NAVAIR Hexavalent Chromium Minimization Status

SERDP/ESTCP Symposium 2010
Cr\textsuperscript{6+} Session

Bill C Nickerson
AIR 4.3.4
NAVAIR has set forth a comprehensive goal for the development and qualification of nonchromated materials. Candidate alternative materials have often been developed and evaluated to the bare minimum of performance requirements, leading to reduced corrosion protection and loss of robustness in the materials protection scheme. The NAVAIR nonchromated materials goal is to: Identify, test, validate and implement non-chromate, Class N primers and surface preparations which are as broad in capabilities and performance as current chromated, Class C, primers and surface preparations. To this end, demonstrated capabilities must include performance across multiple alloys/substrates, with and without subsequent topcoats and specialty coatings. Alternative materials must perform a variety of uses, such as protection of faying surfaces, galvanic corrosion protection in dissimilar materials interfaces, wet installation of fasteners and bushings, resistance to SCC, embrittlement and exfoliation damage and other traditional uses of chromate primers, metal finishes, and sealants. Coatings must perform over Type I and Type II conversion coatings per MIL-DTL-81706/MIL-DTL-5541. Additional surface conditions/finishes that the coating must demonstrate compatibility and full performance over are finishes such as: Type I, IC, II, or IIB anodized aluminum per MIL-A-8625, sacrificial coatings (such as IVD-Al, Cd, Zn-Ni, etc.), touch-up on Fe alloys, other conversion coated or anodized light metals such as Ti, Mg, and Zn, and composite substrates. Full implementation will also require demonstration of equivalent performance in adhesion filiform, humidity, and fluid resistance properties for all the materials and applications currently protected by Class C coatings and finishes. This talk will address the current and planned alternative materials efforts, status of approval and technology implementations, and identify gaps and shortfalls in the current technologies.
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<th>16. SECURITY CLASSIFICATION OF:</th>
<th>17. LIMITATION OF ABSTRACT</th>
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<td>b. ABSTRACT unclassified</td>
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NAVAIR NON-CHROMATE MATERIALS STATUS

MR. BILL NICKERSON
NAVAIR 4.3 Air Vehicle Engineering
48066 Shaw Road
Building 2188
Patuxent River, MD 20670
(240) 925-0175
william.nickerson@navy.mil

NAVAIR has set forth a comprehensive goal for the development and qualification of non-chromated materials. Candidate alternative materials have often been developed and evaluated to the bare minimum of performance requirements, leading to reduced corrosion protection and loss of robustness in the materials protection scheme. The NAVAIR non-chromated materials goal is to: Identify, test, validate and implement non-chromate, Class N, primers and surface preparations which are as broad in capabilities and performance as current chromated, Class C, primers and surface preparations. To this end, demonstrated capabilities must include performance across multiple alloys/substrates, with and without subsequent topcoats and specialty coatings. Alternative materials must perform a variety of uses, such as protection of faying surfaces, galvanic corrosion protection in dissimilar materials interfaces, wet installation of fasteners and bushings, resistance to SCC, embrittlement and exfoliation damage, and other traditional uses of chromate primers, metal finishes, and sealants. Coatings must perform over Type I and Type II conversion coatings per MIL-DTL-81706/MIL-DTL-5541. Additional surface conditions/finishes that the coating must demonstrate compatibility and full performance over are finishes such as: Type I, IC, II, or IIB anodized aluminum per MIL-A-8625, sacrificial coatings (such as IVD-Al, Cd, Zn-Ni, etc.), touch-up on Fe alloys, other conversion coated or anodized light metals such as Ti, Mg, and Zn, and composite substrates. Full implementation will also require demonstration of equivalent performance in adhesion, filiform, humidity, and fluid resistance properties for all the materials and applications currently protected by Class C coatings and finishes. This talk will address the current and planned alternative materials efforts, status of approval and technology implementations, and identify gaps and shortfalls in the current technologies.
NAVAIR Non-Chromate Coatings Goal

Identify, test, validate and implement non-chromate, primers and surface preparations which are as broad in capabilities and performance as current chromated primers and surface preparations.

