# FINAL REPORT

## Non-Chromate, ZVOC Coatings for Steel Substrates on Army and Navy Aircraft and Ground Vehicles: Non-Chromate Sealers for Zinc Phosphate

## ESTCP Project WP-200906

FEBRUARY 2015


Jack Kelley
Tom Braswell
Tom Considine
**U.S Army Research Laboratory**

Luwam Hagos
Amy Fowler
**Naval Air Systems Command**

Keith Legg
**Rowan Technology Group**



*Distribution Statement A*

*This document has been cleared for public release*

# REPORT DOCUMENTATION PAGE

*Form Approved*
*OMB No. 0704-0188*

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. **PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.**

**1. REPORT DATE** *(DD-MM-YYYY)*

**2. REPORT TYPE**
Final Report

**3. DATES COVERED** *(From - To)*
2009-2012

**4. TITLE AND SUBTITLE**
Final report
"Non-chromate Sealers for Zinc Phosphate"

**5a. CONTRACT NUMBER**

**5b. GRANT NUMBER**

**5c. PROGRAM ELEMENT NUMBER**

**6. AUTHOR(S)**
Jack Kelley, Tom Braswell (US ARL) Amy Fowler, Luwam Hagos (NAVAIR)

**5d. PROJECT NUMBER**
WP200906

**5e. TASK NUMBER**

**5f. WORK UNIT NUMBER**

**7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)**
US Army Research Laboratory

**8. PERFORMING ORGANIZATION REPORT NUMBER**

**9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)**

**10. SPONSOR/MONITOR'S ACRONYM(S)**

**11. SPONSOR/MONITOR'S REPORT NUMBER(S)**

**12. DISTRIBUTION / AVAILABILITY STATEMENT**
DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited

**13. SUPPLEMENTARY NOTES**

**14. ABSTRACT**
The objective of this demonstration/validation was to determine the use and viability of non-hexavalent chrome sealers for zinc phosphate as a base for organic coatings. By itself zinc phosphate does not provide adequate corrosion protection. It is a porous coating that requires a sealer to enhance the corrosion protection and provide a good surface for paint adhesion. Currently zinc phosphate requires a chromate seal post treatment and the health and environmental risks associated with hexavalent chromium based compounds have necessitated reductions and even eliminations of their usage on U.S. Army weapon systems. U.S. Army Research Laboratory conducted laboratory tests on two alternative sealers and demonstrated the best performed at Anniston Army Depot on armored vehicle and miscellaneous parts. Selected parts processed at Anniston were evaluated at ARL. The results showed that the nonchromate alternative had comparable performance to the chromate baseline. In some cases, the alternatives outperformed the baseline in laboratory tests. The demonstrated product is approved for use IAW TT-C-490.

**15. SUBJECT TERMS**

| **16. SECURITY CLASSIFICATION OF:** | | | **17. LIMITATION OF ABSTRACT** | **18. NUMBER OF PAGES** | **19a. NAME OF RESPONSIBLE PERSON** |
|---|---|---|---|---|---|
| | **b. ABSTRACT** | **c. THIS PAGE** | | | John Kelley |
| | U | U | U | 64 | **19b. TELEPHONE NUMBER** *(include area code)* 410-306-0837 |

**Standard Form 298 (Rev. 8-98)**
**Prescribed by ANSI Std. Z39.18**

## TABLE OF CONTENTS


TABLE OF FIGURES ..... 3
TABLE OF TABLES ..... 5
LIST OF ACRONYMS ..... 6

**1.0 INTRODUCTION ..... 7**
1.1 BACKGROUND ..... 7
1.2 OBJECTIVE OF THE DEMONSTRATION ..... 9
1.3 REGULATORY DRIVERS ..... 10
1.4 STAKEHOLDER/END-USER ISSUES ..... 11

**2.0 TECHNOLOGY ..... 12**
2.1 TECHNOLOGY DESCRIPTION: SurTec 580 (TCP) ..... 12
2.2 TECHNOLOGY DESCRIPTION: PPG Chemseal 100 ..... 14
2.3 TECHNOLOGY DEVELOPMENT: SurTec 580 (TCP) ..... 14
2.4 TECHNOLOGY DEVELOPMENT: PPG Chemseal 100 ..... 16
2.5 ADVANTAGES AND LIMITATIONS OF THE TECHNOLGY ..... 16

**3.0 PERFORMANCE OBJECTIVES ..... 18**

**4.0 SITES/PLATFORM DESCRIPTION ..... 20**
4.1 TEST PLATFORMS/FACILITIES ..... 20
4.2 PRESENT OPERATIONS ..... 21
4.3 SITE-RELATED PERMITS AND REGULATIONS ..... 22

**5.0 TEST DESIGN ..... 23**
5.1 CONCEPTUAL EXPERIMENTAL DESIGN ..... 23
5.2 Laboratory Experimental Procedure ..... 23
5.3 PHOSPHATE LINE COMPONENT DEMONSTRATION ..... 28
5.4 FIELD TESTING ..... 30

**6.0 PERFORMANCE ASSESSMENT ..... 33**
6.1 Laboratory Results ..... 33
6.2 Component Demonstration Results ..... 47

**7.0 COST ASSESSMENT ..... 54**

**8.0 IMPLEMENTATION ..... 59**

**9.0 REFERENCES ..... 61**

**APPENDICES ..... 63**
Appendix A: Joint Test Protocol ..... 63
Appendix B: Points of Contact ..... 107

## TABLE OF FIGURES

FIGURE 1-1: MILD STEEL ZINC PHOSPHATE SURFACE AT 500X ILLUSTRATES THE POROSITY OF THE CONVERSION COATING AND THE NEED FOR A SEALER. ..... 7
FIGURE 1-2: FUEL CAPS IN THE REFINISHING LINE AT ANAD. ..... 10
FIGURE 2-1: SCHEMATIC OF A PHOSPHATED METAL SURFACE SHOWING EXPOSED METAL SURFACE AT THE BASE OF THE PORES. ..... 13
FIGURE 2-2: VIEW OF THE REACTIONS THAT OCCUR AT THE EXPOSED METAL BETWEEN THE PHOSPHATE CRYSTALS. ..... 13
FIGURE 2-3: FLOW DIAGRAM FOR SURTEC 580 MANUFACTURER RECOMMENDED PROCESS STEPS 14
FIGURE 2-4: POLYMERIZATION OF EPOXY PRECURSORS WITH AMINE CATALYST TO FORM THIN-FILM ..... 14
MATRIX. ..... 14
FIGURE 4-1: M1 TANKS LINING THE RAILROAD TRACK AT ANAD ON THEIR WAY BACK TO SOLDIERS AFTER BEING REPAIRED AND OVERHAULED BY DEPOT PERSONNEL. ..... 20
FIGURE 4-2: FLOW DIAGRAM OF A TYPICAL PHOSPHATE LINE CURRENTLY EMPLOYED AT ANAD. 21
FIGURE 5-1: ELCOMETER MODEL 108 HYDRAULIC ADHESION TEST EQUIPMENT (HATE) PULL-OFF TEST APPARATUS. ..... 24
FIGURE 5-2: CROSS-CUT AREA RATINGS ..... 25
FIGURE 5-3: EXAMPLE OF THE Q-LAB GRAVELOMETER USED TO MEASURE CHIP RESISTANCE PER SAE J400 AND THE AREA OF A PANEL EVALUATED. ..... 26
FIGURE 5-4: SURTEC 580 VAT NEAR PHOSPHATE LINE USED IN DEMONSTRATION ..... 28
FIGURE 5-5: M1 ABRAMS FUEL CAPS AND TEST PANELS ZINC PHOSPHATE AND SEALED ..... 29
FIGURE 5-6: STRYKER CUPOLA RISING OUT OF THE SURTEC 580 VAT AFTER SEAL ..... 29
FIGURE 5-7: ROUTING TAG INDICATING ALL STEPS REQUIRED TO RECONDITION FUEL CAPS ..... 30
FIGURE 6-1: PULL-OFF AND DRY TAPE ADHESION STRENGTH ON MILLED FINISH LCS ..... 34
FIGURE 6-2: PULL-OFF AND DRY TAPE ADHESION STRENGTH ON ABRASIVE BLASTED HHA ..... 34
FIGURE 6-3: ASTM-D3359A RATINGS FOR WET TAPE ADHESION FOR LCS AT 24 HOURS IMMERSION ..... 35
FIGURE 6-4: ASTM-D3359A RATINGS FOR WET TAPE ADHESION FOR ABRASIVE BLASTED HHA AT 24 HOURS IMMERSION ..... 35
FIGURE 6-5: ASTM D 1654 RATINGS FOR LOW CARBON STEEL PANELS THROUGH 1000 HOURS OF ASTM B117 SALT FOG EXPOSURE ..... 37
FIGURE 6-6: ASTM D 1654 RATINGS FOR ABRASIVE BLASTED HHA PANELS THROUGH 1000 HOURS OF ASTM B117 SALT FOG EXPOSURE ..... 38
FIGURE 6-8: LOW CARBON STEEL WITH MIL-DTL-53022/MIL-DTL-53039 SCRAPED AFTER 1000 HRS B117 EXPOSURE. PERFORMANCE RATINGS DETERMINED BY CREEP-BACK FROM SCRIBE. . 38
FIGURE 6-8: ABRASIVE BLASTED HHA WITH MIL-DTL-53022/MIL-DTL-53039 SCRAPED AFTER 1000 HRS B117 EXPOSURE. PERFORMANCE RATINGS DETERMINED BY CREEP-BACK FROM SCRIBE. ..... 39
FIGURE 6-9: LOW CARBON STEEL WITH MIL-DTL-53022/MIL-DTL-53039, SCRAPED AFTER 80 CYCLES OF GM 9540P EXPOSURE ..... 40
FIGURE 6-10: ABRASIVE BLASTED HHA WITH MIL-DTL-53022/MIL-DTL-53039, SCRAPED AFTER 80 CYCLES OF GM 9540P EXPOSURE ..... 41
FIGURE 6-11: LOW CARBON STEEL WITH MIL-DTL-53022/MIL-DTL-64159, SCRAPED AFTER 80 CYCLES OF GM 9540P EXPOSURE ..... 42

FIGURE 6-12: ABRASIVE BLASTED HHA WITH MIL-DTL-53022/MIL-DTL-64159, SCRAPED AFTER 80 CYCLES OF GM 9540P EXPOSURE ..... 42
FIGURE 6-13: LOW CARBON STEEL PANELS WITH MIL-DTL-53022/MIL-DTL-53039, WITH RATINGS INDICATED AFTER 3 YEARS IN OUTDOOR EXPOSURE AT CAPE CANAVERAL ..... 43
FIGURE 6-14: LOW CARBON STEEL PANELS WITH MIL-DTL-53022/MIL-DTL-64159, WITH RATINGS INDICATED AFTER 3YEARS IN OUTDOOR EXPOSURE AT CAPE CANAVERAL ..... 44
FIGURE 6-15: ASTM D1654 RATINGS WITH STANDARD DEVIATION FOR LCS AFTER 3 YEARS IN OUTDOOR EXPOSURE AT CAPE CANAVERAL ..... 44
FIGURE 6-16: ABRASIVE BLASTED HHA PANELS WITH MIL-DTL-53022/MIL-DTL-53039, WITH RATINGS INDICATED AFTER 3 YEARS IN OUTDOOR EXPOSURE AT CAPE CANAVERAL ..... 45
FIGURE 6-17: ABRASIVE BLASTED HHA PANELS WITH MIL-DTL-53022/MIL-DTL-64159, WITH RATINGS INDICATED AFTER 3 YEARS IN OUTDOOR EXPOSURE AT CAPE CANAVERAL ..... 45
FIGURE 6-18: ASTM D1654 RATINGS WITH STANDARD DEVIATION FOR HHA AFTER 3 YEARS IN OUTDOOR EXPOSURE AT CAPE CANAVERAL ..... 46
FIGURE 6-19: AVERAGE $K_{1EAC}$ VALUES OF MIL-A-46100 COMPARED TO HISTORICAL RANGE ..... 46
FIGURE 6-20: ANAD PHOSPHATE PROCESS WITH SURTEC 580 MODIFICATION. ..... 47
FIGURE 6-21: ARL M1 FUEL CAPS IMMEDIATELY FOLLOWING ZINC PHOSPHATE AND SEALING AT ANNISTON ARMY DEPOT ..... 48
FIGURE 6-22: PICTURES SHOWING BOTH SIDES OF THE ARL M1 FUEL CAPS AFTER PROCESSING AT ANNISTON ARMY DEPOT ..... 48
FIGURE 6-23: CLOSE UP VIEW OF THE CUPOLA (FIGURE 5-6). ON THE LEFT SHOWS FLASH RUST WHEN COLD RINSED AND NO FLASH RUSTING WHEN HOT RINSED ..... 49
FIGURE 6-24: REPRESENTATIVE STEEL TEST PANELS FROM ANAD TESTED THROUGH 1008 HOURS OF ASTM B117 SALT SPRAY. CAPTIONS CONTAIN THE ASTM D 1654 RATING ..... 50
FIGURE 6-25: REPRESENTATIVE CRS PANELS AFTER 60 CYCLES OF GM 9540P CYCLIC CORROSION TEST. CAPTIONS CONTAIN ASTM D 1654 RATING ..... 50
FIGURE 6-26: REPRESENTATIVE HHA STEEL PANELS AFTER 60 CYCLES OF GM 9540P CYCLIC CORROSION TEST. CAPTIONS CONTAIN ASTM D 1654 RATING ..... 50
FIGURE 6-27: REPRESENTATIVE M1 ARMOR STEEL FUEL CAPS AFTER 60 CYCLES OF GM 9540P CYCLIC CORROSION TEST WITH SSPC VIS 2 RATINGS ..... 51
FIGURE 6-28: REWORKED M1 ARMOR STEEL FUEL CAPS AFTER 40 CYCLES OF GM 9540P CYCLIC CORROSION TEST. ..... 52
FIGURE 7-1: CHROMIC ACID RINSE TANK IN PHOSPHATE LINE AT (ANAD). ..... 54
FIGURE 7-2: TOP: CURRENT PROCESS - ZINC PHOSPHATING AND CHROMIC ACID SEALING. BOTTOM – CHROMATE-FREE SEALER REPLACEMENT ..... 55
FIGURE 7-3: DIRECT COST OF CHROMATE PROCESSING AS FUNCTION OF BATH TEMPERATURE AND HEATING EFFICIENCY ..... 57

## TABLE OF TABLES

TABLE 1-1: TARGET HAZARDOUS MATERIAL (HAZMAT) SUMMARY. ..... 9
TABLE 3-1 PERFORMANCE OBJECTIVES FOR ALTERNATIVE SEALERS FOR ZINC PHOSPHATE ..... 19
TABLE 5-1: TEST MATRIX FOR BOTH STEEL SUBSTRATES ..... 23
TABLE 5-2: SAE J400 RATINGS FOR NUMBER OF CHIPS IN A 4 INCH X 4 INCH AREA..... 26
TABLE 5-3: SAE J400 RATINGS FOR SIZE OF CHIPS IN A 4 INCH X 4 INCH AREA ..... 26
TABLE 5-4: CYCLE DETAILS FOR THE GM 9540P CYCLIC CORROSION TEST ..... 27
TABLE 5-5: LIST PARTS AND TEST PANELS PROCESSED DURING DEMONSTRATION AT ANAD ..... 30
TABLE 5-6: SSPC-VIS 2 RATINGS FOR PERCENT CORROSION OF PAINTED SURFACES. ..... 32
TABLE 6-1: CHIP RESISTANCE OF PRETREATMENTS ON ABRASIVE BLASTED HHA STEEL ..... 36
TABLE 6-2: ASTM-D1654 RATINGS FOR GM9540P CYCLIC CORROSION TEST OF MIL-DTL-53022/MIL-DTL-53039 COATED STEEL ..... 40
TABLE 6-3: ASTM-D1654 RATINGS FOR GM9540P CYCLIC CORROSION TEST OF MIL-DTL-53022/MIL-DTL-64159 COATED STEEL ..... 41
TABLE 6-4: SUMMARY OF AVERAGE RATINGS AT 60 CYCLES OF GM9540P INDICATING SUCCESS CRITERIA ..... 43
TABLE 6-5: SUMMARY OF AVERAGE ASTM 1654 RATINGS AFTER 60 CYCLES OF GM 9540P EXPOSURE ..... 51
TABLE 6-6: VALIDATION METHODS AND EXPECTED PERFORMANCE METRICS ..... 53
TABLE 7-1: COST FACTORS – CHROMIC ACID SEAL VS. SURTEC 580 ..... 56
TABLE 7-2: CALCULATION OF EVAPORATION AND WATER USAGE..... 58

## LIST OF ACRONYMS

| | |
|---|---|
| ANAD | Anniston Army Depot |
| APG | Aberdeen Proving Grounds |
| ARL | Army Research Laboratory |
| ASTM | American Society of Testing and Materials |
| CARC | Chemical Agent Resistant Coating |
| CCPE | Corrosion Control and Prevention Executive |
| CTC | Concurrent Technologies Corporation |
| DoD | Department of Defense |
| DRCF | Depot Repair Cycle Float |
| EAC | Environmentally Assisted Cracking |
| ECAM | Environmental Cost Analysis Methodology |
| ESTCP | Environmental Security Technology Certification Program |
| FY | Fiscal Year |
| HHA | High Hard Armor |
| IR | Infrared |
| JTP | Joint Test Protocol |
| LCS | Low Carbon Steel |
| MRAP | Mine Resistant Ambush Protected Armored Vehicles |
| MSDS | Material Safety Data Sheet |
| NAVAIR | Naval Air Warfare Center |
| NDCEE | National Defense Center for Energy and Environment |
| NSN | National Stock Number |
| OEM | Original equipment manufacturer |
| OQE | Objective Quality Evidence |
| OSD | Office of the Secretary of Defense |
| OSHA | Occupational Safety and Health Administration |
| PEL | Permissible Exposure Limit |
| PMO | Program Managers Office |
| PPG | Pittsburgh Plate Glass industries |
| SAE | Society of Automotive Engineers |
| SCC | Stress Corrosion Cracking |
| SERDP | Strategic Environmental Research and Development Program |
| SPOTA | Sustainable Painting Operations for Total Army |
| SSPC | Society for Protective Coatings |
| TCP | Trivalent Chrome Pretreatment |
| VOC | Volatile Organic Compound |
| ZVOC | Zero-Volatile Organic Compound |

# 1.0 INTRODUCTION

## 1.1 BACKGROUND

There are three basic types of phosphate coatings: Iron (TT-C-490 type II or IV), Zinc (TT-C-490 type I)[1] and heavy Zinc or Manganese phosphate (DoD-P-16232)[2]. Zinc Phosphate is a steel conversion coating that provides a crystal structure that is often used as a base for organic coatings such as paints and powder coatings. The zinc phosphate coating by itself does not provide adequate corrosion protection. It is a porous coating that is also commonly used to absorb lubricant like oils and waxes to reduce friction is some applications. Figure 1-1 gives an example of what the surface morphology of zinc phosphate steel would look like. Although a porous surface allows for better absorption of lubricants and for increased adherence to paint, the pores present many pathways that allow for corrosives to attack the base metal substrate. To enhance the corrosion protection and provide a good surface for paint to adhere, a sealer or rinse in necessary. Prior to painting, zinc phosphate is typically sealed with a chromate sealer using chromic acid. The associated health and environmental risks associated with hexavalent chromium based compounds have necessitated reductions and even eliminations of usage of these compounds on U.S. Army weapon systems. These compounds are primarily associated with pretreatments and conversion coatings such as phosphates and electroplated zinc that is applied prior to the primers and topcoats that make up the Chemical Agent Resistant Coating (CARC).

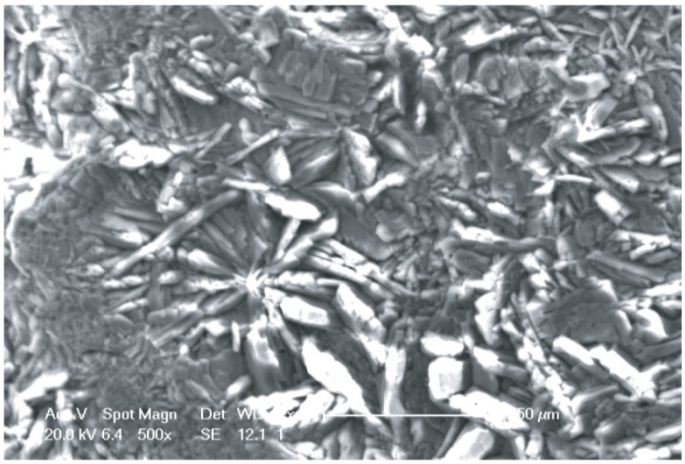



**Figure 1-1: Mild steel zinc phosphate surface at 500X illustrates the porosity of the conversion coating and the need for a sealer.[3]**

Significant progress has been made during the execution of SERDP Project WP1521, "Non-Chromate, Non-VOC Coating Systems for DoD Applications." This project was completed in FY 2008 and assessed a number of promising coatings and pretreatments in the laboratory by themselves or in combination with each other, with the ultimate goal of elimination and/or reduction of VOCs and hexavalent chromium based processes. However, these systems require additional demonstration on Army weapons systems before they can be considered ready for full implementation. The purpose of this demonstration was to validate the early successes of the

SERDP project by sealing zinc phosphate parts with a non-hexavalent chrome sealer. The intent is to indentify one or more replacements for the chromic acid sealer for zinc phosphate.

## 1.2 OBJECTIVE OF THE DEMONSTRATION

Although the overall goal of this project is to investigate non-chromate zero-volatile organic compound (ZVOC) coatings for steel substrates, the specific objective of this demonstration is to determine the viability of a non-hexavalent chrome sealer for zinc phosphate conversion coated steel. A successful demonstration of a non-hexavalent chrome sealer will reduce and eventually eliminate the use of hexavalent chrome in the phosphating process.

As mentioned earlier, zinc phosphate by itself does not provide adequate corrosion protection. Zinc phosphate (TT-C-490) is a porous coating that requires a sealer to enhance the corrosion protection and provide a good surface for paint adhesion. Currently, zinc phosphate is usually sealed with a chromate sealer using chromic acid. The health and environmental risks associated with hexavalent chromium based compounds have necessitated reductions and even eliminations of their usage on U.S. Army weapon systems.

Table 1-1 summarizes the hazards targeted and components that will be used for the demonstration of an alternative sealer for zinc phosphate. To validate performance of the proposed coating systems, The Army Research Laboratory (ARL) was given the opportunity to modify an existing zinc phosphate production line at Anniston Army Depot (ANAD). The phosphating phase of the conversion coating was carried out as usual with the exception of the chromic acid seal. The sealing of these parts was carried out using the alternative non-hexavalent chrome SurTec 580. All of the SurTec 580 sealed parts were logged for tracking purposes.

**Table 1-1: Target Hazardous Material (HazMat) Summary.**

| Target HazMat | Current Process | Applications | Current Specifications | Affected Programs | Candidate Parts and Substrates |
|---|---|---|---|---|---|
| Hexavalent Chromium | Zinc Phosphate with chromic acid seal | Steel and armor steel substrates | TT-C-490 SSPC-SP10 MIL-DTL-53072[4] | All military systems using zinc phosphate | NSN 5340-01-460-5277, Cover Access (Fuel Cap), Stryker Cupola |

An example of one of the parts used in the demonstration can be seen before phosphating in figure 1-2. These are steel fuel caps NSN 5340-01-460-5277 that are mounted on the outside of the vehicle and exposed to environmental elements.

**Figure 1-2: Fuel caps in the refinishing line at ANAD.**

## 1.3 REGULATORY DRIVERS

The Occupational Safety and Health Administration (OSHA) Final Rules effective May 30, 2006, Federal Register #71:10099-10385 states in part that OSHA has amended the standard limiting occupational exposure to hexavalent chromium ($Cr^{6+}$). OSHA has determined that at the current permissible exposure limit (PEL) for $Cr^{6+}$ workers face is a significant risk to material impairment of their health. The evidence in the record for this rulemaking indicates that workers exposed to $Cr^{6+}$ are at an increased risk of developing lung cancer. The record also indicates that occupational exposure to $Cr^{6+}$ may result in asthma and damage to the nasal epithelia and skin. The final rule establishes an 8-hour time-weighted average (TWA) exposure limit of 5 micrograms of $Cr^{6+}$ per cubic meter of air (5 $\mu g/m^3$). This is a considerable reduction from the previous PEL of 1 milligram per 10 cubic meters of air (1 mg/10 $m^3$, or 100 $\mu g/m^3$) reported as $CrO_3$, which is equivalent to a limit of 52 $\mu g/m^3$ as $Cr^{6+}$. The final rule also contains ancillary provisions for worker protection such as requirements for exposure determination, preferred exposure control methods, including a compliance alternative for a small sector for which the new PEL is infeasible, respiratory protection, protective clothing and equipment, hygiene areas and practices, medical surveillance, recordkeeping, and start-up dates that include four years for the implementation of engineering controls to meet the PEL. The PEL established by this rule reduces the significant risk posed to workers by occupational exposure to $Cr^{6+}$ to the maximum extent that is technologically and economically feasible.

In April of 2009, a memo from OSD signed by Mr. Young was released outlining a new policy for reducing the use of $Cr^{6+}$ for DOD applications. The memo specifically directs the military to do the following:

- Approve the use of alternatives where they can perform adequately for the intended application and operating environment.
- Update relevant technical documents and specifications to authorize the use of qualified alternatives.

- Require Program Executive Office (PEO) or equivalent in coordination with the Military Department's Corrosion Control and Prevention Executive (CCPE) to certify that there is no acceptable alternative to the use of $Cr^{6+}$ on a new system taking into account several factors including cost effectiveness of alternative ·materials or processes, environment, safety, and occupational health risks, corrosion performance difference of alternative materials or processes as determined by agency corrosion subject matter experts.

Effectively, the memo directs DOD Military Departments to restrict the use of $Cr^{6+}$ unless no cost-effective alternative with satisfactory performance has been identified.

## 1.4 STAKEHOLDER/END-USER ISSUES

The process has the potential to be transitioned to any DOD facility that processes steel based systems, as well as to Original Equipment Manufacturers (OEM) and their subcontractors. The business case for each location will have to be completed depending on the process and coatings of interest. Benefits to the stakeholders include the reduction or elimination of $Cr^{6+}$ in the zinc phosphating process. This demonstration plan will benefit all military systems utilizing zinc phosphate as a pretreatment for steel as a base for organic coatings.

The primary challenges facing those users who continue to use chromic acid sealers for zinc phosphate is to find effective, low cost, easy to use (drop-in replacements) for their existing process. An alternative that would require very little modification to their facilities would be desirable. SurTec 580 has been proven to be effective in a laboratory environment using the performance requirements in table 3-1. However, other factors have been investigated during the demonstration: ease of use, process time, and toxicity. The objective is to provide the end-user with the relevant data to make an informed decision prior to implementation.

Moreover, ARL has revised TT-C-490 (revision F) which now provides more detail and guidance in cleaning and processing of metallic substrates. It describes the value for objective quality evidence (OQE) during each phase of processing to assure compliance to the standards set forth in the document and the requirements designed into the supplier's materials. These OQE will be collected during the demonstration to ensure that the SurTec 580 is being used as specified.

The requirements listed in Table 3-1 parallels TT-C-490 and MIL-DTL-53072. Therefore the materials and processes have undergone a thorough review during this demonstration and will be well into the approval process at ARL for acceptance on the TT-C-490 product QPL.

# 2.0 TECHNOLOGY

The alternative sealers tested, Surtec 580 and Chemseal 100, can be used as drop-in replacements for chromate sealers for zinc phosphate. The actual application of the alternative is similar to using chromic acid rinse with some advantages. In addition to being non-chromate, both processes are carried out at ambient temperature. This reduces the utility burden on the facility. No capital improvements or modifications to the existing phosphate lines are needed to accommodate either of the technologies described below.

## 2.1 TECHNOLOGY DESCRIPTION: SurTec 580 (TCP)

The Trivalent Chromium Process (TCP) was developed by NAVAIR in an effort to replace chromated sealers, post-treatments, and conversion coatings. It was subsequently evaluated as part of SERDP project WP-1521 "Non-Chromate/No VOC Coating System for DOD Applications"[5] for use on steel. The majority of non-chromate conversion coating work thus far has focused on the use of TCP on aluminum alloys. In recent years TCP has exhibited success for use on aluminum substrates. However, for steel alloys and phosphated surfaces, further development is needed. One of the key advantages to using TCP is that the processing and maintenance requirements are similar to currently used technologies, thus making them favorable alternatives for depots and OEMs. This transition eliminates the need for additional training of personnel and large equipment purchases. TCP is based on a fluorozirconate complex with a trivalent chromium salt. TCP contains significantly less total chromium than the current hexavalent chromium conversion coatings and has no hexavalent chromium. The use of TCP eliminates personnel exposure to hexavalent chromium thus saving labor reporting costs associated with PPE and worker safety regulations. Additionally, it saves time and money by eliminating the need to treat the waste stream for hexavalent chromium.

Through the prior effort funded by SERDP, it was established that TCP forms predominately zirconium oxide/fluoride, chromium oxide conversion coating on the aluminum alloy surface. Previous work has been conducted on hexavalent chromium films suggesting a film backbone consisting of a polymerized trivalent chromium hydroxide species with a loosely hydrogen-bonded active chromate inhibitor species. Chromate films tend to be very thin over precipitates and inter-metallics, only releasing the inhibitor species after the film has broken down and substrate metal is exposed. Electrochemical evidence suggests that the TCP forms a much more uniform film thickness across these inter-metallic sites with improved barrier coating properties from the denser zirconium oxide and localized corrosion inhibition through the ability of the trivalent chromium species to bind up attacking anions such as chlorides.

The technology demonstrated, SurTec 580, is a trivalent chrome passivating product that has been modified from the original SurTec 650 TCP for use as a post-treatment for phosphating prior to the application of organic coatings. It intensifies phosphate layers by filling the pores and enhances the adhesion of the primer to the substrate thereby improving the corrosion protection and preventing under film corrosion. Figure 2-1 illustrates the phosphate crystals and the characteristic porosity. The bare metal (in this case steel) is exposed making it vulnerable to corrosive attack.

