



IAEG® WG2 Technical Exchange Project Summary Report, Non Chrome 6 Anodize Seals

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Version 2

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Acronyms

BSAA	Boric-Sulfuric Acid Anodize
CAA	Chromic Acid Anodize
DiCr	Dirchromate Seal, (Potassium or Sodium)
IAEG	International Aerospace Environmental Group
OEM	Original Equipment Manufacturer
OSHA	Occupational Safety & Health Administration
REACH	Registration, Evaluation, Authorisation and Restriction of Chemicals
SAA	Sulfuric Acid Anodize
SOW	Statement of Work
TFSAA	Thin-Film Sulfuric Acid Anodize
TSAA	Tartaric-Sulfuric Acid Anodize
TCP	Trivalent Chromium Pretreatment (Trivalent Chrome Conversion Coating)
VOC	Volatile Organic Compound
WP	Work Package

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1 Purpose

The Non-Chrome 6 Anodize Seal project was proposed and executed as a performance screening exercise of non-hex-chrome (with no nickel or cobalt) sealing solutions used for anodized aluminium, with and without paint coatings, (organic finishes, chromated and non-chromated), either developed by the project members or proposed by the major suppliers identified by the project members.

The outcome of this screening exercise is tabular test results summarizing performance levels for the agreed criteria of each proposed anodize seal solution, including the results communicated by project participants.

2 Scope

2.1 Project Scope

Sodium or potassium dichromate, or dilute chromic acid seals, heated to near 100°C, are common in the aluminum finishing industry and used to seal all anodize types. This project focused on less toxic alternatives acceptable for use with SAA (Sulfuric Acid Anodize), BSAA (Boric Sulfuric Acid Anodize), TSAA (Tartaric Sulfuric Acid Anodize) and TFSAA (Thin Film Sulfuric Acid Anodize), used by the aerospace industry. The project will not include seals used for Hardcoat, (MIL-PRF-8625 Type III Hard Anodize), Dyed Anodize coatings, (MIL-PRF-8625, Type II, Class 2) or Chromic Acid Anodize, (CAA or MIL-PRF-8625 Type I, Class 1). One or more of the less toxic anodic seals identified may also be applicable for CAA, but data development and collection efforts are directed to finding alternatives to CAA.

2.2 Project Structure

The project uses three (3) work packages (WP), each with an IAEG member company as the work package lead. The project lead for each work package was responsible for creating detailed project plans. Furthermore, the project lead responsibilities included producing deliverables, accomplishing milestones, and reporting progress. Figure 1 outlines project structure.

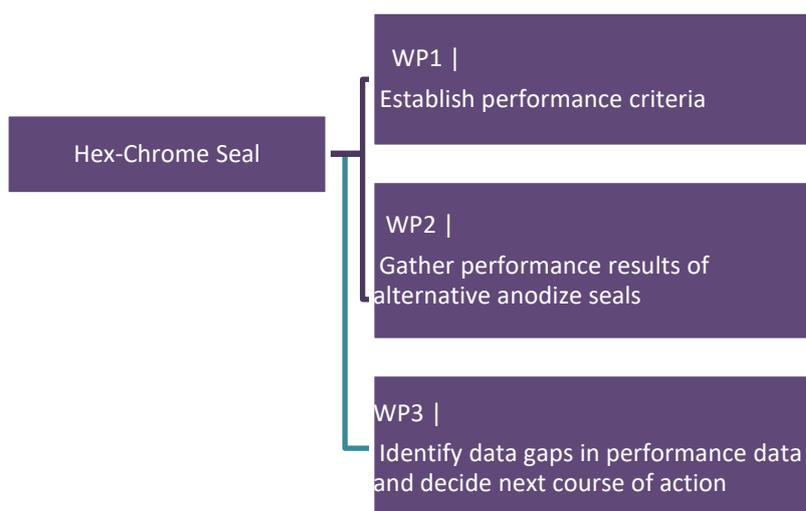


Figure 1. Project Structure Schematic

2.2.1 Work Package 1 (WP1)

This package established the composition of the alternative anodize seals considered for this project, appropriate performance criteria used to screen the alternative seals for the different anodize coatings, including unpainted and painted anodize, (chromated and non-chromated primers), and a format used to gather performance test results used for WP2.

Deliverables

- List of seal composition and performance criteria to replace hex-chrome seals
- List of performance tests required to determine if performance criteria have been met

2.2.2 Work Package 2 (WP2)

Tasks within this package included obtaining reports (or any form of data) on alternative anodize seals tested by project members or suppliers and align these results with the criteria established in WP1. A compiled list of performance results and a summary of those results in a table form (or other media) easily reviewed by the project members was developed. After determining the need for any clarification or additional results the team made adjustments as required to the data table.

Deliverables

- Reports on alternative anodize seals
- Summary of performance results for alternative anodize seals

2.2.3 Work Package 3 (WP3)

The team reviewed the performance results obtained from WP2, identified the data gaps from the list of performance results, and determined recommendations for the next course of action. If the course of action was to discontinue the project, then the team would prepare a final report. If the course of action was to continue the project, the team would prepare an interim report and a draft of the revised statement of work for the anodize seal project denoting the additional work packages required to continue the project.

Deliverables

- A presentation summarizing performance results and data gaps from results presented to project members to decide on the next course of action
- A Final or Interim Report (as determined by the meeting with the project members)
- Draft of revised SOW (if required)

3 Objectives

- Identification of environmentally friendly alternatives to common hexavalent chromium anodic seals, such as, dichromate and dilute chrome sealing solutions.
- Exchanging test data on anodic seal alternatives and using that data as a basis for implementation/substitution of said alternatives for hexavalent chromium anodic seals.
- Identification of any operational or other advantages to hexavalent chromium free anodic seals.

4 Background

The aerospace industry has been successfully replacing chromic acid anodizing with various non-chrome alternative anodize processes such as thin film sulfuric acid anodize, boric sulfuric acid anodize, and tartaric sulfuric acid anodize. However, most of these anodize processes still use a hex-chrome seal to pass rigorous aerospace corrosion resistance requirements for anodized aluminium with and without paint (particularly with scribed non-chromated paint systems). Non-hex-chrome seals, without nickel or cobalt, capable of compliance with performance requirements, are needed for aluminum anodize. Nickel and cobalt sealing solutions were eliminated from further consideration due to current and pending environmental regulatory regulations.

This project consisted of a performance screening exercise of non-hex-chrome sealing solutions used for anodized aluminium, with and without paint coatings (chromated and non-chromated), either

developed by the project members or proposed by the major suppliers identified by the project members.

Project results are found in Section 6 where a summary of each Work Package is provided; test data in table and figure form aligns required performance levels with measured performance of each proposed solution. Data includes test results communicated by the suppliers and test results provided by the participating project members. The project members will use results to determine the next course of action.

5 Participating Companies

COMPANY	REPRESENTATIVES
BAE Systems	Gareth Whittle
Bell Flight	Katlin Booth Brooklyn Pearson Thomas Sosa
Collins Aerospace, (Subsidiary of RTX)	Kevin Bordage Blair Smith Steven Poteet Steve Sproson Michael Metzger Olivier Brucelle Mark Brege
Honda Jet	Magdalena Michalowska
Pratt & Whitney, (Subsidiary of RTX)	Poulomi Sannigrahi
Pratt & Whitney Canada, (Subsidiary of RTX)	Marie-Claude Caplette Siliu Chen Marianne Guindon Daniel Meilleur
Rolls Royce	Laura Wilkinson
SAAB Group	Ronja Flink Nicola Naujoks Lars Olsson
Raytheon Technologies Corporation (RTX)	Michelina Molongoski
Textron Aviation	Stacey Sullivan April Sawyer J R Burgett
Thales Group	Quachvu Ngoc-Chang
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6 Project Results

6.1 Work Package 1 – Performance Criteria

Military specification, MIL-PRF-8625F w/Amendment 2 – ANODIC COATINGS FOR ALUMINUM AND ALUMINUM ALLOYS, Types I and II, serve as the basis for establishing performance requirements for anodize types and classes, and as a benchmark for comparison with Original Equipment Manufacturer (OEM) performance requirements, which may be further defined by their particular customer base.

Anodize processes employ electromotive force to convert some of the base metal to a dense oxide film or coating, useful for corrosion prevention and protection against abrasion, whereas conversion coating converts some of the native metal oxide via chemical reaction to a more protective corrosion resistant coating with little abrasion resistance.

Detail parts progress from fabrication to finishing by a variety of manufacturing processes, which may include one or more of the following: forming, heat-treatment & aging, chemical milling, or machining. Before execution of anodizing, oils, soils and heat scale must be removed, and if non-destructive testing is required, smeared metals must also be removed. Figure 2 provides a general flow chart for aluminum anodizing, and for most parts, anodize is followed by application of an organic finish, (e.g., paint). MIL-PRF-8625 provides latitude for execution of the process, and Figure 2 captures a description of the entire process as practiced by project participants with options as noted.

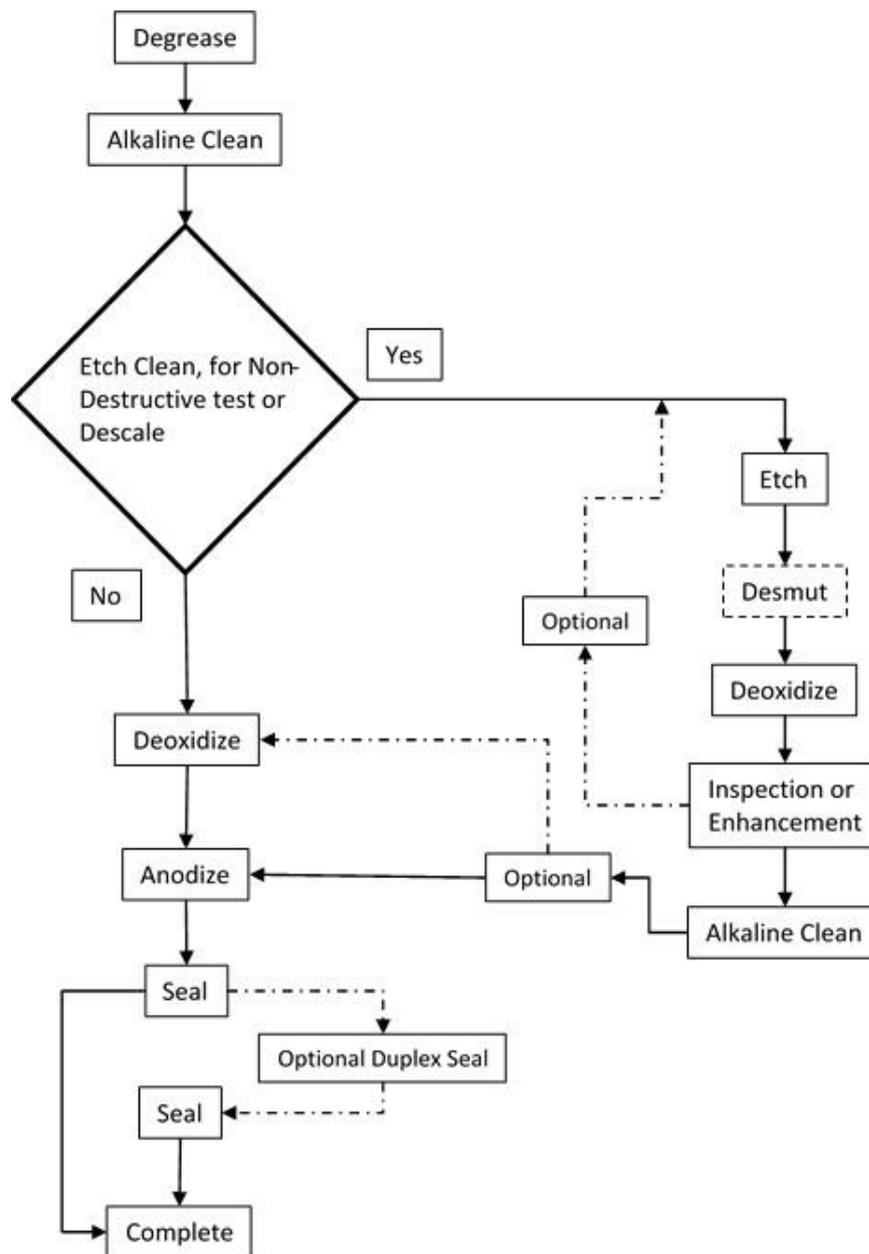


Figure 2. Anodize Process Flow

MIL-PRF-8625F defines types of anodize coatings. The name of each coating refers to the electrolyte employed for creation of the anodic film. MIL-PRF-8625 Type I and Type IB employ chromic acid in the electrolyte and all other types employ sulfuric acid with or without modifications. Anodic seal solutions employed for Class 1 anodize are generally applicable for all types. Table 1 provides a summary of coating types.

