Hazmat Alternatives

DoD Metal Finishing Workshop - Chromate Alternatives for Metal Treatment and Sealing

DoD EPA DOE SERDP Strategic Environmental Research and Development Program

ESTCP Environmental Security Technology Certification Program

DEPARTMENT OF DEFENSE MERIT Materials of Evolving Regulatory Interest Team

Davis Conference Center
Layton Utah
May 15 - 17, 2007

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Report Documentation Page

Form Approved
OMB No. 0704-0188

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1. REPORT DATE	2. REPORT TYPE	3. DATES COVERED
01 MAY 2007	**N/A**	**-**

4. TITLE AND SUBTITLE	5a. CONTRACT NUMBER
DoD Metal Finishing Workshop - Chromate Alternatives for Metal Treatment and Sealing (May 2007)	5b. GRANT NUMBER
	5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S) **SERDP ESTCP**	5d. PROJECT NUMBER
	5e. TASK NUMBER
	5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) **Strategic Environmental Research & Development Program 901 N Stuart Street, Suite 303 Arlington, VA 22003**	8. PERFORMING ORGANIZATION REPORT NUMBER
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) **Strategic Environmental Research & Development Program 901 N Stuart Street, Suite 303 Arlington, VA 22003**	10. SPONSOR/MONITOR'S ACRONYM(S)
	11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release, distribution unlimited

13. SUPPLEMENTARY NOTES
The original document contains color images.

14. ABSTRACT
This technical workshop was designed to bring together DoD and industry engineers to identify specific DoD needs, commercial solutions, and engineering data for replacing chromate processes used for overhaul and new weapons systems in vehicles, aircraft and vessels: DoD needs for chromates and their alternatives; Commercial and military experience with alternatives; Specific COTS treatments to meet DoD requirements; Data and specifications for making engineering decisions; Data gaps are and how they can best be filled; Options where no potential COTS products are available.

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			
unclassified	**unclassified**	**unclassified**	**UU**	**47**	

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CLEARED
For Open Publication

NOV 14 2007 8

Office of Security Review
Department of Defense

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08-S-0337

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This report contains hyperlinks to the briefings and other documents. Briefings can be accessed from the agenda, from links within the text or by clicking on the paper clips. Other documents such as vendor literature can be accessed from Appendix 2.

EXECUTIVE SUMMARY

Workshop sponsors: The workshop was sponsored by SERDP/ESTCP and the DoD Office of Emerging Contaminants.

Workshop purpose: This technical workshop was designed to bring together DoD and industry engineers to identify specific DoD needs, commercial solutions, and engineering data for replacing chromate processes used for overhaul and new weapons systems in vehicles, aircraft and vessels:

- DoD needs for chromates and their alternatives
- Commercial and military experience with alternatives
- Specific COTS treatments to meet DoD requirements
- Data and specifications for making engineering decisions
- Data gaps are and how they can best be filled
- Options where no potential COTS products are available.

The workshop included briefings from DoD and commercial organizations (including summaries of available COTS technologies), panel discussions and breakout sessions.

DoD usage of chromates: Chromates are used both for corrosion protection and for adhesion. The major usages are:

- Corrosion resistant chromate conversion coatings on Al and Mg alloys, including aircraft skins, gearboxes and structural components
- Chromated primers and chromated metallic-ceramic paints (paints and primers were not covered in this workshop to permit a sharp focus compatible with a two-day meeting)
- Chromic acid anodizing
- Chromate washes for steels.

DoD usage of chromate alternatives: There are several ways in which non-Cr^{6+} alternatives differ from chromates:

- We are now using 4th generation Cr^{3+} treatments, which often (but not always) contain Co inhibitors and nanophase SiO_2 to fill scratches. For many materials trivalent processes are now as good as (and in some cases better than) hexavalent processes.
- Non-Cr^{6+} treatments are significantly more process sensitive than chromates. Even rinse water quality must be carefully controlled. This has often made them less reliable in production.
- Non-Cr^{6+} treatments are still not adequate for 2024 and 7075 Al.

There have been a number of adoptions of chromate alternatives by DoD and prime contractors:

- NAVAIR Tri-Chrome Pretreat (**TCP**) is now available commercially from several vendors, and has proved to work well for most Al alloys, for Cd-alternatives (Al and ZnNi coatings), for sealing anodize layers, and for Mg alloys.
    - TCP has been approved by NAVAIR for some Al alloys.

- The various commercial TCP materials are expected to appear on the QPL for Mil-DTL-5541F, Type II, Class 1A and 3 shortly.
- AMRDEC Materials has authorized TCP for implementation on Army helicopters

- NADEP Cherry Point Implemented **Alodine T5900** Jan. 2006.
- The Air Force has authorized **PreKote** under Tech Order 1-1-8.
- The US Army Tank Automotive Command (TACOM) has issued an Authorization Letter for **Alodine 5700**.
- The Marine Corps DRPM AAA has authorized **Alodine T5900** as well as **Alodine 5700** wipes.
- Boegel adhesion promoter is now available commercially as **AC 130, Alodine 6000 SG**.
- The Stryker program has adopted **Alodine 5200/5700** for Al roadwheels as well as eliminating wash primers for some high-hard steels.
- The Air Force has successfully demonstrated **PreKote** in place of chromate conversion for C130 aircraft skins.
- The F-35 has adopted **Deft non-Cr^{6+} primer** in combination with a chromate pretreat for all internal painted spaces.

COTS products: There are a large number of COTS products for Zn and Zn alloys, which are widely used commercially, especially in the automotive industry (see table below). In addition there are various products for Al and Mg:

- TCP products – Alodine T5900, Aluminescent, ChromitAl TCP, TCP-HF
- NCP products – Iridite NCP
- Numerous products for Zn alloys (see table below).
- Anodize alternatives – Thin Film Sulfuric, Boric Sulfuric, Keronite, Tagnite.
- Non-Cr primers from Deft, Akzo-Nobel, PPG.

Outstanding issues:

- Chromate seals on Cd and Zn plated **fasteners**. Needs coverage identification method (a color indicator to show degree of protection, which current Cr^{6+} rinses provide as yellow, olive, black, etc.).
- Still need replacement for **Cr^{6+} wash primer** for steel and aluminum.
- There are still few treatments that work well for **7075 and 2024 Al**.
- There is still a need for chromate alternative treatments for Al electrical boxes that are not painted.

Workshop product: In addition to this report, users agreed to supply their requirements for various applications, and vendors to identify COTS products that meet those requirements, match against commercial specs, and provide supporting data if it can be made available. This information will be incorporated into a database of information on chromate alternatives.