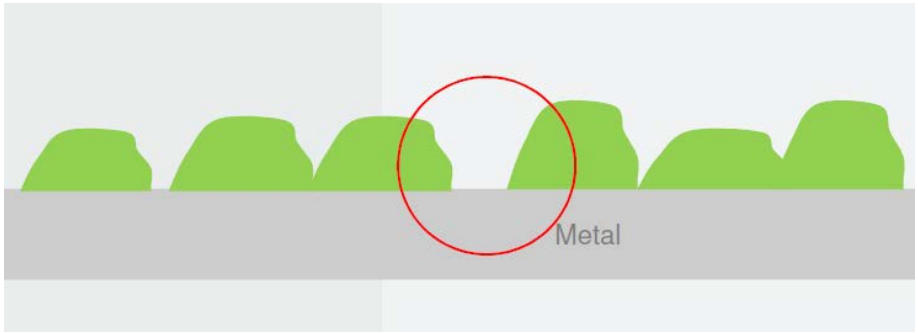



**Figure 2-1: Schematic of a phosphated metal surface showing exposed metal surface at the base of the pores.**

The SurTec 580 is manufactured by SurTec International (TCP licensee). When a fresh phosphated surface is immersed in the SurTec 580 there are two reactions that take place. First, a localized chemical reaction at the metal/solution interface which forms a hydroxide. As the pH increases locally a CrOH is formed. Figure 2-2 demonstrates the progression of the reaction.

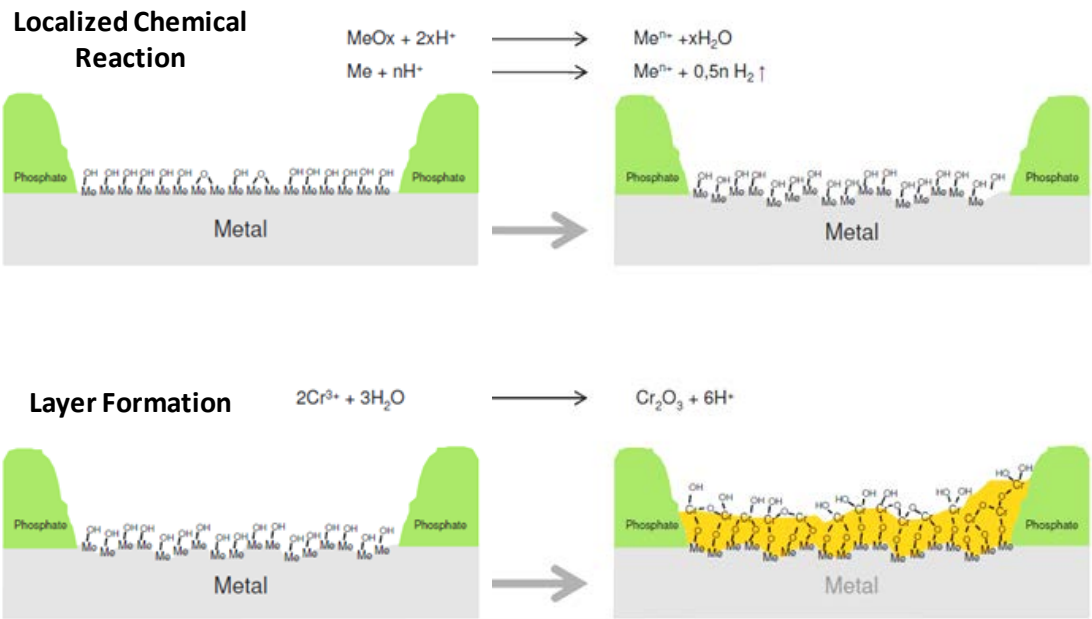



**Figure 2-2: View of the reactions that occur at the exposed metal between the phosphate crystals.**

The demonstrated technology is a green, clear-turbid liquid with a density of 1.00-1.02 g/ml and an approximate pH of 3.8. The SurTec 580 is purchased as a concentrate. Using deionized water, it has a make-up value of 7 ml per liter of solution. Steps for make-up:

1. Dissolve SurTec 580 in deionized water portion by portion with strong agitation.
2. Adjust the pH-value to pH 3.8 (if necessary) using sodium hydroxide solution (1 % solution).

The manufacturers recommended process steps are given in the process flow diagram in Figure 2-3. The SurTec operating temperature range is 20-40°C.

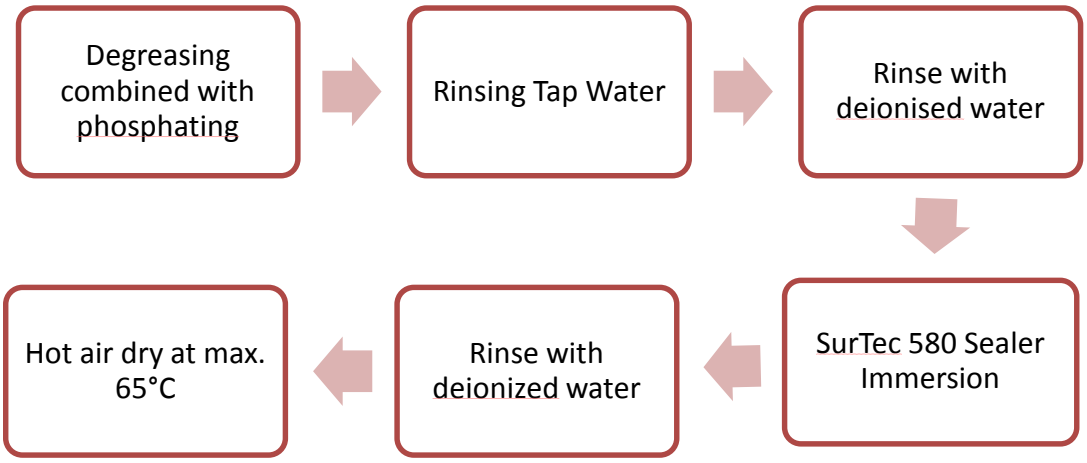



**Figure 2-3: Flow diagram for SurTec 580 manufacturer recommended process steps**

## 2.2 TECHNOLOGY DESCRIPTION: PPG Chemseal 100

Chemseal 100 is a post-phosphate, passivating sealer formulated to improve adhesion and corrosion protection of either zinc- or iron-phosphate. It incorporates organic and inorganic materials according to the following scheme. The backbone of the product is an aminated epoxy resin with the structure illustrated below:

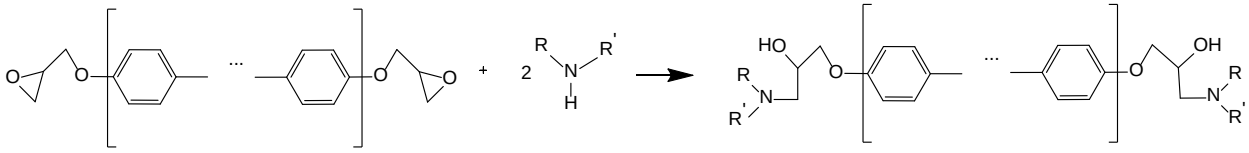


**Figure 2-4: Polymerization of epoxy precursors with amine catalyst to form thin-film matrix.**

The aminated epoxy resin is then reacted with a slight excess of fluorozirconic acid and a strong mineral acid to create water-soluble, protonated ammonium salts in mildly acidic solution. When phosphated parts are immersed into, or sprayed with a dilute solution of this product, mild etching of the phosphated steel results in both the deprotonation of the organic molecule onto the surface and the deposition of zirconium oxides.
Zirconium oxides serve to “seal” tiny voids in the phosphate surface, while the aromatic, organic molecule provides excellent opportunities for strong hydrogen bonding to both the underlying, “sealed” phosphate layer and the primer or single-coat finish applied after pretreatment.

## 2.3 TECHNOLOGY DEVELOPMENT: SurTec 580 (TCP)

The primary motivation for this project is the promise of transitioning the success of the TCP technology to steel. Trivalent chrome pretreatments were studied for use on steel substrates as part of the SERDP project WP1521. Trivalent chromium compositions and processes were originally developed as a chromate conversion coating alternative for aluminum alloys, and the vast majority of research has been focused on non-ferrous applications.

Dr. Vinod Agarwala is the original inventor of the TCP technology. In 1994 he studied the electrochemical impedance of trivalent chrome pretreatments on aluminum. The results showed a 10 to 100 fold increase in the polarization resistance of the surface films compared to the untreated aluminum alloy. These electrochemical results compared well with the corrosion behavior in B117 salt fog testing. The trivalent chromium treated surfaces showed no corrosion for up to 200 hours in 5% salt spray. A post-treatment with an oxidizer even further raised the coating's resistance due to an improved corrosion protection.[6]

A modified version of the trivalent chrome was later developed by NAVAIR, Patuxent River Maryland. Among the inventors are Dr. Michael Kane and Craig Matzdorf. Mr. Matzdorf and Dr. Kane conducted a demonstration of the technology on the aft section of two S-3 U.S. Navy Aircraft using a spray on process at the Naval Aviation Depot (NADEP), North Island California. The report included toxicology information consistent with what is presently stated in the current materials safety data sheets (MSDS). The report discusses trivalent chrome and the events leading up to the demonstration, including the application of the pretreatment on the aircraft. However, the final performance results were not available at the time of their report.[7]

"Trivalent Chrome Process as a Sealer for MIL-A-8625F Type II, IIB, and IC Anodic Coatings"[8] documents evaluations of TCP as sealers for various anodic coatings conducted by Materials Engineering, NAWCAD Patuxent River, Maryland. The performance of TCP as a sealer was compared to standard sealers like dichromate and water which are commonly used in aerospace and other industries. Paint adhesion was performed with commonly used high-solids and water-borne chromated and chromate-free primers qualified to MIL-PRF-23377 and MIL-PRF-85582. In these evaluations, TCP performs as good as or better than chromate in corrosion resistance and equal to chromate in paint adhesion. TCP is far superior to water for sealing. An additional benefit is that the TCP is applied at ambient conditions for 5 to 10 min. Chromate and Water sealers are applied at 190°F to 200°F for up to 25 min.

Many other studies have been conducted by the U.S. Army Research Laboratory to validate the performance of TCP on various aluminum substrates. One such study focused on aluminum alloy 5059-H131 under different surface treatment conditions. The surface treatment conditions included abrasive blasted substrate with and without a commercial TCP. Corrosion resistance was evaluated using GM 9540P and ASTM B 117 neutral salt fog methods. Dry adhesion was assessed using ASTM D 4541 pull-off and wet adhesion was assessed using ASTM D3359A. TCP showed excellent performance and was recommended as the pretreatment of choice based upon its qualification with the conversion coating specification MIL-DTL-81706 and its ability to sustain performance under bare conditions.[9]

In recent years, TCP has been considered for use in ferrous and multi-metal applications. The literature that can be found for steel pales in comparison to that found for use on aluminum.

However, a logical application of TCP for steel would be for use as a zinc phosphate sealer. NAVAIR Indian Head Division qualified TCP as an alternative to hex-chrome sealers for propellant and cartridge actuated devices. TCP was qualified to replace hexavalent chromate conversion coating on zinc-nickel plated steel. Unpainted test panels exhibited at least 42 days of resistance to cyclic salt fog. These panels lasted at least 4 days when subjected to cyclic sulfur dioxide and cyclic salt fog testing with full red rust evident on the seventh day. Painted and scribed TCP panels previously subjected to 10 days of humidity and 120 days of salt cyclic fog were subject to 78 days of cyclic sulfur dioxide and salt fog and paint was still largely intact with only moderate scribe corrosion and paint blistering near the scribe.[10]

## 2.4 TECHNOLOGY DEVELOPMENT: PPG Chemseal 100

Chemseal 100 was originally developed in the early 1990s to enhance the corrosion resistance of *iron* phosphated steel, specifically under high-solids, polyester, solvent-borne liquid paint systems. It was formulated to be a high-performing, environmentally sound alternative to established, $Cr^{6+}$ or $Cr^{3+}$ sealers. After evaluation of multiple epoxy and amine chemistry options, the final version of CS100 was commercialized and demonstrated equivalent performance to chrome-based products. Further investigation showed that CS100 benefits could also be realized over zinc phosphated steel, but that performance of the product could be coating-specific. Under some coating technologies, corrosion resistance enhancements are realized, while under others, minimal improvement relative to no phosphate sealer at all is seen. Although CS100 is not universally effective with all coatings technologies, it remains a useful tool in the modern landscape of environmentally sound, corrosion enhancing pretreatment sealers. The chemistry is patent-protected under US Patent Nos. 5,565,823 and 5,585,695.

## 2.5 ADVANTAGES AND LIMITATIONS OF THE TECHNOLGY

In this section, the advantages and limitations of the demonstrated technology are listed as compared to the current zinc phosphate coating process that uses chromic acid rinse.

**SurTec 580 (TCP)**

Advantages (Technical):

- TCP proven effective as a conversion coating on aluminum alloys
- Adds a layer of corrosion protection while improving coating adhesion
- Tests show that performance is comparable to hex chrome sealer
- Easy to apply drop-in replacement for existing sealer.

Advantages (Safety and Environmental):

- No hexavalent chromium
- Non-irritant to skin, or eyes.

Limitations:

- Little historical data for use on steel

**PPG Chemseal 100**

Advantages (Technical):

- Contains no chromium component
- Adds a layer of corrosion protection while improving coating adhesion
- Effectively used to seal phosphate coatings commercially for years
- Easy to apply drop-in replacement for existing sealer.

Advantages (Safety and Environmental):

- No chromium
- Non-irritant to skin, or eyes.

Limitations:

- Not universally effective with all coatings technologies
- Only slight color change to substrate surface to indicate full coverage

# 3.0 PERFORMANCE OBJECTIVES

The performance objectives with acceptance criteria for the demonstrated technology were evaluated in accordance with the tests delineated in the JTP provided in Appendix A. The functional performance objectives are summarized in Table 3-1. Specifically, this effort is meant to focus on sealing technologies for zinc phosphate. To that end, performance objectives were evaluated using steel as the base metal in all testing. The existing zinc phosphate with a hex chrome passivating rinse is the baseline process. As the demonstration progressed it became evident that some performance objectives established in the demonstration plan had to be modified. Many of these objectives were established for WP200906 as a whole and were not modified when the project was broken down into three distinct technology areas. All performance objectives applied to technology area 1: Pretreatments for Armor Steel, but not all are relevant for zinc phosphate sealers. For example, the metric for outdoor exposure was 25% less creep from scribe than baseline. The reasoning was that the "addition" of a chemical preatreatment on armor steel vs. direct-to-metal should yield a conservative 25% improvement. However, this technology area seeks to "replace" the existing hexavalent chrome sealer with a non-hexavalent chrome sealer. Therefore we consider a more reasonable objective to be; alternative shall meet or exceed the performance of the baseline chromate sealer. All of the performance objectives were evaluated by substrate and coating system. The modifications are reflected in table 3-1 below.

## Table 3-1 Performance objectives for alternative sealers for zinc phosphate

| Performance Objective | Data Requirements | Success Criteria | Substrate | Results | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Quantitative Performance Objectives | | | | SurTec 580 | | Chemseal 100 | | Chromic Acid | |
| | | | | CARC MIL-DTL- | | CARC MIL-DTL- | | CARC MIL-DTL- | |
| | | | | 53039 | 64159 | 53039 | 64159 | 53039 | 64159 |
| Adhesion Test | ASTM-4541 Pull-off Adhesion | Minimum average 30 events rating of 1200 PSI on 1.5 mil profile surface | LCS | Met | Met | Met | Met | Met | Met |
| | | | HHA | Met | Met | Met | Met | Met | Met |
| | ASTM- D3359 Dry Adhesion | Adhesion rating (steel) >4B; adhesion rating | LCS | Not Met | Not Met | Not Met | Met | Met | Met |
| | | | HHA | Met | Met | Met | Met | Met | Met |
| | ASTM- D3359 Wet Adhesion | Scribed area rating (steel) ≥ 3A after 24 hours at ambient; | LCS | Met | Met | Met | Met | Met | Met |
| | | | HHA | Met | Met | Met | Met | Met | Met |
| Chip Resistance | SAE-J400 | After one cycle, chip rating NLT 5B for steel | LCS | Not Met | Not Met | Met | Met | Met | Not Met |
| | | | HHA | Met | Not Met | Not Met | Not Met | Met | Met |
| Accelerated corrosion | ASTM-B117 Salt Fog | After 500 hours of exposure: steel substrate rating ≥6 scribed | LCS | Met | N/A | Met | N/A | Met | N/A |
| | | | HHA | Met | N/A | Met | N/A | Met | N/A |
| | GM-9540P Cyclic Corrosion ASTM D 1654 | After 60 cycles: steel substrate rating ≥ 4 | LCS | Met | Not Met | Met | Met | Not Met | Met |
| | | | HHA | Met | Met | Met | Not Met | Met | Met |
| Outdoor Exposure* | Tropical climate exposure at Cape Canaveral Air Force Base FL ASTM D 1654 ASTM G50 | Three years of exposure: meet or exceed baseline performance | LCS | Met | Met | Met | Not Met | N/A | N/A |
| | | | HHA | Not Met | Met | Not Met | Met | N/A | N/A |
| Hydrogen Embrittlement | ASTM E 399-97 | No detrimental effect to K1c of substrate. High Hard K1c @ 48-51Rc shall maintain $K_{1EAC}$ ≥ 19 (ksi√in) | LCS | N/A | N/A | N/A | N/A | N/A | N/A |
| | | | HHA | Met | Met | Met | Met | Met | Met |
| Field Testing** | TT-C-490 | Equivalent or less than existing process | — | N/A | N/A | N/A | N/A | N/A | N/A |
| **Qualitative Performance Objectives** | | | | | | | | | |
| Toxicity Clearance | Toxicity clearances and full disclosure from CHPPM | Approved by processing facility | — | Met | | Met | | | |
| Processing time | TT-C-490 | Equivalent or less than existing process | — | Met | | N/A | | | |
| Ease of use | Feedback from field technician on usability of technology and time required during demonstration | Minimal operator training required | — | Met | | N/A | | | |
| Monitor Solution collect data for OQE | TT-C-490 and manufacturers recommended operating parameters | No increase in downtime | — | Met | | N/A | | | |

* Modified outdoor exposure success criteria from 25% less creep from scribe to meet or exceed baseline performance
** Despite repeated efforts by ARL and ANAD, the parts that were fielded could not be located.

# 4.0 SITES/PLATFORM DESCRIPTION

## 4.1 TEST PLATFORMS/FACILITIES

The demonstration of the zinc phosphate sealer was carried out at ANAD in Building 114. The demonstration involved one of phosphate coating lines that support the Cleaning and Finishing Branch. Building 114 supports all vehicle programs that require phosphate coating of reclaimed parts.

There were several candidate parts and platforms considered. The small fuel caps for the M1 Abrams tank described in section 1.2 is one of the parts used. They were tagged using a routing tag, and tracking number and all information recorded for tracking location of fielded parts. During reassembly of the vehicle, the serial and vehicle number was to be recorded and matched with the part tracking number. Vehicle tracking is done by TACOM using the vehicle serial number. Below is a photograph of M1 tanks lining the railroad track at ANAD. These combat vehicles are on their way back to soldiers after being repaired and overhauled by depot personnel[11].

**Figure 4-1: M1 tanks lining the railroad track at ANAD on their way back to Soldiers after being repaired and overhauled by depot personnel.**

The Anniston site was selected for three reasons: 1) It was the site where the high hard armor steel pretreatment demonstration was initiated as the first phase of this ESTCP project thus the location allows us to consolidate our efforts 2) ANAD conducts a significant amount of phosphating and has been exploring opportunities to evaluate alternatives to hexavalent chrome for many parts of their finishing operations, and finally 3) ARL, through the Sustainable Painting Operations for Total Army (SPOTA) program, has enjoyed a long standing productive working relationship with ANAD to eliminate methylene chloride in ANAD depainting operations. These factors have provided the program ample opportunity for success. All of the work was performed

on site by ANAD personal and SurTec International representatives. The selected parts were zinc phosphated in accordance with normal operating conditions; however representative parts were redirected to the SurTec 580 sealer while baseline parts continued through the chromic acid sealer. All of this was documented in order to attempt to track each part and platform in the field for periodic inspections.

## 4.2 PRESENT OPERATIONS

Zinc phosphating has been used for many years as an effective pretreatment for steels. It produces a consistent, fine-grained protective coating and is commonly used as a pretreatment prior to painting and powder coating. ANAD uses zinc phosphating on parts that require a final corrosion protective coating before storage and on parts prior to painting. The current process utilized at ANAD begins with preparing the parts by abrasive blasting with steel grit, aluminum oxide, glass bead, plastic bead, or other ceramic type abrasives. Alternatives to abrasive blasting may be used provided they do not adversely affect coating weight, salt spray, or hydrogen embrittlement requirements. Once the parts are properly cleaned and prepared, they are immersed in zinc phosphate and subsequently rinsed in cold water. The next step is critical for corrosion inhibition. The phosphate parts are dipped in a solution of 4 ounces chromic acid and 4 ounces of phosphoric acid per 100 gallons of solution at a temperature of 150-200'F. Once sealed, drying with compressed hot air or a centrifugal drier is performed. The parts are now ready to be primed and painted. If the unpainted parts will be stored long term, a corrosion preventive compound or preservative lubricating oil is applied. Below is a flow diagram of a typical zinc phosphating line currently employed at ANAD.

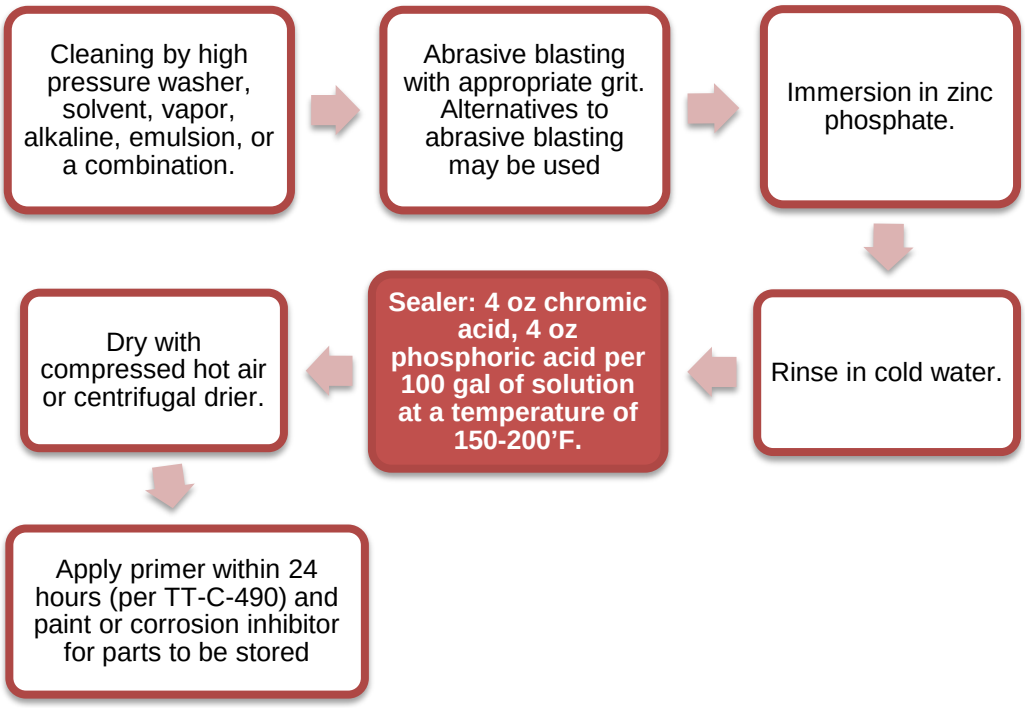



**Figure 4-2: Flow diagram of a typical phosphate line currently employed at ANAD**

The demonstrated technology is intended to replace the sealer in step 5 (highlighted in red) in the above flow diagram. Since the SurTec 580 is a drop-in replacement for the chromic acid solution, it does not add additional steps to the current process.

## 4.3 SITE-RELATED PERMITS AND REGULATIONS

Additional site related permits or regulations were not required for the demonstration to be conducted at ANAD. The facility has had the capability to process and apply pretreatments, including hexavalent chrome pretreatments, and holds the necessary documentation to perform the demonstrated chemical pretreatments and dispose of any waste if necessary.

# 5.0 TEST DESIGN

## 5.1 CONCEPTUAL EXPERIMENTAL DESIGN

The details of the laboratory testing are provided in the JTP (appendix A). Although significant testing and evaluation of trivalent chrome pretreatments on steel substrates was performed as part of the SERDP project WP1521, trivalent chrome sealers for zinc phosphate were not adequately explored. For this reason, the demonstrated technology, SurTec 580 was laboratory validated and demonstrated on zinc phosphate steel samples and components in accordance with the JTP provided in Appendix A. In addition to the laboratory validation described in the JTP, field testing on parts processed in the zinc phosphate lines in building 114 at ANAD were planned.

## 5.2 Laboratory Experimental Procedure

**Sample Preparation**

The experiments were conducted using 4 inch x 6 inch x 3/16 inch steel test panels fabricated from HHA steel MIL-A-46100 and low carbon steel (LCS). All of the HHA test panels were abrasive blasted to a 1.5 mil surface finish using aluminum oxide blast media, while all LCS panels had a milled surface finish of approximately 100-63 micro-inches (μ in). All of the pretreatments were applied by the vendors in order to eliminate inconsistencies in the processes. All primer and topcoats were applied by the Army Research Laboratory. The coatings used were MIL-DTL-53022 Type II primer[12], solvent borne MIL-DTL-53039 Type III topcoat[13], and water borne MIL-DTL-64159 Type II topcoat[14] all manufactured by Hentzen. The test matrix is shown below in Table 5-1.

**Table 5-1: Test Matrix for Both Steel Substrates**

| Coating System | Tests | Low Carbon Steel (4 x 6 panels) | | | High Hard Armor Steel (4 x 6 panels) | | |
|---|---|---|---|---|---|---|---|
| | | Dip Zn Phos w/ Chromate Seal Baseline | Spray Zinc Phosphate Sealers | | Dip Zn Phos w/ Chromate Seal Baseline | Spray Zinc Phosphate Sealers | |
| | | | SurTec 580 | PPG Chemseal 100 | | SurTec 580 | PPG Chemseal 100 |
| Low VOC MIL-DTL-53022 / MIL-DTL-53039 | ASTM D 4541 | 3 | 3 | 3 | 3 | 3 | 3 |
| | ASTM D 3359 Dry | 1 | 1 | 1 | 1 | 1 | 1 |
| | ASTM D 3359 Wet | 2 | 2 | 2 | 2 | 2 | 2 |
| | SAE J400 | 1 | 1 | 1 | 1 | 1 | 1 |
| | ASTM B 117 | 5 | 5 | 5 | 5 | 5 | 5 |
| | GM9540P | 3 | 3 | 3 | 3 | 3 | 3 |
| | Outdoor Exposure | 5 | 5 | 5 | 5 | 5 | 5 |
| Low VOC MIL-DTL-53022 / MIL-DTL-64159 | ASTM D 4541 | 3 | 3 | 3 | 3 | 3 | 3 |
| | ASTM D 3359 Dry | 1 | 1 | 1 | 1 | 1 | 1 |
| | ASTM D 3359 Wet | 2 | 2 | 2 | 2 | 2 | 2 |
| | SAE J400 | 1 | 1 | 1 | 1 | 1 | 1 |
| | ASTM B 117 | 5 | 5 | 5 | 5 | 5 | 5 |
| | GM9540P | 3 | 3 | 3 | 3 | 3 | 3 |
| | Outdoor Exposure | 5 | 5 | 5 | 5 | 5 | 5 |

**Pull-off Adhesion (ASTM D 4541)**
An Elcometer Model 108 Hydraulic Adhesion Test Equipment (HATE) was used to obtain the pull-off adhesion strength in pound per in$^2$ (psi). The apparatus included a loading fixture commonly referred to as a "dolly" which was secured to the coating normal to the coating surface using Instabond™ S-100 cyanoacrylate adhesive. After allowing the adhesive to cure for 24 hours at 25ºC at 50% RH, the attached dolly was inserted into the test apparatus. The load applied by the apparatus was gradually increased and monitored on the gauge until a plug of coating was detached. The failure tension in pounds per square inch was recorded and the failure mode and location within the coating system was recorded. The pull-off test apparatus and dolly configuration are illustrated in Figure 5-1.

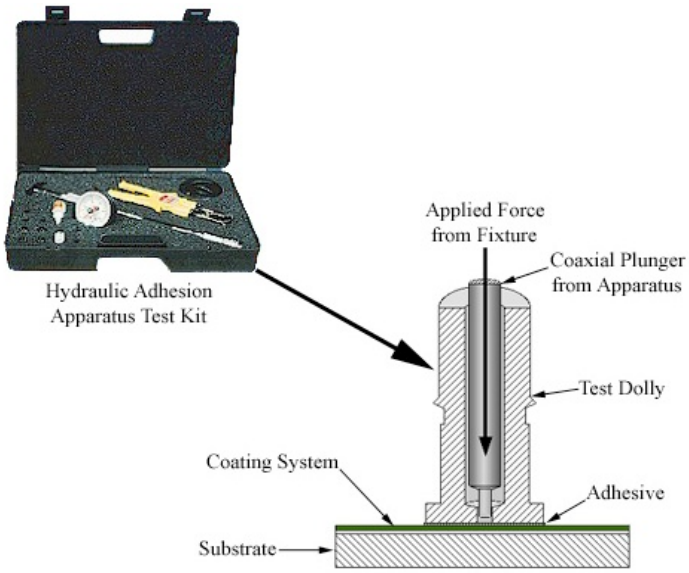



**Figure 5-1: Elcometer Model 108 Hydraulic Adhesion Test Equipment (HATE) Pull-off Test Apparatus.**

Dry Tape Adhesion:
Tests were conducted at room temperature as defined in ASTM D 3924. An area of the panel free of blemishes was selected. Using a sharp cutting tool, 6 parallel cuts @2mm spacing through the paint film to the metal substrate were made. A second series of cuts at 90 degrees to the initial set were then made. Both cuts were made ensuring that they were long enough to make a complete set of 6x6 grid lines. The grids were repeated in two other areas on test coupons in order to obtain 3 data points per coupon. The grid lines were then brushed lightly with a stiff brush to remove any detached flakes or ribbons of coating. A piece of 3M 396 tape was used to further clean off the area by lightly touching it to the grid lines to remove any detritus that would interfere with the full application of the test tape. A complete lap of tape was removed from the roll and discarded prior to removing the length of tape used for the test. A length of tape was removed at a steady rate and cut about 75 mm (3 in.) long. The center of the tape was placed over the grid and the area of the grid smoothed into place by a fingernail. To ensure good contact with the film, the tape was rubbed firmly with the eraser on the end of a pencil. The tape was removed by seizing the free end and rapidly pulling (not jerked) back upon itself at as close to an angle of 180° as possible. Following the tape pull off, each grid was rated using the classification in ASTM D 3359 shown below in Figure 5-2.

| Surface of cross-cut area from which flaking has occured. (Example for 6 parallel cuts). | None | | | | | Greater than 65% |
|---|---|---|---|---|---|---|
| **Classification** | **5** | **4** | **3** | **2** | **1** | **0** |

**Figure 5-2: Cross-cut area ratings**

Wet Tape Adhesion:
Wet tape adhesion test evaluates the coating's ability to resist penetration by water. This test was performed in accordance to Method 6301 of FED-STD-141 (Paint, Varnish, Lacquer and Related Materials; Methods of Inspection Sampling and Testing) and rated per ASTM D 3359. An "X" scribe was required on all test panels. The high hard armor steel and low carbon steel panels were evaluated in the 24 and 96 hour wet tape adhesion test. The samples were immersed in distilled water for 24 and 96 hours at room temperature and 120 degree F, respectively. The panels were then removed from the water and dried by wiping with a soft cloth. Two parallel lines were scribed approximately one inch apart with an "X" scribed between the two parallel lines making sure that the coating had been scribed all the way through. A piece of tape was placed over the scribes and smoothed out by rolling with a 3-lb roller. The tape was then removed at an angle of approximately 180-degrees (parallel) to the surface. The areas around the scribes were inspected for peel-away/delamination and the unscribed immersed area for blisters. Each panel was rated IAW ASTM D3359 and photo-documented.