• Performance across multiple alloys/substrates, with and without topcoats per MIL-PRF-85285 and TT-P-2760; in combination with specialty coatings
• Across all exposure conditions for all the materials currently protected by Class C materials.
• Galvanic Corrosion Protection – faying surfaces, dissimilar materials interfaces, wet installation of fasteners and bushings, SCC, exfoliation, etc.
• Surface Prep/Primer Compatibility –
  – Type I and Type II conversion coatings per MIL-DTL-81706/MIL-DTL-5541
  – Type I, IC, II, or IIB anodized aluminum per MIL-A-8625
  – Sacrificial coatings (such as IVD-Al, Cd, Zn-Ni, etc.)
  – Fe alloys, other conversion coated or anodized light metals such as Ti, Mg, and Zn, and composite substrates
  – Adhesion, filiform, humidity, and fluid resistance properties
Policy and Technology Drivers

- Environmental Regulatory –
  - OSHA PEL, RoHS, WEEE, REACH, EO
- DoD Policy –
  - DUSD AT&L Policy Memo Minimizing the use of Hexavalent Chromium (Cr$^{6+}$) – 8 Apr 2009
- Technology –
  - Performance, Durability, Reliability, Maintainability
- Cost –
  - Cost of Corrosion – Increasingly complex, expensive systems
    - Focus solely on ESOH reduced robustness of protective materials and coatings
  - Aging Aircraft – Flying beyond predicted service life
Operational Reality

- Extremely Harsh Environment = High Life Cycle Cost
  - True for current environmentally unfriendly yet robust materials
  - Factors: TMS, # in inventory, average age/flight hours, airframe corrosion ~7X
NAVAIR Transition Strategy

- Engage all relevant levels and user communities –
  - Military & Commercial OEM’s
  - Depot/Manufacturing Sites
  - Industry Partners, Chemical Manufacturers
  - O-level activities
  - Research & Development, Demonstration/Validation, Specifications, Technology Transition

- Implementation Path –
  - Lab validation – process and product performance
  - Field validation – process and product performance

- Risk Analysis & Mitigation – Application Axis vs. Platform/Basing Axis
Proposed NAVAIR Policy/Procedure

- Take a Command-Wide Approach
  - Command Response with PEO concurrence
  - Establish a Cr\(^{6+}\) Usage Certification Process
  - Communicate state-of-the-art alternatives
  - Establish risk reduction process that accounts for performance/technical, environmental and logistics factors

- Establish a NAVAIR Cr\(^{6+}\) Elimination Task Force Team
  - Develop Action Plan (Command)
  - Identify command use, applications, and current/future mitigation strategies

- Document, assess, monitor, track the use of Cr\(^{6+}\)
  - Program and budget for mitigation of environmental regulations

- AIR 4.3 Engineering Circular –
  - Uniform guidance for PMA’s
Risk Analysis for Implementation of Non-Chromate Technology

<table>
<thead>
<tr>
<th>Probability of Failure for Non-Chromate Technology vs. Chromate*</th>
<th>Impact of Non-Chromate Technology Failure</th>
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<tbody>
<tr>
<td>High</td>
<td>Mishap, Replacement</td>
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<td>Reduced Service Life, High Repair Costs</td>
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<td>Increased Maintenance Activities</td>
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<td>Medium</td>
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<td>Low</td>
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<td>Same as Chromate</td>
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* Probability of failure of non-chromate technology based on sufficient laboratory testing, comparison to current chromate technology for a particular application, and AIR-4.3.4 endorsement.

** Note: Factors such as platform/component operational environment and inspection intervals must be considered and may justify adjustment to the risk analysis level. Ex. Trainer aircraft operate in a less severe environment than ship based aircraft.
Accelerated Testing