Application	Vendor	Product	Cr^{3+}	Non-Cr	Notes. Approvals
Al zincate pre-plate	Atotech	TriBond® II			Non-cyanide
Al zincate pre-plate	Atotech	AlumSeal® 650			
Al zincate pre-plate	MacDermid	Bondal CF			
Al zincate pre-plate	MacDermid	Metex 6811			
Passivation - Al pretreat	Atotech	Interlox® 338 (ZrCr)	✓		
Passivation - Al pretreat	Atotech	UniPrep® PP (borate ester)		✓	Pretreat for powder coat
Passivation - Al pretreat	Atotech	Interlox® 5704(ZrMn)		✓	
Passivation - Al pretreat	Atotech	Interlox 5705(ZrMg)		✓	
Passivation - Al pretreat	MacDermid	Iridite TCP	✓		NAVAIR TCP licensee
Passivation - Al pretreat	MacDermid	Iridite NCP		✓	NAVAIR NCP licensee
Passivation - Al pretreat	Pantheon	PreKote		✓	Paint adhesion promoter
Passivation - Al, Ti, Mg	Henkel	Alodine T5900, T5900 RTU	✓		
Passivation - Al, Ti, Mg	Henkel	Alodine 871 Touch-N-Prep	✓		Touch-up
Passivation - Al, Ti, Mg	Henkel	Alodine 5200, 5700		✓	Non-phosphate
Passivation - Al, Ti	Henkel	Alodine 6000 SG		✓	Sol-gel (Boegel); adhesion promoter
Passivation - Al	AC Tech	AC 130		✓	Sol-gel (Boegel); adhesion promoter
Passivation - Al	Henkel	Alodine EC[2]		✓	Electrodepos TiO_2
Passivation - Al, Mg	Luster-On	Aluminescent	✓		NAVAIR TCP licensee; QPL listed for Mil-DTL-5541F, Type II, Class 1A and 3
Passivation - Al	SurTec	ChromitAl TCP	✓		NAVAIR TCP licensee
Passivation - Al	Metalast	TCP-HF	✓		NAVAIR TCP licensee; also used as anodize seal
Passivation - Zn, Zn alloy	Atotech	Ecotri®	✓		
Passivation - ZnNi	Atotech	Tridur®	✓		
Passivation - Zn, Zn alloy	Atotech	Rodip® ZnX	✓		Co-free
Passivation - Zn	MacDermid	Tripass, Tripass ELV	✓		Clear, blue, iridescent, yellow, black
Passivation - Zn, Zn alloy	Enthone	Zinchrolyte	✓		
Passivation - Zn, Zn alloy	Enthone	Perma Pass	✓		Nanoparticle, clear, blue, iridescent, yellow, black
Passivation - Zn, Zn alloy	Taskem	Trimate	✓		
Passivation - Zn (alk)	Taskem	Lanthane	✓		
Passivation - Zn, Zn alloy	Taskem	Finidip	✓		
Passivation - Zn, Zn alloy	Taskem	TR 175, 173	✓		Includes nanoparticles
Passivation - Zn, Zn alloy	Taskem	TC-HP, TC-SA	✓		Blue
Passivation - Zn, Zn alloy	Luster-On	Tri-blue, Tri-descent, Tri-black TZT	✓		
Passivation - Zn, Zn alloy	SurTec	Chromiting	✓		
Mg zincate pre-plate	MacDermid	Bondal Mg			Non-cyanide
Sn-Zn electroplate	Atotech	Reflectalloy® SnZn			Non-cyanide
Sn-Zn electroplate	MacDermid	Enviralloy SN			
Zn (12-16)Ni electroplate	Atotech	Reflectalloy® ZnA			Bright
Zn (12-16)Ni electroplate	Atotech	Reflectalloy® HD			Ductile
Zn (12-16)Ni electroplate	Atotech	Reflectalloy® TF			For subsequent organic coating
Zn-Ni	MacDermid	IsoBrite 570			Acid process
Zn (12-15)Ni electroplate	MacDermid	Enviralloy NI 12-15			Alkaline, for rack and barrel plating
Zn (8-12)Ni electroplate	MacDermid	Nyzin 1200			Alkaline
Zn-Ni	Enthone	Enthobrite CLZ 933, 941, 953			Acid, bright
Zn-Ni	Enthone	Enthobite NCZ			Alkaline non-cyanide; ductile and bright versions
Inorganic seal	Atotech	Rogard®			
Inorganic seal	MacDermid	JS 500, 600, 1000, 2000			
Inorganic seal	MacDermid	Ultraseal			
Inorganic seal	MacDermid	Hydroklad SI			
Organic seal	Atotech	Corrosil®			
Organic seal	MacDermid	Torque n' Tension			
Organic seal	MacDermid	HydroKlad			
Organic seal	MacDermid	Hydrolac			
Organic seal	MacDermid	Aqualac			
Organic/Inorganic seal	Enthone	Enseal			Various
Electroless Ni	Atotech	Nichem® 11			Pb and Cd-free
Electroless Ni	MacDermid	NiKlad ELV			Pb and Cd-free; low, medium, high phos
Electroless Ni	MacDermid	Niklad ELV LMO			Pb and Cd-free; organic stabilizers
Electroless Ni	Enthone	Enplate EN333LF			High phos, Pb-free
Electroless Ni	Enthone	Enplate EN-806			Mid phos, Cd and Pb-free
Electroless Ni/PTFE	MacDermid	Niklad ICE with ELV 811			High phos
Torque-tension modifier	Taskem	Finigard			Zn and Zn alloys

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ACRONYMS

AAA	Advanced Amphibious Assault
AFB	Air Force Base
AFRL	Air Force Research Lab
ALC	Air Logistics Centers
AMCOM	Aviation and Missile Command
AMRDEC	Aviation and Missile Research, Development and Engineering Center
AMS	Aerospace Material Specification (SAE)
ARL	Army Research Lab
ASTM	American Standard Test Method
AVCRAD	Aviation Classification Repair Activity Depot
AVTB	Amphibious Vehicle Test Branch
BSAA	Boric Sulfuric Acid Anodizing
CANMET-MTL	Canada Centre for Mineral and Energy Technology - Materials Technology Lab
CARC	Chemical Agent Resistant Coating
CCC	Chromate conversion coating
COTS	Commercial off-the-Shelf
Cr^{6+}, CrVI, hex chrome	Synonyms for hexavalent chromium
DLA	Defense Logistics Agency
DoD	Department of Defense
DSP	Defense Standardization Program
DSTO	Defence Science & Technology Organisation (Australia)
DTL	Detail
EFV	Expeditionary Fighting Vehicle
ELV	End of Life Vehicles
ESOH	Environment, Safety and Occupational Health
ESTCP	Environmental Security Technology Certification Program
FRC	Fleet Readiness Center
GSA	General Services Administration
GTE	Gas turbine engine
HVOF	High velocity oxygen fuel
IVD	Ion Vapor Deposition
LCS	Littoral combat ship
MERIT	Materials of Evolving Regulatory Interest Team
MRO	Maintenance repair and overhaul
NADEP	Naval Aviation Depot (now called Fleet Readiness Centers, FRCs)
NAVAIR	Naval Air Systems Command
NAVSEA	Naval Sea Systems Command

NCP	Non-chrome pretreatment
NDI	Non Developmental Item
NDMA	N-nitrosodimethylamine
NGS	Non Government Standard
NIOSH	National Institute for Occupational Safety and Health
NSN	National Stock Number
OEC	Office of Emerging Contaminants
OEM	Original Equipment Manufacturer
OO-ALC	Ogden Air Logistics Center
OSD	Office of the Secretary of Defense
OSHA	Occupational Safely & Health Administration
PAX	NAVAIR Putaxent River
PBB	Polybrominated Biphenyl
PBDE	Polybrominated Diphenyl Ether
PDM	Periodic Depot Maintenance
PEL	Permissible Exposure Limit
PFOA	Perfluorooctanoic Acid
PFOS	Perfluorooctyl Sulfonate
PM	Project manager
PPE	Phenylene Ether Co-polymer
PRF	Performance
QPL	Qualified Products List
RDE	Responsible Design Engineer
RDX	Cyclotrimethylenetrinitramine
REACH	Registration, Evaluation, Authorization and Restriction of Chemicals
RoHS	Restriction of Hazardous Substances.
SAA	Sulfuric Acid Anodize
SAE	Society of Automotive Engineers
SERDP	Strategic Environmental Research and Development Program
TACOM	Tank Automotive Command
TCE	Trichloroethylene
TCP	Tri-Chrome Pretreat (developed by NAVAIR)
TFS(AA)	Thin Film Sulfuric (Acid Anodizing)
TSAA	Thin Film Sulfuric Acid Anodize
USAF	US Air Force
USAMP	United States Automotive Materials Partnership
USCG	United States Coast Guard
USMC	US Marine Corps
WEEE	Waste Electrical and Electronic Equipment
ZAP	ZinKlad Approved Plater

1. Agenda

Wednesday May 16, 2007 Click blue text to go to item in document. Paper clips open files.