**IAW SAE-J400 Chip resistance test**
Prior to beginning the tests, each panel was digitally photo documented. The panels were then subjected to chip resistance testing IAW SAE J400 at ambient temperature using a Q-Lab Gravelometer (Figure 5-3). The panels were held in a 45° angle specimen holder and air pressure (70 psi +/- 0.30 psi) was used to propel gravel at the sample. The test sample was then removed and gently wiped off with a clean cloth. Tape (3M #898 filament strapping tape as specified in SAE J400) was then applied to the entire tested surface in order to remove any loose fragments of the coating. The tested panel was then compared to standard SAE transparencies to determine a chipping rating.

Panels were again digitally photographed following tests and rated using IAW SAE J400 and ratings for each panel was recorded. The total number of chips inside a 4"x4" grid (16 $in^2$ area) using a transparency overlay was counted and the rating obtained using Table 5-2. The average size of the chips was measured and rated using Table 5-3. For panels without a dominant chip size, the second most prevalent chip size was included (for example, a "$^{B}/_{A}$" rating had at least $^{2}/_{3}$ chips of size "B" and $^{1}/_{3}$ chips of size "A").

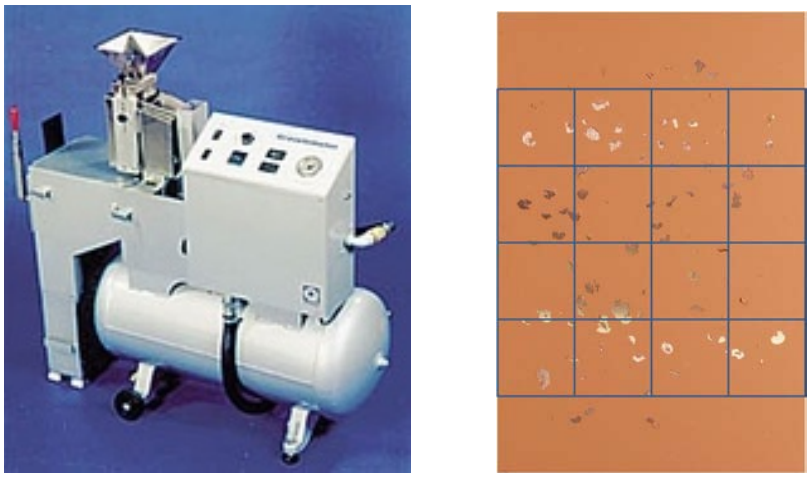


**Figure 5-3: Example of the Q-Lab Gravelometer used to measure chip resistance per SAE J400 and the area of a panel evaluated.**

**Table 5-2: SAE J400 Ratings for number of chips in a 4 inch x 4 inch area**

| SAE J400 Rating | 10 | 9 | 8 | 7 | 6 | 5 | 4 | 3 | 2 | 1 | 0 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Number of Chipps in 4"x 4" Grid | 0 | 1 | 2-4 | 5-9 | 10-24 | 25-49 | 50-74 | 75-99 | 100-149 | 150-250 | >250 |

**Table 5-3: SAE J400 Ratings for size of chips in a 4 inch x 4 inch area**

| | | |
|---|---|---|
| A | < 1mm | (approximately < 0.03") |
| B | 1-3mm | (approximately 0.03-0.12") |
| C | 3-6mm | (approximately 0.12-0.25") |
| D | > 6mm | (approximately > 0.25") |

**Accelerated Corrosion**

Two accelerated corrosion test chambers were used to evaluate the steel test panels. The Harshaw Model 22 for standard neutral salt fog and an Atotech Model CCT-NC-30 for cyclic corrosion using GM9540P shows both test chambers in the laboratory where the testing was carried out. The test panels evaluated in neutral salt fog had a single diagonal scribe while the test panels exposed to GM9540P were "X" scribed. In each case, the panels were scribed completely through the coating making sure that the substrate was exposed. The samples were then placed in their respective chambers, tilted at an angle between no more than 15º from the vertical with the scribed surface facing upwards.

The ASTM B 117 neutral salt fog conditions are 95°F with saturated humidity and an atomized fog of 5% NaCl solution. The GM 9540P test consists of 18 separate stages per cycle that

include the following: saltwater spray, humidity, ambient, and heated drying. The environmental conditions and duration of each stage for one complete 9540P cycle are provided in Table 5-4. The standard 0.9% NaCl, 0.1% CaCl2, 0.25% NaHCO3 test solution was used. In addition, the cyclic chamber was calibrated with standard steel mass-loss calibration coupons as described in the GM 9540P test specification.

**Table 5-4: Cycle details for the GM 9540P cyclic corrosion test[15]**

| Interval | Description | Time (min) | Temperature (±3 °C) |
|---|---|---|---|
| 1 | Ramp to salt mist | 15 | 25 |
| 2 | Salt mist cycle | 1 | 25 |
| 3 | Dry cycle | 15 | 30 |
| 4 | Ramp to salt mist | 70 | 25 |
| 5 | Salt mist cycle | 1 | 25 |
| 6 | Dry cycle | 15 | 30 |
| 7 | Ramp to salt mist | 70 | 25 |
| 8 | Salt mist cycle | 1 | 25 |
| 9 | Dry cycle | 15 | 30 |
| 10 | Ramp to salt mist | 70 | 25 |
| 11 | Salt mist cycle | 1 | 25 |
| 12 | Dry cycle | 15 | 30 |
| 13 | Ramp to humidity | 15 | 49 |
| 14 | Humidity cycle | 480 | 49 |
| 15 | Ramp to dry | 15 | 60 |
| 16 | Dry cycle | 480 | 60 |
| 17 | Ramp to ambient | 15 | 25 |
| 18 | Ambient cycle | 480 | 25 |

**Outdoor Exposure Testing:**
Test panels were prepared as described above. The 4x6" coupons of both HHA and LCS were transferred to Cape Canaveral Air Force Station in Florida. These coupons were scribed with a carbide scribe all the way through the coating to the substrate as described in ASTM D1654 before being mounted to racks using Teflon fixtures, scribed side up on a 30º angle to the vertical. The racks are set parallel to the Atlantic Ocean and are approximately 100 yards inland from the water. The coupons continue to be inspected and evaluated biannually until failure in accordance with ASTM D1654. Weather data is collected utilizing a data-logging weather station and downloaded annually. Mass loss coupons placed with the test coupons are analyzed annually.

**Stress Corrosion Cracking Evaluation:**
The resistance to environmentally assisted cracking was assessed using the rising step load method for determination of $K_{1EAC}$. For this procedure, CV2 Charpy specimens of MIL-A-46100D were machined in the longitudinal-transverse (L-T) orientation IAW ASTM E 399-97. The Charpy specimens were spray zinc phosphate and subsequently sealed prior to testing. The phosphate systems which included the sealers were evaluated to determine if they would have

any detrimental effect on the $K_{1EAC}$ of the HHA steel. The specific procedure and specimen fatigue pre-cracking of each of the samples is described in the JTP and elsewhere [15].

### 5.3 PHOSPHATE LINE COMPONENT DEMONSTRATION

The non-chromate sealer for zinc phosphate demonstration was initiated on parts processed at ANAD. All of the chemicals for the demonstration were provided by the manufacturers along with specific instructions on the application process. These can be seen in the process flow diagrams in section 2.0. A vat near the zinc phosphate line was cleaned and made available for the SurTec 580 sealer. All wastes generated from the clean out were disposed of by ANAD's waste contractor in accordance with all state and federal regulations.

The SurTec 580 sealer is essentially a drop-in replacement for the chromic acid solution. No additional modifications to the vats were necessary. Representatives from SurTec International worked closely with ARL and ANAD to properly mix the SurTec 580 solution, and carry out the sealing of the demonstration parts. In order to minimize disrupting the throughput on the phosphate lines, a 450 gallon vat located one row over from an active phosphate line was used. Figure 5-4 shows the SurTec 580 vat used for the demonstration in building 114, at ANAD.

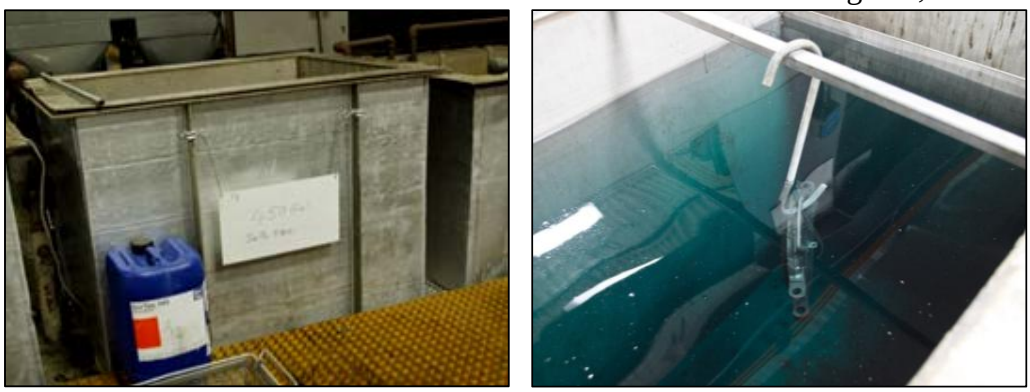


**Figure 5-4: SurTec 580 vat near phosphate line used in demonstration**

The M1 Abrams small fuel cap NSN 5340-01-460-5277 was the primary candidate part in the demonstration. Some miscellaneous parts and test panels were also treated for the purpose of evaluating at the ARL. Below in figure 5-5 are the fuel caps and test panels treated in the same zinc phosphate line during the demonstration. The caps were first steam cleaned, and the paint chemically removed. They were then abrasive blasted and repaired if necessary before phosphate and painting completed. The test panels were used to obtain the weight of the zinc phosphate coatings for the lines as well as corrosion tests at ANAD. ARL was also given the opportunity to treat a cupola from Stryker. The cupola shown in figure 5-6 was the largest part that would fit in the 450 gallon SurTec vat.

**Figure 5-5: M1 Abrams Fuel Caps and test panels zinc phosphate and sealed**

**Figure 5-6: Stryker cupola rising out of the SurTec 580 vat after seal**

According to section 3.5.5 of TT-C-490, the organic coating shall be applied to thoroughly dried surfaces within 24 hours after pretreatment. The research team was careful to follow this specification in order to minimize any variables during the demonstration. The information on the routing tickets for each part was recorded in order to track their location for inspection at a later date. An example of the routing tags can be seen in figure 5-7. This was done for both the parts sealed with SurTec 580 as well as the representative baseline (hex-chrome sealed) fuel caps. Additional caps and test panels were prepared in the same phosphate line for laboratory testing and validation by ARL at Aberdeen Proving Ground (APG). ASTM D 4541, ASTM B117 and GM 9540P was used to test the additional parts returned from ANAD.

**Figure 5-7: Routing tag indicating all steps required to recondition fuel caps**

## 5.4 FIELD TESTING

A variety of parts and components were identified for the demonstration. The parts that are processed in the phosphate line are typically small miscellaneous parts and components. These parts, by themselves are usually not easily identified for tracking. The M1 small fuel cap was selected because it was thought it could be tracked as part of a major platform by matching it with the serial number of the vehicle. Table 5-5 below lists all of the parts that were processed in the phosphate line used in the demonstration in building 114.

**Table 5-5: List parts and test panels processed during demonstration at ANAD**

| QTY | Substrate | Sealer | Testing Facility |
|---|---|---|---|
| 4 | Steel (ANAD) | SurTec 580 | ANAD |
| 4 | Steel (ANAD) | Chromate | ANAD |
| 4 | Steel (ANAD) | SurTec 580 | ARL |
| 4 | Steel (ANAD) | Chromate | ARL |
| 3 | HHA | SurTec 580 | ARL |
| 2 | HHA | Chromate | ARL |
| 3 | LCS | SurTec 580 | ARL |
| 2 | LCS | Chromate | ARL |
| 1 | Fuel Cap | SurTec 580 | ARL |
| 1 | Fuel Cap | Chromate | ARL |
| 5 | Fuel Cap | SurTec 580 | Field |
| 5 | Fuel Cap | Chromate | Field |
| 1 | Copula | SurTec 580 | Field |

ARL was also given a variety of miscellaneous parts not listed in table 5-5. These parts receive zinc phosphate and were sealed with both baseline hex chrome and the SurTec 580 and tested and analyzed at the ARL.

As previously mentioned, identifying information of the demonstrated parts was recorded in an effort to track the parts (and respective platform) in the field. The tracking information was taken from routing tags similar to the one in figure 5-7. Below is a list of the "fielded" parts along with the identifying information recorded during the demonstration.

5 - ZnPhos/SurTec 580 M1 Fuel Caps: Basket# 3096587, 3100357, 3100369

5 - ZnPhos/Cr6+ M1 Fuel Caps: Basket# 3095674, 3089137, 3096764

1 - ZnPhos/SurTec 580 Cupola: Basket# 3092341, WBS Code 10MFE001-K0

Once the parts were completed, the path taken for reassembly is monitored by Mr. Jeb Nabors. Therefore, all tracking of processed parts was attempted through him. Mr. Nabors contact information is as follows:

Jeb Nabors
Systems Branch, DPM
Anniston Army Depot
COM: 256-240-3620
jeb.nabors@us.army.mil

Parts that are immersion phosphate are typically "miscellaneous parts" and many cannot be tracked once they've left the depot. In discussions with ANAD, we were hopeful that the M1 fuel caps were substantial enough parts that they could be matched with a vehicle serial number and located for inspection. Unfortunately, that was not the case. Despite repeated efforts by ARL and ANAD, the parts that were fielded could not be located. Several parts were however, retained and brought back to ARL laboratories to evaluate the performance of the SurTec 580 against the baseline chromate sealer. These parts were assessed using the same success criteria: performance greater than or equal to the baseline system using SSPC-VIS 2. Table 5-6 quantifies the degree of rusting on painted steel surfaces with a zero to ten scale based on percentage of visible rust present on the surface. Visible rust includes rust blisters and undercutting of the coating.

**Table 5-6: SSPC-VIS 2 Ratings for Percent Corrosion of Painted Surfaces.**

| Rust Grade | Percent of Surface Rusted | Photographic Standard | | |
|---|---|---|---|---|
| | | Spot | General | Pinpoint |
| 10 | Less than or equalt to 0.01 percent | NONE | | |
| 9 | Greater than 0.01 percent to 0.03 percent | 9-S | 9-G | 9-P |
| 8 | Greater than 0.03 percent to 0.1 percent | 8-S | 8-G | 8-P |
| 7 | Greater than 0.1 percent to 0.3 percent | 7-S | 7-G | 7-P |
| 6 | Greater than 0.3 percent to 1 percent | 6-S | 6-G | 6-P |
| 5 | Greater than 1 percent to 3 percent | 5-S | 5-G | 5-P |
| 4 | Greater than 3 percent to 10 percent | 4-S | 4-G | 4-P |
| 3 | Greater than 10 percent to 16 percent | 3-S | 3-G | 3-P |
| 2 | Greater than 16 percent to 33 percent | 2-S | 2-G | 2-P |
| 1 | Greater than 33 percent to 50 percent | 1-S | 1-G | 1-P |
| 0 | Greater than 50 percent | NONE | | |

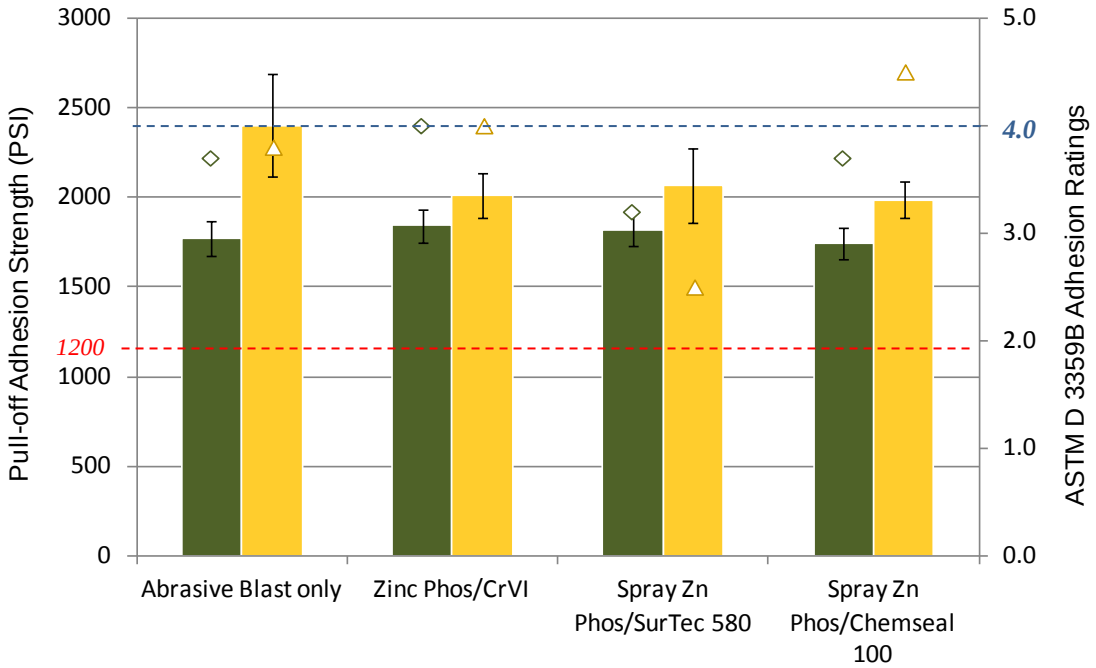
## 6.0 PERFORMANCE ASSESSMENT

### 6.1 Laboratory Results

Candidate zinc phosphate sealers have undergone a comprehensive evaluation as determined by the JTP provided in appendix A. The target substrate material for this demonstration is steel and therefore all tests were validated on two steel alloys; HHA and LCS. Table 3-1 represents an overview of the performance requirements of the technology being demonstrated.

**Adhesion Performance:**

The purpose of zinc phosphate as a base for paint is to enhance the adhesion of the primer and topcoat to the substrate. Figure 6-1 through Table 6-1 are indicators of adhesion provided by the zinc phophated and sealed surface. Figure 6-1 contains both pull-off strength and dry tape adhesion ratings on milled finish (63-125µ inch) LCS surface. The success criteria for pull-off adhesion was set at 1200 psi. A 1200 psi threshold was selected because it represents the average pull-off strength achieved for DoD-P-15328 wash primer on low carbon steel with a milled finish (63-125µ inch) and was considered to be an ample pull off strength for an organic coating. All of the tested surface conditions shown in figure 6-1 met the pull off strength criteria; therefore we must consider the dry adhesion tape test results overlaying the pull off results. This test was done in accordance with ASTM D3330 and ASTM D3359. A roll of 1" wide LA-26 Intertape was selected as the pull-off adhesion tape since it meets the TT-C-490F requirement of 80oz-force over CARC. The threshold for success is a 4B rating. Only the chromate seal met the tape test requirement of a 4B with both CARC systems. The PPG Chemseal 100 met the requirement with the MIL-DTL-53022/ MIL-DTL-64159 paint system only and was the only instance of a tape with greater than a 4B rating. A rating of 4B is required by TT-C-490F for inclusion on the Qualified Products Database (QPD). As such, only the zinc phosphates with Chemseal 100 or the chrome seal would meet the success criteria for the QPD.



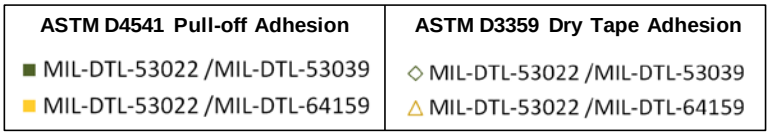



**Figure 6-1: Pull-off and dry tape adhesion strength on milled finish LCS**

All of the HHA test panels were abrasive blasted to remove mill scale. A surface finish of approximately 1.5 mils is expected to improve adhesion of all systems. This is typically how HHA surfaces are prepared in both production and during rework, so it is considered an accurate representation of HHA surfaces with zinc phosphate. Figure 6-2 is interesting in that the only samples to fail any of the adhesion tests were the panels that were only abrasive blasted without zinc phosphate or sealer. All passed the pull-off requirement, but it is clear that the phosphate surface improves adhesion regardless of the sealer used on abrasive blasted surfaces. All sealers met the performance objectives for adhesion on HHA. There is no specific requirement for pull-off adhesion testing for inclusion on the TT-C-490F QPD.

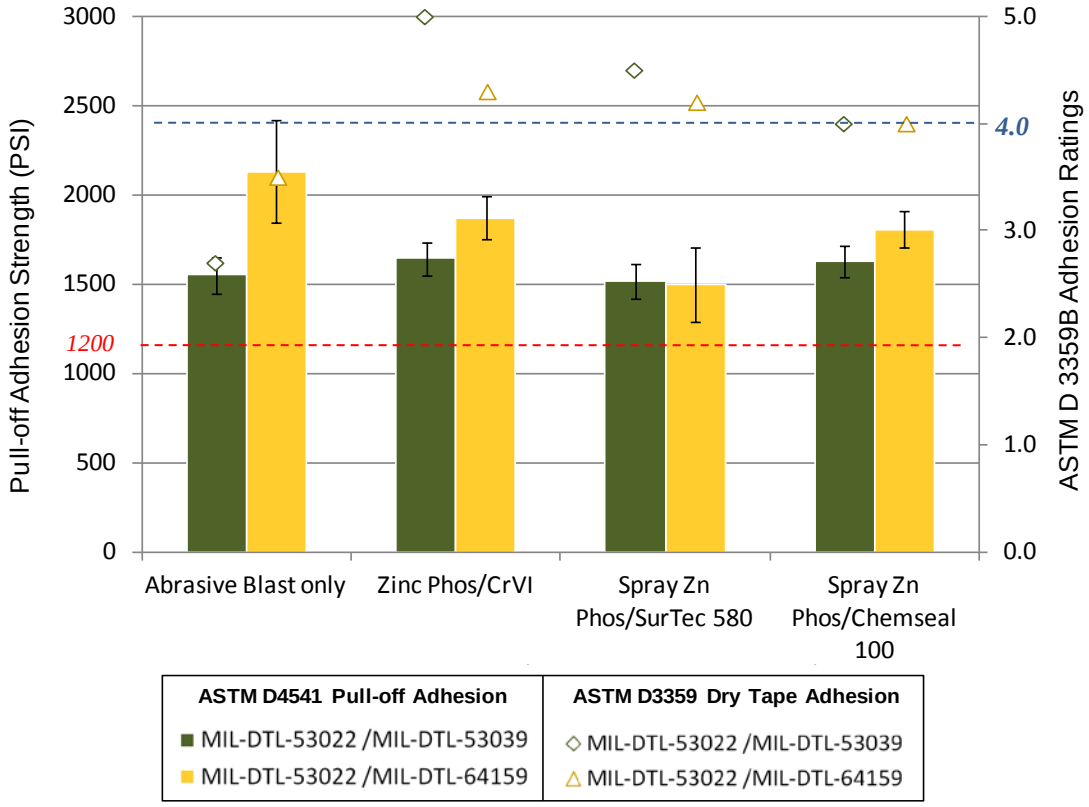



**Figure 6-2: Pull-off and dry tape adhesion strength on abrasive blasted HHA**

Wet adhesion tests were carried out according to ASTM D3359, Method A's scribing technique with this caveat: The specification does not prescribe water, temperature or duration. The success criteria was derived using NAVAIR requirements. The wet tape adhesion test results are shown in Figure 6-3 and 6-4. The success criterion here is a rating of 3A after 24 hours immersed in ambient DI water. All of the samples tested easily met the minimum 3A rating. The abrasive

blasted surfaces with or without zinc phosphate provided the most consistent results. There is no specific requirement for wet tape adhesion testing for inclusion on the TT-C-490F QPD

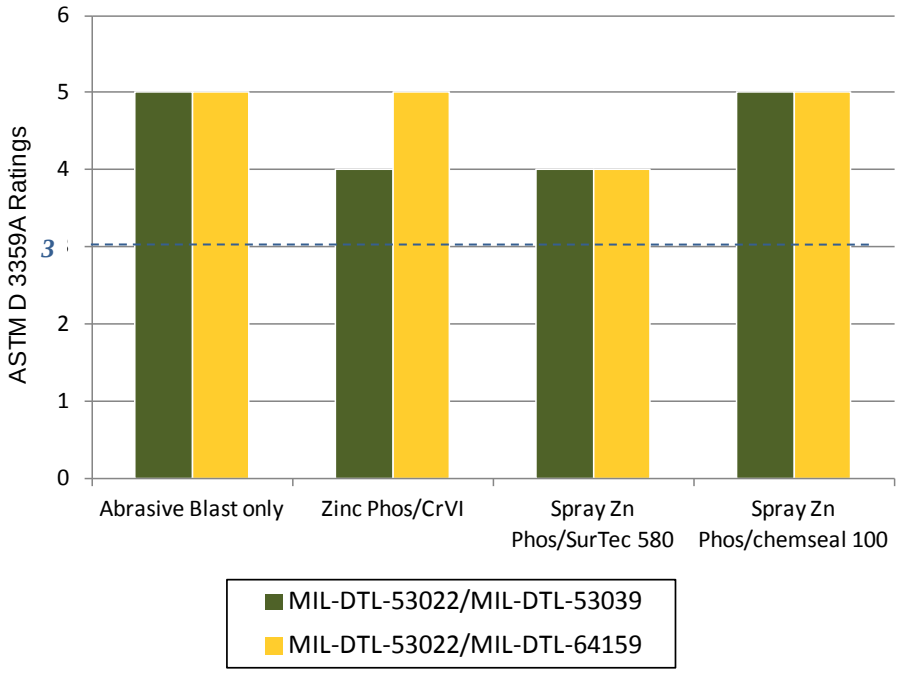



**Figure 6-3: ASTM-D3359A ratings for wet tape adhesion for LCS at 24 hours immersion**

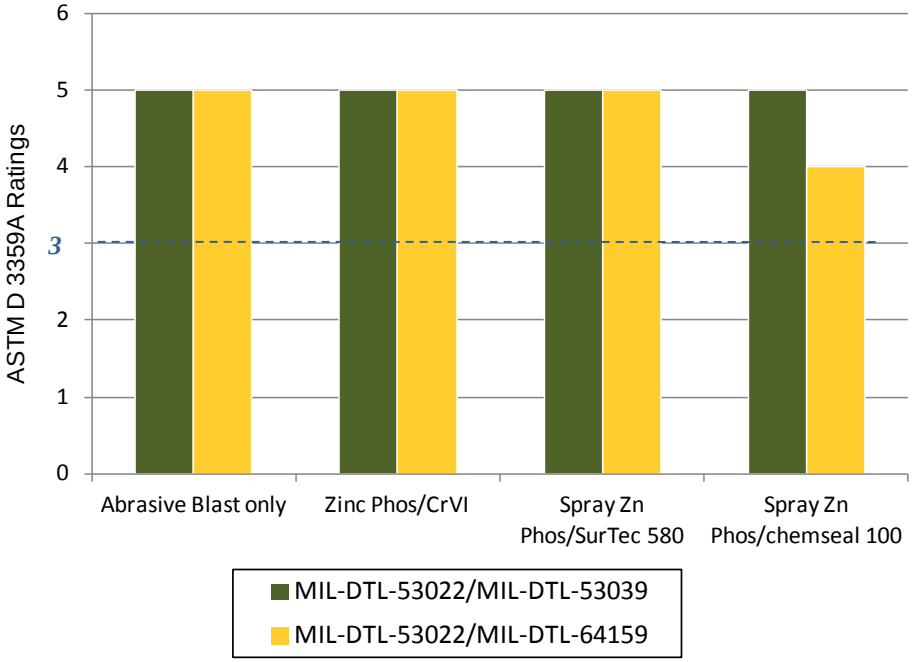



**Figure 6-4: ASTM-D3359A ratings for wet tape adhesion for abrasive blasted HHA at 24 hours immersion**

Another indication of proper adhesion is the ability of the coating system to resist chipping. This is particularly important for military ground vehicles that are navigated through rough terrain. Table 6-1 shows the results of the SAE J400 gravelometer test for chip resistance. The test samples are zinc phosphate sealed with the baseline chrome sealer, zinc phosphate sealed with the two alternative candidate sealers and an abrasive blasted-only control. All panels were coated using two different primer/topcoat combinations. The pass criterion for chip resistance is a rating of 5B. The baseline chromate sealed zinc phosphate met the 5B requirement in 3 of the 4 systems. The LCS with MIL-DTL-53022/MIL-DTL-64159 was the only chromate sealed system rated below a 5B. Of the two alternative sealers, the Chemseal 100 provided the best chip resistance with 5B/A ratings on LCS. When compared with the baseline, both alternatives performed relatively well and provided adequate adhesion and chip resistance. However, in terms of meeting the performance objectives for chip resistance, the sealers vary by application. SurTec 580 met the objective on HHA with the MIL-DTL-53022/MIL-DTL-53039 CARC system, while Chemseal 100 did better on LCS. None of the sealers, including the baseline, exceeded the performance objectives. The chromate sealed zinc phosphate systems didn't achieve a 5B rating in all cases. Therefore, it could be argued that a 4B may be a sufficient threshold for chip resistance on a non-abrasive blasted surface. There is no specific requirement for chip resistance testing for inclusion on the TT-C-490F QPD

**Table 6-1: Chip resistance of pretreatments on abrasive blasted HHA steel**

| Surface Treatment | Milled Finish Low Carbon Steel | | | | Abrasive Blasted High Hard Armor | | | |
|---|---|---|---|---|---|---|---|---|
| | MIL-DTL-53022/ MIL-DTL-53039 | | MIL-DTL-53022/ MIL-DTL-64159 | | MIL-DTL-53022/ MIL-DTL-53039 | | MIL-DTL-53022/ MIL-DTL-64159 | |
| Abrasive Blasted Only | 4 | B | 4 | B | 6 | A/B | 5 | B/A |
| Dip Zinc Phosphate/Cr6+ | 5 | B | 4 | B | 5 | B | 5 | B/A |
| Spray Zinc Phos/Surtec 580 | 4 | B | 4 | B | 5 | B | 4 | B/A |
| Spray Zinc Phos/Chemseal 100 | 5 | B/A | 5 | B/A | 4 | B/A | 4 | B |

**Accelerated Corrosion:**

Only test panels coated with CARC system MIL-DTL-53022/MIL-DTL-53039 were tested in ASTM B117. The primary mode of failure for all of the test panels was creepage from the scribe. The ratings of five replicates of each pretreatment were averaged and presented in Figure 6-5 and 6-6. The success criterion for salt fog test is a scribed rating of ≥ 6 as per ASTM D1654, Method A after 500 hours of exposure. The requirement for ASTM B117 testing for inclusion on the TT-C-490F QPD for steel is a rating of ≥ 6 at 336 hours. As such, each system tested met the success criteria for the QPD.