- High-Solids / Water-Reducible Chromate Primers – initial 1000 hours in ASTM B117 (MIL-P-23377 rev F/1989)
  - Based on QC of established chromate pigments/coating performance on aircraft
    - Class N products in each spec, 2000 hours for both specs, all primers
    - Change made without field data to establish correlation to accelerated tests
- Presumed performance to spec tests would yield good field performance based on known chromate chemistry –
  - Galvanic couples, beach/real world exposure, barrier properties, substrate/surface preparation compatibility – Not reflected in specification testing
  - Minimum performance based on quality control does not equal similar performance when trying to validate and authorize new coatings
  - Unknown correlation of performance of new non-chromate inhibitor chemistries in field compared to accelerated corrosion tests
  - Large differences in performance for solvent-borne vs. water-reducible primers in galvanic tests regardless of inhibitors – Solvent-borne typically better in lab test data
  - Non-chromate primers tend to rely more on pretreatment performance
  - No general guidance exists on how to use tests, suggested combinations of alloys and tests, comparative data of accelerated tests versus beach exposure
Test to Failure vs. Qualification Criteria

Weeks in ASTM B 117

- MIL-PRF-23377 & MIL-PRF-85582 Performance Range, no Topcoat
- MIL-PRF-23377 & MIL-PRF-85582 Performance Range, with Topcoat
- MIL-A-8625 Type II Performance Range
- MIL-A-8625 Type IIB and IC Performance Range
- MIL-PRF-23377 and MIL-PRF-85582 Qualification Requirement
- MIL-DTL-81706 Performance Range
Non-chromate Coatings Test Protocol

ESTABLISH STANDARD PRACTICE – Minimize or eliminate false positives and negatives in accelerated testing

- Use AA2024-T3 and AA7075-T6 aluminum panels. Use 1 sacrificial coating plated over high strength steel, such as IVD-AI/4340. Use standard 1018/1020 LC steel panels.


- 1+ year minimum beach exposure test – at a facility with a documented salt-laden, corrosive local environment, such as the Kennedy Space Center corrosion test facility.

- Evaluate coatings in faying surface and fastener dissimilar metal couples, as well as with any specialty coatings, always with a known chromate control.

- Test all non-chromate primers in conjunction with currently authorized and promising non-chromate metal finishing technologies, i.e. MIL-DTL-81706, MIL-A-8625, TT-C-490, MIL-DTL-84388, etc.

- Evaluate alternatives with and without topcoat and with simulated damage (scribes) through the coating systems.

- As improved corrosion test methods become available, combine the test protocol with improved accelerated exposures.

- Test in faying surface and fastener dissimilar metal couples

- Evaluate compatibility with composites substrates, ceramics, and other electroplated/mechanically deposited coatings
Application Areas for Chromate Alternatives

Hexavalent chromium alternatives

- Aluminum
- Avionics/Electronics
- Components/Structure
- Chrome Plating
- Cadmium
- Sacrificial Coating Post Treatment
- Zinc-Nickel
- Aluminum
- Prep
- Bonding
- Wash Primer
- Primer
- Aircraft
- Vehicles
- Ships
- Support Equipment
- Type IC Seal
- Type IIB Seal
- MIL-A-8625 Anodize
- Type II Seal
- Magnesium/Titanium Anodize/Conversion
- Aluminum Anodizing
- Sealants
- Phosphating
- Rinse (Aluminum)
- Rinse (Steel)
- Primer
- Prep
- Equipment
Finishing Applications

• Conversion Coatings – Aluminum, Magnesium, Titanium, Zinc
• Anodizing Sealing – Aluminum Anodizing Sealing
• Sacrificial Coatings – Cd, Zn/Zn Alloys, IVD-Al/Aluminum Plate
• Phosphate Rinsing – Zn/Mn/Fe Phosphate
• Rust Inhibiting Coatings – Fe & Steel Alloys
• Wash Primers – Multiple Substrate Processing
• Mechanical Coatings – Hard Cr, HVOF, CS
• Process Chemicals – Cleaners, Degreasers, Etchants, Deoxidizers
• Process Solutions – Chemical Conversion, Anodize/Anodize Rinsing
• Organic Films – Epoxy Primer, Alkyd Primer, Polyurethane Primer
• Adhesive Structural Bonding – Sol-gel, Anodize, Bond Primers
• Sealants – Polysulfide, Polythioether, Sealants, Polyurethane
# Authorization/Implementation Status