	Subject	Name	Organization
	Welcome and logistics	**Bruce Sartwell**	ESTCP, SERDP
	Workshop introduction and aim	**Keith Legg**	Rowan Technology
	OSD and Emerging Contaminants	**Carole LeBlanc**	MERIT team, DoD OEC
	Global Chromate Requirements & Trends	**Mike Wyrostek**	MacDermid
	Pretreatments for Al on aircraft	**Bill Nickerson**	NAVAIR
	Cr^{6+} and COTS options for vehicles	**John Beatty**	ARL
	Hex Chrome Conversion Coatings at Hill AFB	**Nate Hughes**	Hill AFB
	Application of PreKote at Robins AFB/C-130	**Todd Lavender**	WR ALC
	Current Status of Trivalent Passivates	**Mike Kelly**	Taskem
	Available Non Hexavalent Chromate Products	**Craig Bishop**	Atotech
	Hex-Cr Free Zn & Zn Alloy Technology	**Jim Kloeckener**	Enthone
	Chromate Alternatives for Metal Protection	**Bill Wittke**	Henkel
	Managing the Transition to Hexavalent Chromium Free Anti-Corrosion Coatings	**Rob Berry, Mike Wyrostek**	MacDermid
	Other COTS alternatives: specialist vendors Cjejka Radzvilowicz Buchi Lindeman Kelly	**Joe Ciejka** (Luster-On), **Joe Radzvilowicz** (Metalast), **Richard Buchi** (Pantheon Chemical), **Ray Lindeman** Sur-Tec International, **Mike Kelly** (Taskem)	
	Sol-gel Conversion coatings, and anodizing	**Joe Osborne**	Boeing
	Coatings for Fasteners	**Rick Delawder**	SWD
	Ce-based Conversion Coatings	**Matt O'Keefe**	Univ MO/Rolla
	Issues with use of Chromate Alternatives	**Steve Gaydos**	Boeing
	Panel Discussion #1 - COTS Cr^{6+} alternatives:		Chair**: Steve Brown**
	Available products and experience with commercial Cr^{6+} alternatives (for Al, steels, coatings)	**Steve Brown** (NAVAIR, PAX), **Craig Bishop** (Atotech), **Jim Kloeckener** (Enthone), **Todd Lavender** (WR ALC), **Bill Wittke** (Henkel), **Mike Wyrostek** (MacDermid), **Mary Traficante** (Atotech)	
	Advanced Aluminum Anodizing Technology	**Ruben Prado**	FRC Southeast
	Modern Facilities Design for Surface Finishing	**Peter Gallerani**	Integrated Tech
	Panel discussion #2 - Running a non-Cr^{6+} line		Chair**: Bill Emery**
	What does it take to put a non-Cr system into practice?	**Bill Emery** (SW United), **Peter Gallerani** (Integrated Technologies), **Nate Hughes** (Hill AFB), **John Lindstedt** (Artistic Plating)	

Thursday May 17, 2007

	Subject	Name	Organization
	Corrosion Protection of Structural Mg Alloys in Automotives	**Wenyue Zheng (K. Legg)**	CANMET-MTL
	Cr-free conversion Coatings for Aerospace - the European Perspective	**Nieves Lapena-Rey (Steve Gaydos)**	Boeing Europe/Spain
	Electroplated AlumiPlate Al & TCP	**Gus Vallejo**	Alumiplate
	ESTCP Hydrogen Embrittlement testing study	**Steve Gaydos**	Boeing St Louis
	Breakout Sessions:		
	1. Pretreatment (conversion coating) of Al and Mg alloys – structural and electronic		Chair: **Joe Osborne**
	2. Chromate treatment of steels (e.g. chromate wash primer)		Chair: **John Beatty**
	3. Chromate treatment of coatings (IVD Al, electroplated Al, Zn, ZnNi)		Chair: **Steve Gaydos**
	Outbriefs from Breakouts		
	Introducing Products to DoD Utilizing OSD's New Specs & Standards On-line Tool	**Robert Herron**	Redstone Arsenal
	Panel discussion #3 - Getting alternatives into production		Chair**: Robert Herron**
	Qualification, Authorization, Implementation Paths & Drivers (who authorizes for different services and applications)	**Robert Herron** (Redstone), **Steve Battle** (WR-ALC), **John Beatty** (ARL), **Elizabeth Berman** (AFRL), **Mike Hanson** (Coast Guard), **Nate Hughes** (OO-ALC), **Ruben Prado** (FRC SE), **Steve Brown** (NAVAIR PAX)	
		Open Discussion – Action Items	

2. Background and purpose of the workshop

DoD spends $10-20 billion/year on corrosion control[1]. To reduce these costs we need to improve the performance of our corrosion control systems, but at the same time environmental and health pressures are forcing a move away from the existing corrosion control technologies of Cd and Cr^{6+}.

The new OSHA PEL for Cr^{6+} and growing restrictions on the use of chromates around the world are driving manufacturers and overhaulers to find non-toxic alternatives. The most cost-effective approach is to use commercial off the shelf (COTS) products wherever possible, while working with suppliers to modify products that do not adequately meet DoD's needs. This technical workshop was intended to bring together DoD and industry engineers to identify specific DoD needs, commercial solutions, and engineering data for replacing chromate processes used for metal finishing in the overhaul and new weapons systems in vehicles, aircraft and vessels:

- DoD needs for chromates and their alternatives
- Experience with alternatives in DoD and the supplier base
- What specific COTS treatments are available to meet DoD needs and requirements
- What data and specifications are available for making engineering decisions
- Where data are insufficient, where the gaps are and how they can best be filled
- Where there are no potential COTS products, what should be done to fill the need
- Since the workshop was intended to focus on metal finishing uses only it did not cover Cr^{6+} use in primers, sealants, etc.

The meeting was held near Hill AFB (Ogden Air Logistics Center), and attendees toured the plating and finishing shops at Hill to provide commercial suppliers with a better understanding of how DoD uses chromates.

2.1. SERDP, ESTCP

***Keith Legg*, Rowan Technology Group:** This briefing provided a quick introduction to the purpose and expected outcome of the workshop.

The purpose of this workshop was to improve collaboration between DoD and the supply base. Ultimately the aim is to develop an Engineering Database for finishing that will contain the data that engineers require for informed decision-making.

Figure 1 illustrates how such an Engineering Database would be used to support the development and adoption of surface engineering technologies. Such a database will save DoD a great deal of time and cost by making it much easier to adopt commercial processes that meet DoD requirements.

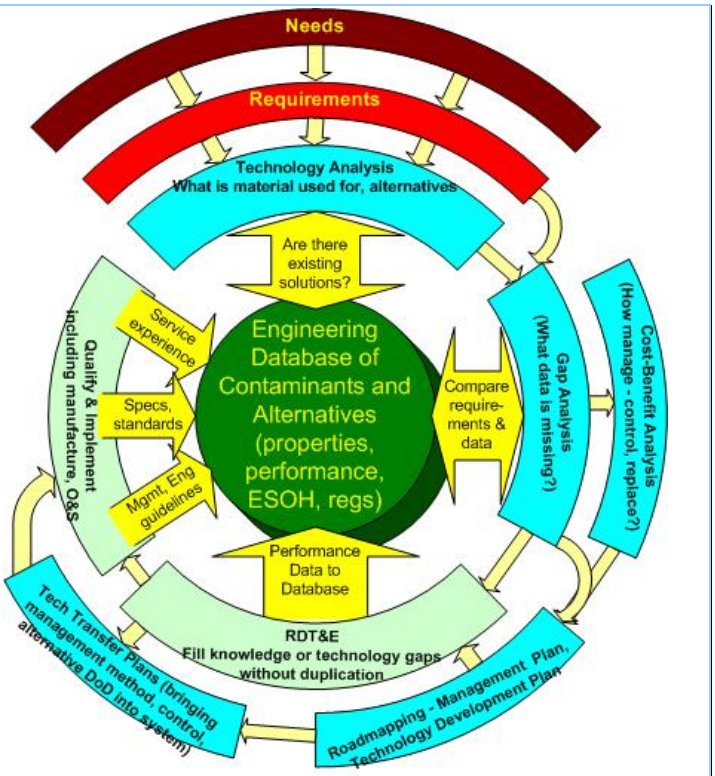


Figure 1. Purpose of a Surface Engineering Database.

2.2. DoD Office of Emerging Contaminants

Carole LeBlanc*, *MERIT: The DoD Materials of Evolving Regulatory Interest Team (MERIT)[2] identifies Emerging Contaminants and seeks ways to manage the risk to DoD operations that they represent.

Emerging Contaminants are chemicals whose regulatory standards are non-existent or evolving.

- The Watch List comprises those materials with a potential for regulatory change that could impact DoD. This list includes lead, beryllium, tungsten and nanomaterials.
- The Action List comprises those materials with a significant potential for regulatory action that will impact DoD, as determined from a Phase I Impact Assessment. Items in this list will undergo a Phase II Impact Assessment, which will generate options for risk management that may include substitution of materials, processes modifications, better controls or PPE, etc. Chemicals currently on the Action List are:
  - Cr^{6+}
  - RDX explosive (Cyclotrimethylenetrinitramine)
  - Trichloroethylene (TCE)
  - Perchlorate
  - Naphthalene.

Carole handed out a questionnaire covering uses of Cr^{6+}, roadblocks and risk management options.