At 500 hours of exposure, all of the alternatives met this performance objective. The SurTec 580 just achieved a rating 6.0 on LCS at 500 hours. Beyond that, the SurTec 580 provided slightly more protection for the LCS panels that were abrasive blasted. The SurTec 580 performed far better on the abrasive blasted HHA panels. In figure 6-6, the SurTec 580 performed as well as the chromate sealer throughout 1000 hours of exposure. On the abrasive blasted HHA, both the

SurTec 580 and Chemseal 100 easily met the success criterion. After all samples completed 1000 hours, they were scraped with a 2" putty knife before the final measurements were made. Representative panels from each set of replicates are shown in Figure 6-8 and 6-9. Each panel is captioned with sealer identification and average rating. These panels were selected because they best represent the average of the set. A visual comparison in figure 6-8 shows that on milled finished LCS, chromate zinc phosphate provided better corrosion protection throughout the 1000 hours of exposure. However, for the abrasive blasted HHA panels in figure 6-9, after 1000 hours the two alternatives were still comparable to chromate.

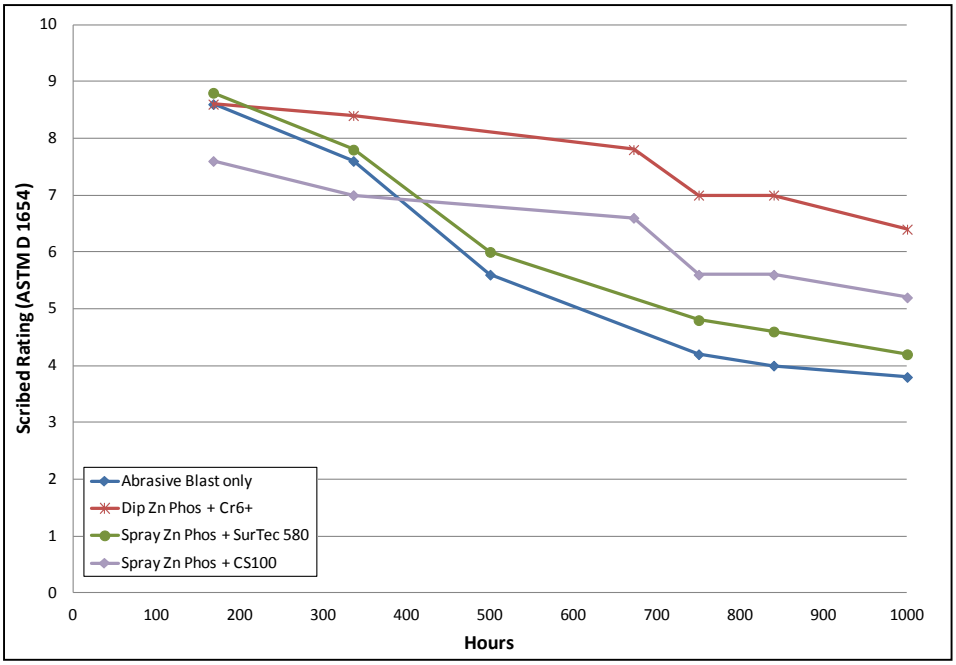



**Figure 6-5: ASTM D 1654 ratings for low carbon steel panels through 1000 hours of ASTM B117 salt fog exposure**

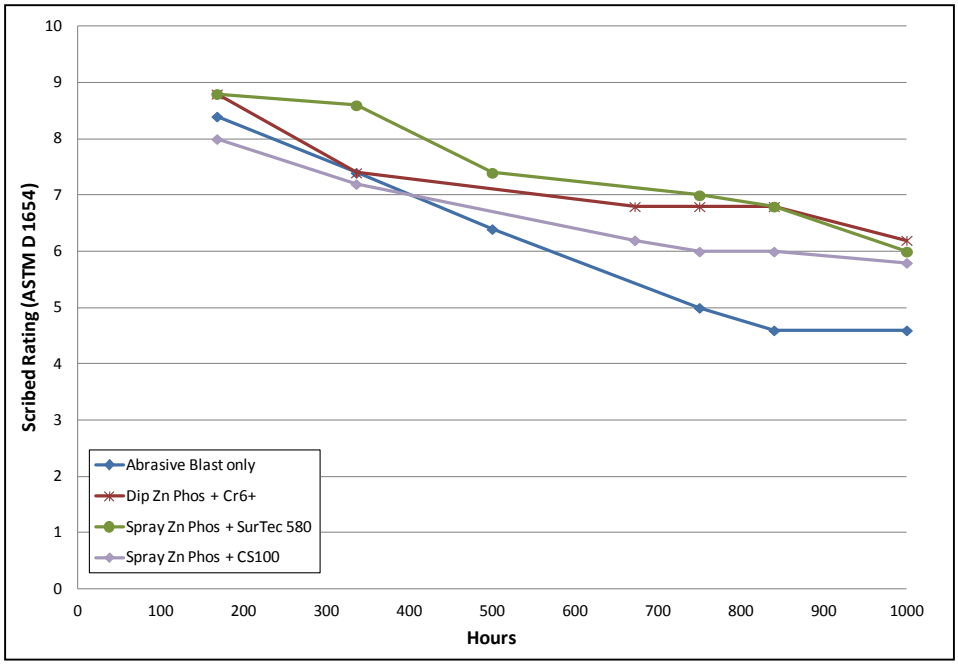
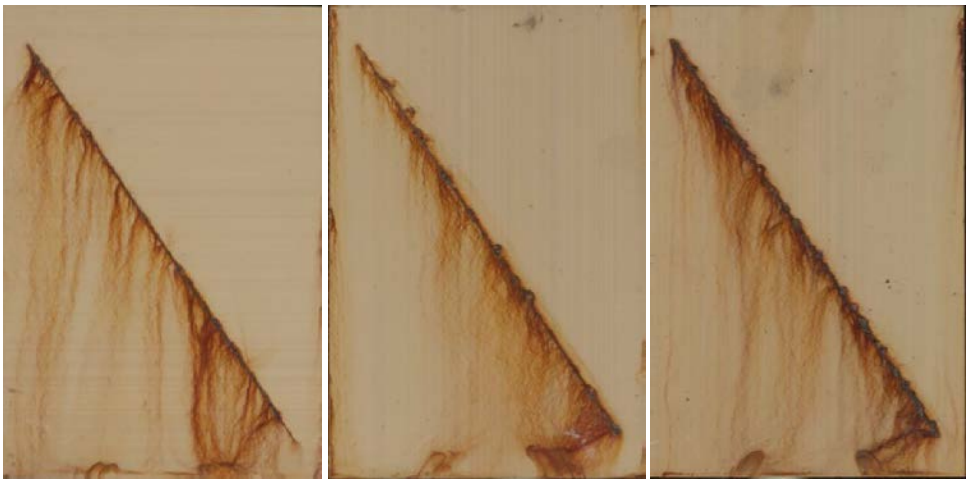



**Figure 6-6: ASTM D 1654 ratings for abrasive blasted HHA panels through 1000 hours of ASTM B117 salt fog exposure**



Cr6+ seal – 6.4 SurTec 580 – 4.2 Chemseal 100 – 5.2

**Figure 6-8: Low carbon steel with MIL-DTL-53022/MIL-DTL-53039 scraped after 1000 hrs B117 exposure. Performance ratings determined by creep-back from scribe.**

Cr6+ Seal – 6.2 SurTec 580 – 6.0 Chemseal 100 – 5.8

**Figure 6-8: Abrasive blasted HHA with MIL-DTL-53022/MIL-DTL-53039 scraped after 1000 hrs B117 exposure. Performance ratings determined by creep-back from scribe.**

Sets of panels with two CARC coating systems, MIL-DTL-53022 / MIL-DTL-53039 and MIL-DTL-53022 / MIL-DTL-64159 were tested in GM 9540P cyclic corrosion on both LCS and HHA. Replicates of 3 were used for the GM 9540P tests for each pretreatment and baseline. The results of the GM9540P tests are presented in Table 6-2 through Table 6-4. The success criteria is an average ASTM D1654, Method A rating of ≥ 4 for "X" scribed panels after 60 hours of exposure. The test was carried out to 80 cycles before scraping the panels for a final rating. There is no specific requirement for cyclic testing for inclusion on the TT-C-490F QPD for MIL-DTL-53022 Type II.

Table 6-2 and 6-3 lists the ratings for each test panel (not averaged), and illustrates the benefits of zinc phosphate with and without an abrasive blasted surface. The abrasive blasted only samples, also known as direct-to-metal, is clearly inferior to zinc phosphate for preparing steel surfaces for paint. None of the direct-to-metal panels could meet the success criterion of ≥ 4 rating at 60 cycles. All of the zinc phosphate panels outperformed the direct-to-metal with both paint system on both substrates.

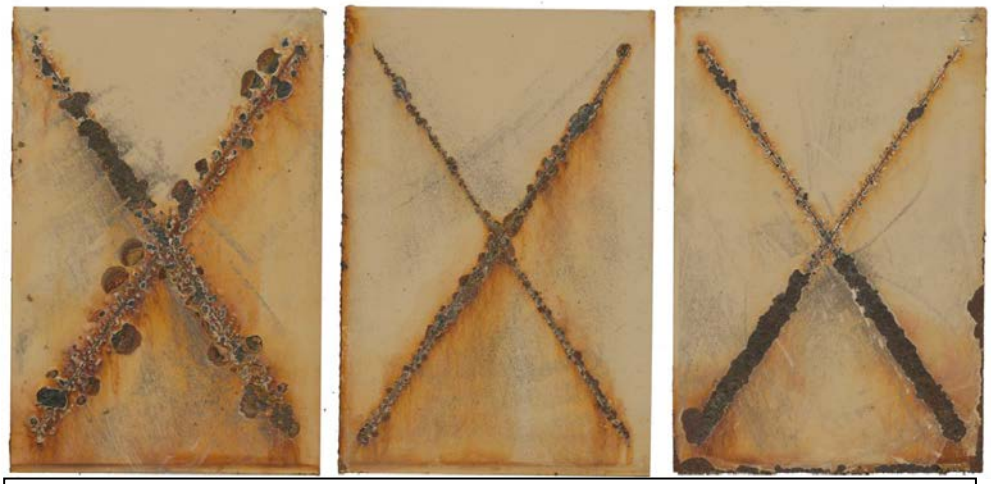
The baseline chromate sealed zinc phosphate panels met the success criterion on only 3 of the 4 substrate/coating combinations, failing on the LCS with MIL-DTL-53022 / MIL-DTL-53039. The ratings in table 6-2 show that both alternatives met the average rating required to pass the success criterion. Although success is determined after 60 cycles, table 6-2 and 6-3 illustrate the extended performance at 80 cycles. The SurTec 580 was the only seal to score a zero on all 3 LCS replicates with the MIL-DTL-53022 / MIL-DTL-64159 CARC system after 80 cycles. The 80 cycles of GM9540P is a challenging metric for even a chromated zinc phosphate to endure without the benefit of an abrasive blasted surface.

Representative test panels after 80 cycles are shown in figure 6-9 through figure 6-12 to better illustrate the performances of the alternatives vs. the baseline chromate sealer. Although the ASTM D1654 rating numbers are the success criteria, the creepage measurements are taken at in

the worst area, but the majority of the coating around the scribe may still be well adhered. For example, in figure 6-9 the Chemseal 100 sample failed primarily in the lower half of the panel, while the SurTec 580 showed more uniform corrosion along the scribe. Both outperformed the chromate phosphate in this case. In figure 6-11, the SurTec 580 panel shown was rated a 0. The low rating here was due to the blister in the lower left corner of the panel. By comparison, this SurTec panel is obviously in better condition than the 0 rated chromate sealed panel in figure 6-9. All of these photographs show that the alternatives performance was very comparable to the chromate, and in some cases, performed better.

**Table 6-2: ASTM-D1654 Ratings for GM9540P Cyclic Corrosion Test of MIL-DTL-53022/MIL-DTL-53039 Coated Steel**

| | | Low Carbon Steel (LCS) | | | | | High Hard Armor (HHA) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | GM 9540P Cycles | | | | | GM 9540P Cycles | | | | |
| Panel | Pretreatment | 10 | 20 | 40 | 60 | 80 | 10 | 20 | 40 | 60 | 80 |
| 1 | Abrasive Blast Only | 8 | 7 | 4 | 2 | 0 | 7 | 6 | 4 | 2 | 0 |
| 2 | | 8 | 7 | 4 | 2 | 0 | 8 | 7 | 4 | 0 | 0 |
| 3 | | 8 | 5 | 3 | 1 | 0 | 8 | 6 | 5 | 2 | 0 |
| 1 | Dip Zn Phos/Cr6+ | 8 | 6 | 5 | 3 | 2 | 8 | 8 | 6 | 6 | 4 |
| 2 | | 8 | 7 | 5 | 4 | 2 | 7 | 7 | 6 | 6 | 4 |
| 3 | | 7 | 6 | 5 | 2 | 0 | 7 | 7 | 6 | 6 | 5 |
| 1 | Spray Zn Phos + SurTec 580 | 7 | 7 | 6 | 3 | 3 | 8 | 7 | 5 | 5 | 3 |
| 2 | | 7 | 7 | 5 | 5 | 4 | 7 | 7 | 6 | 4 | 2 |
| 3 | | 7 | 6 | 5 | 5 | 5 | 6 | 6 | 5 | 5 | 5 |
| 1 | Spray Zn Phos + Chemseal 100 | 8 | 7 | 5 | 4 | 3 | 8 | 7 | 6 | 6 | 5 |
| 2 | | 8 | 7 | 7 | 5 | 3 | 7 | 7 | 6 | 5 | 3 |
| 3 | | 8 | 8 | 7 | 5 | 3 | 8 | 8 | 7 | 6 | 5 |



Cr6+ Seal SurTec 580 Chemseal 100

**Figure 6-9: Low carbon steel with MIL-DTL-53022/MIL-DTL-53039, scraped after 80 cycles of GM 9540P exposure**

| Cr6+ Seal | SurTec 580 | Chemseal 100 |
|---|---|---|

**Figure 6-10: Abrasive blasted HHA with MIL-DTL-53022/MIL-DTL-53039, scraped after 80 cycles of GM 9540P exposure**

**Table 6-3: ASTM-D1654 Ratings for GM9540P Cyclic Corrosion Test of MIL-DTL-53022/MIL-DTL-64159 Coated Steel**

| | | Low Carbon Steel (LCS) | | | | | High Hard Armor (HHA) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | GM 9540P Cycles | | | | | GM 9540P Cycles | | | | |
| Panel | Pretreatment | 10 | 20 | 40 | 60 | 80 | 10 | 20 | 40 | 60 | 80 |
| 1 | Abrasive Blast Only | 8 | 5 | 2 | 0 | 0 | 7 | 5 | 5 | 2 | 0 |
| 2 | | 8 | 5 | 3 | 1 | 0 | 8 | 7 | 5 | 3 | 0 |
| 3 | | 6 | 4 | 3 | 2 | 0 | 7 | 7 | 4 | 1 | 0 |
| 1 | Dip Zn Phos/Cr6+ | 7 | 7 | 5 | 3 | 0 | 7 | 7 | 5 | 5 | 3 |
| 2 | | 7 | 7 | 6 | 4 | 3 | 7 | 7 | 6 | 5 | 4 |
| 3 | | 8 | 7 | 6 | 6 | 3 | 7 | 7 | 5 | 5 | 4 |
| 1 | Spray Zn Phos + SurTec 580 | 7 | 7 | 5 | 2 | 0 | 8 | 7 | 6 | 5 | 3 |
| 2 | | 7 | 7 | 6 | 4 | 0 | 9 | 8 | 6 | 6 | 3 |
| 3 | | 8 | 7 | 5 | 2 | 0 | 8 | 8 | 7 | 7 | 7 |
| 1 | Spray Zn Phos + Chemseal 100 | 7 | 7 | 6 | 5 | 4 | 7 | 7 | 4 | 0 | 0 |
| 2 | | 7 | 7 | 6 | 3 | 1 | 7 | 7 | 6 | 4 | 3 |
| 3 | | 8 | 7 | 5 | 5 | 4 | 7 | 7 | 6 | 5 | 3 |

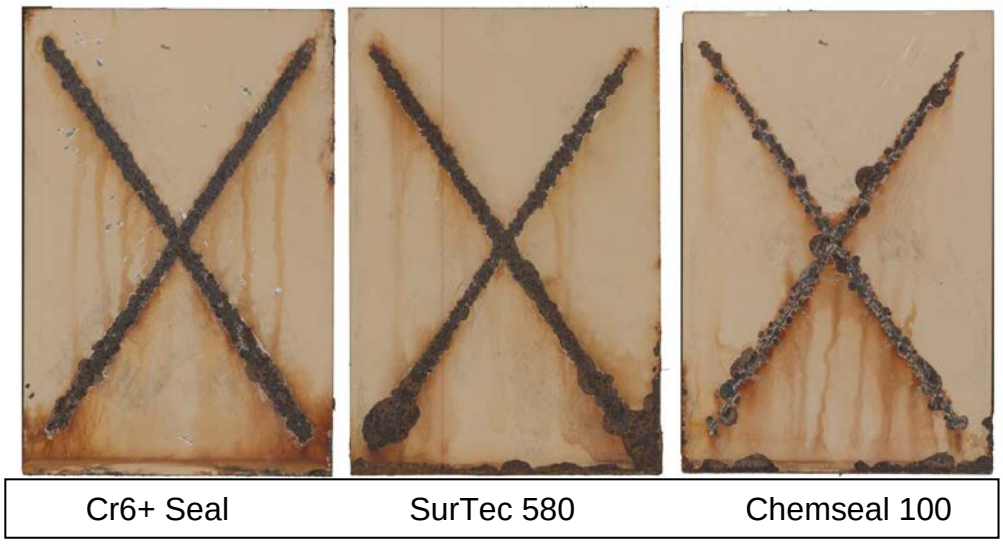



**Figure 6-11: Low carbon steel with MIL-DTL-53022/MIL-DTL-64159, scraped after 80 cycles of GM 9540P exposure**

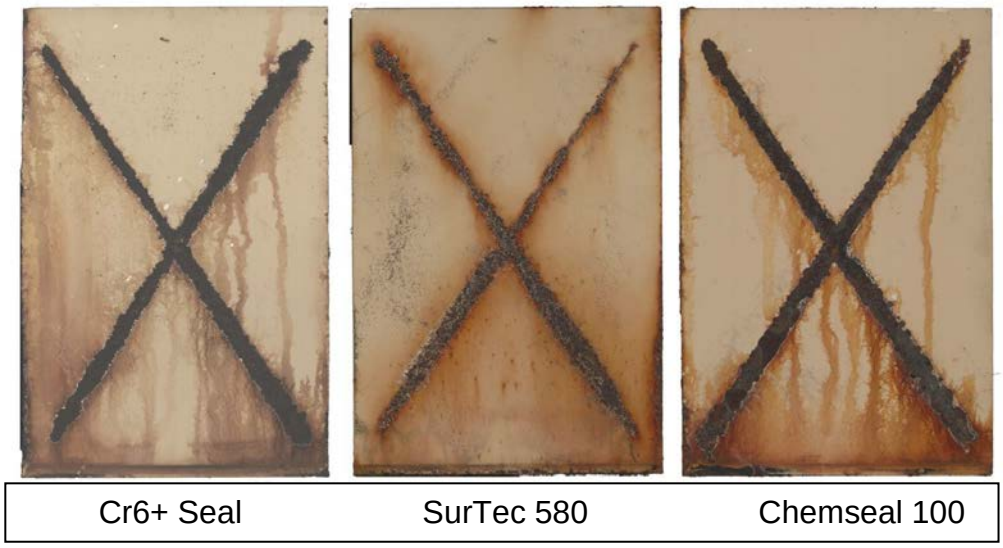



**Figure 6-12: Abrasive blasted HHA with MIL-DTL-53022/MIL-DTL-64159, scraped after 80 cycles of GM 9540P exposure**

Table 6-4 is a summary of the average ratings for GM 9540P testing. This table illustrates how effective zinc phosphate is as a base for organic coatings. The abrasive blasted only (direct-to-metal) was not able to rate batter than a 2.0 average. All of the alternatives and chromate outperformed the direct-to-metal on abrasive blasted and non-abrasive blasted surfaces. However, with the exception of the Chemseal 100, all benefited from the 1.5 mil profile provided by abrasive blasting prior to zinc phosphating. This shows that abrasive blasting coupled with zinc phosphate has superior performance in GM9540P testing than each process individually.

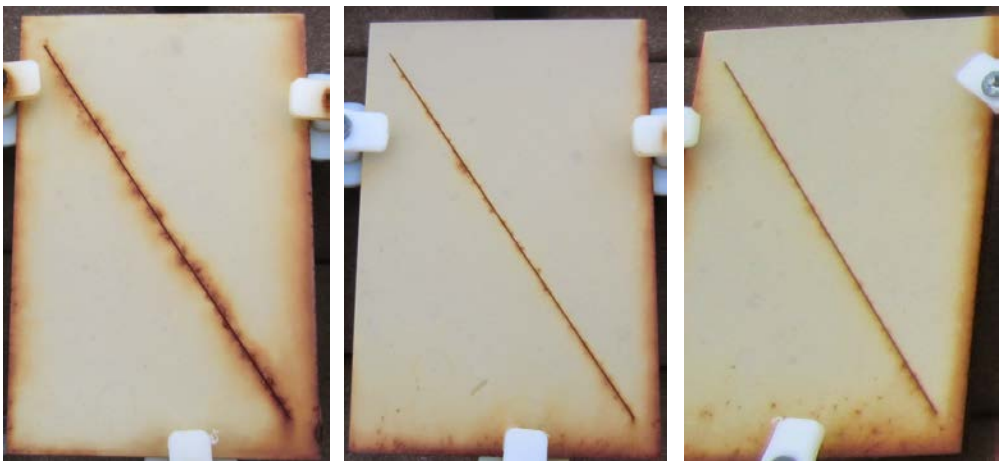
**Table 6-4: Summary of average ratings at 60 cycles of GM9540P indicating success criteria**

| Pretreatment | MIL-DTL-53022/MIL-DTL-53039 | | MIL-DTL-53022/MIL-DTL-64159 | |
|---|---|---|---|---|
| | LCS | HHA | LCS | HHA |
| Abrasive Blast Only | 1.7 | 1.3 | 1.0 | 2.0 |
| Dip Zn Phos/Cr6+ | 3.0 | 6.0 | 4.3 | 5.0 |
| Spray Zn Phos + SurTec 580 | 4.3 | 4.7 | 2.7 | 6.0 |
| Spray Zn Phos + Chemseal 100 | 4.7 | 5.7 | 4.3 | 3.0 |

Outdoor Exposure:
As previously discussed in section 3.0, the success criteria of 25% less creepage from the scribe was modified because this technology area seeks to "replace" the existing technology rather than provide an additional level of protection. For this reason the success criteria is to meet or exceed the performance of the baseline hexavalent chrome sealer after 3 years of outdoor exposure at Cape Canaveral. Inspections and ratings were conducted at 3 years and those results presented in Figures 6-13 through 6-18. The photographs of the representative test panels in 6-13 and 6-14 help illustrate the overall performance of the candidates versus the baseline. In almost all cases the alternatives SurTec 580 and Chemseal 100 met or exceeded the performance of the chromate seal. Figure 6-15 is a side-by-side bar chart rating comparison on low carbon steel. Both SurTec 580 and Chemseal 100 provided better corrosion protection over the 3 years and were more consistent from panel to panel which is indicated by a significantly lower standard deviation.

Cr6+ Seal: 5.3 SurTec 580: 7.0 Chemseal 100: 6.7

**Figure 6-13: Low carbon steel panels with MIL-DTL-53022/MIL-DTL-53039, with ratings indicated after 3 years in outdoor exposure at Cape Canaveral**



| Cr6+ Seal: 5.0 | SurTec 580: 7.0 | Chemseal 100: 6.0 |
|---|---|---|

**Figure 6-14: Low carbon steel panels with MIL-DTL-53022/MIL-DTL-64159, with ratings indicated after 3years in outdoor exposure at Cape Canaveral**

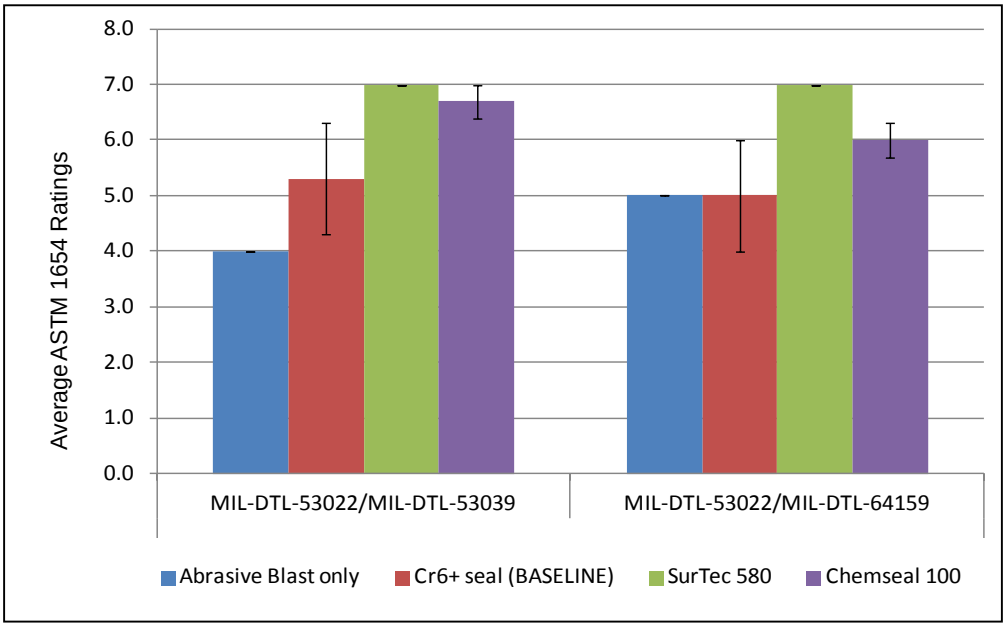


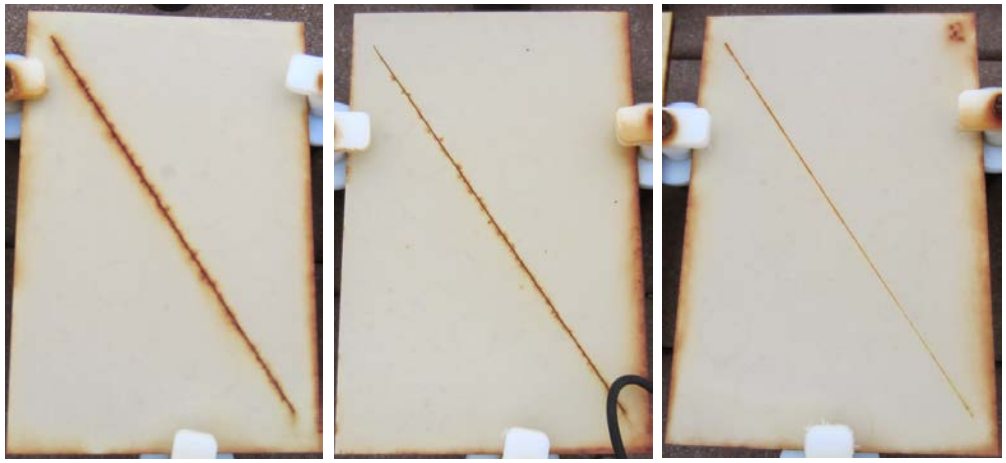

**Figure 6-15: ASTM D1654 Ratings with standard deviation for LCS after 3 years in outdoor exposure at Cape Canaveral**

The abrasive blasted HHA panels in Figure 6-16 show the only case where the alternative sealers did not exceed the performance objective set by the chromate baseline. However, one can clearly see that the performances were actually comparable with all rating a 7.0 or higher. The standard deviation of a 0.6 for the chromate set indicates that the alternatives performed as well, and perhaps more consistently than the chromate. For this reason the performance objectives are considered met for both alternative sealers used on abrasive blasted HHA with the MIL-DTL-53022/MIL-DTL-53039 coating system. It is more evident in figure 6-17 that both alternatives met the performance objectives by exceeding the performance of the chromate sealed panels.