<table>
<thead>
<tr>
<th>M&amp;P Area</th>
<th>Sub Area</th>
<th>Location</th>
<th>Process Status</th>
<th>wt % of Cr6+ in NAE</th>
<th>% of Cr6+ eliminated</th>
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</thead>
<tbody>
<tr>
<td><strong>Painting/Primer</strong></td>
<td>Support</td>
<td>Depot and Field</td>
<td>Non-chromate primer in use (MIL-DTL-53022) for all applications</td>
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<td>100</td>
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<td>Aircraft</td>
<td>Depot and Field</td>
<td>Non-chromate primer authorized scuff sand and overcoat of chromate primer. Limited demonstrations underway direct to metal on fleet C-2s. More demos planned for FY11+.</td>
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<td><strong>Adhesive Bonding</strong></td>
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<td>Depot and Field</td>
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<td><strong>Sealing</strong></td>
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<td>Depot and Field</td>
<td>Non-chromated sealants are available but have not been transitioned to depot and field applications (field introduction controlled through new acquisition programs)</td>
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<td><strong>Aluminum Pretreatment</strong></td>
<td>Avionics/Electrical</td>
<td>Depot and Field</td>
<td>Alternative not authorized. Class 3 demonstration and validation efforts proposed for FY11. Leveraged with NASA effort.</td>
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<td>Components</td>
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<td>Sealing Type II &amp; III</td>
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<td>Authorization of alternative (MIL-DTL-81706 Type II TCP products) pending authorization letter in FY10. Jacksonville planning to implement TCP as soon as authorization is issued.</td>
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<td><strong>Sealing Type IIB</strong></td>
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<td>Type IIB not authorized currently. Dem/val underway to produce data for potential authorization as Type IC alternate. Being considered by Jacksonville as part of single tank Type II, IIB and III anodize system. Authorization of alternative (TCP) pending.</td>
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<td><strong>Hard Chrome Plating</strong></td>
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<td>WCCo and WCCoCr HVOF coatings authorized; limited to low stress/spalling risk applications. CoP plating in dem/val through ESTCP project.</td>
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<td>Depot and Field</td>
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<td>Mn-phosphate process with chromate rinse. New alternative being assessed (ChromiPhos) as part of ESTCP project with the Army.</td>
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R&D Efforts and Needs

• Correlation of accelerated corrosion test results with results of natural, beach front and ship-board exposures
  – “Cherry Picking” alloys and accelerated tests leads to strong false positives and negatives
• Aerospace is last “hold-out” for Cr\(^{6+}\) use in protective coatings
  – Navy and Marine Corps operating requirement is most challenging
    • Alternatives acceptable for automotive, commercial aviation, land-based DoD are typically not good enough
    • Simulation of operating environment a challenge for RDT&E and acquisition/design trade studies
• Additional challenges – Mechanisms, HAP/VOC, Logistics
  – Basic understanding of corrosion protection mechanisms for qualified and emerging non-chromated, proprietary inhibitors
  – Basic understanding of inhibitor effects on stress-corrosion cracking and corrosion fatigue
  – Primers with improved flexibility, high adhesion and easy removal, improved processing characteristics
  – Reduce or eliminate VOCs/HAPs/TRI chemicals from current coating systems
Assessment of Accelerated Tests Compared to Beachfront Test and Proposed Evaluation Method

<table>
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<tr>
<th>Tests</th>
<th>Correlation</th>
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<tr>
<td>Beach vs B117</td>
<td>0.915</td>
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<tr>
<td>Beach vs 9540P</td>
<td>0.94</td>
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<tr>
<td>Beach vs SO2</td>
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<td>Beach vs B117/SO2</td>
<td>0.908</td>
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<tr>
<td>Beach vs B117/9540P</td>
<td>0.943</td>
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Dynamic Accelerated Corrosion Testing Methodology

**Deliverables**
- Accelerated corrosion test method that more accurately predicts component failure modes as a function of environment and can discriminate relative performance of material systems
- A means to implement mechanical loading into accelerated corrosion tests to simulate coating and structural material failures
- Improved understanding of the relationship between environmental parameters and corrosion failure modes
- A data mining tool that can be used to tailor laboratory tests to excite various combinations of failure modes
- 1 MS and 2 Post Docs from UVA
- Peer reviewed publications