Table 1. MIL-PRF-8625 Anodize Types

Type	Description	Requirement		Comment	Design Notes		
		Film Weight			Thickness	mils	µm
		mg/cm ²	mg/ft ²				
I	Chromic Acid Anodize (CAA)	0.22	200	Minimum	0.02–0.1	0.5–2.5	
IB	Low Voltage CAA	0.22	200	Minimum	0.02–0.1	0.5–2.5	
IC	Boric/Sulfuric Acid Anodize (BSAA); Tartaric Acid Anodize (TSAA)	0.22-0.75	200-700 (FL1)	Min-Max	0.02–0.2	0.5–5.1	
II	Sulfuric Acid Anodize (SAA)	1.1	1,000	Minimum	0.07-1.0	1.8-25	
IIB	Thin-Film SAA	0.22-1.1	200-1,000	Min-Max	0.02-0.7	0.5-18	
III	Hard Coat or Hard Anodize for Wear Applications (FL2)	4.7-mg/cm ² (4,320-mg/ft ²) for every 0.001-inch of coating		Minimum	0.5-4.5	13-114	
FL1: May exceed 700-mg/ft ² if defined in procurement document. FL2: Type III is identified as a MIL-PRF-8625 anodize type, but Type III is not included in this study as Type III is typically not sealed.							

MIL-PRF-8625 defines anodize class as follows, (definitions from MIL-PRF-8625 are not verbatim):

- Class 1:
 - Sealed anodize for best corrosion resistance performance and suitable as foundation for an organic finish, (paint)
 - When Class 1 is specified, sealing shall be accomplished by immersion in a sealing medium such as 5% aqueous solution of sodium or potassium dichromate, (pH 5.0 to 6.0), for 15-minutes at 90°C to 100°C, (194°F to 212°F), in boiling deionized water, cobalt or nickel acetate, or other suitable chemical solutions. Please note the underscore of, “such as,” in the previous sentence. MIL-PRF-8625 does not require use of dichromate sealing media and only references dichromate sealing media while allowing use of alternate sealing media.
- Class 2:
 - Dyed anodize, (for uniform colour saturation, Type II anodize is typically selected for Class 2 applications).
 - When Class 2 is specified, sealing shall be accomplished after dyeing by immersion in a sealing medium, such as a hot aqueous solution containing 0.5% nickel or cobalt acetate, (pH 5.5 to 5.8), boiling deionized water, duplex sealing with hot aqueous solutions of nickel acetate and sodium dichromate or other suitable chemical solutions.
- Type III Anodize, for maximum wear resistance, is not sealed. Type III anodize may be sealed if requested by the customer, but wear resistance will be diminished.

Anodic seal solutions, commonly used in aluminum finishing and commonly defined in original equipment manufacturer’s process specifications, and typical process control requirements are found in Table 2:

Containing Hexavalent Chromium:

- Sodium or potassium dichromate, 5% by weight, (process temperature 90°C to 100°C)

- Dilute Chrome, 40 to 100-ppm as Cr⁺⁶, (90°C to 100°C)

Free of Hexavalent Chromium

- Hot deionized water, (90°C to 100°C)
- Nickel Acetate, (90°C to 100°C)
- Permanganate, (e.g., SanChem Safeguard™ CC-5000), (90°C to 100°C)
- Magnesium and Nickel Fluoride, (~60°C to 70°C, and typically used to seal Type III anodize)

Seals for consideration – Containing Trivalent Chromium

- Trivalent chromium conversion coating, approved in accordance with MIL-DTL-81706B – CHEMICAL CONVERSION MATERIALS FOR COATING ALUMINUM AND ALUMINUM ALLOYS, (21°C to 41°C).
- Trivalent chromium conversion coating, approved in accordance with MIL-DTL-81706B – CHEMICAL CONVERSION MATERIALS FOR COATING ALUMINUM AND ALUMINUM ALLOYS, (60°C to 70°C).
- Proprietary trivalent chromium coatings or coating systems used in accordance with the manufacturer’s recommendations, for those original equipment manufacturers fabricating hardware untethered to military specifications.

Table 2. MIL-PRF-8625 Process Control Requirements

Class	Corrosion Resistance	Paint Adhesion
Class 1	336-hours exposure to neutral salt fog; total 150-in ² exposure, fewer than 15-pits; fewer than 5-pits/panel (FL1) (FL2)	Wet Scribe Paint Adhesion; No loss of organic finish along scribe line, pass equals coating loss ≤ 0.8-mm beyond scribe line (FL3)
Class 2	Corrosion resistance performance equivalent to Class 1	N/A (FL4)

FL1: Corrosion pit defined in accordance with MIL-DTL-5541F: pit or spot exhibiting white corrosion product and a discernable tail.
 FL2: MIL-A-8625F and now MIL-PRF-8625F add requirements for corrosion resistance performance: dark or stained areas of corrosion resistance panels must be examined at 10X magnification and any pits noted must be added to total number of pits found elsewhere on the panel.
 FL3: Reference test method in accordance with FED-DTD-141, #6301 or ASTM D 3359
 FL4: Light Fastness Resistance not required, unless specified as a requirement in procurement documents. Reference test methods in accordance with ASTM G 23, ASTM D 822 and ASTM G 26.

Class 5 Seal Designation: MIL-PRF-8625 includes no-seal instructions for Type III anodize only; however, some original equipment manufacturers include a no-seal, or Class 5, designation for their internal process specification(s). A common requirement for no-seal anodize is primer application, within time limits, 100% of surface area. Reference to a Class 5 seal condition is included as, “no seal,” with a required organic finish is a no-chrome seal option; however, 100% organic finish is required.

Of the non-hexavalent chromium seals identified, trivalent chromium conversion coatings are commercially available from a variety of suppliers; most are approved as Type II conversion Coatings in accordance with MIL-DTL-81706B and provide the greatest potential as drop-in replacements for seals containing hexavalent chromium.

6.2 Work Package 2 – Test Results

Results of performance testing provided by project participants are collected in table form. Table 3 captures corrosion resistance performance data developed by original equipment manufacturers for specific applications. Table 4 captures corrosion resistance performance data observed for low temperature trivalent chrome conversion coating used as anodic seal for Type II anodize. Table 5 captures performance data observed for organic finish applied over Type IIB anodize with trivalent chrome conversion coating as anodic seal.

Table 3. OEM Corrosion Resistance Performance Requirements, Unpainted

MIL-PRF-8625 Finish	Substrate (FL1)	Neutral Salt Fog Hours @ Failure for Sealing Media					Requirement, NSF Exposure Hours (FL5)
		(FL2)			TCP	Duplex	
		Hot Water	Dilute Chrome	Dichromate			
Type I	2024-T3	700	700				336
	7075-T6	1800	1800				
	7050-T7451 (machined plate)	400	400				
Type IC, (TSAA)	2024-T3	336					336, 504 and 750 depending on application.
Type IC, (TSAA)	2024-T3					408 (FL3)	336
Type IC, (TSAA)	2024-T3					> 1,100 (FL4)	336
Type II	7050-T7451 (machined plate)		620		1,000+		336
Type IIB	2024-T3			1,200	1,400		336
	7075-T6			1,600	1,200		
	7050-T7451 (machined plate)		450	800	850		
Type IIB	2024-T3					> 2,000 (FL3)	336
	7075-T6					> 2,000	
Type IIB	2024-T3				750		> 500
	5086 H111				750		
	6061-T6				750		
	7075-T7351				750		

FL 1: All bare substrate material, no clad.
 FL 2: Hot water, Dilute Chrome and Dichromate Seal Temperatures 85°C to 94°C; TCP seal temperature 22°C minimum; seal immersion times in accordance with OEM requirements.
 FL 3: Duplex Sealing consists of ambient temperature trivalent chrome pre-treatment, (TCP), followed by hot deionized water, 74°C minimum temperature. Immersion times not provided.
 FL 4: Duplex sealing consists of sealing in a coating system, (TCP treatment and supplementary coating), and hot deionized water seal.
 FL 5: Neutral Salt Fog Testing, controlled in accordance with ASTM B 117. Test specimens exposed until failure occurs, i.e., number of pits observed is greater than number of pits allowed.

Table 4. Corrosion Resistance Performance, 2024-T3 Bare, Sulfuric Acid Anodize

Substrate: 2024-T3 Bare				Low Concentration TCP, 22°C, 3 to 5-min Immersion (FL1)			
Film Weight		Thickness		Neutral Salt Fog Hours			
mg/ft ²	mg/cm ²	mils	µm	336	504	672	1008+
200	0.22	0.02	0.51	Fail	Fail	Fail	Fail
250	0.27	0.025	0.64	Fail	Fail	Fail	Fail
300	0.32	0.034	0.86	Pass	Fail	Fail	Fail
351	0.38	0.049	1.24	Pass	Fail	Fail	Fail
400	0.43	0.051	1.30	Pass	Fail	Fail	Fail
500	0.54	0.072	1.83	Pass	Pass	Fail	Fail
616	0.66	0.107	2.72	Pass	Pass	Pass	Fail
687	0.74	0.129	3.28	Pass	Pass	Pass	Fail
690	0.74	0.126	3.20	Pass	Pass	Pass	Pass
817	0.88	0.159	4.04	Pass	Pass	Pass	Pass
1019	1.10	0.225	5.72	Pass	Pass	Pass	Pass
1023	1.10	0.218	5.54	Pass	Pass	Pass	Pass
1083	1.17	0.236	5.99	Pass	Pass	Pass	Pass
1903	2.05	0.52	13.21	Pass	Pass	Pass	Pass
	Approximate minimum film weight for MIL-PRF-8625, Type IIB						
	Approximate film weight range for MIL-PRF-8625, Type II						
Pass	Pass						
Fail	Marginal Pass						
Fail	Fail						
FL1: TCP seal solution performance data from one TCP product found on QPL-81706B.							