More importantly, over the 3 years of outdoor exposure at Cape Canaveral, both alternatives, SurTec 580 and Chemseal 100 exceeded the performance objective of the chromate in 3 of the 4 cases reported here. Only abrasive blasted HHA panels with MIL-DTL-53022/MIL-DTL-53039 (fig 6-16) failed to exceed, but still met the objectives. The requirement for outdoor exposure testing for inclusion on the TT-C-490F QPD for steel is a rating of ≥ 6 at 2 years. As such, each system tested met the success criteria for the QPD.

| Cr6+ Seal: 7.3 | SurTec 580: 7.0 | Chemseal 100: 7.0 |
|---|---|---|

**Figure 6-16: Abrasive blasted HHA panels with MIL-DTL-53022/MIL-DTL-53039, with ratings indicated after 3 years in outdoor exposure at Cape Canaveral**



| Cr6+ Seal: 5.7 | SurTec 580: 7.0 | Chemseal 100: 7.3 |
|---|---|---|

**Figure 6-17: Abrasive blasted HHA panels with MIL-DTL-53022/MIL-DTL-64159, with ratings indicated after 3 years in outdoor exposure at Cape Canaveral**

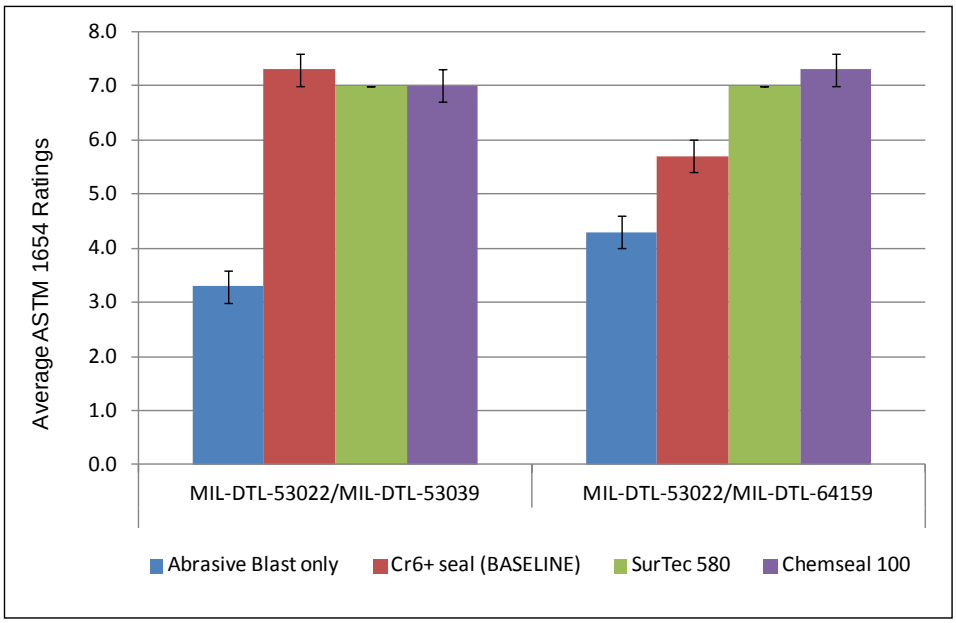



**Figure 6-18: ASTM D1654 Ratings with standard deviation for HHA after 3 years in outdoor exposure at Cape Canaveral**

Hydrogen Embrittlement:
It was important to determine if either of the proposed sealers would have a detrimental effect on the HHA resistance to environmentally assisted cracking. Figure 6-19 shows the $K_I$EAC results that were measured using the rising step load method. When the empirical data for $K_I$EAC is compared with that found in the literature, it is clear that the alternatives had no influence on the MIL-A-46100 resistance to environmentally assisted cracking[16],[17].

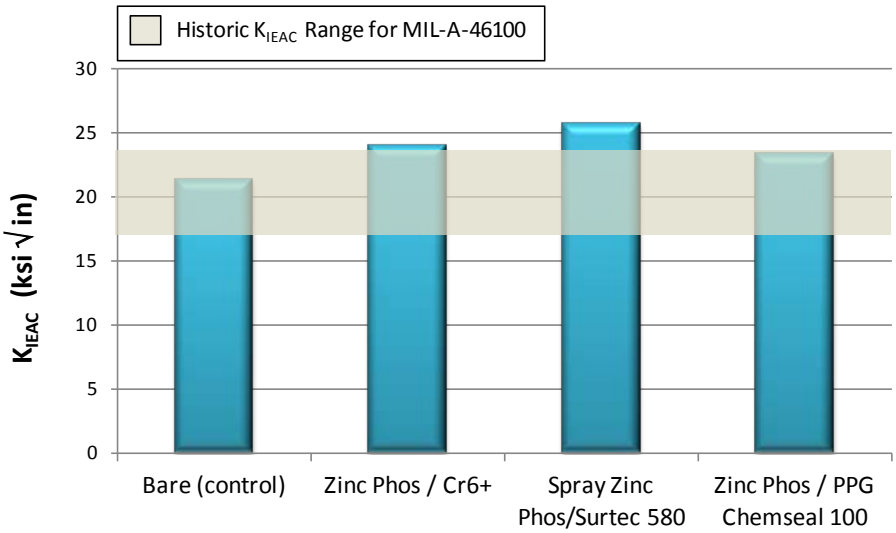



**Figure 6-19: Average $K_{1EAC}$ values of MIL-A-46100 compared to historical range**

## 6.2 Component Demonstration Results

This section discusses the results of the demonstration in a full scale production environment. The demonstration required no major capital investment. The manufacturers of the SurTec 580 were consulted and provided recommended parameters for the application of their product. Step-by-step instructions for mixing the concentrate and the operating parameters were provided to ARL and SurTec technical representatives were on site at ANAD to guide the process. As previously mentioned, the SurTec 580 is essentially a drop-in replacement for the chromic acid solution used at ANAD. Therefore, no changes to the existing zinc phosphate line were necessary. A separate sealing tank was set up adjacent to the phosphate line and parts (see Table 5-5). Panels proceeded through the traditional zinc phosphating process and then were redirected to the SurTec 580 tank for sealing. The zinc phosphating process that was performed at ANAD including the modifications suggested by SurTec International is as follows:

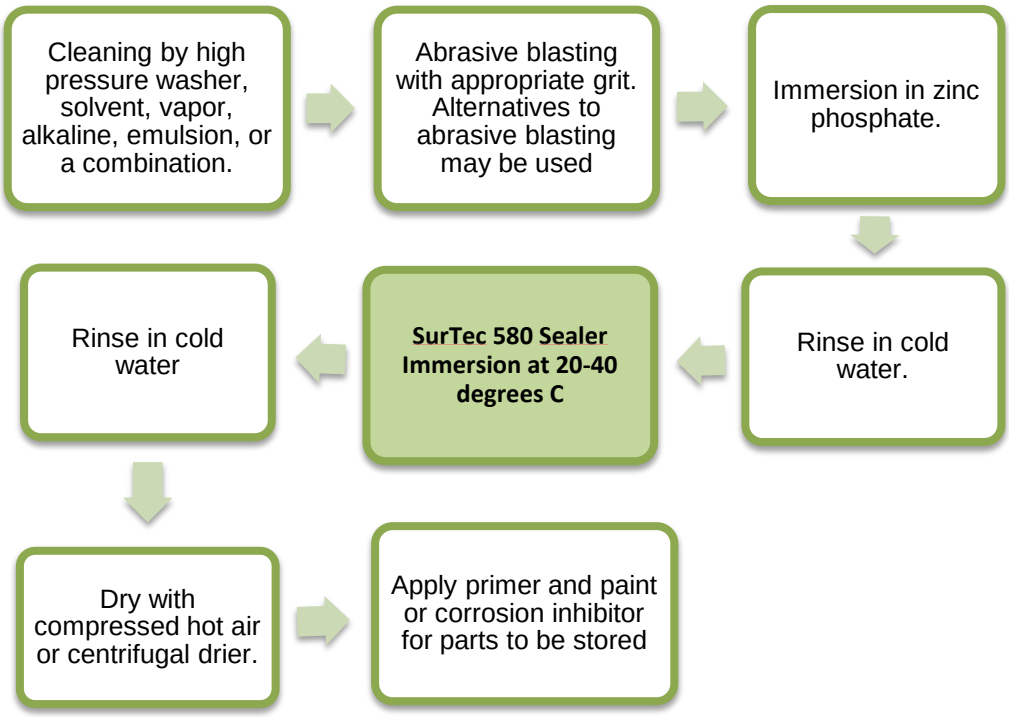



**Figure 6-20: ANAD phosphate process with SurTec 580 modification.**

As mentioned in section 5.4 repeated efforts at demonstration of parts by ARL and ANAD were unsuccessful. Fortunately, representative parts and test panels were retained and brought back to ARL for validation. The primary example of the parts tested at ARL is in figure 6-21 and 6-22. These are two of the M1 fuel caps shortly after being zinc phosphated, sealed (figure 6-21), and following the application of the CARC system MIL-DTL-53022 / MIL-DTL-53039 (figure 6-22). For consistency, the fuel caps remained together throughout the process with the exception of the sealing step.

Cr6+ Seal SurTec 580

**Figure 6-21: ARL M1 fuel caps immediately following zinc phosphate and sealing at Anniston Army Depot**

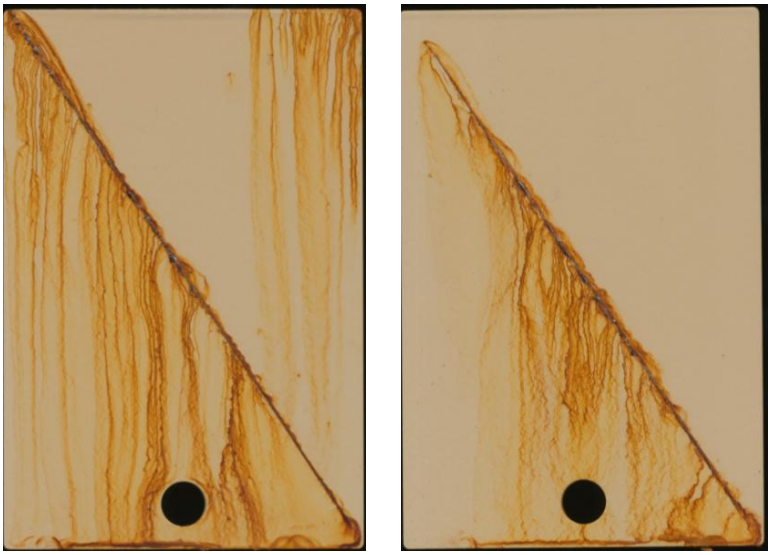
**Figure 6-22: Pictures showing both sides of the ARL M1 fuel caps after processing at Anniston Army Depot**

The largest parts treated in the demonstration were the cupola rings for Stryker. Figure 6-23 shows two cupola rings treated with SurTec 580 each having a slight variation in the rinse process. The cold water rinse resulted in flash rusting, while the hot water flashed off quicker

which reduced the likelihood of corrosion. Although the cold water rinse was adequate for processing smaller parts such as the fuel caps, larger parts with complex geometries should be rinsed using hot water.

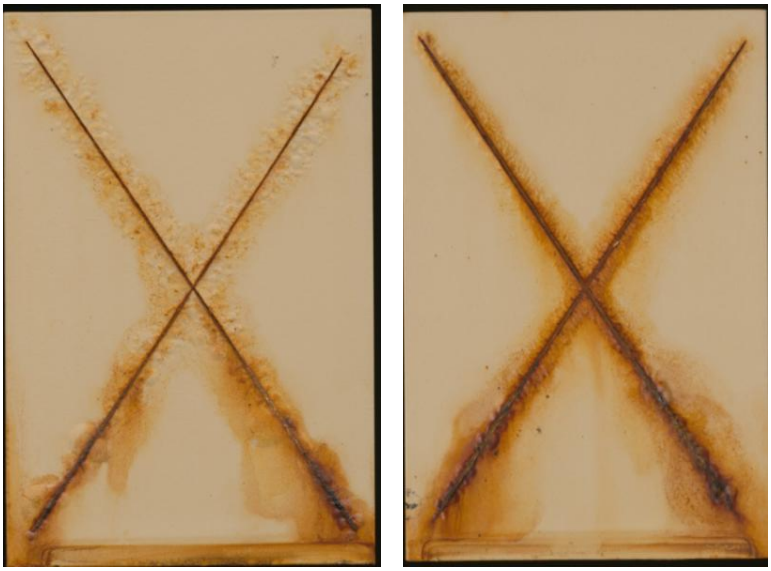
**Figure 6-23: Close up view of the cupola (figure 5-6). On the left shows flash rust when cold rinsed and no flash rusting when hot rinsed.**

Figure 6-24 through 6-26 show the accelerated corrosion test results for test panels processed at ANAD along with the demonstration parts. . In each case, the SurTec 580 met or exceeded the baseline chromate seal. All three panel sets are essentially different substrates. The steel panels in figure 6-24 were provided by ANAD and represent the test panels they use for periodic measurements of phosphate weights, while the other two sets were provided by ARL and are identical to the substrates used in the laboratory evaluations. The results here are also consistent with the accelerated corrosion testing of the laboratory prepared samples reported earlier in Section 6.0. One example is the poor performance of the chromate panel shown in figure 6-25 ( 2.0 rating) mimics the performance observed for same substrate/coating stack-up seen in table 6-2.



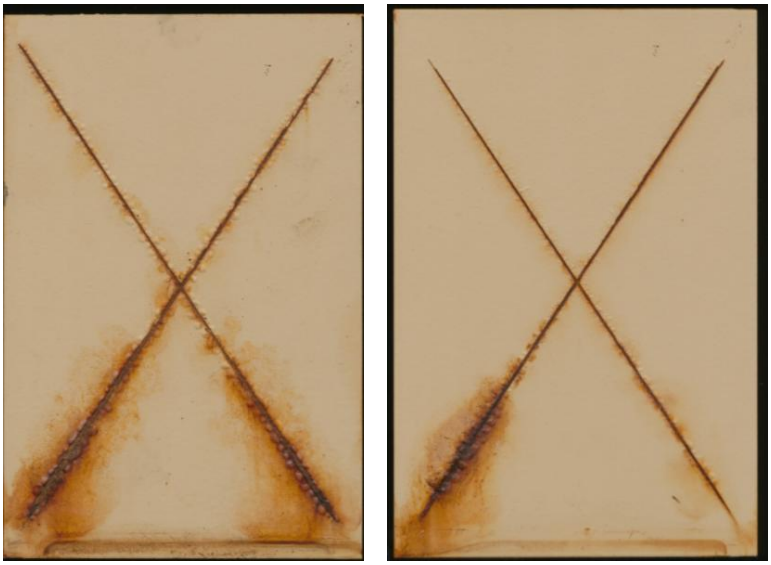
Cr6+ seal – 7.2 SurTec 580 – 7.4

**Figure 6-24: Representative steel test panels from ANAD tested through 1008 hours of ASTM B117 salt spray. Captions contain the ASTM D 1654 rating**



Cr6+ seal – 2.0 SurTec 580 – 5.0

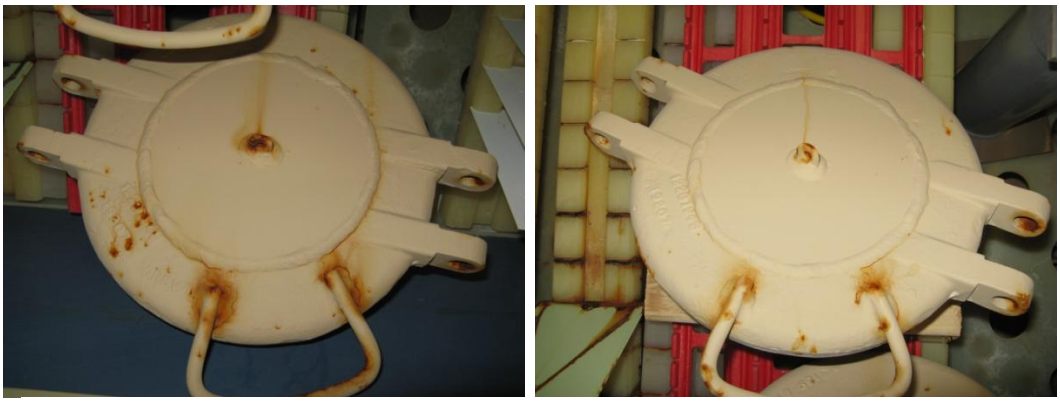
**Figure 6-25: Representative CRS panels after 60 cycles of GM 9540P cyclic corrosion test. Captions contain ASTM D 1654 rating**



Cr6+ seal – 6.0 SurTec 580 – 6.0

**Figure 6-26: Representative HHA steel panels after 60 cycles of GM 9540P cyclic corrosion test. Captions contain ASTM D 1654 rating**

Two of the fuel caps processed at ANAD were brought back the ARL Rodman Laboratory for further testing. The intent was to validate the laboratory results using actual components on platforms in the field as well as using laboratory tests. Figure 6-27 show the components tested at ARL. One chromate sealed fuel cap was compared with one that was sealed with SurTec 580. Prior to exposing the caps to the GM 9540P test chamber, pull off adhesion was performed. The adhesion strength for the SurTec 580 sealed cap was nearly identical to the chromate cap. Both measured in excess of 1670 psi and within 2 psi of each other (1672 psi and 1674 psi). Both fuel caps then completed 60 cycles of cyclic corrosion tests. Examining the caps in 6-27 one may conclude that the SurTec 580 provided better corrosion protection than the chromate. We understand that this is one component and the statistical sample is very small. However, when considering all of the data presented in this report, it is evident that the performance of the SurTec 580 overall is "comparable" to the chromate seal. Figure 6-27 and the results presented in table 6-5 serve as validation for the laboratory and outdoor exposure results compiled thus far. In each case the SurTec 580 met or exceeded the performance of the baseline and met the performance objectives stated in table 6-6.



Cr6+ seal: 4-S rating SurTec 580: 6-S rating

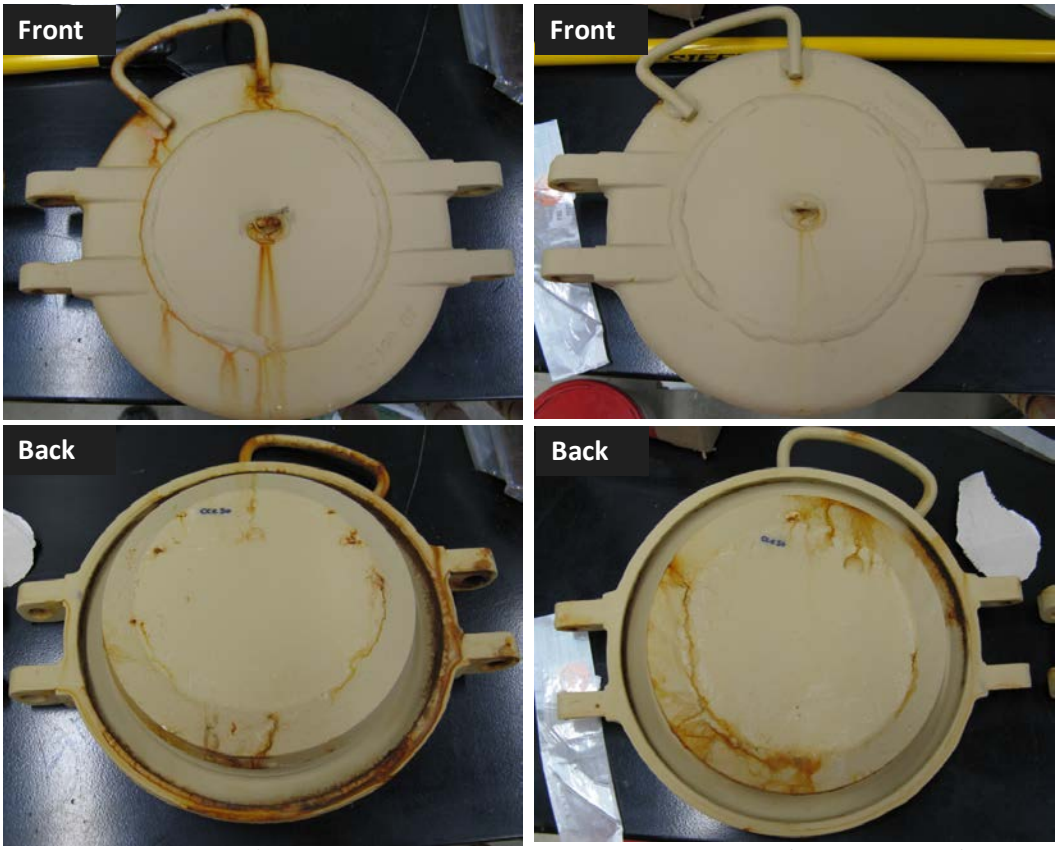
**Figure 6-27: Representative M1 armor steel fuel caps after 60 cycles of GM 9540P cyclic corrosion test with SSPC VIS 2 Ratings**

**Table 6-5: Summary of average ASTM 1654 ratings after 60 cycles of GM 9540P exposure**

| QTY | Substrate | Sealer | Average Rating |
|---|---|---|---|
| 4 | Steel (ANAD) | Chromate | 7.2 |
| 4 | Steel (ANAD) | SurTec 580 | 7.4 |
| 3 | LCS | Chromate | 2.0 |
| 2 | LCS | SurTec 580 | 5.0 |
| 3 | HHA | Chromate | 6.0 |
| 2 | HHA | SurTec 580 | 6.0 |
| 1 | Fuel Cap | Chromate | 4-S* |
| 1 | Fuel Cap | SurTec 580 | 6-S* |

*Corrosion of the irregular surface of the fuel cap was estimated using SSPC-VIS 2 (Table 5-6)

In order to mimic a rework scenario, follow on tests were conducted using the same fuel caps. Each of the fuel caps were reworked by abrasive blasting the corroded areas to bare metal while the well adhered coated areas were left intact. Both fuel caps were pretreated differently and subsequently primered and painted using the CARC system MIL-DTL-53022 / MIL-DTL-53039. The fuel cap that was previously zinc phosphated and sealed with chromate was pretreated with DOD-P-15328 chromated wash primer (figure 6-28, left). The fuel cap that was previously zinc phosphate and sealed with SurTec 580 was pretreated with Oxsilan 9810/2 (figure 6-28, right). The Oxsilan 9810/2 was one of the products demonstrated as part of WP200906[18] and added to the TT-C-490 QPD. Once the coatings were fully cured, both caps were subjected to GM 9540P testing. After only 40 cycles, the chromated wash primer cap was noticeably more corroded than the one pretreated with Oxsilan 9810/2. Although this was rather anecdotal, it was interesting to see that the combination of the two alternatives (SurTec 580 sealed zinc phosphate / Oxsilan 9810/2) held up better that a double treatment of chromate (chromate sealed zinc phosphate / DOD-P-15328).




Cr6+ sealed / DOD-P-15328 SurTec 580 / Oxsilan 9810/2

**Figure 6-28: Reworked M1 armor steel fuel caps after 40 cycles of GM 9540P cyclic corrosion test.**

The primary and secondary performance criteria evaluated in a production setting during the demonstration are listed in Table 6-6, while the metrics for evaluating the SurTec 580 sealer are described in the JTP. The metric for evaluating the fuel caps during the field inspections is a visual comparison with the base vehicle using the Society for Protective Coatings SSPC-VIS-2

"Standard Method for Evaluating the Degree of Rusting on Painted Steel Surfaces". The success criteria for the fielded fuel caps is performance greater than or equal to that of the baseline phosphate system, as well as a the results of a separate test of the parts that are treated and subjected to accelerated corrosion testing at ARL-APG. Any fielded demonstration parts located for inspection and assessed as "failed" will be field repaired using a CARC field repair kit developed by ARL and placed back in service.

**Table 6-6: Validation methods and expected performance metrics**

| Performance Criteria | Expected Performance Metric (Pre-Demonstration) | Performance Evaluation Method | Actual Performance (Post-Demonstration)** |
|---|---|---|---|
| **Primary Performance Criteria** | | | **SurTec 580** |
| Product Testing | The performance of the alternative technology will meet or exceed the current process employed on zinc phosphate lines at ANAD as defined in the JTP in Appendix A. | Laboratory results of demonstration components processed at ANAD | Met |
| | | Field Testing* | N/A |
| Hazardous Materials | Maintains a hex-chrome free platform | Assessment of product constituents and previous studies | Met |
| Hazardous Waste | Meets or exceeds current process used at ANAD | Operating experience and assessments | Met |
| Factors Affecting Technology Performance | Comparison of alternatives in identical operating conditions | Operating Experience | Met |
| **Secondary Performance Criteria** | | | |
| Ease of Use | Man hours and training shall be equivalent to current process. | Operating experience | Met |
| Maintenance | Requirements for record keeping for storage, and clean up shall be equivalent to current process | Compare records | Met |
| Scale up capability | Identify additional equipment, if any, necessary to scale up process for full vehicle treatment. | Operating experience and investigation | Met |

* Data not available. Despite repeated efforts by ARL and ANAD, the parts that were fielded could not be located.

# 7.0 COST ASSESSMENT

This project is unique in that it has three technology areas being demonstrated: Pretreatments for Armor Steel, ZVOC Topcoats for Ground Support Equipment, and Non-Hexavalent Chrome Sealers for Zinc Phosphate. It would be expensive and time consuming to conduct a comprehensive cost assessment on all of the technology areas. An attempt was made earlier in the project to conduct the cost analysis during the MRAP demonstration at Camp Lejeune, but events occurring during that demonstration prevented us from making a reasonable cost and performance assessment. Therefore, an economic and environmental impact study was performed for this demonstration by Dr. Keith Legg of Rowan Technology Group and presented here.

*Technology*

Zn Phosphate coatings are sealed with a dilute chromic acid passivate. The alternative tested in this program is an non-chromate product, SurTec 580, a derivative of the trichrome Zr passivated NAVAIR TCP chemistry. The phosphate rinse tank at Anniston is shown in Figure 7-1.
The primary processing differences are:

1. Chromic acid is applied at 175 -200°F, while the alternative is applied at ambient temperature.
2. The alternative requires a post-process rinse not used for chromate. In some cases a hot water spray rinse may be used for a better visual appearance of the product, but this is not a standard process requirement.
3. The chromate tank requires ventilation, but no additional PPE beyond rubber gloves and aprons.

**Figure 7-1: Chromic acid rinse tank in phosphate line at (ANAD).**

The chromic acid process and its alternative are compared in Figure 7-2.

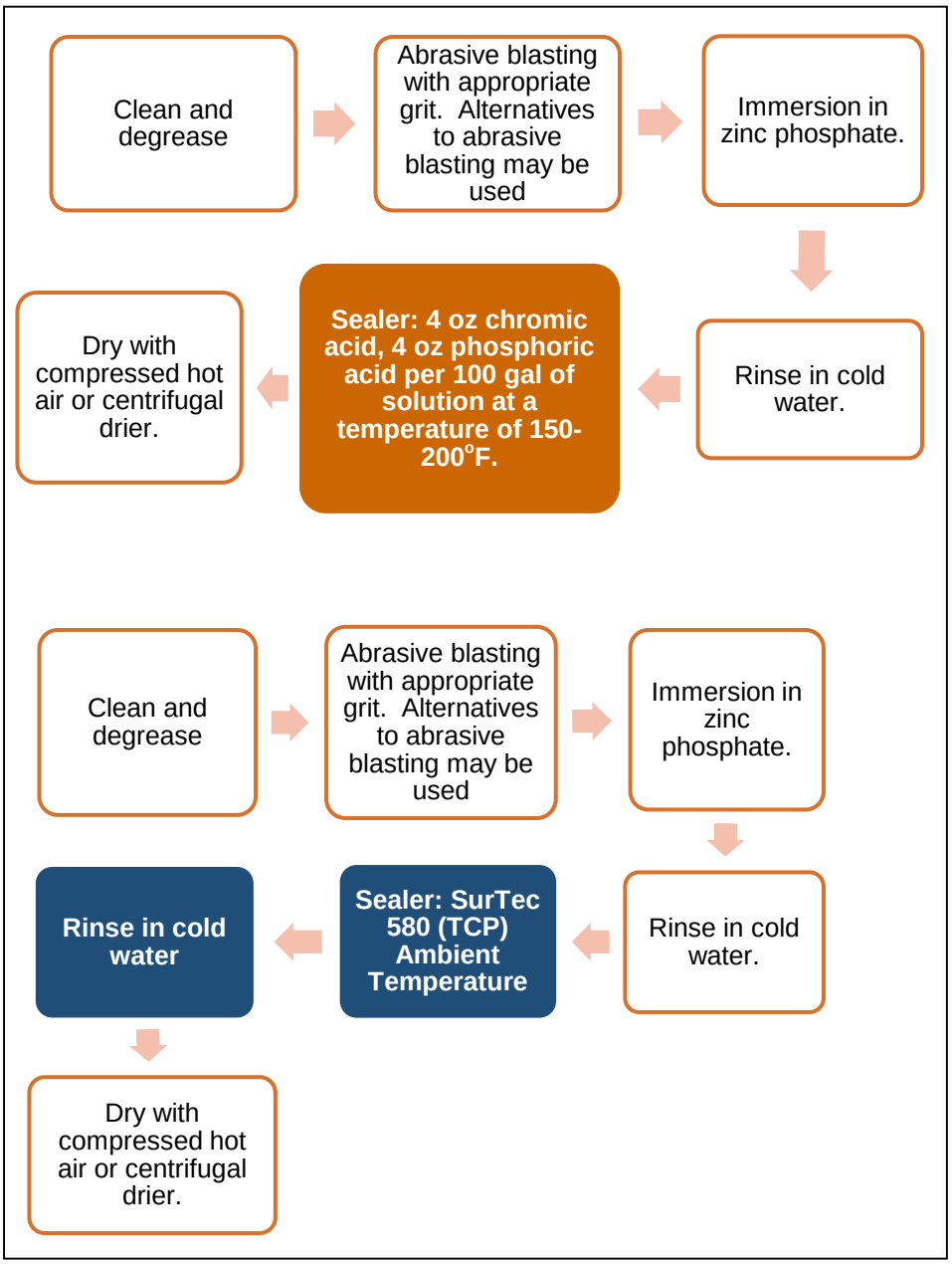



**Figure 7-2: Top: Current process - Zinc phosphating and chromic acid sealing. Bottom – Chromate-free sealer replacement**

*Cost Benefit*

The costs for the two processes are compared in Table 7-1. This table shows the primary costs. Costs that are unaffected or largely unchanged are not included in the analysis. It is immediately clear that the primary difference in cost comes from the difference in bath temperatures.