**Technical Objectives**
- Integrate representative sample designs into accelerated corrosion testing
- Integrate mechanical loading into accelerated corrosion testing
- Characterize and compare the development of the corrosion electrolytes for “real world” and current accelerated corrosion tests
- Determine the effect of critical environmental and mechanical parameters on degradation modes of system components
- Develop an AI framework to tailor the accelerated corrosion test to real world failure modes

**Problem Statement**
- Advanced materials for improved performance are being implemented on new and legacy systems
- Limitations of current corrosion testing
  - Long term testing is rate limiting for technology implementation
  - Short term laboratory testing is proving inadequate for predicting long term performance (i.e. promising technology fails lab testing while poor technology passes lab testing)
- Need to improve accelerated corrosion testing
  - Differentiate candidate materials
  - Accelerate “real world” failure modes

**Task 1 - Laboratory Exposure Testing**
- How are current laboratory environments different from real world?

**Task 2 - Outdoor Exposure Testing**
- How do accelerated and outdoor environments compare?

**Task 3 - Laboratory studies of critical variables**
- What are the critical variables that drive corrosion?
- What are the relationships between these critical variables and corrosion processes?

**Task 4 - Sample design - Data mining**
- What geometry can be used for a universal corrosion test samples?
- Can we develop test criteria that can be used to tailor lab test methods to replicate damage in the field?

**Task 5 - Measure the evolution of chemistry and morphology**
- Measure the relationship between RH, temp., and TOW
- Identify differences between accelerated and outdoor environments

**Task 6 - Determine critical electrolyte chemistry**
- Chemical composition
- RH effects
- UV effects
- Determine effects of environment
- Environmental cracking
- Corrosion at a coating defect
- Coating fracture
- Coating adhesion

**Task 7 - Develop a corrosion test that simulates component geometries and materials**
- Use data mining tools to model relationships between environment and corrosion failure modes
- Based on these models, develop an accelerated corrosion tests
NAVAIR Coating Stack-up Effects

• **Aluminum Surface Preparation** – Anodize > A1200S > TCP > A5200/5700
  – Most consistent performance with/across substrates, primers, materials and exposures – No interest in “muddled middle” or sub-tier adhesion promoters

• **Adhesion is not the only story** –
  – Clear positive impact of “passivation” character of CC on surface
  – Clearly seen in galvanic and beach exposures

• **Ferrous Alloy Preparation** –
  – Most demonstration and validation of chromate alternatives has focused only conversion coating of aluminum and outer mould-line primers
  – There remains a need to demonstrate and validate effective replacement technology for applications on steel
  – Elimination of chromated wash primers and many chromate-rinsed phosphate processes reduced overall protection of coating systems
NAVAIR Primer Efforts

- "Silver" Standard – MIL-DTL-5541 Type II/MIL-PRF-23377 Type I –
  - Most applications covered – 95+% solution – Next Gen Primer needed
  - Robustness is Key – Most robust surface preparations + most robust organic coatings = Most robust coating systems
  - Misconception regarding resins – 340 g/L

- Resin Properties often overlooked –
  - Inhibitor is not the only functional component, adhesion and barrier properties controlled by resin system
  - Also impacts pigment loading and inhibitor release function
  - 23377 High-solids “solvent-borne” superior resin system for total protection
  - 85582 “Waterborne” inferior galvanic corrosion protection, better application characteristics
  - Effect more pronounced in Class N primers
    - Rely more on surface preparation performance
  - Example – Deft 02GN084 > Deft 44GN098 > Deft 65GN015D
NAVAIR Non-Cr6+ Efforts

• AERMIP – GSE focused – Dem/Val Class N primer/ZVOC topcoat

• NESDI – Current “silver-standard” approach – support DEM/VAL
  – Best available technology, limited/lower risk implementation

• ESTCP WP 0731 – Project ended FY09 – Magnesium-rich Primer
  – Performance issues in primer only condition, galvanic assemblies (with or without topcoat), and beach exposures that show rupturing/tunneling failures through the coating and self-corrosion of the primer itself

• ESTCP WP-1010 – Project scaled back in scope – E’coat Primer
  – Promising barrier properties and coating uniformity for complex geometries. Uses an organic inhibitor in a conductive polymer resin. Performance issues for protection of scribed/damaged areas in initial corrosion testing.