The Watch and Action Lists are illustrated in Figure 2, which shows how the Watch List funnels into the Action List

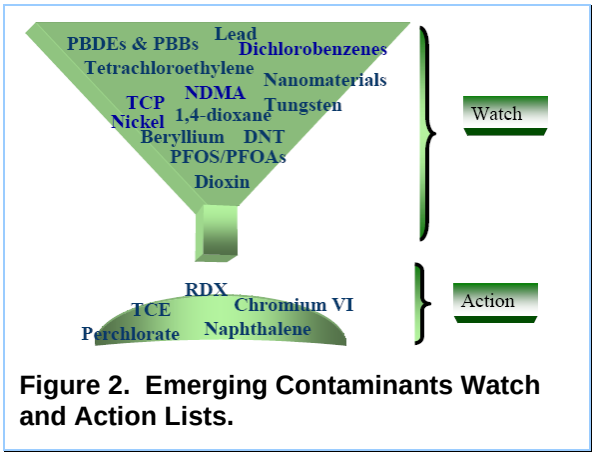


Figure 2. Emerging Contaminants Watch and Action Lists.

2.3. Global trends

Mike Wyrostek, MacDermid: In industry, non-chromate chemistries are generally termed passivates, not conversion coatings, since different chemistries work in different ways.

There is increasing regulation of Cr^{6+} both as a worker health (OSHA and NIOSH) issue and as a pollutant that poses problems for end-of-life disposal. The primary drivers for its elimination are the European Union (EU) regulations:

- ELV – End of Life Vehicles
- WEEE – Waste Electrical and Electronic Equipment
- RoHS – Restriction of Hazardous Substances.

These rules cover all consumer electrical and related equipment sold in the EU, and so affect US manufacturers and their suppliers. RoHS regulations are under development by 23 states (notably CA), China, Canada, Taiwan and Japan. As a result industry is shifting to Cr^{3+} and non-Cr technologies for Al, Zn and Mg alloys and for Zn and Zn alloy coatings. In fact, the US has moved more rapidly than Europe or Asia, where the rules were developed (Figure 3).

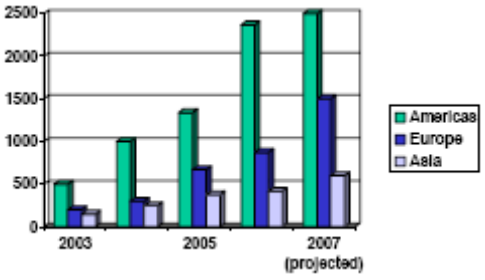


Figure 3. Trivalent passivate market (1000 gals).

It was brought out in various discussions throughout the meeting that this change means that suppliers are installing non-Cr^{6+} lines and decommissioning their Cr^{6+} lines. Although this does not mean that Cr^{6+} processes will be unavailable for DoD, it does mean that the cost of both Cr^{6+} chemicals and processes will rise as availability declines.

European usage, Nieves Lapena-Rey: As we have seen in above the European Union has instituted the ELV, WEEE and RoHS regulations. Boeing Research and Technology Europe has examined the various technologies and materials being offered in the EU. Products offered for sale in Europe are based on phosphates, permanganates, Ti/Zr treatments, cerium-based treatments, molybdates, sol-gel and Cr^{3+} inhibitors. These are in general the same (or similar) products as those used in the US. Because the aerospace sector is exempt from the Cr^{6+} regulations, they continue to use chromate conversion coatings. They are, however, moving towards the use of Cr^{6+}-free primers from Akzo Nobel, Deft and PPG.

2.4. Trivalent and non-chrome technology

It is generally accepted wisdom that Cr^{3+} passivates are not as effective as Cr^{6+} chemistries. However, the performance of trivalent and non-Cr treatments has greatly improved over the past few years. The protection mechanisms are very different, however.

Mike Kelly, Taskem: In traditional chromates oxidation of the surface is carried out by Cr^{6+} ions, while in trivalent passivates oxidation is done by nitrates. As a

result Cr^{3+} coatings are thinner and typically clear or iridescent. This thinner coating requires additional help in protecting the substrate, and we are now in the fourth generation of Cr^{3+} passivates, each generation supplying better protection packages. Typical Cr^{3+} passivates now contain Co and silica nanoparticles. Rather than Cr^{6+} precipitating into any scratch in the coating, the SiO_2 nanoparticles fill the exposed area to prevent further corrosion (Figure 4). As a result of these improvements, 4th generation TASDIP 175 Cr^{3+} on Zn coating is practically the same as Cr^{6+} (Figure 5).

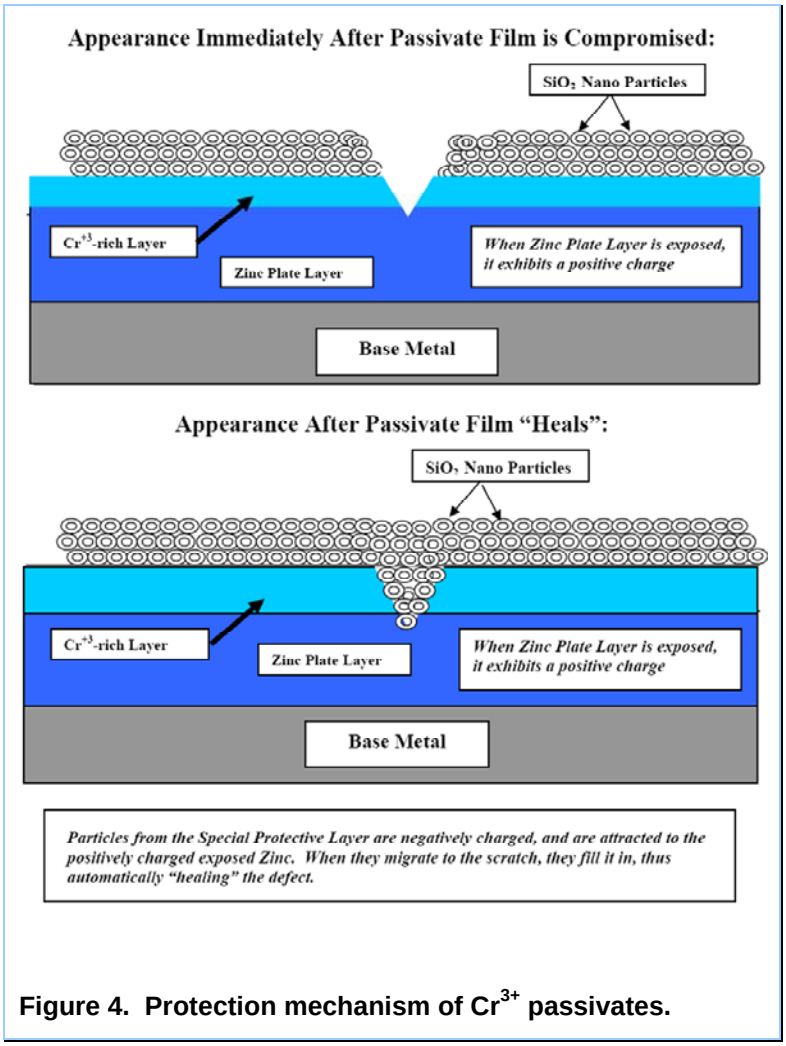


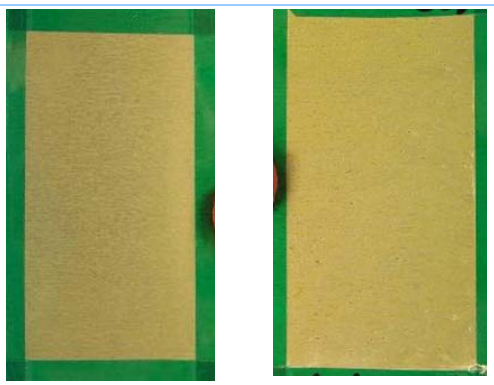
Figure 4. Protection mechanism of Cr^{3+} passivates.

Figure 5. Comparison of Cr^{3+} generation corrosion performance with Cr^{6+} for Zn coatings on steel.

Note: It was brought up numerous times during the workshop that, whereas chromates are very easy to use, Cr^{3+} and non-Cr chemistries are highly dependent on proper surface preparation.

Matt O'Keefe*, *University of Missouri - Rolla: There is now a class of inhibitors based on rare earths (primarily Ce) that is used in various commercial passivates and primers. Development has focused on the most difficult Al alloys 2024 and 7075 and has achieved successful protection (Figure 6).

CeO_x-based protection systems are not inherently protective as Cr^{6+} is, and the mechanism of Ce protection is not well understood. Surface cleaning and preparation are critical with Ce, just as with other non-Cr^{6+} inhibitors, and it is essential for the coating to have the correct structure and thickness. Phosphating improves the protection by changing Ce(IV) to Ce(III).