## Table 7-1: Cost factors – Chromic acid seal vs. SurTec 580

| Direct Manufacturing | Annual or Per-item cost Chromate | Annual or Per-item cost SurTec 580 | Notes |
|---|---|---|---|
| **Chemicals** | | | |
| Tank volume | 2,400 | 2,400 | |
| percentage sealer required (wt%) | 0.03% | 1.5% | |
| Total volume of chemical requirement to fill tank | 6.00 | 36 | |
| Chemical cost per gal | $ 1.10 | $ 25.00 | |
| Bath replenishment chemical volume | $ 6.61 | $ 900.00 | |
| Dragout rate | 1 | 1 | |
| Dragout losses, annual | 2,000 | 2,000 | |
| Bath replenishment chemical frequency | | | |
| Annual chemical usage | $ 6.61 | $ 900.00 | |
| Annual water usage for plating | | | |
| **Utilities** | | | |
| Bath temperature, mid-range (deg F) | 175 | 85 | |
| Evaporation rate (l/hr) | 81.7 | 4.56 | |
| Evaporation rate (l/year) | 715,692 | 39,946 | |
| Makeup water cost | $ 379 | $ 21 | |
| Evaporation energy (kWh/year) | 515,298 | 0 | SurTec is ambient |
| Bath heating cost (steam) | $ 30,918 | $0 | SurTec is ambient |
| Bath heating cost (electrical) | $ 36,071 | $0 | SurTec is ambient |
| **Labor** | | | Same |
| **Indirect Manufacturing** | | | Same |
| **Environmental and Health Costs** | | | |
| **Regulatory compliance** | | | Minimal change |
| **Pollution prevention equipment** | | | Minimal change |
| **Worker health** | | | Minimal change |
| **Waste management** | | | Minimal change |
| **Adoption Costs for New Technology** | | | |
| Additional equipment | | | |
| Drain and dispose of chromate | | $ 1,000 | |
| Tank cleaning and liner replacement | | $ 1,000 | |
| Recharge with SurTec 580 | | $ 900 | |
| Control equipment, filter | | | Probably not needed |
| Paperwork changes | | | Unknown |
| **Year 1 cost** | **$ 31,303.82** | **$ 3,821.17** | |
| **Annual cost (steam heat)** | **$ 31,303.82** | **$ 921.17** | |

The high temperature chromic acid bath must be maintained at temperature 24/7. During this time water is evaporated and makeup water must be added. The evaporation rate depends on temperature, bath surface area and air flow rate over the water surface. The evaporation rate for the two baths was calculated using the NMFRC Plating Tank Evaporation Calculator[19]. This makeup water must be raised to bath temperature. The evaporating water carries off latent heat of vaporization, which must also be replaced to maintain bath temperature. Since the SurTec 580 bath operates at ambient, it only requires water replenishment (to compensate for evaporation and dragout), not water heating. This calculation is shown in Table 7-2.
The annual saving is strongly dependent on the chromate bath temperature used and the efficiency of the heating system (see Figure 7-2). Baths are usually steam heated by steam piped throughout the plant. If the energy conversion and transmission efficiency were both 100% the cost would be very low. If the efficiency is in the region of 50% for converting gas thermal energy to water thermal energy, and about 50% for transmission and bath heating (overall thermal efficiency 25%), this makes gas 10-15% more lower in cost than electrical immersion heating, which is close to 100% efficient (Figure 7-3).

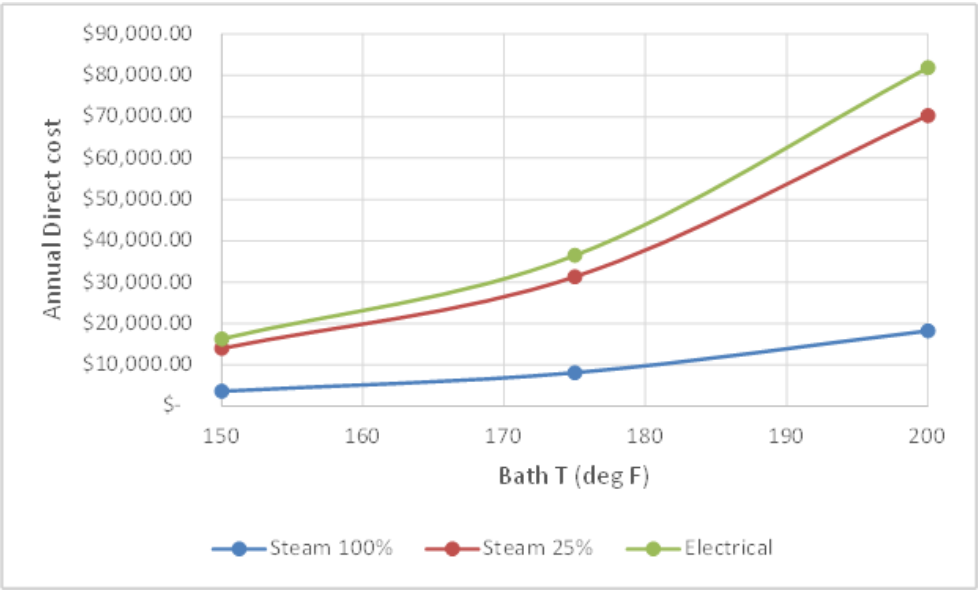



**Figure 7-3: Direct cost of chromate processing as function of bath temperature and heating efficiency.**

Assuming gas heating with overall efficiency of 25% (very similar to electrical heating) the annual saving achieved by replacing chromic acid passivation with SurTec 580 is about $30,000/year. This produces a 15-year NPV of $270,600, with an immediate payback since the changeover will cost only $3,000 plus any costs for changes to paperwork, local specs, and the cost of disposing of the chromic acid in the bath to the base POTW. Since there is no capital cost and the adoption is so low, the annualized ROI is about 1,000%, making measures such as ROI and IRR essentially meaningless. The annual cost saving and the NPV roughly double if the chromate bath typically operates at the upper end of its range (200°F), and are roughly halved if it typically operates at the lower end (150°F).

## Table 7-2: Calculation of Evaporation and Water Usage

| Item | Amount | Units | Chromic acid | SurTec 580 | Units | Notes |
|---|---|---|---|---|---|---|
| Bath dimensions | | | | | | |
| width | 60 | inch | 152 | 152 | cm | approx |
| height | 40 | inch | 102 | 102 | cm | approx |
| length | 240 | inch | 610 | 610 | cm | approx |
| Bath liquid volume | 2,400 | gal | 9,120 | 9,120 | liters | |
| Surface area | 14,400 | in2 | 92,903 | 92,903 | cm2 | |
| Temperature | | | 175 | 85 | deg F | Cr 150-200, SurTec 70-104 |
| Air flow velocity | | | | | | Not known |
| | | | | | | |
| Water balance | Amount | Units | Chromic acid | SurTec 580 | Units | |
| Evaporation rate (avg T) | | | 81.7 | 4.56 | l/hr | http://nmfrc.org/subs/evapcalc.cfm |
| | | | 715,692 | 39,946 | l/yr | |
| Water makeup cost | 0.0005263 | $/l | $377 | $21 | per yr | |
| 85F | | | | 4.56 | l/hr | |
| 150F | | | 36.48 | | l/hr | |
| 175F | | | 81.7 | | l/hr | |
| 200F | | | 183.54 | | l/hr | |
| Dragin and dragout rate | | | | | | Not known |
| Energy balance | | | | | | |
| Heat | 0.09 | kWh/l | | | | Heat makeup water 60 to 175F |
| Evaporation | 0.63 | kWh/l | | | | Evaporate at 175F |
| Heat + Evaporation | 0.72 | kWh/l | 58.64 | | kW | power to replace evaporation |
| Evaporation power/year | | | 5.1E+05 | | kWh | power to replace evaporation/year |
| Natural gas heating cost | | | $ 7,706 | | per year | Natural gas heating plant |
| Cost inc energy efficiency | Steam 25% | | $ 30,822 | | per year | 50% generation and 50% transmissions losses |
| Electrical heating cost | | | $ 35,960 | | per year | 100% efficiency for immersion heater |
| Heat loss from surface | | | | | | Small |
| Heat loss from walls | | | | | | Small |
| Heat loss from parts | | | | | | Small |
| Chemical balance | | | | | | |
| Initial charge Surtec 580 | 1.5% | | | 136.8 | kg | |
| | $ 6.60 | /liter | | $903 | | Total cost for tank charge |
| Initial charge CrO3 | 0.03% | | 2.7 | | kg | |
| | $ 2.22 | per kg | $ 6.08 | | | Total cost for tank charge |
| Annual chemicals | | | $ 6.08 | $903 | | One bath turnover/year |

# 8.0 IMPLEMENTATION

The implementation of non-hexavalent chrome sealers for zinc phosphate will be expedited by the recent publication of the reconstructed Federal specification TT-C-490F. This specification has been the overarching document referenced in dozens of military coating specifications and tens of thousands of military drawings for the cleaning and pretreatment of (only) ferrous substrates prior to the application of organic finishes such as CARC. It has been the primary reference preferred by engineers to specify cleaning, pretreatment, and subsequent testing. It is widely used by all OEMS and Services for finishing steel. However, technical major gaps existed in this specification that motivated the significant changes. First, previous versions of TT-C-490 continued to specify the use of hexavalent chromium in surface finishing although hex chromium has been targeted for elimination for years. With many other specifications that continue to require hexavalent chrome usage, there was no official mechanism to validate, approve and implement alternative surface finishing operations except through contract waivers, drawing changes and engineering change notices, which can be an expensive and a cumbersome process. Also, no comprehensive specification existed governing the cleaning and pretreatment of DoD relevant metallic and multimetal substrates.

The U.S. Army Research Laboratory recognized the synergy that existed between the TT-C-490 reconstructions and the ESTCP funded project WP200906 to examine alternative steel pretreatments and sealers for zinc phosphate. Several of the candidates evaluated were found to at least achieve the performance requirements and in some cases exceed the performance of existing hexavalent chrome pretreatments. The reconstruction of TT-C-490 adopted much of the JTP and success criteria developed under WP200906 as a basis for the performance specification. With this improved testing regimen, ARL can transition pretreatment and sealers that meet ARL's established performance criteria into use through the Qualified Product Database in a seamless structure that will eliminate the costly time consuming and expense of waivers and engineering change notices. This procedure will encourage innovation because of a well-defined path to approval for qualified products.

Details of TT-C-490 Revision F can be seen in the specification in Appendix C. The revisions enable many improvements to multiple alloy finishing operations within industry and the DoD. These improvements include:

1. Provides new commercially available technologies, such as those used by the automotive industry, a pathway for implementation and use on military systems and reduces bureaucracy.
2. ARL provides stewardship of TT-C-490F and monitors approval process.
3. Includes a plan for Objective Quality Evidence that will improve overall quality of new and legacy processes.
4. Governs pretreatments for aluminum and multi-metal applications, (TT-C-490 no longer limited to steel).
5. Establishes a qualified products database (QPD) that will include new Types and Classes.
6. Updates and provides better detail cleaning requirements.
7. Encourages innovation and promotes low-energy and green technologies.

The revised TT-C-490 includes new Types and Classes and ties them to specific cleaning Methods to accommodate steel, aluminum and multi-metal substrates as well as corrosion resistant metal-rich coatings.

The revised document is being adopted by entire DoD and beyond (i.e.: industry) for surface finishing of alloys – TACOM has adopted the language and principles of Objective Quality Evidence in the new TT-C-490F specification and has begun placing it in their Procurement Automated Data and Document System (PADDS clause) for pretreatments and CARC on all new contract requirements that requires all DoD and DoD contractors to follow the doctrine of the newly revised TT-C-490F specification.

The QPD has been populated by two of the products evaluated in WP200906: SurTec 650 and Oxsilan 9810/2. These two spray applied pretreatments have been approved for abrasive blasted steel substrates. Additionally, SurTec 580, the product demonstrated in this report, has met or exceeded the success criteria and has been qualified for inclusion on the TT-C-490F qualified product database. It will be available for users as a qualified alternative to chromic acid rinse for zinc phosphate.

# 9.0 REFERENCES


1 TT-C-490 Cleaning Methods for Ferrous Surfaces and Pretreatments for Organic Coatings

2 MIL-DTL-16232, Detail Specification: Phosphate Coating, Heavy, Manganese Or Zinc Base

3 Effect of zinc phosphate chemical conversion coating on corrosion behavior of mild steel in alkaline medium: protection of rebars in reinforced concrete, Florica Simescu and Hassane Idrissi, 2008 Sci. Technol. Adv. Mater. 9 045009 (http://iopscience.iop.org/1468-6996/9/4/045009)

4 MIL-DTL-53072: U.S. Department of Defense, "Chemical Agent Resistant Coating (CARC) System Application Procedures and Quality Control Inspection", Washington, DC, June 2003.

5 Non-Chromate/No VOC Coating System for DoD Applications, Strategic Environmental Research and Development Program (SERDP) final report March 31, 2009, Dr. John LaScala, US Army Research Laboratory.

6 Electrochemical Impedance Spectroscopy of Trivalent Chromium Pretreated Aluminum Alloys, Vinod S. Agarwala NAVAIR, Dieter W. Beckert, Wehrwissenschaftliches Institut fur Materialuntersuchungen, December 14 1993, TR No. NAWCADWAR-94014-60

7 Demonstration and Validation of Trivalent Aluminum Pretreatment on U.S. Navy S-3 Aircraft, Craig Matzdorf, Michael Kane, James Green, NAVAIR

8 Trivalent Chromium Process (TCP) as a Sealer for MIL-A-8625F Type II, IIB, IC Anodic Coatings, Craig Matzdorf, Erin Beck, Amy Hilgeman, Ruben Prado, NAVAIR, August 29, 2008 TR No. NAWCADPAX/TR-2008/104

9 Extended Performance Assessment in Accelerated Corrosion and Adhesion of CARC Prepared Aluminum Alloy 5059-H131 for Three Different Pretreatment Methods, Brian Placzankis, ARL, Amy Hilgeman, NAVAIR, NACE Paper No 08208, 2008

10 Qualification of Trivalent Chrome as a Hexavalent Chromate Alternative for Propellant and Cartridge Actuated Devices, Harry L. Archer, NAVAR, Indian Head Division, Final Report IHTR 2922, July 28 2008

11 Anniston Army Depot Programming Compliance, Robyn Kreimborg, Charlotte Pugh, Environmental Program Management. http://www.careers.ch2m.com/assets/pdfs/military/army_engineer_anniston_final_nov09.pdf

12 MIL-DTL-0053022: “Primer, Epoxy Coating, Corrosion Inhibiting, Lead and Chromate Free,” U.S. Department of Defense, Washington DC, January 2007

13 MIL-DTL-53039: “Coating, Aliphatic Polyurethane, Single Component, Chemical Agent Resistant,” U.S. Department of Defense, Washington DC, June 2005.

14 MIL-DTL-64159 Chemical Agent Resistant Coating (CARC), polyurethane, water dispersible

15 Examination of Alternative Pretreatments to Hexavalent Chromium-Based DOD-P-15328D Wash Primer for MIL-A-46100D High Hard Steel Armor, Brian E. Placzankis, Chris E. Miller, and John V. Kelley

16 Herman, W. A.; Campbell, G. M. Environmental Assisted Cracking in High Hardness Armor Steel. TR-85-28; U.S. Army Materials and Mechanics Research Center: Watertown, MA, 1985.

17 Herman, W. A.; Campbell, G. M. Environmental Assisted Cracking in High Hardness Armor Steel. TR-85-28; U.S. Army Materials and Mechanics Research Center: Watertown, MA, 1985.

18 Jack Kelley, Tom Braswell, “Non-Chromate, ZVOC Coatings for Steel Substrates on Army and Navy Aircraft and Ground Vehicles: Technology Area 1-“Demonstration of Steel Pretreatments on High Hard Armor Steel” Army Research Laboratory, ESTCP Project 200906 Final Report, 2014

19 Available at http://nmfrc.org/subs/evapcalc.cfm

# APPENDICES

## Appendix A: Joint Test Protocol

# Joint Test Protocol

# Validation of Pretreatment Sealers for Steel

# Draft

# May 18, 2011

Distribution Statement "A" applies.
Approved for public release; distribution is unlimited.

*Prepared and Submitted by the*
*U.S. Army Research Laboratory,*
*Corrosion Science and Engineering Team,*
*Aberdeen, Md.*

*Format and context of this report were developed using*
**Joint Test Protocol**
**J-01-GV-002-P2**
**Validation of Corrosion Protection for**
**Ground Vehicle Frame Structures**
**Draft**
**July 20, 2007**

JTP Approved & Authorized By: _____

*Name, Organization, Date*

## TABLE OF CONTENTS

**Page**


**1.0 INTRODUCTION ..... 1**
1.1 Scope ..... 1
1.2 Execution ..... 1
1.3 Document Maintenance ..... 3
**2.0 JTP DOCUMENT GUIDE ..... 4**
**3.0 APPLICATION SCENARIOS ..... 8**
3.1 Guidelines ..... 8
3.2 Methodology ..... 9
**4.0 TESTING REQUIREMENTS, DESCRIPTIONS AND PROCEDURES ..... 11**
4.1 Screening Testing Requirements ..... 12
4.2 Performance Testing Requirements ..... 13
4.3 Special Testing Requirements ..... 16
4.4 Test Descriptions ..... 17
4.4.1 Adhesion (Dry) (ASTM D3359) ..... 17
4.4.2 Wet Adhesion (ASTM D3359, as modified by NAVAIR) ..... 19
4.4.3 Adhesion (Pull off) (ASTM D-4541) ..... 20
4.4.4 Corrosion Resistance (Cyclic) (GM 9540P) ..... 22
4.4.5 Corrosion Resistance (Neutral Salt Spray (Fog)) (ASTM B117) ..... 24
4.4.6 Chip Resistance (SAE J400) ..... 26
4.4.7 Rising Step Load (Stress Corrosion Cracking) ..... 28
4.4.8 Field Exposure, Static (ASTM G50) ..... 30
4.4.9 Field Exposure, On-Vehicle (ASTM D 1654) ..... 31
**5.0 FAILURE ANALYSIS ..... 33**
**6.0 REFERENCE DOCUMENTS ..... 34**


## LIST OF FIGURES


Figure 1. Test Flow Diagram ..... 5
Figure 2. Generic Substrate and Corrosion Protection System Model (not to scale) ..... 8


## LIST OF TABLES


Table 1. Test Method Matrix ..... 9
Table 2. Screening Testing Requirements ..... 13
Table 3. Performance Testing Requirements ..... 15
Table 4. Special Testing Requirements ..... 17
Table 5. Rating for Number of Chips Within 4" x 4" Grid Lines ..... 27
Table 6. Rating for Size of Chips ..... 27
Table 7. Point of Failure ..... 27
Table 8. Reference Documents ..... 34

## APPENDIX

APPENDIX: List of Stakeholders and List of Contributors ..... A-1

## ACRONYM LIST

| | |
|---|---|
| A2LA | American Association for Laboratory Accreditation |
| ACDT | Accelerated Corrosion Durability Test |
| AISI | American Iron and Steel Institute |
| ANSI | American National Standards Institute |
| ARDEC | U.S. Army Armament Research, Development and Engineering Center |
| ARL | U.S. Army Research Laboratory |
| ASTM | American Society for Testing and Materials |
| ATC | U.S. Army Aberdeen Test Center |
| BP | Best Performance |
| CARC | Chemical Agent Resistant Coating |
| cfm | Cubic Feet per Minute |
| COTS | Commercial Off-the-Shelf |
| CPC | Corrosion Preventive Compound |
| *CTC* | Concurrent Technologies Corporation |
| DoD | Department of Defense |
| VPS | Validation of Pretreatments for Steel |
| HSLA | High-Strength Low-Alloy |
| IP | Improved Performance |
| JG-PP | Joint Group on Pollution Prevention |
| JTP | Joint Test Protocol |
| JTR | Joint Test Report |
| kPa | Kilopascal |
| lb | Pound |
| mg | Milligram |
| mm | Millimeter |
| MMC | Metal-Matrix Composite |
| MP | Minimum Performance |
| NLT | Not Less Than |
| NSS | Neutral Salt Spray |
| NVLAP | National Voluntary Laboratory Accreditation Program |
| PFL | Product Failure Laboratory |
| psi | Pounds per Square Inch |
| QPL | Qualified Products List |
| SAE | Society of Automotive Engineers |
| SS | Salt Spray |
| TACOM | U.S. Army Tank-automotive and Armaments Command |
| TDPMD | Technology Demonstration for Prevention of Material Degradation |
| U.S. | United States |
| VOC | Volatile Organic Compound |

# PREFACE

This Joint Test Protocol (JTP) was prepared by the Army Research Laboratory Corrosion Science and Engineering Team. The objective of this JTP is to select and implement the most appropriate approaches for the improvement of the control of material degradation on Army materiel and assets, thereby reducing life cycle operational costs and maximizing equipment sustainability for the warfighter.

Format and context of this report were developed using Joint Test Protocol J-01-GV-002-P2 Validation of Corrosion Protection for Ground Vehicle Frame Structures (Draft), July 20, 2007. The depth of technical content of this JTP was determined by technical associates, pertinent United States (U.S.) Army personnel, government contractors, and other government and commercial technical representatives (hereafter referred to as "stakeholders") who are participants in the Integrated Product Team (IPT) of the ESTCP funded project for Non-Chromate Zero-VOC Coatings for Army and Navy Ground Vehicles.

## JTP REVISIONS HISTORY

This section will serve as a means to document revisions and discussions regarding this JTP only. It is intended to help the reader identify updated versions of the JTP, and to organize periodic updates of the JTP as new materials and techniques become available. If the latest entry on the JTP Revisions History is more than two (2) years old, the entry "No revisions have been made for the year 20xy" will be entered where appropriate.

| Letter | Pages Changed | Pages Added | Description | Date | Approved by (Signature/Division) |
|---|---|---|---|---|---|
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

## 1.0 INTRODUCTION

This JTP contains the critical requirements and tests necessary to evaluate sealers for pretreatment technologies such as phosphate for use on U.S. military steel components. The JTP provides a standard set of tests and test conditions that the manufacturers, the U.S. military, and third-party testing organizations may use to fairly gauge how the technology compares to existing pretreatment sealer technologies. With the test results presented in a Joint Test Report (JTR), the manufacturer and military can make an informed decision with regard to subjecting the technology to qualification testing for inclusion on the Qualified Products List (QPL). This document is a protocol for testing and assessing the performance of any potential pretreatment sealers for corrosion prevention, or any repair process or maintenance process involving pretreated steel. The potential technologies for consideration will hereafter be referred to simply as "candidates." Candidate steel pretreatment sealer processes shall not exceed 160$^{o}$C in order to qualify for testing.

### 1.1 Scope

This JTP establishes the corrosion-resistance performance requirements that must be met for a candidate to be considered for use on military steel components. Other properties of potential candidates will also be considered (see Feasibility Study discussion in the next section). However, evaluations of these properties are specific to the application, and will be considered acceptable based only upon equal or improved performance when compared to the corrosion protection system currently being used.

**It must be emphasized that this JTP document is not a process, material, or product specification, nor is it intended to address ongoing quality issues.** The testing outlined in this document confirms the technical capabilities of the candidate for the particular application with respect to corrosion resistance, and qualifies the candidate for consideration for military use by the relevant armed services' Corrosion Office invoking the JTP (e.g., the Army Corrosion Manager) or the relevant Program Manager (hereafter referred to as the "invoking authority"). ***It should also be emphasized that successful completion of the procedures outlined in this JTP does not obligate the U.S. Army or any other DoD organization to procure or use the candidate.***

### 1.2 Execution

This document is organized in such a manner to aid the user during the corrosion study planning stage, through the testing activity, and during the data reporting and interpretation phases. This section describes the use of this document by outlining the steps that will guide the user through the process of extracting and utilizing the corrosion data. Section 2.0 describes a logical flow to the process of evaluating the results of the corrosion tests and comparing the properties of the candidate with the established criteria necessary to qualify the candidate for potential military use. Section 2.0 also provides a test flow diagram and examples of situations in which the JTP could be used. Section 3.0 discusses application scenarios, the test method matrix, and methodology. Section 4.0

describes test requirements (acceptance criteria) and procedures. Section 5.0 discusses failure analysis. Finally, Section 6.0 provides a list of reference documents that were utilized in the preparation of this JTP.

The corrosion-resistance performance of candidates evaluated using this JTP will be determined through a series of tests. These tests have been derived from engineering, performance, and operational impact (supportability) standards defined by a consensus of government and industry participants. The tests in this document are based upon recognized commercial and military test standards that are currently in use by established test facilities. In instances where the JTP test method conflicts with the reference standard on which it is based, the JTP test method will take precedence. This JTP also provides guidelines for the screening of candidates (Screening Tests), in cases where initial viability must be assessed before conducting the Performance and Special Tests or for urgent short-run applications.

Prior to conducting the required tests, a candidate must undergo a preliminary Feasibility Study, in which the following considerations shall be addressed:

- The candidate must be evaluated using those tests that define the performance levels of Chemical Agent Resistant Coatings (CARCs) , per MIL-DTL-53072, Chemical Agent Resistant Coating CARC) System Application Procedures And Quality Control Inspection. The candidate must demonstrate compatibility with the existing CARC system, with no adverse effects on the CARC properties. Relevant test methods and military standards are defined in MIL-DTL-53072 . Since CARC compatibility testing involves the use of chemical agents, the U.S. Army Research Laboratory (ARL) will conduct these tests on test specimens supplied by the vendor, at the vendor's expense.
- The candidate must conform to current military environmental regulations and concerns, such as atmospheric and groundwater impact, volatile organic compound (VOC) content, waste disposal, etc.
- Procurement of the candidate must be compatible with standard military business procedures. Considerations include, but are not limited to: distribution status (domestic/offshore), product cost analysis, and vendor capability, reputation, and reliability.

**The Feasibility Study shall be conducted prior to the execution of the test program contained in this JTP**. The business issues assessment shall be conducted again at the completion of the JTP testing if business issues have changed as a result of product and/or financial changes. The actual implementation of the Feasibility Study shall be conducted under the authority of the invoking authority, and is outside the scope of this JTP.

The tests outlined in this JTP are organized into three general areas, Screening, Performance, and Special Testing. Screening Testing involves those tests the vendor may decide to perform if limited data exists to determine the candidate's ability to pass the Performance Tests, or tests that the invoking authority may require for urgent short-run

applications. Performance Testing involves those tests required for evaluating any pretreatment candidate for use on steel components. Special Testing includes those tests identified by some (but not all) stakeholders for evaluating any pretreatment candidate for use on steel in special applications, such as exposure to particularly unusual environments. The candidate must meet both Performance and applicable Special Testing requirements to be considered for special applications.

A JTR will document the testing conducted on each candidate in accordance with this JTP. The JTR will provide a record of test specifics, such as candidate test specimen and substrate preparation, application process, test equipment model and calibration, laboratory environmental conditions, and test results. If planned execution of the tests varies from that described in this JTP, test procedure modifications must be approved by the stakeholders and the invoking authority in advance and documented in the JTR. The JTR will be used as a reference for future corrosion-prevention endeavors by other DoD and commercial users to minimize duplication of effort.

## 1.3 Document Maintenance

Annual updates and general maintenance of this document will be the responsibility of a committee chaired by the Army Corrosion Office or designee. The document will be reviewed and updated on an annual basis with changes being noted on the "JTP Revisions History" page. If no changes have been made, the entry "no revision has been made for the year 20xy" will be entered where appropriate. This document is considered to be obsolete if the latest entry on the JTP Revisions History is more than two years old. In this case, contact the Army Corrosion Office or designee for the most recent revisions before conducting testing in accordance with this JTP.

## 2.0 JTP DOCUMENT GUIDE

This section of the JTP facilitates the use of this document by providing a logical implementation flow process as well as examples of JTP evaluations for several candidates. Use of this document for military consideration of a candidate utilizing the Performance and Special Testing sections, and the preliminary screening of untried candidates using the Screening Tests, is described and demonstrated.

Figure 1 illustrates the process flow for conducting Screening, Performance, and Special Tests, as well as the retesting of candidates that have failed one of the aforementioned tests. The evaluation process begins with the Feasibility Study. If the candidate conforms to current military environmental regulations, and procurement of the candidate is compatible with standard military business procedures, the testing required is determined via the Test Method Matrix (presented later in Table 1).

The Screening Tests have been established so that preliminary screening of newer, unproven candidates can be conducted. The decision of whether to utilize Screening Tests before conducting Performance and Special Tests lies solely with the invoking authority. Successful completion of the Screening Tests qualifies a candidate for continued testing under the Performance Tests or evaluates the performance for qualitative purposes only. Screening Tests can also be used in instances where a small production run is required, and/or where expedited use is required for a limited application. However, consideration of a candidate for generalized military use, as defined in this JTP, can be accomplished only by successful completion of the Performance Tests, and Special Tests for special applications.

Any candidate that is to be considered technically acceptable must meet at least the Minimum Performance (MP) criteria for each Performance or Special Test, as established in Section 4.0, Testing Requirements, Descriptions and Procedures.

A failure analysis can be performed on any test specimen that fails Screening, Performance, or Special Tests to determine the cause of failure (see Section 5.0). Failure in any test does not necessarily disqualify a candidate for use in all possible applications; however, use of a candidate that has failed Screening, Performance, or Special Tests is at the discretion of the invoking authority, and is outside the scope of this document. Following completion of testing and/or failure analysis, the JTR is forwarded to the vendor for transmittal to the invoking authority for review.

Note that, in Figure 1, there are potential “infinite loops” that might occur due to continual testing failures. To resolve this, the following procedure is to be followed. If failure is still occurring after the third cycle for any of the Screening Tests, the testing process is to end, the failures are to be documented in the JTR, and the JTR is to be forwarded to the vendor for transmittal to the invoking authority for review and response. This procedure is likewise applicable for the Performance and Special Tests.