• NISE/NASA – Type II, Class 3 Conversion Coatings
  – Lab Validation and Dem/Val for Electronics Applications
MIL-PRF-23377 Class N Dem/Val

- Objective: Transition Type I non-chrome primers across all gloss paint T/M/S to eliminate Cr6+ in primers by FY12
- DEM/VAL four E-2 Hawkeyes using the leading Class N primer
  - Follow on – Obtain authorization to DEM/VAL on F/A-18s
  - E-2/C-2 DEM/VAL with Type II conversion coating + Class N primer
Performance Evaluation of a Magnesium-Rich Primer (Project WP 0731)
Most demonstration and validation of chromate alternatives has focused only conversion coating of aluminum and outer mould-line primers.

- There remains a need to demonstrate and validate effective replacement technology for applications on steel.
Advanced Anodizing using Process Control Technology

- NESDI N-0086-02: Low HAP Coatings, Solvents and Strippers.
  - Integration of Metalast Process Control technology for producing Type II, IIB & III coatings within one tank system for Depot-Level maintenance
  - Metalast Process Control Technology to include Interface Controller, Process Controller & Bath Additive
  - Evaluate TCP as a non Cr+6 post anodize sealer for all coating types.
    - ROI: 30.7 or Payback Period of 2.1 Yrs

- Capabilities gained:
  - Reduces Operator error and Supervision of Process
  - Improved quality, accuracy and repeatability
  - Reduces defects and rejects
  - Accountability of Work Performed

- Efficiencies achieved:
  - Reduces cycle & throughput times
  - At least 15% more efficient than conventional anodizing

- Environmental benefits achieved:
  - Extends life of bath chemistry/ Reduced Waste
  - Energy savings due to use of aluminum cathodes
  - Allows for consolidation of anodizing processes
  - Elimination of Hexavalent Chromium

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Potential Replacement with TCP

NESDI Project will lead to >35% volume reduction of Cr+6 usage in FRC-SE Treatment Shop

- FRC-SE (JAX) Fully Integrated
- FRC-E (CP) Fully Integrated
- FRC-SW (NI) Integration in Process
Advanced Anodizing using Process Control Technology

TCP shows better performance than Dichromate Sealing

Dichromate Seal (5% wt)
Panels A2-BS1C (1 – 5)
15 minute seal @ 203°F

2,033 Hrs NSF
Average Coating Weight: 450 mg/ft² (~2.6 µm)
Current Density used: 8 ASF for 13 min

TCP-HF (1:1)
Panels A2-BS1T (1 – 5)
10 minute seal @ 80°F

Type IIB TCP sealed coupons went well beyond 3,000 hrs before significant pitting corrosion was visible

Dichromate Seal (5% wt)
Panels A2-B2C (1 – 5)
15 minute seal @ 203°F

7,272 Hrs NSF
Average Coating Weight: 2,880 mg/ft² (~1 2.7 µm)
Current Density used: 12 ASF for 40 min

TCP-HF (1:1)
Panels A2-B2T (1 – 5)
10 minute seal @ 80°F
ESTCP Comprehensive Evaluation and Transition of Non-chromated Primers

**Technical Objectives**

- Develop an application matrix to map candidate technologies to proposed uses, and provide design space guidance to researchers and developers.
- Provide a “top down” assessment of current NC primer technology, including coating process manufacturing readiness level and coating technology readiness level.
- Develop and publish DoD/service testing guidelines which will support authorization and implementation by appropriate technical authorities.
- Demonstrate and validate NC primers and processes with sufficient process and coating maturity.
- Invest in development of promising newer technologies which have the potential to exceed the performance of today’s best NC primers or provide significant cost savings to standard “wet” paint processes.
- Modify MIL-PRF-23377 and MIL-PRF-85582 to account for improved testing methods and potential new types of primers like metal rich, e-coat, and UV-cured products.