Table 5. Organic Finish Flexibility Testing

Test	Finish	Substrate	Organic Finish	Pass/Fail	Comment
Scribed Corrosion	Type IIB w/TCP Seal	7075-T6 Bare	MIL-PRF-85582, Type I, Class 2	Pass	3,000-hour neutral salt fog exposure; no sign of blistering or lifting or corrosion 0.8-mm beyond scribe
Low Temp Flexibility	Type IIB w/TCP Seal	2024-T3 Bare	MIL-PRF-85582, Type I, Class 2	Pass	Soak Specimens for 5-hours @ -51°C and bend over 25-mm diameter mandrel
Room Temp Flexibility, (GE Impact, 36-inch) (FL1)	Type IIB w/TCP Seal	2024-T0 Bare	MIL-PRF-85582, Type I, Class 2	Pass	Primer shall exhibit a minimum impact elongation of 10% when examined under 10X magnification
Room Temp Flexibility, (Gardner Impact, 30-inch) (FL1)	Type IIB w/TCP Seal	7075-T6 Bare	MIL-PRF-85582, Type I, Class 2	Pass	When examined under 10X magnification any voids exposing the substrate shall be ≤ 1-mm length.
Topcoat Compatibility	Type IIB w/TCP Seal	2024-T0 Bare	MIL-PRF-85582, Type I, Class 2 & MIL-PRF-85285 Flat Gray Topcoat	Pass	Primer and Topcoat shall exhibit a minimum impact elongation of 20% when examined under 10X magnification
Intercoat Adhesion	Type IIB w/TCP Seal	7075-T6 Clad	MIL-PRF-85582, Type I, Class 2 & MIL-PRF-85285 Flat Gray Topcoat	Pass	When examined under 10X magnification any voids exposing the substrate shall be ≤ 1-mm length.
FL1: Elevation of impacter before release; GE Impact (four spherical impacters, each representing percent elongation); Gardner impacter is a single 16-mm diameter spherical impacter.					

6.3 Work Package 3 – GAP Analysis; Additional Test Data

Project participants highlighted two performance attributes for which additional test data is preferred:

- Use of duplex sealing, i.e., TCP followed by hot water seal, provides exceptional corrosion resistance performance in neutral salt fog, but data on organic finish adhesion needed;
- Impact testing – scribed corrosion test data provided, but original equipment manufacturers do not intentionally field products with perfectly scribed groves to demonstrate, “healing,” power of hexavalent chromium; however, products may be impacted and possibly dented by shifting cargo; therefore, need data demonstrating that organic finish adheres to TCP sealed anodize when parts are subjected to impact.

- Table 4 implies a 3 to 5-minute immersion in ambient, (> 22°C), temperature TCP is sufficient for MIL-PRF-8625 neutral salt fog corrosion resistance performance requirements for anodic film weights down to 0.32-mg/cm², (300-mg/ft²). Traditional anodic seals are applied at the boiling point of water; therefore, energy savings/carbon footprint reduction estimates for ambient temperature TCP seal will be valuable for justification of anodic seal conversion from hexavalent chromium to trivalent chromium, or non-chromium.
- Brush anodize is identified by some OEMs as a method of anodize repair. Need data on sealing of brush anodize, or evaluation of the process and potential application of TCP seal.

Work Package 3 Test Data, Addressing GAP Analysis

Duplex Sealing with Primer Adhesion Data:

- Three 2024-T3 bare panels anodized and sealed, (MIL-PRF-8625, Type IIB, Class 1), (Figure 3)
- Seal consists of immersion in TCP; followed by 30-minute immersion in hot, (98°C), deionized water.
- A low VOC, (Volatile Organic Compound), coating applied and cured, (time only, or by time & heat).
- Following 24-hours soak in water, adhesion of the organic finish tested in accordance with FED-STD-141, Method 6301.3/ASTM D 3359, (Table 6)

Table 6. Wet Scribe Adhesion on Type IIB SAA with Duplex Seal

ALLOY	ANODIZE	SEAL	PRIMER	ADHESION, 24-HOUR DI WATER IMMERSION (WET)
				ASTM D 3359
2024-T3 Bare	Thin-Film Sulfuric, (MIL-PRF-8625, Type IIB)	TCP + Hot DI Water, 30-minute Immersion	Low VOC Epoxy	5B



FIGURE 2: Adhesion tests on panel 1

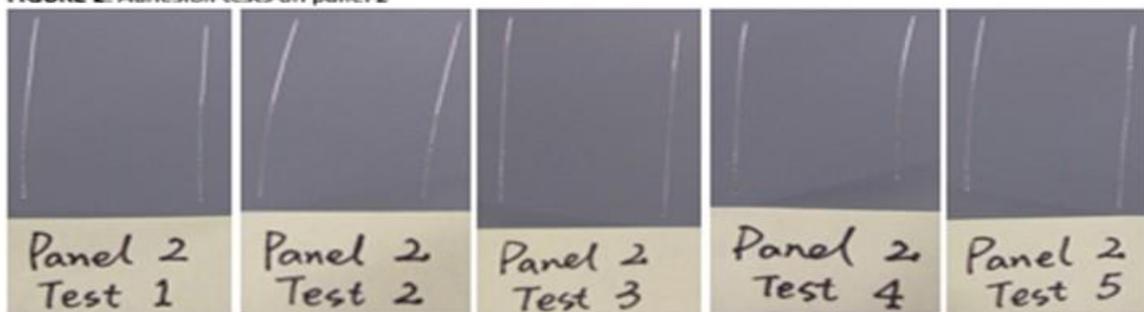


FIGURE 3: Adhesion tests on panel 2



Figure 3. Adhesion Test Panels with Duplex TCP & Hot Water Seal

System Level Testing:

- ASTM D 3359 – STANDARD TEST METHOD FOR RATING ADHESION BY TAPE TEST
- ASTM D 1654 – STANDARD TEST METHOD FOR EVALUATION OF PAINTED OR COATED SPECIMENS SUBJECTED ON CORROSIVE ENVIRONMENTS
- ASTM D 714 – STANDARD TEST METHOD FOR EVALUATING DEGREE OF BLISTERING OF PAINTS.

Tables 7 through 9 and Figures 4 through 7 summarize system level testing of anodize sealed with trivalent chromium conversion coating from two suppliers, and non-chrome primers from two

suppliers; all compared to a control coating system. Performance attributes include organic finish adhesion, scribed corrosion performance and corrosion creep beyond the scribe.

Table 7. ASTM D 3359 Scoring for Primer Adhesion

Status	Alloy	Anodize (FL1)	Seal (FL2)	Primer (FL3)	Adhesion (Dry)	Adhesion (Wet) 24-hour DI Water Immersion	Adhesion (Wet) 14-day DI Water Immersion
					ASTMD3359	ASTMD3359 ISO2812	ASTMD3359 ISO2812
					Method B Score (FL4)	Method B Score	Method B Score
Control	2024	TFSAA	DiCr	C	5B	4B	4B
Control	7075	TFSAA	DiCr	C	5B	5B	5B
Test	2024	TFSAA	TCP1	1	5B	5B	5B
Test	7075	TFSAA	TCP1	1	4B	4B	5B
Test	2024	TFSAA	TCP1	2	5B	5B	5B
Test	7075	TFSAA	TCP1	2	4B	5B	5B
Test	2024	TFSAA	TCP1	3	4B	4B	4B
Test	7075	TFSAA	TCP1	3	5B	4B	4B
Test	2024	TFSAA	TCP2	1	4B	5B	5B
Test	7075	TFSAA	TCP2	1	4B	4B	5B
Test	2024	TFSAA	TCP2	2	4B	4B	5B
Test	7075	TFSAA	TCP2	2	5B	5B	5B
Test	2024	TFSAA	TCP2	3	4B	5B	5B
Test	7075	TFSAA	TCP2	3	5B	5B	5B

FL1: Thin-Film Sulfuric Acid Anodize, (MIL-PRF-8625 Type IIB, Class 1)
 FL2: Seal solution for anodize: DiCr = Dichromate Seal Control, TCP1 and TCP2 are trivalent chrome conversion coating from different manufacturers. Verify QPL-81706B approvals as necessary as not all TCP coatings have gained MIL-DTL-81706B approval.
 FL3: Organic coatings (primer) are: C – Control, BMS10-11, Type 1, Class A, Grade E; Product 1 – MIL-PRF-23377, Type 1, Class N; Product 2 – MIL-PRF-8852, Type I, Class N; Product 3 - MIL-PRF-23377, Type 1, Class N
 FL4: Minimum score for test, 4B.

Table 8. ASTM D 1654 Scoring for Primer, Scribed Corrosion Resistance Performance

Status	Alloy	Anodize (FL1)	Seal (FL2)	Primer (FL3)	ASTM D 1654 (FL4)
Control	2024	TFSAA	DiCr	C	10
Control	7075	TFSAA	DiCr	C	10
Test	2024	TFSAA	TCP2	3	8
Test	7075	TFSAA	TCP2	3	10
Test	2024	TFSAA	TCP1	3	7
Test	7075	TFSAA	TCP1	3	10
Test	2024	TFSAA	TCP2	1	7
Test	7075	TFSAA	TCP2	1	8
Test	2024	TFSAA	TCP2	2	6
Test	7075	TFSAA	TCP2	2	10
Test	2024	TFSAA	TCP1	2	4
Test	7075	TFSAA	TCP1	2	8
Test	2024	TFSAA	TCP1	1	7
Test	7075	TFSAA	TCP1	1	7

FL1: Thin-Film Sulfuric Acid Anodize, (TFSAA), MIL-PRF-8625 Type IIB, Class 1
FL2: Seal solution for anodize: DiCr = Dichromate Seal Control, TCP1 and TCP2 are trivalent chrome conversion coating from different manufacturers. Verify QPL-81706B approvals as necessary as not all TCP coatings have gained MIL-DTL-81706B approval.
FL3: Organic coatings (primer) are: C – Control, BMS10-11, Type 1, Class A, Grade E; Product 1 – MIL-PRF-23377, Type 1, Class N; Product 2 – MIL-PRF-8852, Type I, Class N; Product 3 - MIL-PRF-23377, Type 1, Class N
FL4: Refer to Table 9 – Rating for corrosion creep beneath the scribe; 8-Rating required for passing score.

Table 9. ASTM D 1654 Rating of Failure at Scribe, (for Procedure A)

Representative Mean Creep from Scribe		
Millimetres	Inches, (Approximate)	Rating Number
0.0	0.0	10
0.0 to 0.5	0.0 to 0.016	9
0.5 to 1.0	0.016 to 0.031	8
1.0 to 2.0	0.031 to 0.063	7
2.0 to 3.0	0.063 to 0.125	6
3.0 to 5.0	0.125 to 0.188	5
5.0 to 7.0	0.188 to 0.250	4
7.0 to 10.0	0.250 to 0.375	3
10.0 to 13.0	0.375 to 0.500	2
13.0 to 16.0	0.500 to 0.625	1
16.0 to more	0.625 to more	0

