final data package.

Data Reporting. A report signed and dated by the Laboratory Manager 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, the units and the quality control sample results. The ETV-MF Program Manager shall retain the data packages as required by the ETV-MF QMP [Ref 3].

5.2.3 Calculation of Laboratory Data Quality Indicators

Analytical performance requirements are expressed in terms of precision, accuracy, representability, comparability, completeness, and sensitivity (PARCCS). Summarized below are definitions and QA objectives for each PARCCS parameter. For total metals analysis (Cr, Al & Mg) by ICP-AES, and hexavalent and trivalent chromium measurements by colorimetric methods spikes and spike duplicates will be performed on at least one out of every ten samples for each sample parameter for each sampling day. For example, for each test mode, several different process streams will be sampled as shown in *Table 1* (SP-A through SP-H). The number of samples may vary

with each test mode, but the test parameters (total chromium, aluminum, magnesium, trivalent chromium and hexavalent chromium) will remain the same. For each sampling day at least one sample will undergo spike and spike duplicate procedures for each test parameter. Sample splitting will occur at the analytical laboratory. A blind sample duplicate will be taken from each sampling point at least once during the verification test period. This will ensure the absence of matrix interferences for each sample matrix.

For hexavalent and trivalent chromium analysis by titration, a minimum of two sample duplicates and one sample spike will be performed for each sample collected. Sample duplicate and spike results will be reported by the laboratory on the analytical test result forms. A blind sample duplicate will be sent to the laboratory for each sample matrix at least once during the verification test period for these parameters as well.

For those measurements where duplicates, spikes and spike duplicates are inappropriate (e.g. flowrate, surface tension, cell current, pH and temperature) duplicate measurements will take place at the time of sampling. A minimum of three repetitions will be conducted for each measurement for each sampling day in order to ensure compliance with the QA objectives stated in *Table 2*.

5.2.3.1 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:

$$RPD = \left\{ \frac{|X_1 - X_2|}{\frac{X_1 + X_2}{2}} \right\} \times 100$$

where: X_1 = largest of the observed values and X_2 = smallest of the 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 as specified in the standard test protocols provided in *Table 2*.

5.2.3.2 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:

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

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 the RETEC system verification testing. 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 2**.

5.2.3.3 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 the RETEC technology verification by the use of consistent methods during sampling and analysis and by traceability of standards to a reliable source.

5.2.3.4 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 greater than the value specified in *Table 2*.

5.2.3.5 Representativeness

Representativeness refers to the degree to which the data accurately and precisely represents the conditions or characteristics of the parameter represented by the data. For the purposes of this demonstration, representativeness will be achieved by presenting identical analyte samples to the specified lab(s) and executing consistent sample collection and mixing procedures.

5.2.3.6 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 definition 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:

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

where: MDL = method detection limit
 s = standard deviation of the replicate analyses
 $t_{(n-1, 1-\alpha = 0.99)}$ = students t-value for a one-sided 99% confidence level and a standard deviation estimate with n-1 degrees of freedom

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 as “unqualified” by the laboratory.]

5.3 Quality Audits

Technical System Audits. An audit will be performed during verification testing by the CTC QA Manager according to Section 2.9.3 Technical Assessments of the ETV-MF QMP [Ref 3] to ensure testing and data collection are performed according to the test plan requirements. In addition to the CTC Technical System Audit, the EPA Quality Assurance Manager may also conduct an audit to assess the quality of the verification test.

Internal Audits. In addition to the internal laboratory quality control 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 quality assurance and quality control procedures during verification testing will be performed according to Section 2.10 Quality Improvement of the ETV-MF QMP [Ref 3].

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 analysis is completed. Typically, the Laboratory Manager detects these types of non-conformances. In all cases of non-conformance, the Laboratory Manager 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 Task Leader 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.

6.0 PROJECT MANAGEMENT

6.1 Organization/Personnel Responsibilities

The ETV-MF Project Team that is headed by Concurrent Technologies Corporation (CTC) will conduct the evaluation of USFilter's RETEC system. The ETV-MF Program Manager, Donn Brown, will have responsibility for all aspects of the technology verification, including appointment of a Project Manager, making ETV-

MF Project Team personnel assignments, and coordination of technology testing. The Project Manager assigned to the RETEC verification is Chris Start of the Michigan Manufacturing Technology Center (MMTC). MMTC is one of four partner organizations under contract to CTC for the ETV-MF program. ETV-MF Partner organizations provide the expertise and technical knowledge specific to the metal finishing industry required to design, implement, oversee and publish the ETV testing activities as well as the general ETV-MF Program. In addition, Partners usually identify, research and specify all outsourced activities associated with verification activities (analytical activities, air monitoring, etc.), while CTC handles the administrative paperwork and accounting activities for these services. Mr. Start and/or his staff/subcontractors will conduct or oversee all sampling and related measurements, and insure that required laboratory QC Analyses (spikes/duplicates) are performed.