Before Salt Spray Testing **After 14 Day Salt Spray Testing**

Figure 6. Salt spray testing of Ce conversion coating on 2024-T3 Al.

3. DoD Usage and Needs

DoD has many existing (**legacy**) systems that were all designed with copious usage of Cd plate, Zn plate (in vehicles), hard chrome, chromate conversion coatings and chromated primers. New weapons systems use newer materials, including Al, Mg and Ti alloys, composites, etc. These systems are often now specified to be as "green" as possible, but getting green solutions in place is never an easy task.

Weapons systems are built by OEMs but are mostly maintained at the operational bases for simple maintenance and touch-up. They are sent to military depots (Army depots, Air Force Air Logistics Centers (ALCs), and Navy Yards and Fleet Readiness Centers (FRCs) for major overhaul (often called Periodic Depot Maintenance, PDM).

Additional information can be found in the report of the DoD Metal Finishing Workshop held in May 2006..

3.1. Cr^{6+} and COTS alternatives for vehicles – US Army

***John Beatty*, Army Research Lab:** DoD has a fleet of more than 300,000 vehicles, most of them over 20 years old, with the most common material being carbon steel. In the future we expect much more use of high strength steels, high strength aluminum alloys, lightweight composite armor, titanium and magnesium alloys. Primary new vehicles are in the Stryker family of vehicles (Figure 7).

Figure 7 Stryker infantry armored vehicle.

Usage: Chromates are used very widely, as washes on steels for corrosion and paint adhesion, for sealing of zinc, and for corrosion protection of Al (e.g. electrical cabinets).

Requirements: Chromates (and therefore chromate alternatives) must meet the following requirements:

- Primary requirements:
  - corrosion protection,
  - ease of use
  - conductivity
  - color identification
  - improved processing operational tolerances.
- Other requirements
  - throughput

- process robustness
- coating thickness and uniformity
- wear
- metallurgical bond
- fatigue life
- torsion and tensile strength
- surface condition prior to painting and bonding.

Barriers to adoption of alternatives:

- Changes in overhaul manuals for legacy systems (since these manuals cover everything that must be overhauled or repaired throughout a whole vehicle or aircraft, making changes and getting them approved is a major, very costly and time-consuming task)
- New processes must generally work well for both commercial and DoD applications since both are usually processed in the same equipment by vendors. (For this reason vendors want to have their commercial and military customers adopt the same technologies since it is expensive, and sometimes impractical, to maintain separate processing lines for different customers.)
- New weapons systems use performance-based specs, and the performance of the clean alternative must be equal or better to that of the current technology (without significant cost penalties)
- For depots capital cost is an important issue, and it takes several years to insert new equipment purchases into depot shop budgets.
- New products must have industry specifications so they can be called out in drawings and manuals.

Alternatives adopted:

- Chromates (Cr^{6+})
  - Non-CCC (grit blasting followed by primer and topcoat) on corrosion resistant armor alloys
  - Non-CCC (**Alodine 5200/5700**) on aluminum roadwheels
  - DI water rinse in lieu of chromate seals on zinc phosphate during CARC application process
  - Eliminate Cr^{6+} Wash Primer on some high-hard steel vehicles
  - Navy and Army have achieved good success using Non-CCC coatings on Al 5083 and 7079 armor alloys
  - TCP ready for use.
- Hard Chrome
  - Electroless Ni, Ni-tungsten boron
  - HVOF coatings of tungsten carbide/cobalt coating on M1 Tank GTE components (e.g. compressor bearing housing)
  - Other wear resistant materials in several engine applications.

Applications difficult to change:

- Huge use: Chromate seals on Cd and Zn plated fasteners. Needs coverage identification method, i.e. a color indicator to show the degree of protection, which current Cr^{6+} rinses provide as yellow, olive, black, etc. There is some debate among users as to whether there are different levels of protection with non-Cr^{6+} products, and if so whether they should be color coded to match present chromate colors. Some users prefer non-Cr^{6+} finishes to have a different color.
- Still need replacement for Cr^{6+} wash primer for steel and aluminum.

3.2. TCP and other chromate alternatives in DoD

***Bill Nickerson*, NAVAIR:** NAVAIR controls processes and materials used for Naval aviation (land based and carrier based fixed wings and helicopters). Focusing on ESOH has led to use of some treatments with much worse corrosion protection, which the Navy cannot afford to have. NAVAIR has developed Tri-Chrome Pretreatment (TCP) and licensed to several vendors. This treatment is becoming used in weapons systems across DoD. NAVAIR has also developed a Non-Chrome Pretreatment (NCP), which is currently in test.

Usage: Chromates are used very widely in Navy aircraft

- 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.

R&D work in progress:

Development work is continuing on both Tri-Chrome Pretreatment (TCP) and Non-Chrome Preatreatment (NCP). NCP is looking particularly good for sealing anodized coatings on Al. TCP and NCP are working very well for sealing sulfuric acid anodize, thin film sulfuric and boric sulfuric.

Anodizing and anodize sealing:

- Hot water sealing of anodize coatings is not nearly as effective as chromate sealing.
- TCP is better and more cost-effective than chromate for sealing
- NCP has similar performance as chromate sealing over Type II and IIB sulfuric acid anodize.

Mg pretreatment:

- TCP/NCP improve adhesion and corrosion

Zn pretreatment:

- TCP is looking quite good, but process temperature is very important.

Alternatives adopted or in the works:

See Table 1 below.

Table 1. DoD chromate replacement activities (Bill Nickerson).

Agency	Activity
Naval Aviation (NAVAIR)	Authorization Letter issued for **TCP**
	NADEP Cherry Point Implemented **Alodine T5900** Jan. 2006
	20 aircraft delivered to fleet
	Naval Air Station Kingsville – T-45 trainer aircraft demo planned FY07
US Army Aviation (AMCOM)	AMRDEC Materials authorized **TCP** for implementation
	CH-47 helicopter FY06 Demo (1109th AVCRAD) – In service for 12+ months
	FY07 Transition Planned at Corpus Christi Army Depot
US Air Force	Tech Order 1-1-8 authorizes **PreKote**
	Ongoing demonstration/validation – AC130 aircraft
	SAE Committee Specification for organic surface finishes
US Army Tank Automotive Command (TACOM)	Authorization Letter issued for **Alodine 5700** ❑ 5000/6000 Series – Red River Army Depot
	Project Manager (PM) Combat Systems Environmental Management Team TCP demo ❑ 3 M2A3 Bradley's – 3+ yrs
Marine Corps (USMC)	Direct Reporting PM Advanced Amphibious Assault authorized **Alodine T5900**
	Alodine 5700 Wipes – implemented 2005 ❑ Amphibious Vehicle Test Branch , Camp Pendleton
Naval Sea Systems (NAVSEA)	Technical warrant holder decision on implementation
	New platforms (LCS, Swift, etc) using more "aerospace" materials
General Dynamics	Revised Finish Spec – Expeditionary Fighting Vehicle (EFV) ❑ **Alodine T5900**
BAE Ground Systems	FY06/07 demonstrating **Metalast TCP-HF** ❑ Working with commercial finishing shops
	Evaluating **Nalco TechBond**
Boeing St. Louis	**Alodine 5700 Wipes** for touch-up
	Evaluating TCP products for immersion line
	Working w/ U. Missouri-Rolla for Ce-based conversion processes

***Ruben Prado*, FRC SE, Metalast Sulfuric Acid Anodizing control process:** The Metalast process carries out sulfuric acid anodizing using a process controller and a bath additive to inhibit burning and improve performance. Results have been very good, with accurate thicknesses and higher performance than for standard SAA. In addition, the anodized surface performs better with TCP sealing than with standard dichromate (Figure 8).