Minimum passing score

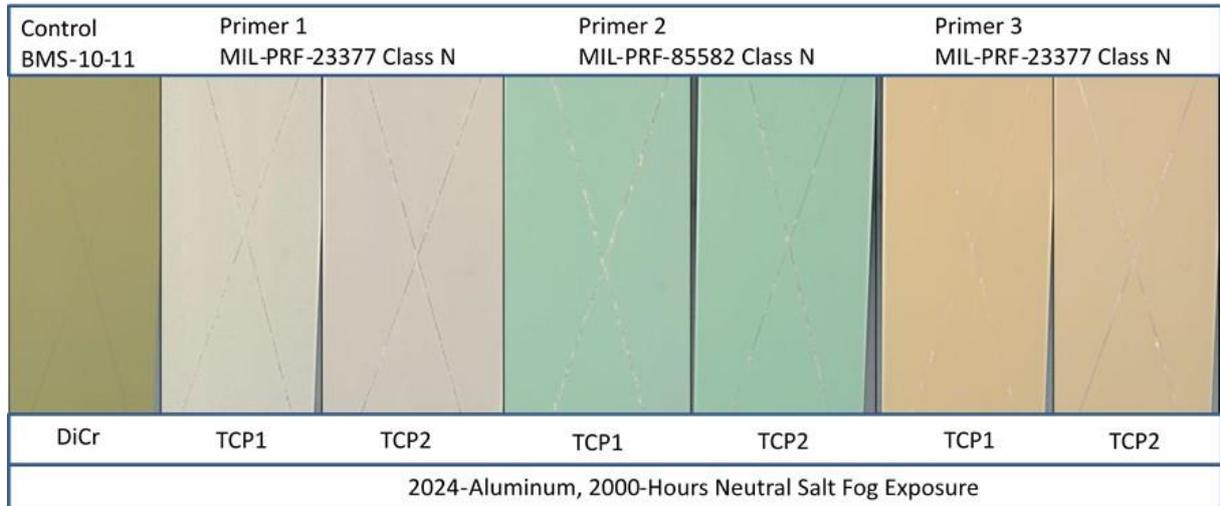


Figure 4. Scribed Corrosion Resistance, Type IIB, TFSAA, ASTM B 117 Controlled Neutral Salt Fog, (NSF)

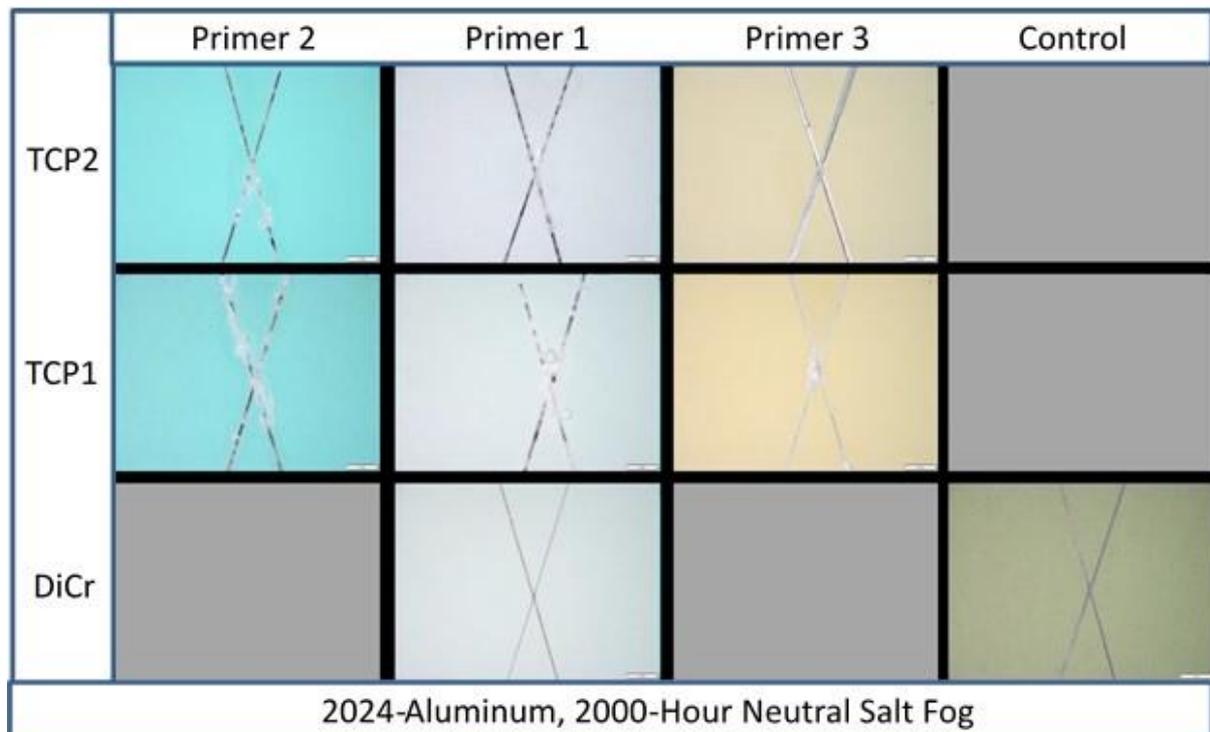


Figure 5. Scribed Corrosion Resistance, Corrosion Product in Scribe, Type IIB, 2000-Hours NSF

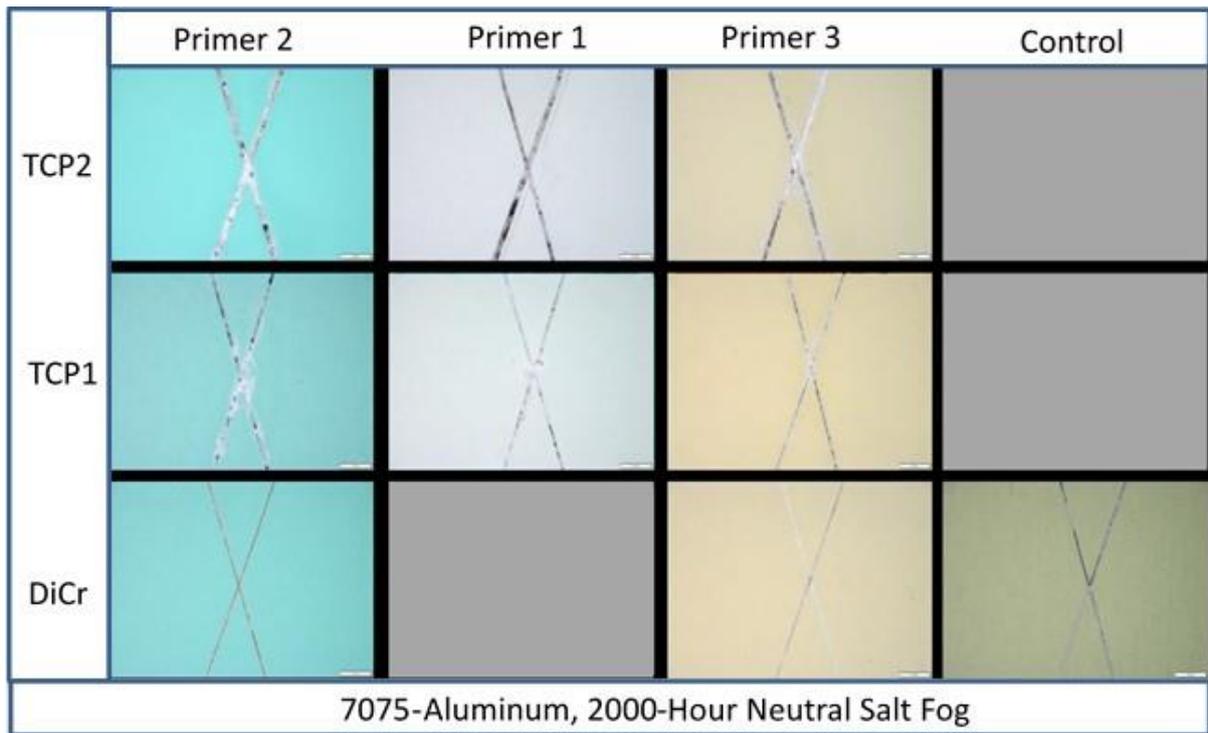


Figure 6. Scribed Corrosion Resistance, Corrosion Product in Scribe, Type IIB, 2000-Hours NSF

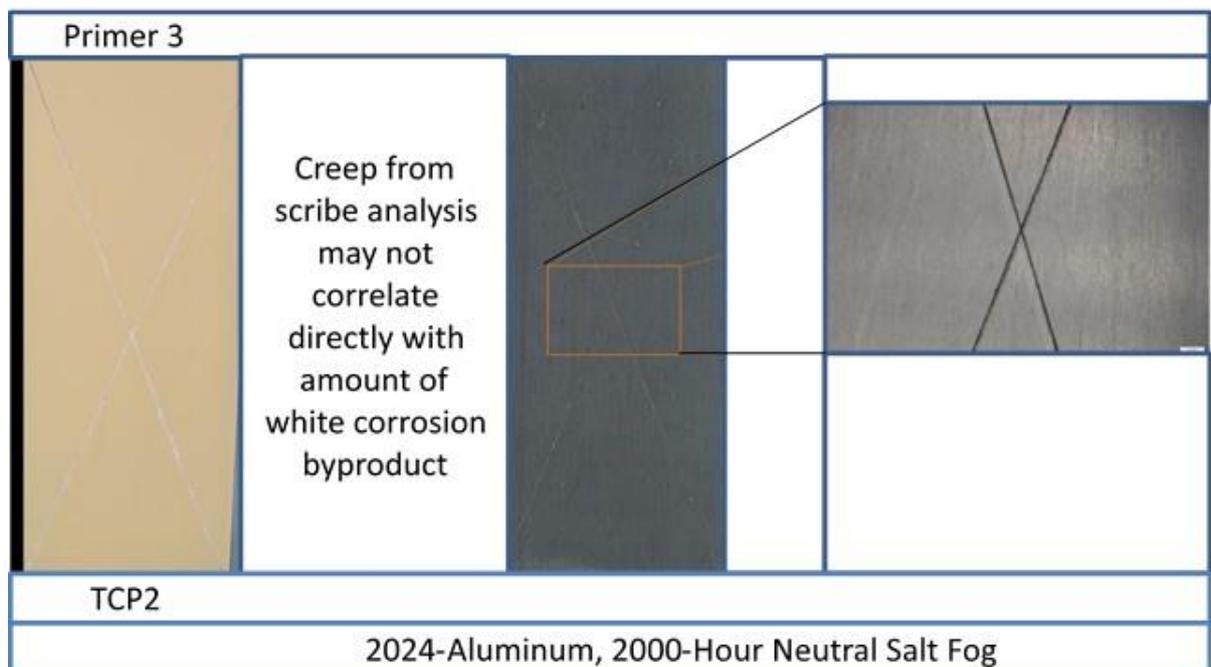


Figure 7. Evaluation of Corrosion Creep Beyond Scribe, (Primer Removed), Type IIB, 2000-Hours NSF

Figures 4, 5 & 6 depict white corrosion product in scribe lines at 2,000-hours neutral salt fog exposure and Figure 7 depicts scribes lines following removal of the organic finish and reveals no corrosion creep beyond the scribe. Figure 8 depicts test panels with primer coating removed following 3,000-hours neutral salt fog exposure. Refer to Table 9, ASTM D 1654 Rating of Failure at Scribe.