A USFilter representative (Dan Hegyan of the Hegyan Corporation) will assist in operating the system and will be on-call during the test period for response in the event of equipment problems.

Columbia Analytical Services (CAS) is responsible for performing aqueous chemical analysis of verification test samples for all rinse water samples and total metals analysis of process bath samples. CAS is accredited by the state of California, Department of Health Services, Environmental Laboratory Accreditation Program for the analyses identified in this Test Plan. Ross Fenstermaker, Project Chemist, will be the CAS laboratory point of contact.

Anachem Labs is responsible for performing aqueous chemical analysis of verification test samples from process baths for hexavalent and trivalent chromium. Anachem Labs is accredited by Boeing and A2LA to conduct the titration methods identified in this Test Plan. Navan Krishnan, Bath Chemist, will be the Anachem Labs point of contact.

Total Air Analysis will collect the air samples for this verification test. Total Air Analysis is certified by the California Air Resources Board (CARB) and the South Coast Air Quality Management District (SCAQMD) to perform sampling of the air pollutants identified in this Test Plan. Dr. Andrew Kitto, President of Total Air Analysis, will be the laboratory point of contact.

West Coast Analytical Services is responsible for performing air sample analysis of verification test samples. West Coast Analytical Services is accredited by the American Industrial Hygiene Association to perform analysis of the air pollutants identified in this Test Plan. Eric Lindsay will be the laboratory point of contact.

If, in the course of verification testing, it is necessary to modify the test plan, Chris Start will initiate the request according to the procedure in Appendix E.

The ETV-MF Project Manager and DVI 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 Pilot Manager.

6.2 Schedule/Milestones

The schedule and milestones will be determined mutually by *CTC* and DVI.

6.3 Documentation/Records

All original documentation generated during verification testing (chain of custody forms, data collection forms, analytical results, etc.) will be maintained at the *CTC* office in Largo, FL.

7.0 EQUIPMENT

7.1 Equipment List and Utility Requirements

Equipment and utility requirements are identified in *Table 5*.

7.2 Monitoring/Sampling Equipment

All monitoring/sampling equipment to be used during testing is identified in *Table 6*.

EQUIPMENT		
Number Req'd	Type of Equipment	Comments
1	Polyvinyl chloride electrolyzer box	Steel frame (anolyte compartment volume is 9.6 gal.)
7	Cathodes	Titanium expanded mesh
6	Anodes	Lead Alloy
6	Anode compartments	Polyvinyl Chloride
12	Anolyte/Catholyte separators	ELRAMIX - 2 per anode chamber
1	Rectifier	(See below)
1	Clarifier	Polypropylene - 681 L/hr
1	Pump - catholyte feed	1/16 HP - Polypropylene
1	Pump - anolyte feed	1/25 HP - Kynar
1	Pump - air sparger system (not in use)	1/8 HP
1	pH meter and electrode	
SPARE PARTS		
5	Anode replacement	
5	Cathode replacement	
2	ELRAMIX membrane replacement	
Required Utilities		
Utilities to include:		
<u>Electrical</u>		
<ul style="list-style-type: none"> Rectifier power supply: 10 VDC, 2500 Amps, 3-Phase Liquid and air transfer pumps: 110 VAC 		
<u>Air</u>		
<ul style="list-style-type: none"> Air sparger system: 22 L/min (not in use) 		

Table 5: Equipment List And Utility Requirements

Equipment	Purpose
Amp/Voltage Meter	Electrolytic Cell Amps and Volts DC
Darrah Electric Company, A.H.M. 500 AMP, 50 Milli-volt Ampere Hour Meter	Electrolytic Cell Cumulative Amp-Hours
Omega Engineering, Inc. Model FD-7000 Multi-Liquid Ultrasonic Flowmeter	Flow of System Liquids
Central Scientific Company, Inc., du Nouy Tensiometer	Surface Tension
Davis Instruments Model # 9214 ATC pH Meter with Integral Temperature Sensor	Process & Rinse Bath pH and Temperature
SKC, Inc., Constant Flow Personal Pump & Cassette Filter Holder w/ Pall Gelman Laboratory PVC Membrane	Personal Exposure to Airborne Hexavalent Chromium
Apex Instruments, Inc., Sampling Pump, Control Box, Impingers & Filter Holder	Process Emissions for Airborne Hexavalent Chromium