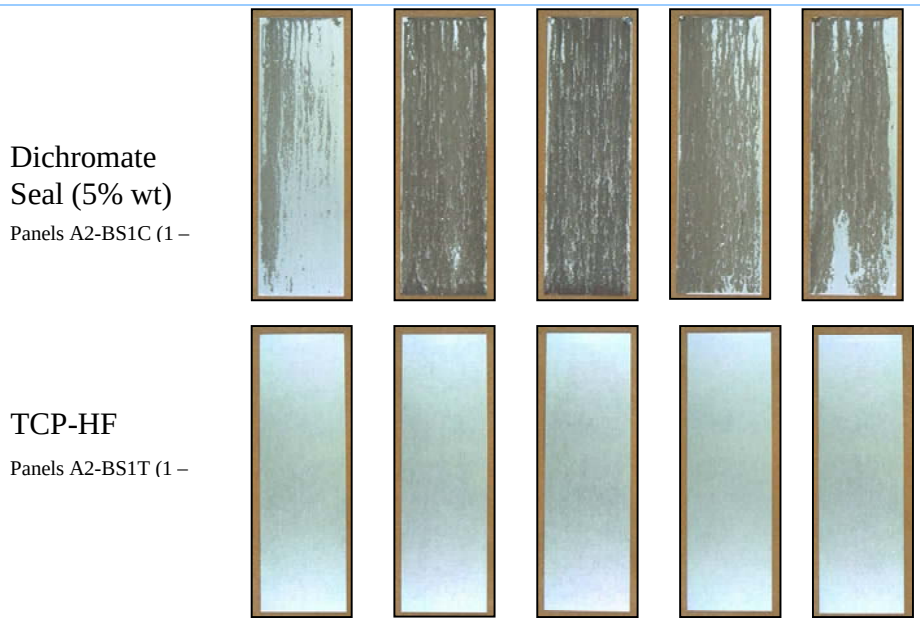


Figure 8 Performance of Metalast sulfuric acid anodize with Metalast TCP-HF sealing vs dichromate sealing.

3.3. Cr^{6+} usage and replacement in the Air Force

***Todd Lavender*, Robins AFB:** Robins overhauls airframes. They have been very successful in replacing alodine chromate conversion coating of Al aircraft skins with PreKote™ paint adhesion promoter. This product is aqueous and improves adhesion on metals and composites. Because it does not provide corrosion protection it cannot be used by itself as a protective coating, but by improving adhesion it makes paint systems much more effective. Because it is also a simpler and faster process (Figure 9) it is more cost-effective. The process has been demonstrated on C130 aircraft.

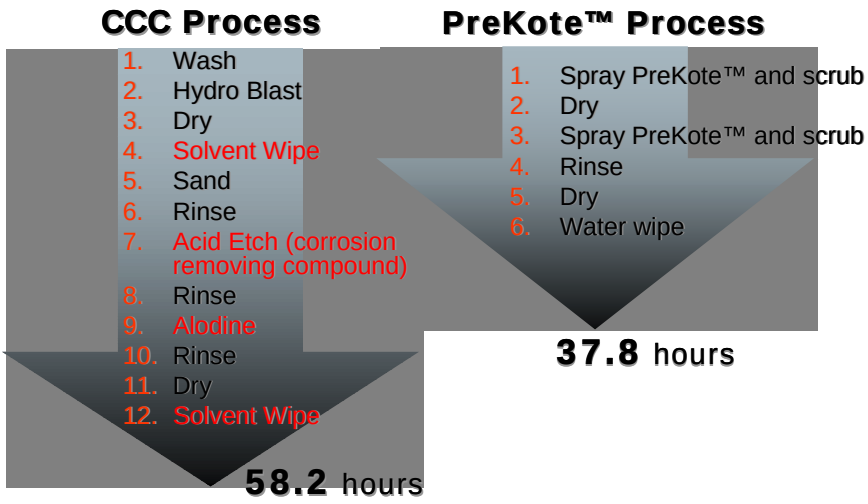


Figure 9. Process comparison between conversion coating and PreKote.

***Nate Hughes*, Hill AFB:**

Usage of Cr^{6+} in landing gear overhaul (Hill AFB)

- Anodize strip
  - For inspection, used to strip anodize under spec MIL-STD-871 (Chromic/Phosphoric acid, 180°F)
  - Proved they do not need 100% inspection, reducing anodize stripping. They now re-anodize partially stripped surfaces so as to avoid stripping and re-anodizing.
- Anodize seal
  - MIL-A-8625F, Type II Class 1 (sodium dichromate), used for wheel and brake components and some struts
- Conversion coat for aluminum
  - MIL-DTL-5541F, Type I Class 1 (Iridite 14-2), used for Al components and IVD Al coatings
- Conversion coat for cadmium
  - MIL-STD-870C, Class 1 Type II, Iridite 80 for sealing of Cd-plated bushings.

4. Commercial Products and Applications

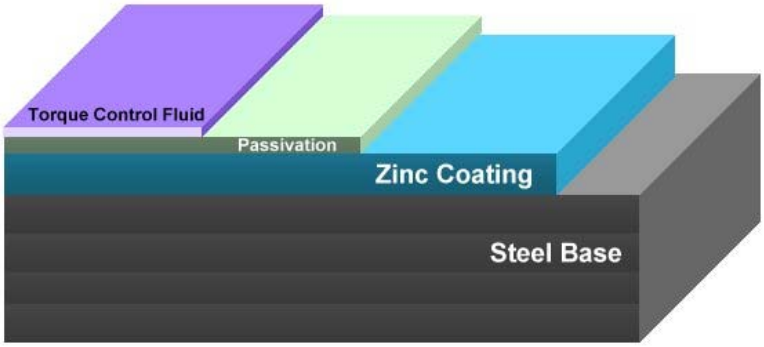


Figure 10. Typical Zn finish for fasteners (Mike Wyrostek, MacDermid).

Commercial products include passivates for Zn, Al, Mg, Ti and other alloys and coatings. In addition some vendors sell plating solutions for Cd alternatives. Figure 10 shows a typical coating for steel fasteners for use in vehicles and electrical equipment. New passivation systems containing nanoparticles eliminate the need for sealing topcoats (Figure 11).

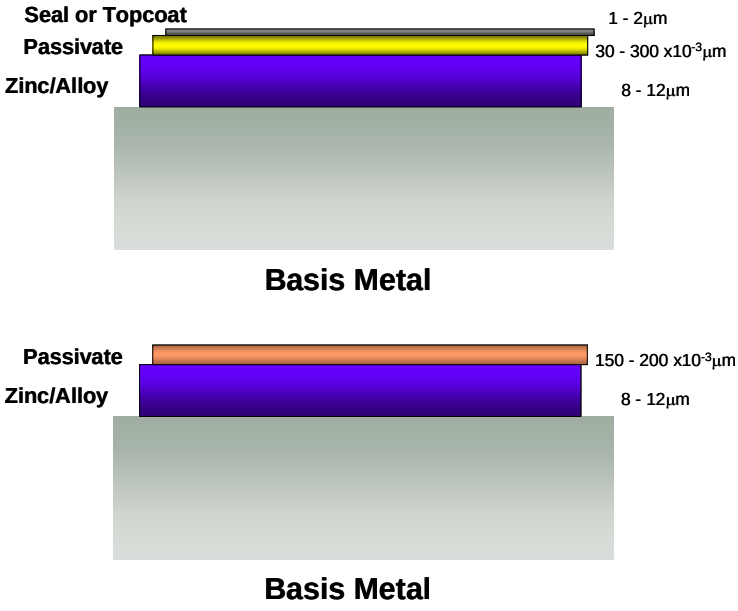


Figure 11. Typical Zn alloy plating with topcoat (top) and new Zn alloy plating system (bottom) (Jim Kloeckener, Enthone).

4.1. COTS products available

Briefings for commercial vendors are linked from Table 2. Table 3 summarizes the COTS products currently available.

Table 2. Vendor briefings; COTS products by vendor.