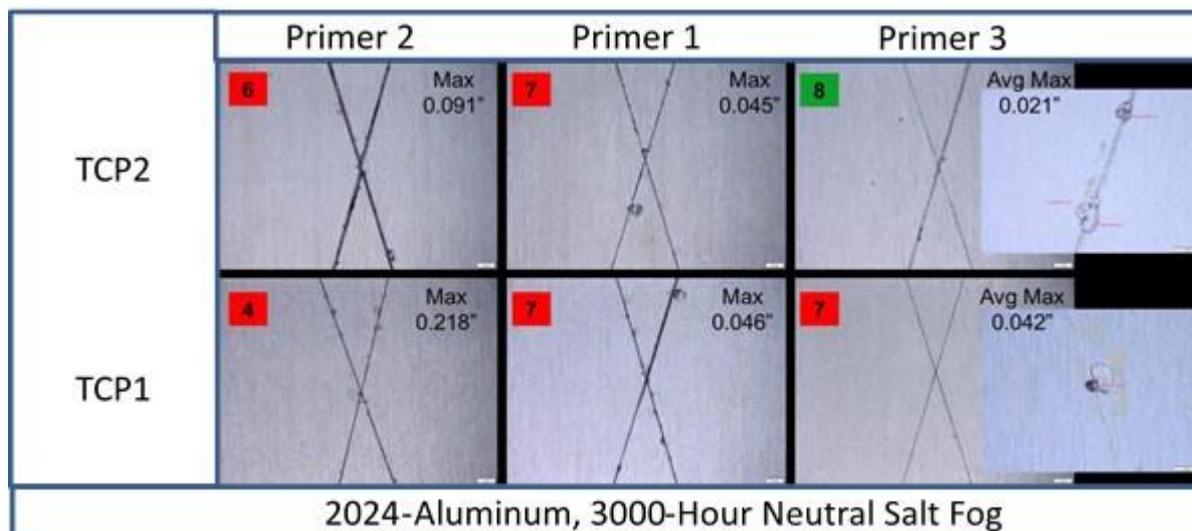


Figure 8. Evaluation of Corrosion Creep Beyond Scribe, Type IIB, 3000-Hours NSF

Table 10 outlines coating system build up and scribed corrosion testing for Tartaric-Sulfuric Acid Anodize, Duplex sealing employing competing TCP coating systems, a TCP conversion coating and epoxy and fuel tank primer organic finishes. TCP coating systems consist of two coatings, the first is a trivalent chromium solution used to form a protective layer, which is then reinforced in a second coating process. Figures 9 & 10 depict scribed corrosion results following 3,000-hours exposure in neutral salt fog.

Table 10. Type IC, (TSAA), System Testing, Coating Build-Up for Scribed Corrosion Testing, (FL1)

Inorganic Finish		Organic Finish
FINISH	SEAL	PRIMER
Control: Type I, CAA	Dichromate	Epoxy, Non-Chromated AMS C 27725 Fuel Tank
Type IC, TSAA	Duplex, TCP Coating System 1 w/Hot DI Water	Epoxy, Non-Chromate AMS C 27725 Fuel Tank
Type IC, TSAA	Duplex, TCP Coating System 1 w/Hot DI Water	Epoxy, Chromated AMS C 27725 Fuel Tank
Type IC, TSAA	Duplex, TCP followed by Hot DI Water	AMS C 27725 Fuel Tank
Type IC, TSAA	Duplex, TCP Coating System 2 w/Hot DI Water	AMS C 27725 Fuel Tank

FL 1: Neutral Salt Fog Exposure, 3,000-hours controlled in accordance with ASTM B 117.

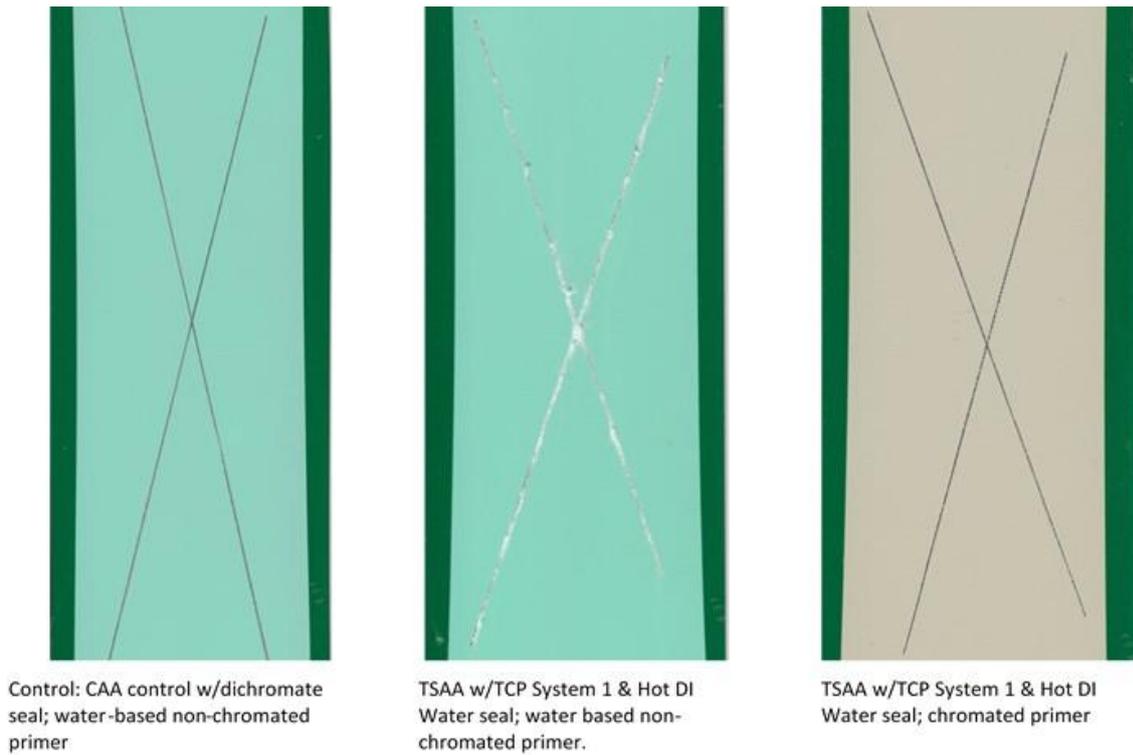


Figure 9. Scribed Corrosion Test Results, Epoxy Primer

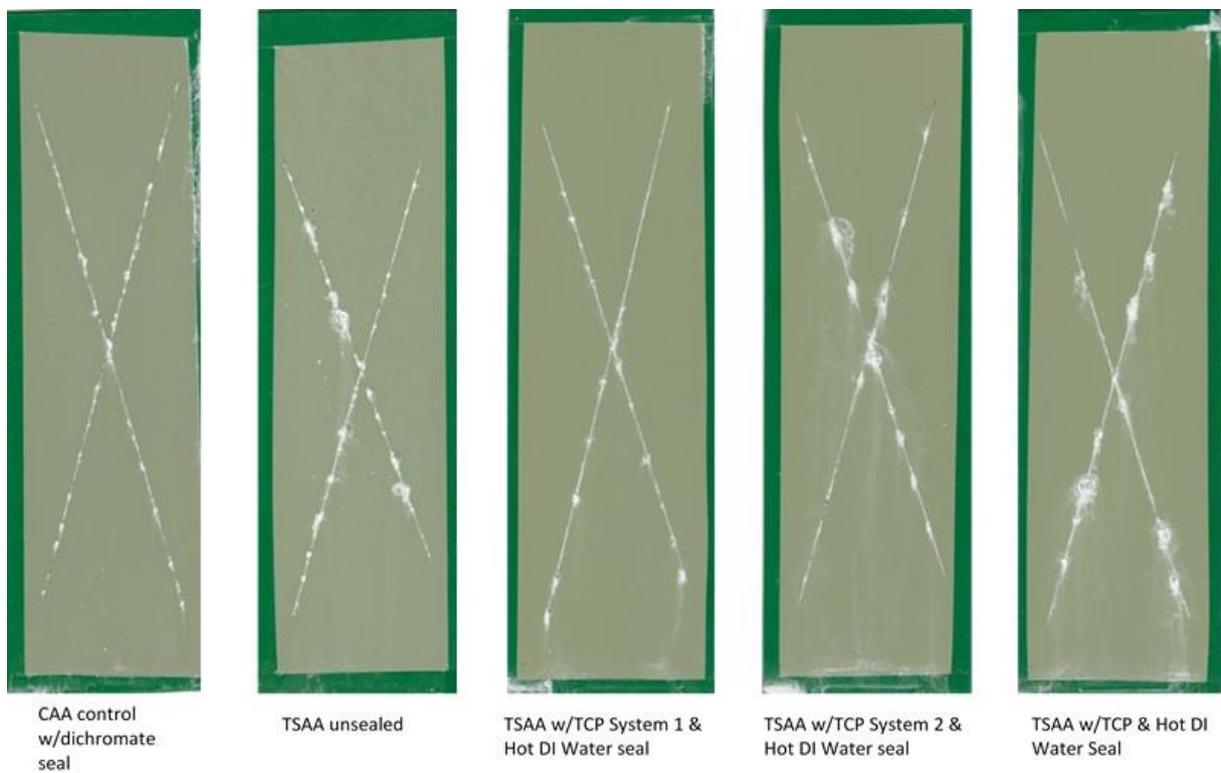


Figure 10. Scribed Corrosion Testing, AMS C 27725 Fuel Tank Coating

Flexibility Testing of Inorganic/Organic Finish Systems by Impact Testing:

- ASTM D 6905 – STANDARD TEST METHOD FOR IMPACT FLEXIBILITY OF ORGANIC COATINGS.
- Place specimen on rubber anvil coating side down
- Raise anvil to the height required to produce a slight imprint of the indenter’s outside diameter in the test specimen,
 - Indenter type is the GE Flexibility Tester
- Report:
 - Maximum percent elongation for flexibility of the finish
 - Height, or elevation of the indenter
 - Thickness of the organic finish
 - Substrate thickness and type of metal
 - Method of panel preparation
 - Atmospheric conditions.

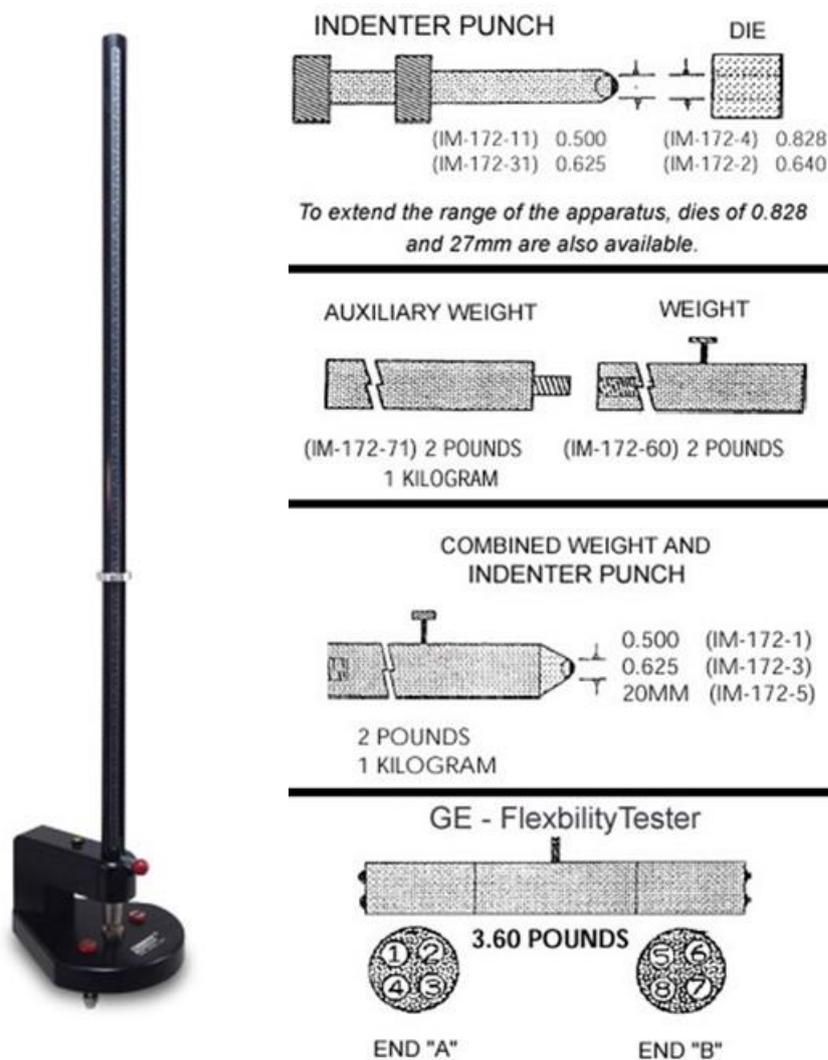


Figure 11. ASTM D 6905 Test Apparatus

Table 11. ASTM D 6905 Scoring Parameters

Indenter Drop, mm, (inch)	Spherical Segment	End	Base Diameter, mm, (inch)	Segment Radius, mm, (inch)	Segment Elevation, mm, (inch)	%Area Increase	Scoring
914-mm (36-inch)	1	A	9.5, (0.375)	4.85 (0.194)	3.65 (0.146)	60	Better ↑
	2	A	9.5, (0.375)	5.2 (0.208)	2.96 (0.119)	40	
	3	A	9.5, (0.375)	6.3 (0.252)	2.10 (0.084)	20	
	4	A	9.5, (0.375)	8.15 (0.326)	1.48 (0.059)	10	Minimum Elongation, by Specification
145-mm (6-inch)	5	B	9.5, (0.375)	11.0 (0.440)	1.05 (0.042)	5	Failure ↓
	6	B	9.5, (0.375)	16.9 (0.676)	0.66 (0.027)	2	
	7	B	9.5, (0.375)	23.7 (0.947)	0.48 (0.019)	1	
	8	B	9.5, (0.375)	33.3 (1.332)	0.32 (0.013)	0.5	