Table 6: Monitoring/Sampling Equipment

8.0 HEALTH AND SAFETY PLAN

This Health and Safety Plan provides guidelines for recognizing, evaluating, and controlling health and physical hazards that could occur during verification testing. More specifically, the Plan specifies for assigned personnel; the training, materials, and equipment necessary to protect them from hazards; and any waste generated. The DVI Hazcom/PPE Plan/Program will be used throughout the RETEC verification testing.

8.1 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 during verification testing. Hazard communication will take place during training and will be reinforced throughout the test period. All appropriate MSDS's will be available for the chemical solutions used during the testing.

8.2 Emergency Response Plan

DVI 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.

8.3 Hazard Controls Including Personal Protective Equipment

All assigned project personnel and visitors 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.

8.4 Lockout/Tagout Program

DVI's lockout/tagout procedure will be implemented when necessary and will be explained to anyone required to perform such duties.

8.5 Material Storage

Any materials used during the project will be kept in proper containers and labeled according to Federal and State law. 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.

8.6 Safe Handling Procedures

All chemicals and wastes or samples will be transported on-site in non-breakable containers used to prevent spills. Emergency spill clean-up will be performed according to DVI procedures.

9.0 WASTE MANAGEMENT

The equipment will be tested on processes already in-place and operating at DVI. Any wastes that may be generated will be no different than those already generated at DVI. Therefore, no special or additional provisions for waste management will be necessary.

10.0 TRAINING

It is important that the verification activities performed by the ETV-MF Pilot 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. 4].

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 Pilot. The JTA Plan describes the method for studying ETV-MF project activity and identifying training needs. The ETV-MF Operation Planning Checklist (*Appendix B*) will be used as a guideline for identifying

potential hazards, and the Job Training Analysis Form (*Appendix C*) 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 D*).

11.0 REFERENCES

1. US EPA Office of Research and Development, “*Waste Reduction in the Metal Fabricated Products Industry*” EPA/600/SR-93/144, September 1993.
2. George C. Cushnie Jr., CAI Engineering, “*Pollution Prevention and Control Technology for Plating Operations*” NCMS/NAMF, 1994.
3. Concurrent Technologies Corporation (CTC), “*Environmental Technology Verification Program Metal Finishing Technologies (ETV-MF) Quality Management Plan*” December 9, 1998.
4. Concurrent Technologies Corporation (CTC), “*Environmental Technology Verification Program Metal Finishing Technologies (ETV-MF) Pollution Prevention Technologies Pilot Job Training Analysis Plan*” May 10, 1999.

12.0 DISTRIBUTION

Alva Daniels, EPA (3)

Mike Chan, USFilter Corporation

Tom Davis, DV Industries, Inc.

Chris Start, MMTC (2)

Donn Brown, CTC (3)