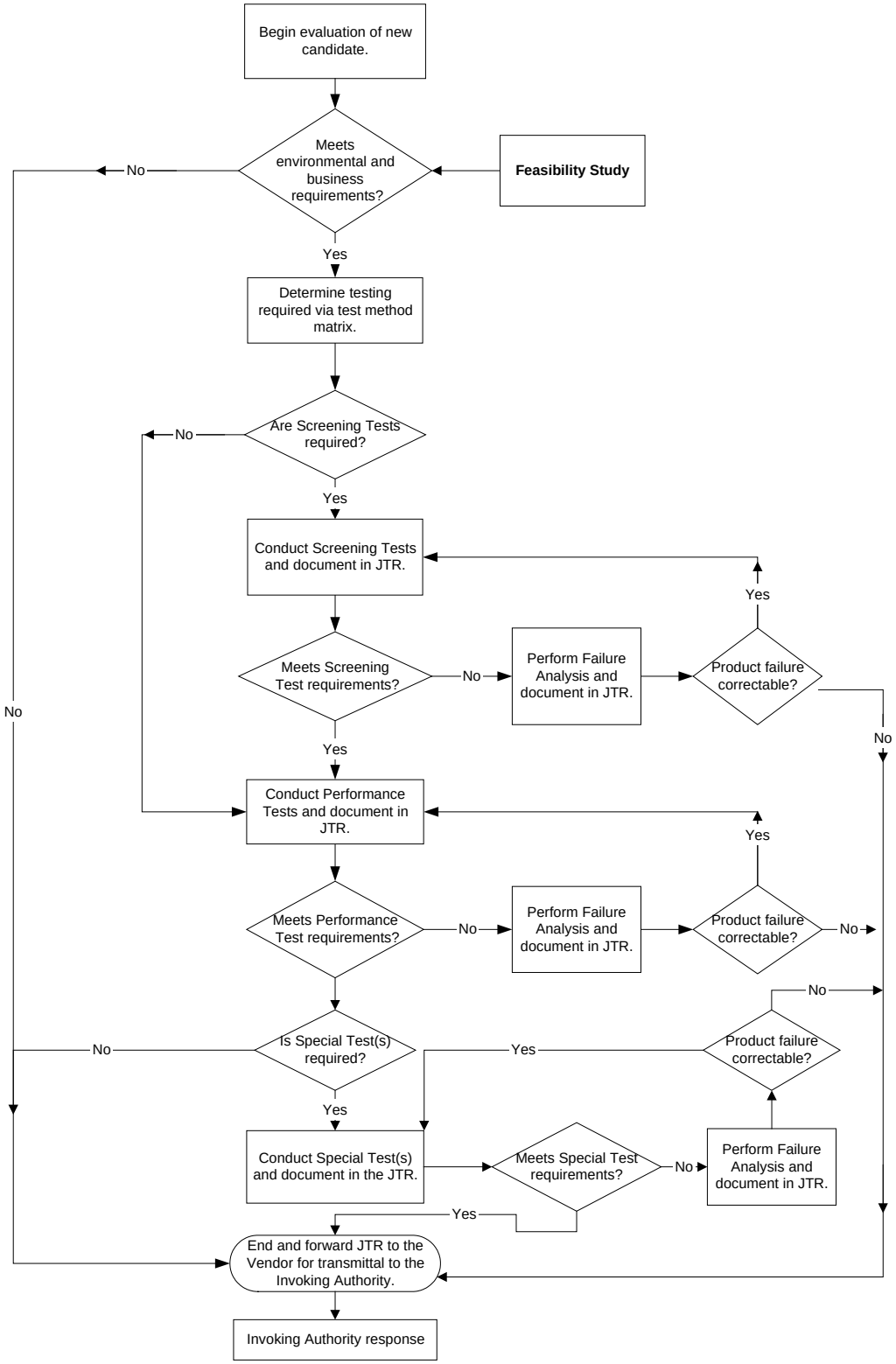



**Figure 1. Test Flow Diagram**

The following three examples are provided to demonstrate how this JTP can be used for Screening, Performance, and Special Testing situations.

Example # 1

SITUATION: A vendor has developed a new conversion coating system to be considered for use on only steel armor for a special urgent short-run application.

EVALUATION:

1. The VPS JTP directs the users to the JTP Test Flow Diagram (Figure 1). The Feasibility Study is conducted, and initial assessments regarding CARC compatibility, environmental concerns, and overall business risk are determined.
2. The invoking authority determines that Screening Tests only will be necessary for this system, and that, if the outcome is positive, qualification will be via a waiver (which is beyond the scope of this document). The JTP Test Flow Diagram leads the users to the Test Method Matrix (Table 1) to determine the testing required for screening.
3. The relevant test lab personnel begin the screening evaluation of the conversion coating.
4. A JTR is written documenting the results of the Screening Tests.
5. Screening Test results demonstrate acceptable performance relative to the other approved coating systems.
6. The JTR is submitted to the vendor for transmittal to the invoking authority for review. The invoking authority, if satisfied, issues a waiver/deviation (which is outside the scope of this JTP) to authorize the new conversion coating for this limited special short-run application.

RESULT: The JTP provides guidelines regarding testing and performance levels for preliminary risk reduction for this urgent short-run requirement.

Example # 2

SITUATION: A vendor proposes a new pretreatment / conversion coating, an inhibitor spray, to be considered for use on steel components.

EVALUATION:

1. The VPS JTP directs the users to the JTP Test Flow Diagram. The Feasibility Study is conducted, and initial assessments are made regarding CARC compatibility, environmental concerns, and overall business risk.
2. The vendor decides that, since the candidate is new, the candidate will be subjected to Screening Tests prior to the initiation of the Performance Tests. The JTP Test Flow Diagram leads the users to the Test Methods Matrix to determine the testing required for effective screening.

3. The relevant test lab personnel begin the screening evaluation of the pretreatment spray.
4. A JTR is written documenting the results of the Screening Tests and is forwarded to the vendor.
5. Screening Test results indicate that the pretreatment shows promise, as the corrosion performance level improved significantly as compared to the current corrosion protection system.
6. The relevant test lab personnel conduct Performance Tests per the Test Method Matrix.
7. A JTR is written documenting the results of the Performance Tests and is forwarded to the vendor for transmittal to the invoking authority for review.

RESULT: The JTP establishes the requirements for consideration, as well as guidelines for preliminary testing (Screening Tests), and provides the methodology for documenting the relative performance of the candidate compared to current corrosion protection systems.

## 3.0 APPLICATION SCENARIOS

### 3.1 Guidelines

This section establishes the guidelines for testing a potential candidate for corrosion protection of steel, given various application scenarios.

A generic model of a candidate and the various layers of materials that may be applied to the substrate to establish a corrosion protection system are shown in Figure 2.

**Figure 2. Generic Substrate and Corrosion Protection System Model (not to scale)**

The model below represents a generic coating system with numerous layers of constituent materials that may be included as part of a candidate corrosion protection system. Using this approach, guidelines for Screening, Performance, and Special Test procedures can be derived, even if the candidate consists of only some of the constituent layers shown in Figure 2.

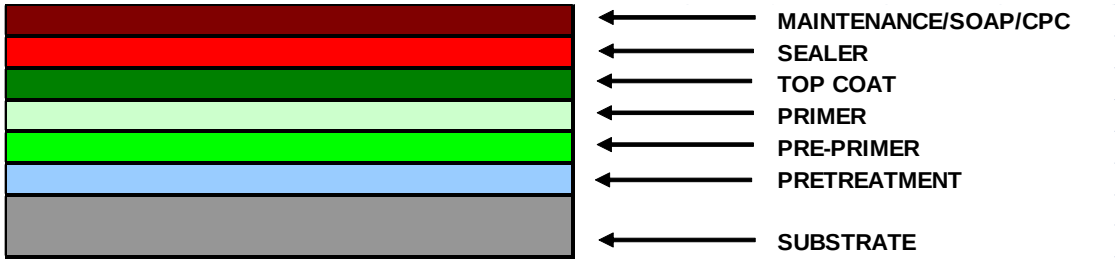



Table 1 lists the tests to be applied for Screening, Performance, and Special Tests, as well as the location of the test procedure within the JTP document.

**Table 1. Test Method Matrix**

| SCREENING TESTS (conducted on coupons, about 1 month in duration) | JTP Section |
|---|---|
| Adhesion (Pull-off) | 4.4.3 |
| Corrosion Resistance (Neutral Salt Spray (Fog)) | 4.4.5 |
| PERFORMANCE TESTS (conducted on actual or simulated parts, about 6 months in duration) | JTP Section |
| Adhesion (Dry) | 4.4.1 |
| Adhesion (Wet) | 4.4.2 |
| Adhesion (Pull-off) | 4.4.3 |
| Corrosion Resistance (Cyclic) | 4.4.4 |
| Corrosion Resistance (Neutral Salt Spray (Fog)) | 4.4.5 |
| Chip Resistance | 4.4.6 |
| Stress-Corrosion Cracking | 4.4.7 |
| SPECIAL TESTS (conducted on actual or simulated parts, up to 5 years in duration) | JTP Section |
| Field Exposure, Static | 4.4.8 |
| Field Exposure, On-Vehicle | 4.4.9 |

The guidelines for testing candidates under this JTP are as follows:

1. Select the test specimens or manufactured parts that accurately simulate current production material, for testing of the candidate.
2. Obtain approval for test procedure modification if applicable.
3. Perform appropriate testing and obtain test results.
4. Submit JTR to the vendor for transmittal to the invoking authority for review.

## 3.2 Methodology

Screening Tests shall be conducted on test panels made from the same material or alloy as the actual steel components. The processes to be used in the preparation of the test panels shall be outlined in the JTR.

Performance and Special Tests shall be conducted on sections of actual manufactured parts, or certified test coupons that accurately simulate current production material and manufacturing processes. Mechanical conditions such as bends, welds, fasteners,

crevices, etc., shall be incorporated when applicable. The actual processes used in the test specimen preparation shall be outlined in the JTR.

## 4.0 TESTING REQUIREMENTS, DESCRIPTIONS AND PROCEDURES

The stakeholders have established the requirements necessary to evaluate corrosion-resistant candidates for use on U.S. military steel components. These requirements have been used to identify test methods, derive test procedures, and establish acceptance criteria.

Screening Test methods are identified along with acceptance criteria in Section 4.1. Performance Test methods are identified along with acceptance criteria in Section 4.2.

Special Test methods are identified along with acceptance criteria in Section 4.3. These are program-specific requirements identified by at least one of the stakeholders. Special Tests are performed on sections of actual vehicles, certified test coupons, or manufactured parts that accurately simulate current production material and manufacturing processes.

It is recommended that different examples of substrates utilizing the candidate, if applicable, be tested concurrently to obtain maximum benefit from the testing effort. Questions regarding the different substrate materials shall be directed to the invoking authority.

The candidate must pass the Performance and applicable Special Tests with at least Minimum Performance (MP) in order to be considered for military use. Acceptance criteria for Improved Performance (IP) and Best Performance (BP) are provided as well, so that improved corrosion resistance with respect to the current corrosion protection system can be quantified.

**In instances where the JTP test method conflicts with the reference standard on which it is based, the JTP test method shall take precedence.**

All testing shall be performed at the vendor's expense by a government or independent testing laboratory, which shall be agreed upon by the stakeholders. The independent testing laboratory must either be accredited by a recognized governing body (such as the American Association for Laboratory Accreditation (A2LA) or the National Voluntary Laboratory Accreditation Program (NVLAP)), or be an ISO 9001 certified company having its own testing laboratory. **Testimonials shall be used for informational purposes only, and are not to be used in lieu of tests required under this JTP.** Incorporation of previous studies performed on the candidate by an outside laboratory, at the request of the vendor, is at the discretion of the invoking authority.

All tests shall be conducted in a manner that will eliminate duplication and maximize the use of each test specimen. Where possible, more than one test shall be performed on each specimen. The number and types of tests that can be run on any one specimen will be dependant upon the degree of alteration imparted to the sample from previous tests. Failure in any test does not necessarily disqualify a candidate for use in all possible applications; however, acceptance of a candidate that has failed Screening, Performance,

or Special Tests is at the discretion of the invoking authority. In this case, use of the candidate will be justified by a special waiver, which is outside the scope of this document.

The tests described in this JTP may involve the use of hazardous materials, operations, and/or equipment. This JTP does not address all safety issues associated with their use. It is the responsibility of each user of this JTP to establish appropriate safety and health practices, and to determine the applicability of regulatory limitations, prior to the use of such materials, operations, and/or equipment.

The following conditions will apply to all Screening, Performance, and Special Testing, unless otherwise specified in an individual test description:

- It is preferred that all test panels be produced from the same material lot.
- It is suggested that at least three specimens be used for Screening Tests, and at least five specimens be used for Performance and Special Tests.
- Unless otherwise specified, all test specimens shall be cleaned prior to pretreatment to ensure surfaces are free of water breaks in accordance with the latest version of American Society for Testing and Materials (ASTM) G1, "Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens."
- Pretreatment of the test specimens will be dependant upon the candidate under scrutiny, and shall be specified in the JTR.

It is recommended that users of this JTP obtain copies of previous JTRs, if available, from the invoking authority for additional test details or minor modifications that were necessary in the execution of previous testing.

## 4.1 Screening Testing Requirements

Table 2 lists all Screening Testing requirements identified by stakeholders for evaluating candidates on steel .

**Table 2. Screening Testing Requirements**

| JTP Section | Test | Acceptance Criteria | Test Method References |
|---|---|---|---|
| 4.4.3 | Adhesion (Pull-off) | Meets or exceeds adhesion strength of DOD-P-15328 on similarly prepared abrasive blasted surface of 1.5 mil profile or 1200 psi | ASTM-4541 Pull-off Adhesion |
| 4.4.5 | Corrosion Resistance (Neutral Salt Spray (Fog)) | After 336 hrs of exposure: Steel substrate rating $\geq$ 7 scribed | ASTM B117<br>ASTM D714<br>ASTM D1654 |

Screening Tests are performed on test panels made from representative steel - substrate material. It is preferred that all test panels be produced from the same material lot, and it is desirable that the processing pedigree be well documented in the JTR. The candidate must pass the acceptance criteria of each Screening Test. Results of the Screening Tests are reported in the JTR and submitted to the vendor for transmittal to the invoking authority.

The Screening Tests (identified in Table 2) are further defined in Section 4.4, with test descriptions, scope, and methodology. Also included are any major or unique equipment and instrumentation requirements, reagents, procedures, and acceptance criteria. The procedure identifies the test specimen preparation, test procedure, and method for collecting and reporting test results.

## 4.2 Performance Testing Requirements

Table 3 lists all Performance Testing requirements identified by stakeholders for evaluating candidates on commonly used steel substrates. The tests (listed below) shall also be conducted for non-traditional candidate substrates such as high-hardness (greater than Rockwell hardness Rc35), (HHA) steels and high-strength low-alloy (HSLA) steels.

A material/corrosion design review will be conducted by the invoking authority to determine if hydrogen embrittlement, corrosion fatigue, or stress-corrosion cracking could occur based on the material and potential exposure environment. However, it shall be known that HHA has hardnesses well over Rc35 and is susceptible to environmentally assisted cracking (EAC) whenever residual stresses are present. The invoking authority will specify the appropriate mechanical stability testing required, and the vendor will contract with an independent, certified lab to perform the required tests.

The criteria for determining a risk candidate for hydrogen embrittlement are as follows:

1. Any ferrous-based alloy exhibiting hardness greater than Rc35 (e.g., high-strength steel) requires testing and heat treatment according to Federal Specification TT-C-490, “Cleaning Methods for Ferrous Surfaces and Pretreatments for Organic

Coatings," . Testing is recommended for materials that will be exposed to an electrochemical environment where hydrogen evolution can occur (e.g., electroplating, pickling).

The basic criteria for determining a risk candidate for stress-corrosion cracking are as follows:

1. Any material that will be exposed to a corrosive environment known to cause stress-corrosion cracking, such as sodium hydroxide for carbon steel or chloride ions for stainless steels, and tensile stress due to applied load or residual stresses such as those produced by welding (e.g., any material that will experience a stress greater than 50% of the yield stress) shall be tested.
2. Any material that is known to be subject to stress-corrosion cracking (determine susceptibility by conducting a literature search or consulting with a corrosion expert) shall be tested.

## Table 3. Performance Testing Requirements

| JTP Section | Test | Acceptance Criteria, Minimum Performance (MP) | Acceptance Criteria, Improved Performance (IP) | Acceptance Criteria, Best Performance (BP) | Test Method References |
|---|---|---|---|---|---|
| 4.4.1 | Adhesion (Dry) | Adhesion rating (steel) ≥ 4B; adhesion rating | N/A | Adhesion rating (steel) = 5B; adhesion rating | ASTM D3359 |
| 4.4.2 | Adhesion (Wet) | Scribed area rating (steel) ≥ 3A after 24 hours at ambient; | Scribed area rating (steel) ≥ 4A after 96 hours at 120°F; | Scribed area rating (steel) ≥ 4A after 168 hours at 150°F; | ASTM D3359 |
| 4.4.3 | Adhesion (Pull-off) | Minimum average 30 events rating of 1200 PSI | Minimum average 30 events rating of 1800 PSI | Minimum average 30 events rating of 2500 PSI | ASTM D 4541 |
| 4.4.4 | Corrosion Resistance (Cyclic) | After 60 cycles: steel substrate rating ≥ 4 scribed | After 60 cycles: steel substrate rating ≥ 6 scribed | After 60 cycles: steel substrate rating ≥ 8 scribed | GM 9540P)<br>ASTM D714<br>ASTM D1654 |
| 4.4.5 | Corrosion Resistance (Neutral Salt Spray (Fog)) | After 500 hours of exposure: steel substrate rating ≥ 6 scribed | After 750 hours of exposure: steel substrate rating ≥ 6 scribed | After 1000 hours of exposure: steel substrate rating ≥ 6 scribed | ASTM B117<br>ASTM D714<br>ASTM D1654 |

NLT = Not Less Than

**Table 3. Performance Testing Requirements (Continued)**

| JTP Section | Test | Acceptance Criteria, Minimum Performance (MP) | Acceptance Criteria, Improved Performance (IP) | Acceptance Criteria, Best Performance (BP) | Test Method References |
|---|---|---|---|---|---|
| 4.4.6 | Chip Resistance | After one cycle, chip rating NLT 5B for steel, | After one cycle, chip rating NLT 7C for steel, | After one cycle, chip rating NLT 9C for steel, | SAE J400 |
| 4.4.7 | RSL Stress-Corrosion Cracking | There shall be no detrimental effect to K1c of substrate. High Hard K1c @ 48-51Rc shall maintain K1eac ≥ 19 (ksi√in) | | | ASTM E 399-97<br>ASTM G30<br>ASTM G38<br>ASTM G39<br>ASTM G47 |

NLT = not less than

Performance Tests are performed on certified test coupons, sections of actual steel components or manufactured parts that accurately simulate current production material and manufacturing processes. Results of the Performance Tests are reported in the JTR and submitted to the vendor for transmittal to the invoking authority.

The Performance Tests (identified in Table 3) are further defined in Section 4.4, with test descriptions, scope, and methodology. Also included are any major or unique equipment and instrumentation requirements, reagents, procedures, and acceptance criteria. The procedure identifies the test specimen preparation, test procedure, and method for collecting and reporting test results.

## 4.3 Special Testing Requirements

Table 4 lists Special Testing requirements identified and required by some (but not all) stakeholders for evaluating candidates.

**Table 4. Special Testing Requirements**

| JTP Section | Test | Acceptance Criteria, Minimum Performance (MP) | Acceptance Criteria, Improved Performance (IP) | Acceptance Criteria, Best Performance (BP) | Test Method References | Branch/ Stakeholders/ Service Requiring Test |
|---|---|---|---|---|---|---|
| 4.4.8 | Field Exposure, Static | Three years of exposure: specimen has a minimum of 25% less creepage from scribe than current corrosion protection system | Four years of exposure: specimen has a minimum of 50% less creepage from scribe than current corrosion protection system | Five years of exposure: specimen has a minimum of 75% less creepage from scribe than current corrosion protection system | Approved test site standard practice ASTM G50 ASTM G7 ASTM D1654 | As required by the invoking authority |
| 4.4.9 | Field Exposure, On-Vehicle | Three years of exposure: steel substrate rating ≥ 5 scribed and ≥ 5M unscribed; | Four years of exposure: steel substrate rating ≥ 5 scribed and ≥ 5M unscribed; | Five years of exposure: steel substrate rating ≥ 5 scribed and ≥ 5M unscribed; aluminum | ASTM D1654 ASTM D714 | As required by the invoking authority |

NLT = not less than

Unless otherwise noted, Special Testing shall be performed on sections of actual steel components including armor or manufactured parts that accurately simulate current production material and manufacturing processes. Results of the Special Tests are reported in the JTR and submitted to the vendor for transmittal to the invoking authority.

The Special Tests (identified in Table 4) are further defined in Section 4.4, with test descriptions, scope, and methodology. Also included are any major or unique equipment and instrumentation requirements, reagents, procedures, and acceptance criteria. The procedure identifies the test specimen preparation, test procedure, and method for collecting and reporting test results.

## 4.4 Test Descriptions

### 4.4.1 Adhesion (Dry) (ASTM D3359)

#### 4.4.1.1 Scope

This test method assesses the adhesion of coatings to substrates by applying and removing pressure-sensitive tape over cuts made in the coating.

#### 4.4.1.2 Equipment

Cutting Tool. A very sharp razor blade, scalpel, knife, or other cutting device having a cutting edge (tip) angle between 15 and 30 degrees.
Cutting Guide. Steel or other hard metal straightedge to ensure straight cuts.
Rule. A steel rule graduated in 0.5-millimeter (mm) (0.02") increments for measuring individual cuts.
Tape. 3M 396 1" wide transparentpressure-sensitive tape, manufactured by 3M, This tape was chosen for its performance over the MIL-DTL-64159 and MIL-DTL-53039 polymer bead versions of CARC.shelf life. Utilizing the tape after the manufacturer's recommended storage may yield inaccurate results.
Pencil with eraser. To be used to make sure the tape is firmly adhered.
Illumination. A light source to determine whether the cuts have been made through the coating into the substrate.
Dry Film Thickness Gage. A device to measure the thickness of the applied coating.

#### 4.4.1.3 Reagents

None.

#### 4.4.1.4 Procedure

Test Specimens. Prepare at least three test specimens for Screening Testing and at least five specimens for Performance Testing. For Screening Testing, use 102 x 152 mm) (4" x 6" test panels, composed of the material that is utilized in the end application. For Performance Testing, sections of actual or simulated steel armororsteel parts shall be used (see Section 3.2).
Preparation. Using test specimens incorporating the candidate, measure the dry film thickness in at least five areas. Make cuts in the coating system per the latest version of ASTM D3359, "Standard Test Methods for Measuring Adhesion by Tape Test – Cross-Cut Tape Test." Remove two laps of tape and discard. Remove an additional length of tape and cut a piece approximately 76 mm (3") long. Place the center of the tape over the grid and smooth into place by firmly rubbing a pencil eraser over the area..
Test Procedure. Within 90 $\pm$ 30 seconds of tape application, remove the tape by holding the free end and rapidly pulling (not jerking) back upon itself at as close to an angle of 180 degrees as possible.
Test Results. Inspect the grid area for removal of coating from the substrate or from a previous coating. Rate the adhesion in accordance with the latest version of ASTM D3359, Test Method B. If ratings differ by more than one rating unit, the results are considered suspect and three additional test specimens for Screening Testing and five additional test specimens for Performance Testing shall be prepared and the tests repeated. If applicable, use these latter ratings in the report.
Report. Report all information per the latest version of ASTM D3359 Test Method B. In addition, report the average of the five dry film thickness measurements (as measured by thickness gauge).

#### 4.4.1.5 Acceptance Criteria

| Substrate | Acceptance Criteria, Minimum Performance (MP) | Acceptance Criteria, Improved Performance (IP) | Acceptance Criteria, Best Performance (BP) |
|---|---|---|---|
| Steel | Adhesion rating ≥ 4B | N/A | Adhesion rating = 5B |

### 4.4.2 Wet Adhesion (ASTM D3359, as modified by NAVAIR)

#### 4.4.2.1 Scope

This test method describes the procedure and conditions for assessing the wet adhesion of coatings to metallic substrates by applying and removing pressure-sensitive tape over cuts made in the coating.

#### 4.4.2.2 Equipment

Tank and Tank Cover. A tank made from corrosion-resistant materials and large enough to hold the required number of test specimens. The tank cover is required to help maintain water temperature and prevent evaporation.

Heaters. Heaters capable of maintaining the required water temperature (see Section 4.4.2.4, Procedure).
Circulation System. A pump or stirrer required for circulating the water in the water tank, capable of low to moderate agitation speeds.
Test Specimen Supports. Supports constructed of nonconductive and corrosion-resistant materials to hold the coated test specimens 30 mm (1.2") apart and at least 30 mm (1.2") from the bottom and sidewalls of the tank.
Cutting Tool. A very sharp razor blade, scalpel, knife, or other cutting device having a cutting edge (tip) angle between 15 and 30 degrees.
Cutting Guide. Steel or other hard metal straightedge to ensure straight cuts.
Rule. A steel rule graduated in 0.5-mm (0.02") increments for measuring individual cuts.
Tape. Permacel 99 (1" wide semitransparent pressure-sensitive tape, manufactured by Permacel, New Brunswick, NJ 08903). (Or equivalent tape) *NOTE*: Permacel 99 tape has a one-year
shelf life. Utilizing the tape after this time may yield inaccurate results.
Roller. A 4.5-lb rubber-covered roller.
Illumination. A light source to determine whether the cuts have been made through the coating to the substrate.
Dry Film Thickness Gage. A device to measure the thickness of the applied coating.

#### 4.4.2.3 Reagents

Distilled Water. Conforming to Type IV water as described in the latest version of ASTM D1193.

#### 4.4.2.4 Procedure

Test Specimens. At least three test specimens shall be used for Screening Testing and at least five specimens for Performance Testing. For Screening Testing, use 102 x 152 mm (4" x 6") test panels, composed of the material that is utilized in the end application. For Performance Testing, use sections of actual or simulated steel components (see Section 3.2).

Preparation. Using test specimens incorporating the candidate, measure the dry film thickness in at least five areas.

Test Procedure. For the Screening and Minimum Performance Tests, immerse the test specimens in ambient (room temperature) distilled water for 24 hours. For Improved Performance, immerse the test specimens in distilled water maintained at 49 ± 2°C (120 ± 4°F) for 96 hours. For Best Performance, immerse the test specimens in distilled water maintained at 66 ± 2°C (150 ± 4°F) for 168 hours. Remove the test specimens from the water and wipe dry with a soft cloth. Within 90 ± 30 seconds after removal from the water, make cuts in the coating system with two parallel lines, 19 mm (0.75") apart, and place an "X" scribe within the parallel lines. Make the "X" lines about 38 mm (1.5") long and intersecting at 30–45 degrees in the center of the parallel lines. Remove two laps of tape and discard. Remove an additional length of tape and cut a piece approximately 75 mm (3") long. Place the center of the 25 mm (1") wide tape over the center of the "X" and smooth into place by passing the roller over the area once. Remove the tape by holding the free end and rapidly pulling (not jerking) back upon itself at as close to an angle of 180 degrees as possible.

Test Results. Rate the adhesion in accordance with the latest version of ASTM D3359, Method A "Measuring Adhesion by Tape Test – X-Cut Tape Test."

Report. Report all information per the latest version of ASTM D3359, Method A. In addition, report the average of the five dry film thickness measurements.

#### 4.4.2.5 Acceptance Criteria

| Parameter | Acceptance Criteria, Minimum Performance (MP) | Acceptance Criteria, Improved Performance (IP) | Acceptance Criteria, Best Performance (BP) |
|---|---|---|---|
| Scribed Area Rating | ≥ 3A | ≥ 4A | ≥ 4A |
| Immersion Period | 24 hours | 96 hours | 168 hours |
| Water Temperature | ambient | 49°C (120°F) | 66°C (150°F) |

### 4.4.3 Adhesion (Pull off) (ASTM D-4541)

#### 4.4.3.1 Scope

This test method covers a procedure for evaluating the pull-off strength (commonly referred to as adhesion) of a coating by determining either the greatest perpendicular force (in tension) that a surface area can bear before a plug of material is detached, or

whether the surface remains intact at a prescribed force (pass/fail). Failure will occur along the weakest plane within the system comprised of the test fixture, adhesive, coating system, and substrate, and will be exposed by the fracture surface. This test method maximizes tensile stress as compared to shear stress applied by other methods, such as scratch or knife adhesion, and results may not be comparable. Further, pull-off strength measurements depend upon both material and instrumental parameters. Results obtained using different devices or results for the same coatings on substrates having different stiffness may not be comparable.

#### 4.4.3.2 Equipment

Adhesion Tester. Commercially available or comparable apparatus as described in Annex A1-Annex A4 of ASTM D 4541.
Loading Fixtures. Device having a flat surface on one end that can be adhered to the coating and a means of attachment to the tester on the other end.
Detaching Assembly. (adhesion tester) A central grip for engaging the fixture.
Base. Part of the detaching assembly, or an annular bearing ring if needed for uniformly pressing against the coating surface around the fixture either directly , or by way of an intermediate bearing ring. A means of aligning the base is needed so that so that the resultant force is normal to the surface and a means of moving the grip away from the base in as smooth and continuous manner as possible so that a torsion-free, co-axial (opposing pull of the grip and push of the base along the same axis) force results between them.
Timer. Means of limiting the rate of stress to less than 150 psi/s (1PPa/s) so that the maximum stress is obtained in less than about 100s. A timer is the minimum equipment when used by the operator along with the force indicator.

#### 4.4.3.4 Procedure

Test Specimens. At least 10 test pulls shall be used for the Screening Testing and at least 30 test pulls shall be used for the Performance Testing.
Preparation. There are a few physical restrictions imposed by the general methods and apparatus. The following requirements apply:
The selected test area must be a flat surface large enough to support the test fixture.
The selected area must have enough perpendicular and radial clearance and be rigid enough to support the counter force.
Test Procedure. Clean the loading fixture and the coating surface to be bonded. Use care to select only those solvents which will not attack the coating and/or leave residues on the fixture. Prepare the adhesive in accordance with the adhesive manufacturer's recommendations. Apply the adhesive to the fixture or the surface to be bonded using a procedure recommended by the adhesive manufacturer being certain the entire bonding surface is covered. Based on the manufacturer's recommendations, allow enough time for the adhesive to cure. Carefully connect the central grip of the detaching assembly to the loading fixture without bumping, bending, or otherwise prestressing the sample and connect the detaching assembly to its control mechanism, if necessary. After setting the

force indicator to zero, increase the load to the fixture in as smooth and continuous manner as possible, at a rate of less than 150 psi/s (1 MPa/s) so that failure occurs or the maximum stress is reached in about 100 s or less.
Test Results. Rate the average results of each set of events.