Coating Flexibility Test Plan:

Table 12. Test Plan for Organic Finish Flexibility

Process Factors		Levels		
		(-)	(+)	(+)'
A	Anodize Seal Time	< 5-min (-1)	> 10-min (+1)	
B	Anodize Time	< 3-min (-1)	> 10-min (+1)	
C	Anodize Voltage	< 15-VDC (-1)	> 18-VDC (+1)	
D	Anodize Seal Type	5% Dichromate @ 93°C	5-Vol% TCP @ 23°C (FL1)	25-Vol% TCP @ 23°C
E	Primer	BMS10-11 (FL2)	AMS3144 (FL3)	
FL1: TCP product selected from QPL-81706B FL2: Spray applied primer; dry film thickness 18 to 43-µm, (Ave. ~31-µm) FL3: Electrocoat primer; dry film thickness 13 to 18-µm.				

Flexibility testing pertains to organic finish; however, the test format also highlights process factors that improve or degrade organic finish flexibility; regarding anodize sealing, the point of interest is whether the choice of anodic seal influences organic finish flexibility.

As highlighted in Table 12 notes, dry film thickness for spray applied primers exhibit greater variance, whereas electrocoat primer thickness exhibits little dry film thickness variance. Application of

electrocoat primers to anodized aluminum is/was thought to be impractical as the anodic film is non-conductive, and that assumption is correct, but not until anodic film thickness exceeds 1.1-mg/cm², (~1,000-mg/ft²).

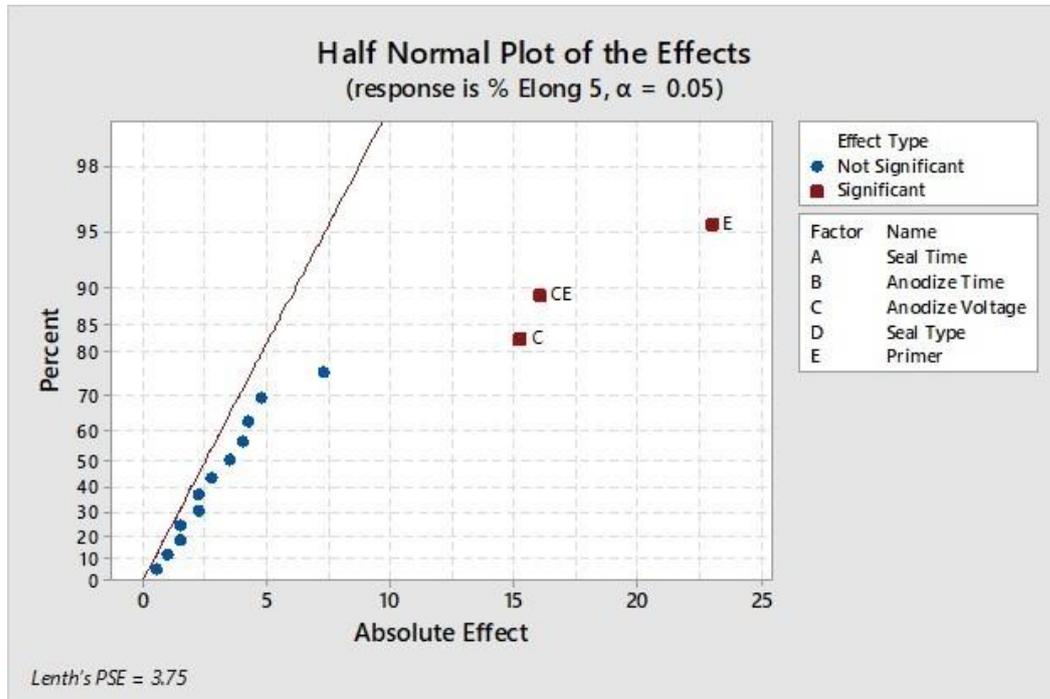


Figure 12. 1/2-Normal Probability Plot of Organic Finish Flexibility Data, 5-Vol% TCP

The probability plot in Figure 12 highlights Primer Type, Anodize Voltage and interaction between primer and anodize voltage as statistically significant in primer flexibility results. The relation is demonstrated again in Figure 13, Main Effects Plot.

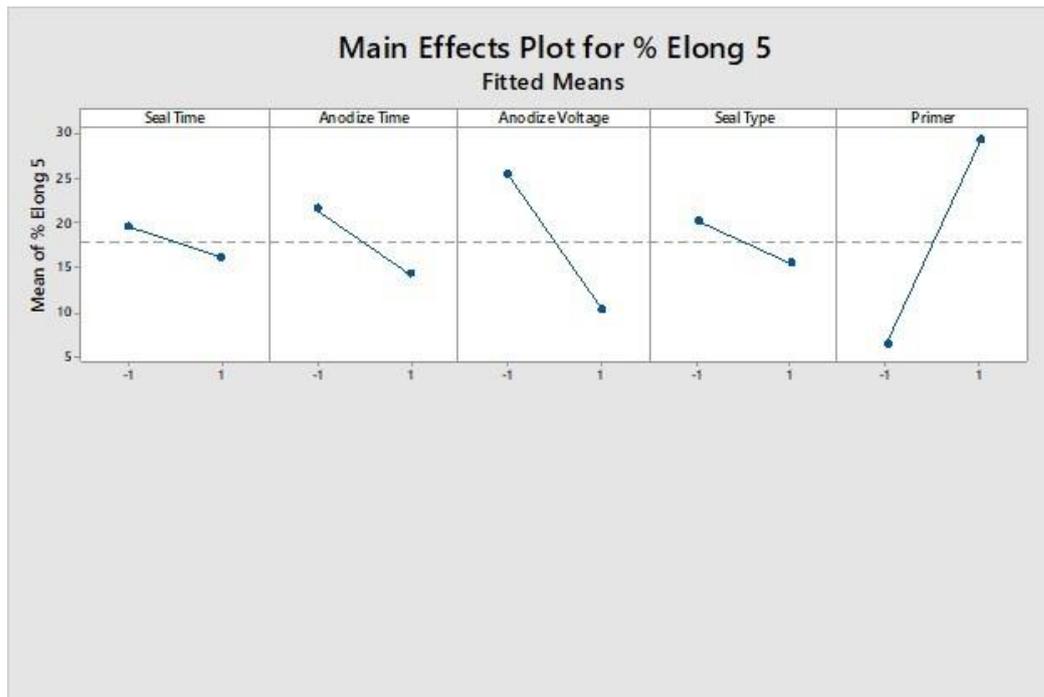


Figure 13. Main Effect Plot, Organic Finish Flexibility, 5-Vol% TCP

Seal Type, 5% Dichromate or 5-Vol% of TCP is statistically insignificant, or may be stated as 5-Vol% TCP provides the same level of performance as 5% Dichromate.

Figure 14, Process Factor Interactions demonstrate that seal type selection does not produce any first order interactions with other process factors beneficial or detrimental to organic finish coating flexibility. Coating flexibility was measured by the test method defined in ASTM D 6905.

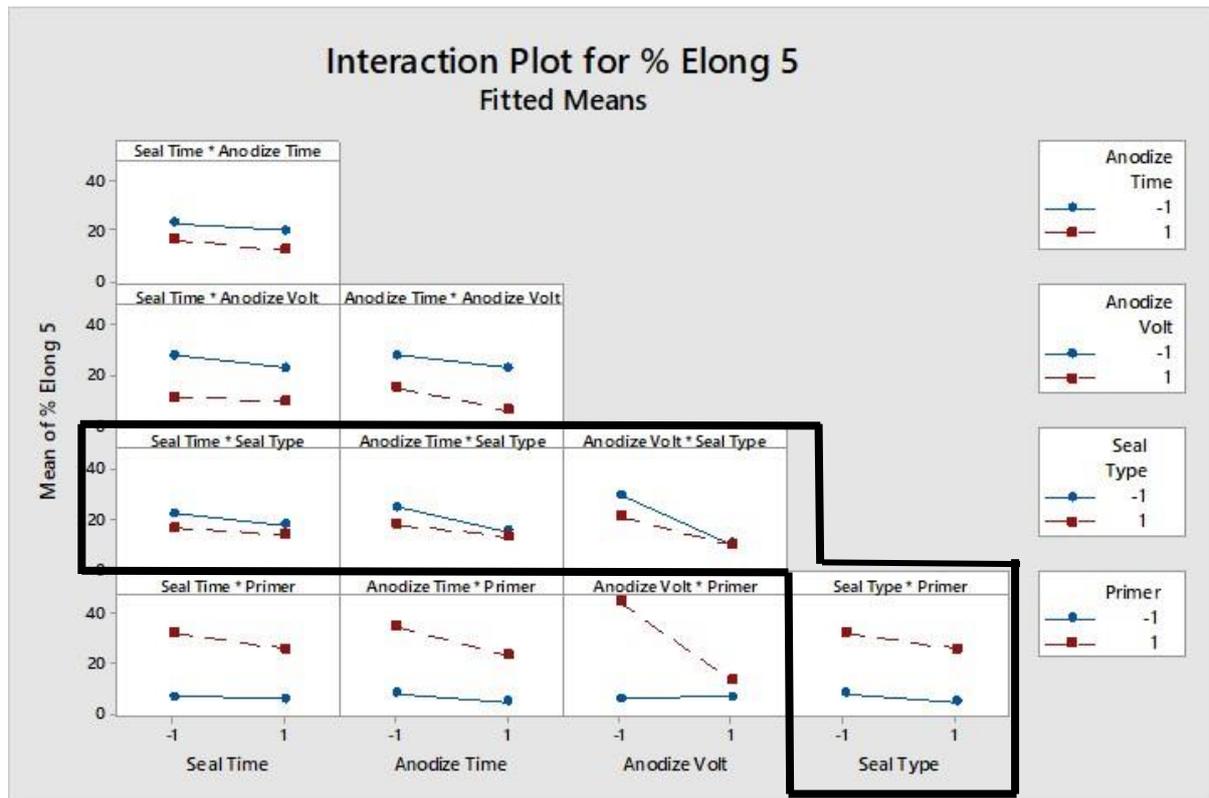


Figure 14. Process Factor Interactions, Organic Finish Flexibility, 5-Vol% TCP

Increasing TCP concentration to 25-Vol%:

Increasing concentration of TCP in the anodic seal to 25-Vol% produces essentially the same results relative to organic finish flexibility, as demonstrated in Figures 15 through 17. Experience with TCP solutions used as conversion coatings and anodic seals includes the observation that TCP coatings leave a deposit on the surface, similar to powdering exhibited by traditional chromated conversion coatings, but with lower impact on organic finish adhesion, unless the powdering becomes excessive. The quantity of powder observed is possibly dependent upon the following process factors:

- pH of the TCP solution
- Immersion time in the TCP solution
- Concentration of the TCP solution
- Filtering of the TCP solution
- Temperature of the TCP solution
- Manufacturer of the TCP solution, and particular modifications, e.g., for colour, corrosion resistance performance, etc.