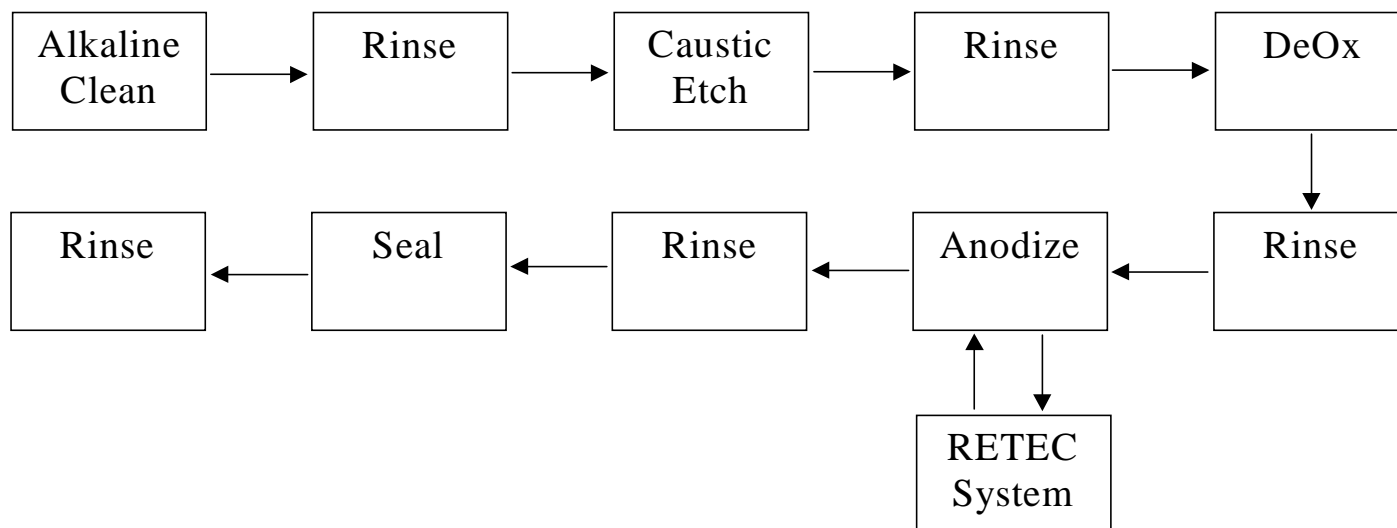
Clinton Twilley, CTC

APPENDIX A

DV Industries, Inc. Anodizing Process Flow

DV Industries, Inc.

Anodizing Process Flow



APPENDIX B

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

1. 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?) <i>Special Procedures for emergency shut-down must be documented in Test Plan.</i>			
2. Equipment requiring special fire prevention precautions? (e.g., Class D fire extinguishers)			
3. Modifications to or impairment of building fire alarms, smoke detectors, sprinklers, or other fire protection or suppression systems?			
4. Equipment lockout/tagout or potential for dangerous energy release? <i>Lockout/tagout requirements must be documented in Test Plan.</i>			
5. Working in or near confined spaces (e.g., tanks, floor pits) or in cramped quarters?			
6. Personal protection from heat, cold, chemical splashes, abrasions, etc.? <i>Use Personal Protective Equipment Program specified in Test Plan.</i>			
7. Airborne dusts, mists, vapors and/or fumes? <i>Air monitoring, respiratory protection, and /or medical surveillance may be needed.</i>			
8. Noise levels greater than 80 decibels? <i>Noise surveys are required. Hearing protection and associated medical surveillance may be necessary.</i>			
9. X-rays or radiation sources? <i>Notification to the state and exposure monitoring may be necessary.</i>			
10. Welding, arc/torch cutting, or other operations that generate flames and/or sparks outside of designated weld areas? <i>Follow Hot Work Permit Procedures identified in Test Plan.</i>			
11. The use of hazardous chemicals? <i>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.</i>			
12. 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**

13. Processing or recycling of hazardous wastes? <i>Special permitting may be required.</i>			
14. Generation or handling of waste?			
15. Work to be conducted before 7:00 a.m., after 6:00 p.m., and/or on weekends? <i>Two people must always be in the work area together.</i>			
16. Contractors working in CTC facilities? <i>Follow Hazard Communication Program.</i>			
17. Potential discharge of wastewater pollutants?			
18. EHS aspects/impacts and legal and other requirements identified?			
19. Contaminants exhausted either to the environment or into buildings? <i>Special permitting or air pollution control devices may be necessary.</i>			
20. Any other hazards not identified above? (e.g., lasers, robots, syringes) <i>Please indicate with an attached list.</i>			

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)

US EPA ARCHIVE DOCUMENT

APPENDIX C

Job Training Analysis Form

US EPA ARCHIVE DOCUMENT

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

US EPA ARCHIVE DOCUMENT

APPENDIX D

ETV-MF Project Training Attendance Form

ETV-MF Project Training Attendance Form

ETV-MF Pilot Project: _____

Date Training Completed	Employee Name Last First	Training Topic

ETV-MF Project Manager: _____

US EPA ARCHIVE DOCUMENT

APPENDIX E

Test Plan Modification

Test Plan Modification Requests

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 insure 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. The *CTC* ETV-MF Program Manager 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 ETV-MF Project Manager (Partner). The ETV-MF Project Manager (Partner) should sign both lines if he is the requestor. 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: _____

ETV-MF Project Manager: _____

CTC ETV-MF Program Manager: _____

US EPA ARCHIVE DOCUMENT