Company	Products	Notes
McDermid (Rob Berry, Mike Wyrostek)	❑ ZinKlad line of Cr^{6+}-free zinc finishes. ❑ Iridite NCP, Al non-chrome passivate	To manage the transition to non-Cr^{6+} products established ZinKlad Approved Plater (ZAP) program to ensure platers are qualified to provide the products. Iridite NCP better than Cr^{6+} except on 7075 and 2024 Al
Atotech (Craig Bishop)	❑ Interlox Al passivates: o Interlox 338, Cr^{3+} o Interlox 5704, 5705 non-Cr ❑ Uniprep iron phosphate paint pretreat for steel ❑ Non-Cr^{6+} Zn passivates ❑ Alkaline ZnNi	Interlox 338 equivalent to TCP but different formulation
Enthone (Jim Kloeckener)	❑ PermaShield processes: o Zincrolyte, Enthbrite Zn and Zn alloys o Permapass Cr^{6+}-free passivates o Enseal sealers	Co and nanomaterials in passivates Zn systems for threaded fasteners
Henkel (Bill Wittke)	❑ TriChrome: o Alodine T 5900 ❑ Non-Chrome o Alodine 5200, 5700 ❑ Touch up o Alodine 871 Touch-N-Prep ❑ Alodine 6000 SG sol-gel ❑ Alodine EC2	Alodine T5900 is a TCP license Alodine 6000 SG is a paint adhesion promoter licensed from Boeing Alodine EC^2 is a new hard oxide coating
Luster-On (Joe Ciejka)	❑ Aluminescent Cr^{3+} for Al and Mg ❑ Tri-Blue Cr^{3+} Zn passivates ❑ Zn alloy passivatesTri-Blue, Tridescent,Tri-Black	Aluminiscent is a TCP license
Metalast (Joe Radzvilowicz)	❑ Cr^{3+} TCP-HF for Al, Zn alloys, Cd, Mg ❑ TCP-HF for anodize seal	TCP-HF is a TCP license
Pantheon Chemical (Richard Buchi)	❑ PreKote for Al, Ti, Mg, steel, composites	Primer adhesion promoter
Sur-Tec International (Ray Lindeman)	❑ ChromitAl TCP for Al, IVD Al, ZnNi, SnZn, Cd	ChromitAl TCP is a TCP license
Taskem (Mike Kelly)	❑ Tasdip TR 184, 185, 186 + FT 190 for Zn and Zn alloys	

Table 3. Cr^{6+}-free COTS products (not necessarily matched to DoD requirements).

Application	Vendor	Product	Cr^{3+}	Non-Cr	Notes. Approvals
Al zincate pre-plate	Atotech	TriBond® II			Non-cyanide
Al zincate pre-plate	Atotech	AlumSeal® 650			
Al zincate pre-plate	MacDermid	Bondal CF			
Al zincate pre-plate	MacDermid	Metex 6811			
Passivation - Al pretreat	Atotech	Interlox® 338 (ZrCr)	✓		
Passivation - Al pretreat	Atotech	UniPrep® PP (borate ester)		✓	Pretreat for powder coat
Passivation - Al pretreat	Atotech	Interlox® 5704(ZrMn)		✓	
Passivation - Al pretreat	Atotech	Interlox 5705(ZrMg)		✓	
Passivation - Al pretreat	MacDermid	Iridite TCP	✓		NAVAIR TCP licensee
Passivation - Al pretreat	MacDermid	Iridite NCP		✓	NAVAIR NCP licensee
Passivation - Al pretreat	Pantheon	PreKote		✓	Paint adhesion promoter
Passivation - Al, Ti, Mg	Henkel	Alodine T5900, T5900 RTU	✓		
Passivation - Al, Ti, Mg	Henkel	Alodine 871 Touch-N-Prep	✓		Touch-up
Passivation - Al, Ti, Mg	Henkel	Alodine 5200, 5700		✓	Non-phosphate
Passivation - Al, Ti	Henkel	Alodine 6000 SG		✓	Sol-gel (Boegel); adhesion promoter
Passivation - Al	AC Tech	AC 130		✓	Sol-gel (Boegel); adhesion promoter
Passivation - Al	Henkel	Alodine EC[2]		✓	Electrodepos TiO_2
Passivation - Al, Mg	Luster-On	Aluminescent	✓		NAVAIR TCP licensee; QPL listed for Mil-DTL-5541F, Type II, Class 1A and 3
Passivation - Al	SurTec	ChromitAl TCP	✓		NAVAIR TCP licensee
Passivation - Al	Metalast	TCP-HF	✓		NAVAIR TCP licensee; also used as anodize seal
Passivation - Zn, Zn alloy	Atotech	Ecotri®	✓		
Passivation - ZnNi	Atotech	Tridur®	✓		
Passivation - Zn, Zn alloy	Atotech	Rodip® ZnX	✓		Co-free
Passivation - Zn	MacDermid	Tripass, Tripass ELV	✓		Clear, blue, iridescent, yellow, black
Passivation - Zn, Zn alloy	Enthone	Zinchrolyte	✓		Automotive company approved
Passivation - Zn, Zn alloy	Enthone	Perma Pass	✓		Nanoparticle, clear, blue, iridescent, yellow, black. Automotive company approved
Passivation - Zn, Zn alloy	Taskem	Trimate	✓		
Passivation - Zn (alk)	Taskem	Lanthane	✓		
Passivation - Zn, Zn alloy	Taskem	Finidip	✓		
Passivation - Zn, Zn alloy	Taskem	TR 175, 173	✓		Includes nanoparticles
Passivation - Zn, Zn alloy	Taskem	TC-HP, TC-SA	✓		Blue
Passivation - Zn, Zn alloy	Luster-On	Tri-blue, Tri-descent, Tri-black TZT	✓		
Passivation - Zn, Zn alloy	SurTec	Chromiting	✓		
Mg zincate pre-plate	MacDermid	Bondal Mg			Non-cyanide
Sn-Zn electroplate	Atotech	Reflectalloy® SnZn			Non-cyanide
Sn-Zn electroplate	MacDermid	Enviralloy SN			
Zn (12-16)Ni electroplate	Atotech	Reflectalloy® ZnA			Bright
Zn (12-16)Ni electroplate	Atotech	Reflectalloy® HD			Ductile
Zn (12-16)Ni electroplate	Atotech	Reflectalloy® TF			For subsequent organic coating
Zn-Ni	MacDermid	IsoBrite 570			Acid process
Zn (12-15)Ni electroplate	MacDermid	Enviralloy NI 12-15			Alkaline, for rack and barrel plating
Zn (8-12)Ni electroplate	MacDermid	Nyzin 1200			Alkaline
Zn-Ni	Enthone	Enthobrite CLZ 933, 941, 953			Acid, bright. Automotive company approved
Zn-Ni	Enthone	Enthobite NCZ			Alkaline non-cyanide; ductile and bright versions
Inorganic seal	Atotech	Rogard®			
Inorganic seal	MacDermid	JS 500, 600, 1000, 2000			
Inorganic seal	MacDermid	Ultraseal			
Inorganic seal	MacDermid	Hydroklad SI			
Organic seal	Atotech	Corrosil®			
Organic seal	MacDermid	Torque n' Tension			
Organic seal	MacDermid	HydroKlad			
Organic seal	MacDermid	Hydrolac			
Organic seal	MacDermid	Aqualac			
Organic/Inorganic seal	Enthone	Enseal			Various
Electroless Ni	Atotech	Nichem® 11			Pb and Cd-free
Electroless Ni	MacDermid	NiKlad ELV			Pb and Cd-free; low, medium, high phos
Electroless Ni	MacDermid	Niklad ELV LMO			Pb and Cd-free; organic stabilizers
Electroless Ni	Enthone	Enplate EN333LF			High phos, Pb-free
Electroless Ni	Enthone	Enplate EN-806			Mid phos, Cd and Pb-free
Electroless Ni/PTFE	MacDermid	Niklad ICE with ELV 811			High phos
Torque-tension modifier	Taskem	Finigard			Zn and Zn alloys

4.2. DoD approvals and specs

The various NAVAIR TCP licensees have applied for QPL listing. Specific items mentioned:

- Luster-On Aluminescent is QPL listed for Mil-DTL-5541F, Type II, Class 1A and 3
- Metalast TCP-HF has passed all QPL testing for MIL-DTL -81706-B and MIL 5541 for immersion and spray applications. QPL approved May 15, 2006

Of the various COTS products the majority are Cr^{3+} based, with a number licensed from the Navy's TCP process. In addition, a number of companies offer non-chrome products.

There are two adhesion promoters, PreKote and Alodine 6000 SC, that do not provide corrosion inhibition, but protect by improving the adhesion of the paint system. PreKote is authorized by Air Force TO (see Table 1).

4.3. Other approaches

Henkel (Bill Wittke): One new system on the market, Alodine EC^2, is a ceramic coating. This is an electrodeposited TiO_2 coating that provides a hard, impervious surface rather than a conversion coating (see Figure 12).

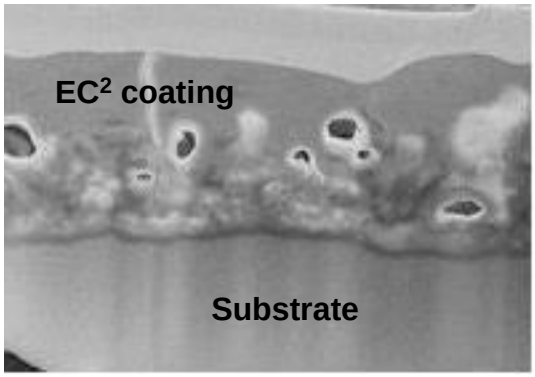


Figure 12. Alodine EC^2 ceramic coating (Bill Wittke, Henkel).