A general heuristic relative to use of TCP solutions for conversion coating and anodic seal is less is better, i.e., use the lowest concentration for the least time and lowest temperature to meet performance requirements, and filter the seal solution with filter media capable of 10-µm or less.

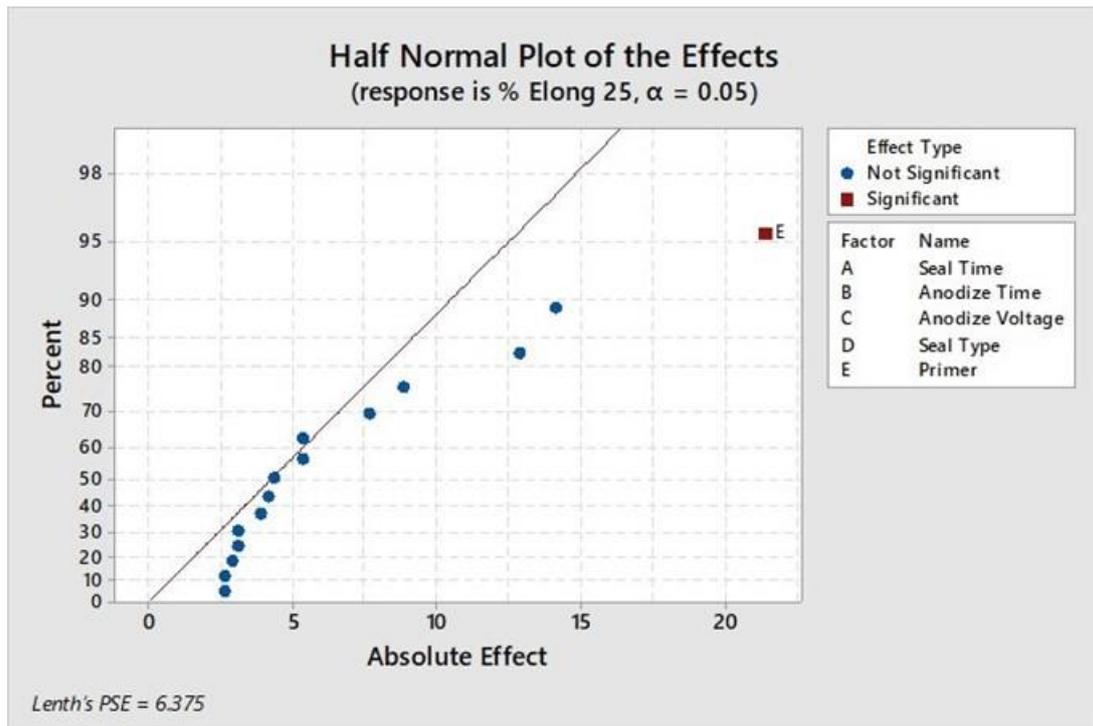


Figure 15. 1/2-Normal Probability Plot, Organic Finish Flexibility, 25-Vol% TCP

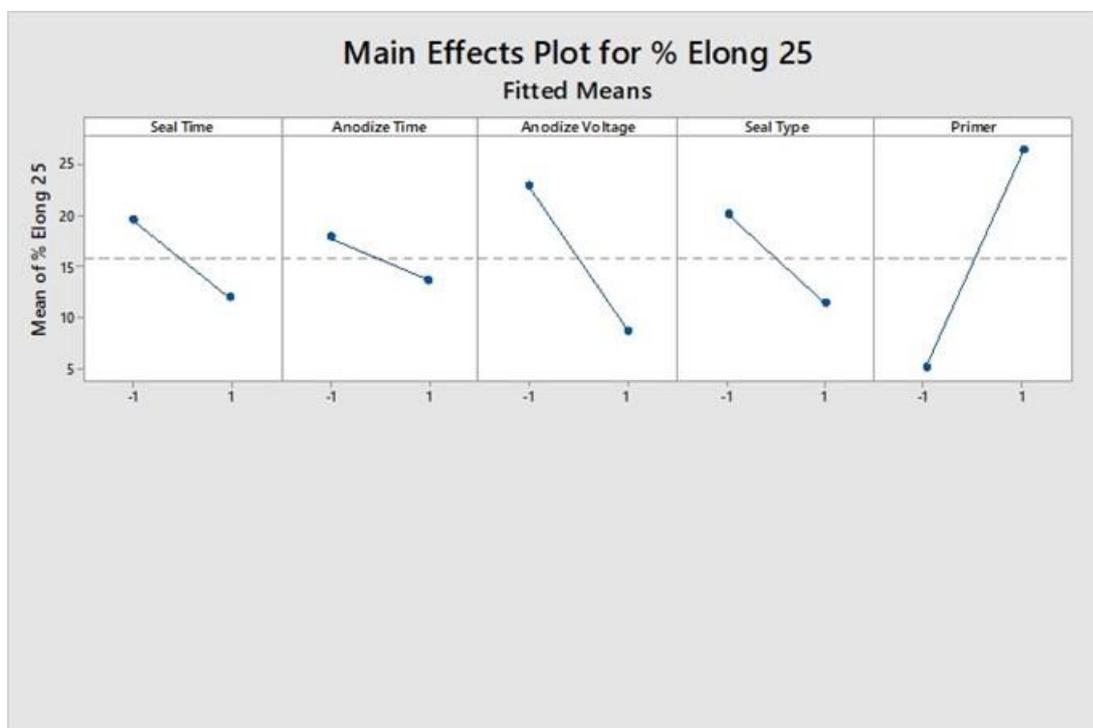


Figure 16. Main Effects Plot, Organic Finish Flexibility, 25-Vol% TCP

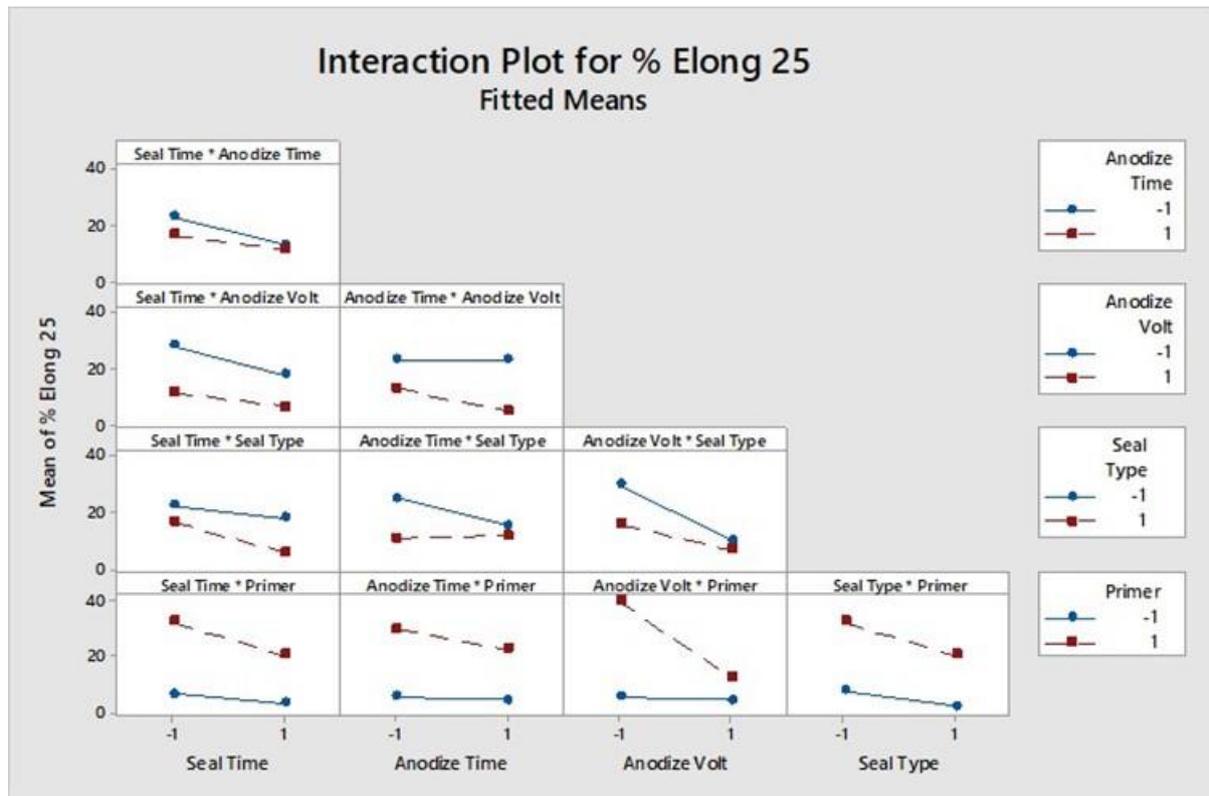


Figure 17. Process Factor Interaction, Organic Finish Flexibility, 25-Vol% TCP

The test format employed allows one additional comparison of TCP performance and that is a statistical comparison of organic finish flexibility for BMS10-11 and AMS3144 primers applied over anodize coatings sealed with TCP at 5% and 25-Vol% concentrations.

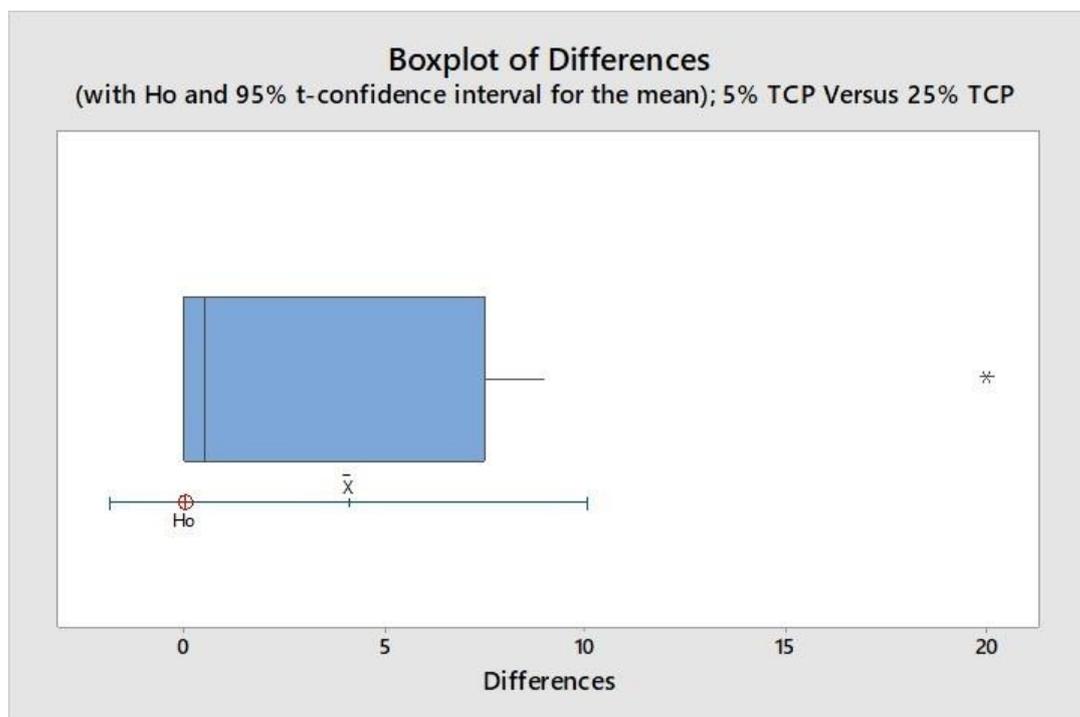


Figure 18. Boxplot of Differences, %Elongation of Primer for 5% versus 25% TCP

Table 13. Paired-T Test and Confidence Interval, Primer %Elongation, 5-Vol% versus 25-Vol% TCP

Seal	N	Mean	Std. Deviation	Std. Error Mean
5-Vol% TCP	8	15.5	16.2	5.73
25-Vol% TCP	8	11.38	14.14	5.00
Difference	8	4.13	7.12	2.52
95% Confidence Interval for mean difference: (-1.83, 10.08)				
T-Test of mean difference = 0 (Versus ≠ 0): T-Value = 1.64; P-Value = 0.145				

Interpretation: Zero (0) falls within the confidence interval and the P-Value is ≥ 0.05 ; therefore, even though the mean value of % elongation of the organic finish is greater for the 5-Vol% TCP solution, no statistically significant difference in performance has been measured, (refer to Table 13 & Figure 18).