#### 4.4.3.5 Acceptance Criteria (See Table 3)

| Substrate | Screening Test | Acceptance Criteria, Minimum Performance (MP) | Acceptance Criteria, Improved Performance (IP) | Acceptance Criteria, Best Performance (BP) |
|---|---|---|---|---|
| Steel | Minimum average 10 events rating of 1200 PSI | Minimum average 30 events rating of 1200 PSI | Minimum average 30 events rating of 1800 PSI | Minimum average 30 events rating of 2500 PSI |

### 4.4.4 Corrosion Resistance (Cyclic) (GM 9540P)

#### 4.4.4.1 Scope

This test method describes a field-correlated, laboratory corrosion test method for determining cosmetic corrosion performance that provides a combination of cyclic conditions (salt solution immersion, temperature, and humidity) to accelerate the corrosion process.

#### 4.4.4.2 Equipment

Test Cabinet. Test cabinet with the ability to obtain and maintain the required environmental conditions as specified in GM9540P.
Scribe Tool. An ANSI B 94.50, style E scriber.
Imaging System. A means of visually recording corrosion effects on all test specimens, such as a camera or scanner/software system.
Air Source. A source of clean, dry compressed air capable of delivering at least 10 cfm at 80 psi.
Scale. A ruler with 1-mm (0.04") divisions.
Balance. A digital electronic balance capable of weighing up to 10,000 mg with an accuracy of ± 1%.
Straightedge. Any straightedge of sufficient length to guide the scribing tool in a straight line.
pH Meter. A meter to measure the pH of the salt solution prior to the start of the test and on a weekly basis thereafter.
Putty Knife. Blunt-edged, 38 mm (1.5") wide.

#### 4.4.4.3 Reagents

Distilled Water. Conforming to Type IV water as described in the latest version of ASTM D1193.
Cleaning Solution. Methanol.
Sodium Chloride. Substantially free of nickel and copper and containing not more than 0.1% sodium iodide and not more than 0.3% total impurities by weight.
Calcium Chloride.
Sodium Bicarbonate.

#### 4.4.4.4 Procedure

Test Specimens. Actual or simulated steel components shall be used for test specimens (see Section 3.2). The number of test specimens depends on the number of cycles selected for the test exposure duration. Use reference coupons consisting of uncoated 25 x 51 x 3 mm (1" x 2" x 1/8") pieces of any alloy American Iron and Steel Institute (AISI) 1006 through 1010 steel to monitor the average general bare steel corrosion produced by the test environment. The coupon weight in milligrams shall be recorded and retained for future reference. The number of coupons also depends on the number of cycles selected for the test exposure duration. Each test specimen and reference coupon shall be permanently identified by stamping numbers onto the surface.
Preparation. Using test specimens incorporating the candidate, scribe an X scribe through the coating, making sure that the scribed line is all the way through the coating to the substrate. Place the scribed test specimens and reference coupons in the chamber, leaning at an angle of at most 15 degrees from the vertical with the scribed surface facing upwards. Prepare the salt solution per GM9540P and measure the pH prior to the start of the test and on a weekly basis thereafter. Do not attempt to adjust the pH. Clean the reference coupons (bare steel bars) thoroughly with the cleaning solution prior to placing them in the exposure chamber.
Test Procedure. For the MP level, use a test duration of 80 cycles; for the IP and BP levels, use a test duration of 120 cycles. After initially weighing each reference coupon and test specimen, install both the reference coupons and test specimens in the exposure chamber. After every 20 cycles, remove two coupons and two test specimens. Weigh each reference coupon (after removal of the rust layers) and determine the average weight loss for that specific number of cycles. For the test specimens, record the scribe creepback values with respect to average, ASTM-D1654. For the interim creepback measurements, conduct them in a rinsed-only condition. At the final number of cycles, two sets of creepback values will be recorded – one set in a rinsed-only condition and one set after the scrape-and-tape process.
Test Results. At the conclusion of the exposure period (or interim period), remove the test specimens and rinse. Scrape the specimens side-to-side with the putty knife at a 30-degree contact angle. Evaluate the creepage of the test specimens per the latest version of ASTM D1654 for scribed areas and D714 for unscribed. Rate the corrosion or loss of coating extending from the scribe mark (using the worst case for the rinsed or scraped methods) and evaluate the unscribed areas for corrosion spots, blisters, and any other types of failure that may occur. Photographically document each of the test specimens and the reference coupons using the imaging system. Clean the reference coupons using a mild sand (or glass bead) blast to remove all corrosion by-products. Once they are

clean, wipe the coupons with methanol and weigh to determine weight loss. Corrosion losses may also be expressed in terms of average corrosion rates from the weight loss, coupon area, test duration, and metal density by use of the calculation described in ASTM G1.

Report. Report all information required in ASTM D714, and ASTM D1654, including the photographs from the imaging system, and weight loss and/or corrosion rate of the reference coupons.

#### 4.4.4.5 Acceptance Criteria

| Parameter | Acceptance Criteria, Minimum Performance (MP) (60 cycles) | Acceptance Criteria, Improved Performance (IP) (60 cycles) | Acceptance Criteria, Best Performance (BP) (60 cycles) |
|---|---|---|---|
| Scribed Area Rating | $\geq$ 4 | $\geq$ 6 | $\geq$ 8 |
| Unscribed Area Rating | $\geq$ 5F | $\geq$ 7F | $\geq$ 9F |

### 4.4.5 Corrosion Resistance (Neutral Salt Spray (Fog)) (ASTM B117)

#### 4.4.5.1 Scope

This test method describes the procedure and conditions required to create and maintain the neutral salt spray (NSS) (fog) test environment and the evaluation of specimens incorporating the candidate with respect to corrosion, blistering associated with corrosion, loss of adhesion at a scribe mark, or other corrosive attack.

#### 4.4.5.2 Equipment

NSS (Fog) Chamber. This equipment shall consist of a heated fog chamber, a salt solution reservoir, a supply of conditioned (oil- and contaminant-free) compressed air, atomizing nozzles, and specimen supports.

Imaging System. A means of visually recording corrosion effects on all tested specimens, such as a digital camera or scanner/software system.

Scribe Tool. An American National Standards Institute (ANSI) B 94.50, style E scriber.

Straightedge. Any straightedge of sufficient length to guide the scribing tool in a straight line across the specimen surface.

Air Source. A source of clean, dry compressed air capable of delivering at least 10 cubic feet per minute (cfm) at 80 pounds per square inch (psi).

Air Gun and Guard. An air dusting gun and nozzle combination to meet the specification in ASTM D1654. A guard to protect the operator, such as a sandblasting cabinet.

Scale. A ruler with 1-mm (0.04") divisions.

Putty Knife. Blunt-edged, 38 mm (1.5") wide.

#### 4.4.5.3 Reagents

Distilled Water. Conforming to Type IV water as described in the latest version of ASTM D1193.
Sodium Chloride. Substantially free of nickel and copper and containing not more than 0.1% sodium iodide and not more than 0.3% total impurities by weight.

#### 4.4.5.4 Procedure

Test Specimens. At least three specimens shall be used for Screening Testing, and at least five specimens shall be used for Performance Testing. Screening Testing shall be conducted with 102 x 152 mm (4" x 6") test panels, composed of the material that is utilized in the end application. Actual or simulated frame structures shall be used for Performance Testing (see Section 3.2). Each test specimen shall contain a clear identification mark.
Preparation. Using test specimens incorporating the candidate, scribe a single diagonal line through the coating making sure that the scribed line is all the way through to the substrate. Place the scribed test specimens in the chambers, leaning at an angle between 15 and 30 degrees from the vertical with the scribed surface facing upwards. Prepare the salt solution as specified in ASTM B117 such that when atomized at 35°C (95°F), the collected solution is in the pH range of 6.5–7.2.
Test Procedure. Conduct the NSS (fog) test in accordance with the latest version of ASTM B117, "Standard Practice for Operating Salt Spray (Fog) Apparatus." The NSS (fog) chamber shall be operated continuously for the specified number of hours, as shown in Section 4.4.3.5, Acceptance Criteria.
Test Results. At the conclusion of the exposure period, remove the test specimens and clean them by gently flushing with running tap water and drying them with a stream of clean, dry compressed air. Allow the test specimens to recover for 24 hours. Scrape the test specimens side-to-side with the putty knife at 30-degree contact angle. Evaluate the corrosion resistance and creepage of the test specimens in accordance with the latest version of ASTM D1654, "Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments." Rate the corrosion or loss of coating extending back from the scribe mark and evaluate the unscribed areas for corrosion spots, blisters, and any other types of failure that may occur. Use the rating system in ASTM D1654 for scribed areas and D714 for unscribed. Photographically document the surface condition of each of the test specimens using the imaging system.
Report. Report all information required in ASTM B117, D714, and D1654, and include the images from the imaging system.

#### 4.4.5.5 Acceptance Criteria

| Parameter | Screening Test | Acceptance Criteria, Minimum Performance (MP) | Acceptance Criteria, Improved Performance (IP) | Acceptance Criteria, Best Performance (BP) |
|---|---|---|---|---|
| **Steel Substrate** | | | | |
| Scribed Area Rating | ≥ 7 | ≥ 6 | ≥ 6 | ≥ 6 |
| Unscribed Area Rating | ≥ 7F | ≥ 7F | ≥ 7F | ≥ 7F |
| Exposure Period | 336 hours | 500 hours | 750 hours | 1000 hours |

### 4.4.6 Chip Resistance (SAE J400)

#### 4.4.6.1 Scope

The Chip Resistance test is designed to reproduce the effect of gravel or other media striking exposed painted and/or coated surfaces of a vehicle and has been correlated to actual field results. The purpose of this test is to evaluate the chip resistance of flat test specimens incorporating the candidate.

#### 4.4.6.2 Equipment

Gravelometer. As specified in SAE J400 with the test panel at a 45-degree angle.
Test Cabinet. Temperature conditioning (usually run at ambient or lower temperature) with the ability to obtain and maintain the required environmental conditions as specified in SAE J400.
Transparent Grid. Approximately 3.2 x 127 x 127 mm (1/8" x 5" x 5") on which a 101.6 x 101.6 mm (4" x 4") grid of 25.4 mm (1") squares has been etched or scribed.
Paint Removal Tape. 100 mm (3.94") wide or 50 mm (1.97") wide, 3M product # 898 filament strapping tape or equivalent. *NOTE*: Note that the tape has a one-year shelf life. Utilizing the tape after this time may yield inaccurate results.
Gravel. Water-worn road gravel, not crushed limestone or rock. The gravel will pass through 15.86 mm (5/8") space screen when graded, but be retained on 9.53 mm (3/8") space screen. Gravel must be changed in accordance with SAE J400 Section 4.2.

#### 4.4.6.3 Reagents

None.

#### 4.4.6.4 Procedure

Test Specimen. Screening and Performance test specimens shall be panels, composed of the material that is utilized in the end application. The chipped area to be evaluated on the tested panel shall be flat and 101.6 x 101.6 mm (4" x 4") square and must be located about the center of the chipped pattern. SAE recommends that three replicates of each test panel be exposed in the Gravelometer. The composition, surface preparation, and size of panels; the type and thickness of the coating and the number and method of

application; and the aging conditions for the coatings shall be agreed upon between the vendor and invoking authority.

Test Setup/Preparation. Condition the test panels incorporating the candidate at the temperature specified in SAE J400 for a minimum of 15 minutes prior to testing. Fill a 0.473-liter (1-pt) container to the top with grated/screened gravel. Gravel must be changed every 3 runs. Adjust air pressure on the Gravelometer to 483 kilopascals (kPa) (70 psi) +/- 21 kPa (0.30 psi) with the air valve open. Set feed rate so that the hopper empties in 7–10 seconds per pint (s/pt). Other air pressures can be used as agreed upon by the vendor and invoking authority.

Test Procedure for Modular Gravelometer with Electronic Feed Mechanism. Insert the test panel into the specimen holder assembly. Clamp panel and close the specimen holder. Pour gravel from the one-pint container into hopper, then set the test timer.

a. Time Test

Set the test timer to the desired test time (typically < 10 s).

Turn the main power switch to ON.

Flip the control switch to TIME START.

b. Manual Test

See SAE J400 Section 4.2.3.2.

Chipping Rating System. The basic structure of the chipping rating system consists of one or more number-letter combinations in which rating values/numbers 10–0 indicate the number of chips of each size with rating letters A–D designating the sizes of the corresponding chips. Tables 5 and 6 provide guidelines for Rating Criteria as stated in SAE J400. A point of failure notation can also be used if desired (see Table 7).

**Table 5. Rating for Number of Chips Within 4" x 4" Grid Lines**

| Rating Number | # of Chips | Rating Number | # of Chips |
|---|---|---|---|
| 10 | 0 | 4 | 50–74 |
| 9 | 1 | 3 | 75–99 |
| 8 | 2–4 | 2 | 100–149 |
| 7 | 5–9 | 1 | 150–250 |
| 6 | 10–24 | 0 | > 250 |
| 5 | 25–49 | | |

**Table 6. Rating for Size of Chips**

| Rating Letter | Size of Chips |
|---|---|
| A | < 1 mm (approximately 0.03") |
| B | 1–3 mm (approximately 0.03–0.12") |
| C | 3–6 mm (approximately 0.12–0.25") |
| D | > 6 mm (> approximately 0.25") |

**Table 7. Point of Failure**

| Notation | Level of Failure | Failure Type |
|---|---|---|
| (S/P) | Substrate to Primer | Adhesional |

| | | |
|---|---|---|
| (S/T) | Substrate to Top Coat | Adhesional |
| (P) | Primer | Cohesional |
| (P/T) | Primer to Top Coat | Adhesional |
| (T) | Top Coat | Cohesional |

Method 1 – Exact Counting Procedure. This very precise method shall be used where definitive accuracy is required or as the referee method in case differences arise between laboratories.

a) Use the transparent overlay onto which has been etched a 101.6 x 101.6 mm (4" x 4") grid of 25.4 mm (1") squares as a location reference to aid the counting/rating process.
b) Examine all chips that are within each 25.4 mm (1") square, and estimate the size of each chip as encountered; examine all 16 squares and record the summed results.
c) Convert the actual number of chips counted for each size into the number-letter combinations utilizing Tables 5 and 6. Then arrange the number-letter ratings in ascending order (by number then letter). Summarize the number-letter ratings to give a condensed single number rating based on the total number of chips of all sizes followed by all applicable letter ratings to indicate the relative number of chips of each size.

Method 2 – Visual Comparison Procedure. This faster method shall be used for many routine laboratory evaluations where accuracy is not required.

a) Visually compare the area to be rated with the standards (SAE J400, Figure 3).
b) As with Method 1, list the ratings in ascending order. Summarize the number-letter ratings to give a condensed single number rating based on the total number of chips of all sizes followed by all applicable letter ratings to indicate the relative number of chips of each size.

Test Results. Visually evaluate the resistance of the coating surface to chipping by gravel impact using the transparent grid and the rating scheme (Tables 5, 6 and 7, and Method 1 and Method 2).

Reports. Report the summarized number-letter rating and all applicable test conditions. In addition, report the substrate material type and thickness; any preliminary surface treatment of test panels; the type of surface coatings; baking/aging or pertinent processing schedules; and the film thickness of the coating system being evaluated.

#### 4.4.6.5 Acceptance Criteria

| Substrate | Acceptance Criteria, Minimum Performance (MP) | Acceptance Criteria, Improved Performance (IP) | Acceptance Criteria, Best Performance (BP) |
|---|---|---|---|
| Steel | After one cycle, chip rating not less than 5B | After one cycle, chip rating not less than 7C | After one cycle, chip rating not less than 9C |

### 4.4.7 Rising Step Load (Stress Corrosion Cracking)

#### 4.4.7.1 Scope

Hydrogen embrittlement testing shall be performed on any candidate that is considered a risk candidate. Resistance to environmentally assisted cracking shall be assessed using the rising step load method for determination of $K_{IEAC}$. For this procedure, CV2 Charpy specimens of MIL-A-46100D shall be machined in longitudinal-transverse (L-T) and transverse longitudinal (T-L) orientations in accordance with ASTM E 399-97. Unlike the steel test panels, the charpy specimens shall not be abrasive blasted prior to pretreatment.

#### 4.4.7.2 Equipment

The equipment shall be determined by the applicable test method.

4.4.7.3 Reagents

The reagents shall be as described in the applicable test method.

#### 4.4.7.4Procedure

Specimen fatigue precracking shall be carried out using three stages, each consisting of decreasing loading levels. In the first precracking stage, the load was maintained to keep stress intensity values below 80% of the estimated experimental critical stress intensity and the stress ratio ( $\sigma_{max}/\sigma_{min}$) was kept between –1 and+0.1. In the intermediate stage, the cycling load shall be reduced to maintain the stress intensity value as crack growth occurred and the intact cross section was reduced. For the final stage of precracking, the load shall be further reduced so the final value of $K_{max}$ will unlikely exceed 60% of the estimated value for $K_I$ during experimentation. Additionally, the final value for $K_{max}/E$ should not exceed 0.0032 $m^{1/2}$, where E is Young's modulus. Precrack length, represented by the dimensionless expression a/W (crack length over specimen width), shall be maintained near 0.5.

Specimens shall be fastened into a double cantilever array test fixture under aqueous conditions with 3.5% NaCl solution at open circuit potential conditions. Specimens shall be loaded by incremental steps in accordance with ASTM F 1624-95 (*26*) using an appropriate load frame apparatus. The specimen load values versus time shall be recorded. The calculation for the onset of environmentally assisted cracking, or KIEAC, is derived as follows for cantilever bending from the four-point bending expression.

$$K_{IEAC} = \left( \frac{6M_{IEAC}\sqrt{\pi a}}{BW^2} \right) \times f(a/W)$$

### 4.4.8 Field Exposure, Static (ASTM G50)

#### 4.4.8.1 Scope

This test method describes a basic procedure for conducting outdoor testing of specimens incorporating of candidates for GVFSs.

#### 4.4.8.2 Equipment

Standard Racks. See Section 5 of ASTM G50.
Scribe Tool. An ANSI B 94.50, style E scriber.
Straightedge. Any straightedge of sufficient length to guide the scribing tool in a straight line.
Balance. A digital electronic balance capable of weighing up to 10,000 mg with an accuracy of +/- 1%.

#### 4.4.8.3 Reagents

Cleaning Solution. Methanol.

#### 4.4.8.4 Procedure

Test Specimens. Prepare at least 10 specimens consisting of sections of actual GVFSs or manufactured parts that accurately simulate current production material and manufacturing processes, incorporating the candidate, and 10 specimens incorporating the current corrosion protection system. Each test specimen and reference coupon shall contain a clear identification mark. Use reference coupons consisting of uncoated 25 x 51 x 3 mm (1" x 2" x 1/8") pieces of any alloy AISI 1006 through 1010 steel to monitor the average general bare steel corrosion produced by the test track environment. The reference coupons shall be thoroughly cleaned using the cleaning solution. The coupon weight in milligrams shall be recorded and retained for future reference.
Test Sites. Test sites shall be chosen at a number of locations representative of the atmospheric environments where the military vehicle is likely to be used.
Preparation. Using test specimens incorporating the candidate and the current corrosion protection system, scribe a single diagonal line making sure that the scribed line is all the way through the coating into the substrate.
Test Procedure. Attach the test specimens and reference coupons to the racks at the approved test site and test in accordance with the test site standard practice, ASTM G50, “Standard Practice for Conducting Atmospheric Corrosion Tests on Metals”. ASTM G50 recommends a multi-year exposure period to minimize the variability of environmental (industrial and natural) factors influencing the atmospheric corrosivity of a test site. Monitor environmental factors in accordance with ASTM G50. Evaluate the performance of the candidate and current corrosion protection system test specimens, and reference coupons at six-month intervals and at the completion of the exposure period. At the end of the exposure period, clean the reference coupons using a mild sand (or glass bead) blast to remove all corrosion by-products.

Test Results. Inspect the test specimens and reference coupons for any signs of degradation. Measure scribe creep in accordance with ASTM D1654. Once clean, wipe coupons with methanol and weigh to determine weight loss. Corrosion losses may also be expressed in terms of average corrosion rates from the weight loss, coupon area, test duration, and metal density by use of the calculation described in ASTM G1.
Report. Report observations in accordance with the test site standard practice, ASTM G50, including environmental factors monitoring, and weight loss and/or corrosion rate of the reference coupons.

#### 4.4.8.5 Acceptance Criteria

| Test | Acceptance Criteria, Minimum Performance (MP) | Acceptance Criteria, Improved Performance (IP) | Acceptance Criteria, Best Performance (BP) |
|---|---|---|---|
| Field Exposure, Static | Three years of exposure: specimen has a minimum of 25% less creepage from scribe than current corrosion protection system | Four years of exposure: specimen has a minimum of 2~~50~~% less creepage from scribe than current corrosion protection system | Five years of exposure: specimen has a minimum of 75% less creepage from scribe than current corrosion protection system |

### 4.4.9 Field Exposure, On-Vehicle (ASTM D 1654)

#### 4.4.9.1 Scope

This test method describes a basic procedure for conducting on-vehicle testing of candidates. This may be performed by selective replacement or refinishing of an appropriate representative substrate/component on a vehicle incorporating the candidate, or by the use of test specimens incorporating the candidate attached to the military ground vehicle.

#### 4.4.9.2 Equipment

Military Ground Vehicle. A vehicle used for standard deployment.

#### 4.4.9.3 Reagents

Cleaning Solution. Materials required as designated by each candidate supplier.

#### 4.4.9.4 Procedure

At a minimum, the process shall be conducted to replace or refinish a part or section of the vehicle in accordance with the suggested finishing parameters and the controls established by the CARC applications specification MIL-DTL-53072. If using test panel, they shall be prepared in accordance with above G50 for static field testing and evaluated using ASTM-D 1654. Representative substrates/components will be pretreated in accordance with pretreatment manufacturers recommended specifications finishing parameters and controls established in MIL-DTL-53072. Components substrates will be evaluated during periodic inspections by visual comparison with the base vehicle or control samples attached to the vehicle. The Society for

Protective Coatings SSPC-VIS-2 “Standard Method for Evaluating the Degree of Rusting on Painted Steel Surfaces” shall be used for evaluating component substrates and control samples. The success criteria for field testing will be performance greater than or equal to the base vehicle (baseline) or control sample.

Report. After a predetermined exposure agreed upon by the stakeholders, the affected vehicles/parts shall be evaluated for coating adhesion, color, and corrosion resistance in accordance with SSPC-VIS-2, MIL-DTL-53072 and ASTM D 1654.

## 5.0 FAILURE ANALYSIS

To be considered for use as a replacement for the current corrosion protection system, a candidate must pass all tests. The failure of any Screening, Performance, or Special Test shall be documented in the JTR. At the candidate vendor's request and expense, a failure analysis procedure can be undertaken to determine the failure mechanisms. Such failure analysis can be a useful vendor option to identify and correct failure mechanisms prior to retesting. However, after failing any of the Screening, Performance, or Special Tests for the third time, further iterations of that test are not permitted. Instead, the JTP process shall be ended and the results noted in the JTR. The JTR shall then be forwarded to the vendor for transmittal to the invoking authority for review.

In the event of any testing-related dispute between vendor and tester, such as causes of premature failure, a third-party testing lab will be mutually agreed upon as a credible testing source by the invoking authority. This Product Failure Laboratory (PFL) must have no pre-existing connections to either the vendor of the candidate or the original laboratory that conducted the testing. The process flow is illustrated in Figure 1, which appears in Section 2.0, JTP Document Guide.

Marginal test results must be either overcome by retesting or documented before rejecting/failing the candidate. Failure in any test does not necessarily disqualify a candidate for use in all possible applications.

The initial JTR and all related JTRs (specifically those documenting failure analyses) shall be submitted to the vendor for transmittal to the invoking authority for review.

## 6.0 REFERENCE DOCUMENTS

The documents listed in Table 8 were referenced in the development of this JTP.

**Table 8. Reference Documents**

| Reference Document | Title | Applicable Section(s) of Reference Document | JTP Test | JTP Section Cross-Reference | Document Source |
|---|---|---|---|---|---|
| ASTM B117 | Standard Test Method of SS (Fog) Testing | All | Corrosion Resistance NSS (Fog) | 4.4.3 | ASTM |
| ASTM D714 | Test Method for Evaluating Degree of Blistering of Paints | All | Corrosion Resistance NSS (Fog)<br>Corrosion Resistance (Cyclic)<br>Field Exposure, on-Vehicle | 4.4.3<br>4.4.4<br>4.4.12 | ASTM |
| ASTM D1193 | Specification for Reagent Water | All | All | All | ASTM |
| ASTM D1654 | Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments | All | Corrosion Resistance NSS (Fog)<br>Corrosion Resistance (Cyclic)<br>Seawater Immersion<br>Field Exposure, Static<br>Field Exposure, On-Vehicle<br>Corrosion Resistance (Modified Salt/$SO_2$ Spray (Fog)) | 4.4.3<br>4.4.4<br>4.4.5<br>4.4.11<br>4.4.12<br>4.4.13 | ASTM |
| ASTM D3359 | Standard Test Methods for Measuring Adhesion by Tape Test | All | Adhesion (Dry)<br>Adhesion (Wet) | 4.4.1<br>4.4.2 | ASTM |
| Federal Specification TT-C-490D | Cleaning Methods for Ferrous Surfaces and Pretreatments for Organic Coatings | 3.5.9<br>3.5.10 | Hydrogen Embrittlement | 4.2 | DoD |
| SAE J400 | Test for Chip Resistance of Surface Coatings | All | Chip Resistance at –29° Celsius | 4.4.6<br>4.4.14 | SAE |
| GM9540P | Corrosion Resistance (Cyclic) | All | Corrosion Resistance (Cyclic) | 4.4.4 | ASTM |
| MIL-DTL-53072 | Chemical Agent Resistant Coating (CARC) System Application Procedures And Quality Control Inspection | All | All | | |

# APPENDIX

## LIST OF STAKEHOLDERS AND LIST OF CONTRIBUTORS

## List of Stakeholders

| FIRST NAME | LAST NAME | ORGANIZATION | ADDRESS | CITY/STATE/ZIP | PHONE | FAX | E-MAIL |
|---|---|---|---|---|---|---|---|
| Steve | Bails | SBCT | | Warren MI 48397-5000 | 586-282-7886 | | Stephen.Bails@us.army.mil |
| Todd | Weimer | PEO-CS-CSS, MRAP | SFAE-CSS-MR-E | Warren MI 48397-5000 | (586) 282-2679 | (586) 282-2978 | todd.p.weimer@us.army.mil |
| Jack | Kelley | ARL | RDRL-WMM-C | Aberdeen Proving Ground, MD 21005-5059 | 410-306-0837 | 410-306-0829 | Jack.kelley1@us.army.mil |
| Thomas | Braswell | ARL | RDRL-WMM-C | Aberdeen Proving Ground, MD 21005-5059 | 410-306-0935 | 410-306-0829 | Thomas.e.braswell@us.army.mil |
| Luwam | Hugos | NAVAIR | NAVAIR, Code 4.3.4.2 48066 Shaw Road, Bldg. 2188 | Patuxent River, MD 20670 | (301) 342-8159 | (301)342-8062 | luwam.hagos@navy.mil |
| Amy | Fowler | NAVAIR | NAVAIR, Code 4.3.4.2 48066 Shaw Road, Bldg. 2188 | Patuxent River, MD 20670 | 301-342-0986 | 301-342-7566 | amy.fowler1@navy.mil |
| Brian | Placzankis | ARL | RDRL-WMM-C | Aberdeen Proving Ground, MD 21005-5059 | * | * | * |
| Andrew | Sheetz | NSWCCD | 9500 MacArthur Blvd, Code 6130 | West Bethesda, MD 20817-5700 | * | * | * |
| John | Escarsega | ARL | RDRL-WMM-C | Aberdeen Proving Ground, MD 21005-5059 | * | * | * |
| Chris | Miller | ARL | RDRL-WMM-C | Aberdeen Proving Ground, MD 21005-5059 | * | * | * |
| Carl | Handsy | TACOM | 6501 E Elenen Neil Rd, AMSTA-TR-E1 MEPS1267 | Warren, MI 48397-5000 | * | * | * |

## Appendix B: Points of Contact

| POINT OF CONTACT Name | ORGANIZATION Name Address | Phone Fax E-mail | Role in Project |
|---|---|---|---|
| Jack Kelley | US Army Research Laboratory<br>B4600 Deer Creek Loop<br>Aberdeen Proving Ground MD | PH: 410-306-0837<br>FX: 410-306-0829<br>BB: 240-429-8485<br>jkelley@arl.army.mil | Project Lead |
| Tom Braswell | US Army Research Laboratory<br>B4600 Deer Creek Loop<br>Aberdeen Proving Ground MD | PH: 410-306-0935<br>FX: 410-306-0829<br>Thomas.e.braswell@arl.army.mil | Testing, and Specifications |
| Amy Fowler | NAVAIR, Code 4.3.4.2<br>48066 Shaw Road, Bldg. 2188<br>Patuxent River, MD 20670 | PH: 301-342-0986<br>FX: 301-342-7566<br>amy.fowler1@navy.mil | Navy Co-Performer |
| Luwam Hagos | NAVAIR, Code 4.3.4.2<br>48066 Shaw Road, Bldg. 2188<br>Patuxent River, MD 20670 | PH: 301-342-8159<br>FX: 301-342-8062<br>luwam.hagos@navy.mil | Navy Co-Performer |
| Patricia Dodson | Patty Dodson<br>Anniston Army Depot<br>7 Frankford Avenue<br>Bld. 106<br>Anniston, AL 36201 | Patricia.dodson@us.army.mil<br>COMM: 256-235-6700 | Coordinate demonstration at Anniston |
| Dr. Keith Legg | Rowan Technology Group<br>1590 South Milwaukee Ave., Suite 205<br>Libertyville, IL 60048 | Telephone: 847-680-9420<br>FAX: 847-680-9682<br>klegg@rowantechnology.com | Cost Assessment |