Results: Flexibility test data provided demonstrates that TCP, when used as anodic seal, does not influence organic finish coating flexibility; therefore, works as well as traditional chromated seals.

Carbon Footprint Reduction:

Project participants provided data demonstrating anodize sealed with trivalent chrome conversion coating used at elevated temperature, or used in combinations with hot water sealing, will meet customer driven requirements for corrosion resistance performance, (reference Table 3); when used at ambient temperature, (ambient temperature $\geq 23^{\circ}\text{C}$), trivalent chrome conversion coating provides Class 1 corrosion resistance performance compliant with requirements found in MIL-PRF-8625, (Reference Table 4).

Practitioners of the art currently use seal solutions containing hexavalent chromium employed at temperatures near 100°C for 15 to 25-minutes; therefore, converting to a low temperature seal will garner a reduction in carbon dioxide emissions, evaporation losses and ventilation requirements.

Several models exist for estimation of energy requirements for an anodic seal tank, including two that are available in the public domain, (internet), and two others were developed from heat transfer text book examples and data tables. All such models are of marginal value without a physical example that provides a reference point. Fortunately, the project was able to obtain seal tank dimensions and boiler operating data for use as a reference for model based estimates.

The anodic seal tank used for reference data is located in the United States of America, in the Mid-West:

- Anodic Seal Tank dimensions: 12.8 Long X 1.2 Wide X 2.7-metres Deep; Solution volume is 43,000-litres.
- Minimum operating temperature is 93°C
- Anodize Seal Solution is heated by a 500-HP boiler fuelled by a renewable energy source, with 15 to 17-KW, (20 to 23HP), of power output to heat the seal solution, based on records/estimates by Plant Engineering. Operating efficiency of boiler assumed to be 35%.
- Tank construction is steel, open top, no insulation, slot-ventilation compliant with requirements developed by the Occupational Safety and Health Administration, (OSHA).
- Operating assumptions: (2) 8-hour work-shifts/day, or 80-hours/week; 51-weeks/year

- Ambient temperature in seal tank area: due to adjacent heated tanks used for other metal finishes, immediate area ambient temperature range is 23°C to 32°C.

Table 14. Carbon Footprint Reduction Facilitated by TCP Anodic Seal versus Hot Seal

CO ₂ Reduction, 23°C Seal Versus 93°C Seal					
	Reference	Model A		Model B	
Power Requirement	17-KW	19-KW		24-KW	
CO ₂ Reduction, 23°C Seal Versus 93°C Seal, Kg-CO ₂ /Year					
Fuel Source		Model A	Model 1A	Model B	Model C
Coal		326,000	216,000	379,000	405,000
Natural Gas		189,000	125,000	220,000	234,000
#4 Fuel Oil		264,000	175,000	308,000	328,000

CO₂ reduction estimation models:

- Model A is an estimate of energy required to heat the volume of seal solution to operating temperature in 4-hours, and maintain energy loss at the solution surface; least complicated model of all employed.
- Model 1A is available in the public domain at http://www.engineeringtoolbox.com/heat-loss-open-water-tanks-d_286.html. Assumptions are not available to the user, but estimates are similar to Model A and the reference.
- Model B is based on Model A, with added considerations for heat loss through tank sides and tank foundation. Model B may be more applicable to process line situations where the anodic seal tank is not surrounded by other warm process tanks, which was the case for the reference.
- Model C is available in the public domain at <https://www.spiraxsarco.com>. The Spirax-Sarco site includes tutorials on heating process tanks with steam coils under tabs *Learn About Steam* and *Heating with Coils and Jackets*. As with Model 1A, assumptions are not available to the user, but assumptions regarding tank ventilation velocities and air velocity around the tank, boiler operating efficiency, for example, might explain the large difference in estimates.

Project participants identified applications requiring corrosion resistance performance beyond MIL-PRF-8625 requirements, and for said applications a trivalent chromium seal heated to 66°C provides additional protection, or 1,500 to 2,000-hours of neutral salt fog exposure. A carbon footprint reduction can also be estimated for reducing seal solution temperature approximately 30°C.

Table 15. Carbon Footprint Reduction Facilitated by Hot TCP Anodic Seal versus Common Hot Seal

CO ₂ Reduction, 66°C Seal Versus 93°C Seal, Kg-CO ₂ /Year				
Fuel Source	Model A	Model 1A	Model B	Model C
Coal	130,000	215,000	132,000	343,000
Natural Gas	75,000	125,000	77,000	199,000
#4 Fuel Oil	105,000	174,000	107,000	279,000

Project participants identified applications requiring corrosion resistance performance beyond that provided by 66°C TCP, which is achieved by following the TCP seal with 93°C deionized water. In the case of duplex seals, both at elevated temperature, there is a carbon footprint penalty.

Brush anodize applications: Data for sealing brush anodize repairs was not available; however, the current process for sealing the anodic film created by brush anodizing is immerse part(s) in a sealing solution if possible, and when immersion sealing is not possible, then swab or pour room temperature sealing solution on the brush anodize area; the sealing solutions defined include dilute dichromate anodize sealing solution, or MIL-DTL-81706 Type I, Class 1A or Class 3 coatings. Trivalent chromium conversion coatings, at standard conditions for temperature and pressure, are applied by immersion and the resulting Class 1 anodize meets MIL-PRF-8625 requirements; therefore, there is no technical reason or basis to assume TCP coatings inadequate for brush anodize sealing, and TCP coatings would be applied in the same fashion/method defined for hexavalent chromium conversion coatings.

7 Conclusions

Data provided by project participants demonstrates performance of trivalent chromium conversion coatings, when used as an anodic seal, as compliant with corrosion resistance requirements defined in MIL-PRF-8625. The data also demonstrated trivalent chromium conversion coatings can serve as part of a modified non-hexavalent chromium sealing process for OEM applications where extreme stand-alone corrosion resistance performance is required.

The majority of test data provided pertains to sulfuric acid anodize; however, testing has also demonstrated compliant performance with Tartaric-Sulfuric Acid Anodize, (Type IC), and additional data pertaining to Type IC anodize will be added to this report as it becomes available. Table 3 provides a reference for corrosion resistance performance of Chromic Acid Anodize, (Type I), sealed with a hot dilute chrome seal, and no effort was made to collect data on Type I anodize sealed with trivalent chrome conversion coating as any such process includes use of hexavalent chrome.

Data provided by project participants demonstrates trivalent chromium conversion coating as anodic seal facilitates organic finish adhesion equivalent with performance observed with hexavalent chromium seals, with one caveat. Non-chrome primer, e.g., products compliant with MIL-PRF-23377J, Type I or II, Class N, and MIL-PRF-85582D, Type I or II, Class N, can be applied over anodize sealed with non-hexavalent chromium seals and comply with scribed corrosion performance requirements, (white corrosion products in the scribe is allowed for Class N primers). However, MIL-PRF-23377K and MIL-PRF-85582E do not include the same allowance for Class N primers, which may be problematic for OEMs building hardware for military customers. Tables 6 through 10 and Figures 3 through 10 demonstrate compliance of an inorganic and organic finish system free of hexavalent chrome with MIL-PRF-8625F, MIL-PRF-23377J and MIL-PRF-85582D. At 2,000-hours exposure in neutral salt fog scribed panels exhibit white corrosion product in scribe lines, but no pitting extending beyond 1-mm, (≥ 8 rating per ASTM D1654), from the scribe line for some organic finishes, (see Figure 7). Impact testing, perhaps a more important performance attribute, demonstrated TCP anodic seal provides equivalent performance to chromate seals.

Process requirements coupled with test data gathered demonstrate non-hexavalent chromium seals and sealing processes are suitable replacements for dichromate seals and dilute chromium seals, and offer equivalent or better performance with good potential for carbon footprint reduction. Careful process tank-line planning may also allow a dual-use tank, used for both Type II conversion coating and anodic sealing.

8 Recommendations

Most OEM process specifications already allow use of non-hexavalent chromium seals, e.g., hot water seals. The same process specifications often discourage use of hot water seals due to unreliable adhesion of organic finishes when said finishes are applied over hot water sealed anodize.

Non-hexavalent chromium seals, such as, trivalent chromium conversion coating provide equivalent or better performance than hexavalent chromium seals, and evaluation data collected from original equipment manufacturers in the European Union and North America supports implementation in process documentation, and on hardware. However, as demonstrated by recent changes to MIL-PRF-23377, (Rev J versus Rev K), and MIL-PRF-85582, (Rev D versus Rev E), customers must be aware of the change/substitution of anodic seals and approve use of non-hexavalent chromium seals on hardware they purchase. Strict interpretation of the primer specifications implies a chromated primer must be coupled with a non-hexavalent chrome seal or a non-chromated primer may be coupled with a hexavalent chromium seal, or the customer must agree to the pairing of non-chrome primer with non-hexavalent chromium anodic seal.

9 References

Public Specifications:

MIL-DTL-81706B – Chemical Conversion Materials for Coating Aluminum and Aluminum Alloys

MIL-DTL-5541F – Chemical Conversion Coatings on Aluminum and Aluminum Alloys

MIL-PRF-8625 – Anodic Coatings for Aluminum and Aluminum Alloys

MIL-PRF-23377 – Primer Coatings: Epoxy, High Solids

MIL-PRF-85582 – Primer Coatings: Epoxy Waterborne

ASTM D 3359 – Standard Test Method for Rating Adhesion by Tape Test

ASTM D 1654 – Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments

ASTM D 714 – Standard Test Method for Evaluating Degree of Blistering of Paints

ASTM B 117 – Standard Test Practice for Operating Salt Spray (Fog) Apparatus

ASTM B 136 – Standard Method for Measurement of Stain Resistance of Anodic Coatings on Aluminum

ASTM B 457 – Standard Method for Measurement of impedance of Anodic Coatings on Aluminum

ASTM B 680 – Standard Method for Seal Quality of Anodic Coatings on Aluminum by Acid Dissolution

ASTM D 4060 – Standard Method for Abrasion Resistance of Organic Coatings by the Taber Abraser