Electroplated Al with TCP (Gus Vallejo): A different approach is that used by AlumiPlate. Their electroplated Al coatings are used to replace Cd on the F-22, F-35, F-18 and F-16. Al + TCP outperforms Cd + Chromate. It also does well in G85 SO_2 salt fog tests. It was pointed out in the workshop that applying electroplated Al + Cr^{3+} passivate over 2024 and 7075 Al is one way to overcome the difficulty with passivating these alloys.

AlumiPlate specifications:

- Custom call outs by Lockheed Martin and Goodrich; Amphenol MIL-DTL-38999K; MIL-DTL-83488D and MIL-DTL-5541F
- MIL-DTL-38999L revision out for comment to include Class P for electroplated Al.

5. Experience with Chromate Alternatives

5.1. Issues with chromate alternatives at Boeing

***Steve Gaydos,* Boeing:** *Requirements for conversion coatings:*

- Non-Chrome Conversion Coating Shall Meet MIL-DTL-81706B, Type II for Class 1a and 3
- Applied by Immersion, Spray and Brush
  - One Tank Operation - No Heat
- Needs to Work on Other Aluminum Alloys Besides 2024, 7075, 6061
- Must Consistently Pass Monthly 1 Week Salt Spray Test on 2024 for Immersion Tank Operations
- Needs to Work with Existing Aluminum Prep Line
  - Must Be Compatible with Boeing Cleaners and Etches.

Boeing has worked on developing and qualifying Cr^{6+} alternatives since the early 1990's and so has evaluated the various generations of non-Cr^{6+} passivates. The early Sanchem 3300 (permanganate seal) and 3400 (silicate seal) and the Alodine 2000 cobalt conversion coatings were not implemented as they were qualified only for 1100 Al. Alodine Safegard CC 3000 was too complex.

Alodine 1132 Touch-&-Prep pens have been MIL-SPEC- qualified and added to the Boeing process spec. They contain Cr^{6+} but are much less hazardous to personnel than chromate brush applications. Boeing is beginning to work on qualifying Alodine 871 Cr^{3+} pens.

Boeing is working with the University of Missouri on Ce-based conversion coatings (see Section 2.4 above). Again, surface preparation is critical, and in addition Boeing needs a non-electrodeposited coating. Boeing is also working to qualify the COTS TCP products. So far corrosion resistance has been poor on 2024 panels. A new additive developed by NAVAIR is being tested in the COTS products.

In all cases the performance of non-Cr^{6+} products is strongly dependent on surface preparation. This was a common theme of the workshop.

Boeing has also found that citric acid is as good as nitric acid for passivating stainless steels and Boeing PS 13001 has been changed to allow citric acid.

5.2. Sol-gel coatings and anodizing at Boeing

***Joe Osborne*, Boeing:** Boeing has developed two non-Cr^{6+} processes for Al:

- Boric sulfuric acid anodizing (BSAA) to replace chromic acid anodizing under MIL-A-8625 Type I, Type IB, Type IC. Boeing requires a dilute Cr^{6+} seal. The process has been used on all Boeing commercial aircraft for the past 15 years with no failures, and has been tested successfully for the C-17. NAVAIR is planning MIL-A-8625 revisions to include it.

Issues: BSAA electrolyte is corrosive to pumps and components. It should not be used where liquid is likely to be trapped. Dissimilar metals may need to be masked.

- Sol-gel coatings provide excellent paint adhesion and can be formulated for different substrate and primer chemistries.

  Boegel (Figure 13) has a large number of Boeing specifications for bonding and paint system adhesion on Al, Ti, NI and stainless steels.

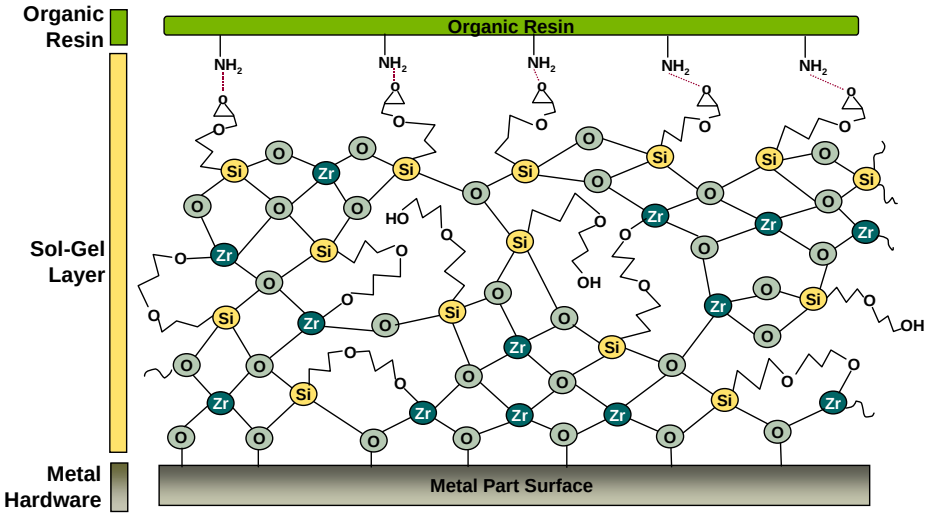


Figure 13. Boeing Boegel paint adhesion system (licensees Henkel, AC Tech, Socomor).

5.3. Coatings for fasteners

***Rick Delawder*, SWD, Inc.:** Most military fasteners are coated with Cd and chromated, except for vehicle fasteners, which are often Zn plated and chromated. However, the automotive industry primarily uses fasteners that are coated with an organic or inorganic system containing Zn flake. The coating method mostly used is dip-spin coating, in which the components are held in a basket, dipped into the solvent- or water-borne topcoat, removed and then spun at high speed to remove the excess.

In some cases the coating is applied directly to the fastener, while in others the fastener is Zn-coated first. Coatings can be applied by dip-spin, dipping or spraying. They can be organic- or inorganic-based. These coatings supply both corrosion protection and torque-tension. Products are supplied by many different vendors, including:

- Dorken MKS Systems
- The Magni Group
- Nippon Dacro Shamrock (Japan)
  - Metal Coatings International (North Am.)

- Dacral (Europe).

- Units Coatings (recently acquired by Atotech).

These types of coatings are not as good for fine fasteners since the coating can tend to fill the threads. For this reason they are not generally used below a size M6. In addition, because the fasteners are coated in a basket there is a possibility of nicking the threads.

Note: Dip-spin coating of fasteners as an alternative to Cd plating has been evaluated by the Army[3], and Magni 555 coating (previously called Dorrltech coating) was found to work well.

5.4. Corrosion protection of structural Mg alloys in automotive

***Wenyue Zheng*, National Resources Canada-CANMET:** Natural Resources Canada's CANMET Materials Technology Laboratory has been evaluating protection schemes for Mg alloys for automotive use under the US Automotive Materials Partnership (USAMP) program. They tested 20 different products including:

- Alodine 5200 with powder coat
- Keronite (early version), Tagnite, Anomag, Magoxid with or without topcoat
- Polyurea topcoat
- Hot wax coating
- Sol Gel coating
- MAGPASS+Topcoat.

Coatings that protected against general corrosion did not always protect against galvanic corrosion. Some of the metal-filled polymer coatings performed well in both overall corrosion and galvanic corrosion protection. For protection against stress-corrosion cracking the best results were with a conversion coat, followed by a powder coating and an anti-chipping topcoat.

To try to limit corrosion, they have developed an Al-Mg alloy, which creates far lower galvanic corrosion currents against steels.

They have begun to see very good results using cold spray Al coatings. This is an approach currently being evaluated in an ESTCP project headed by the Army Research Lab, and also supported by an F-35 Program Office contract with the Australian DSTO.

5.5. Panel Discussions – COTS Cr^{6+} alternatives

The automotive industry had no choice but to eliminate Cr^{6+} finishes, with the passage of the ELV regulations in Europe. RoHS extends this to most electrical equipment except large scale plant. However, even prior to the ELV rules, Cr^{6+} processes were coming under cost pressure. It was pointed out that conversion coatings are actually primarily Cr^{3+}, and therefore what is really needed is to find a route to get to that state without requiring an intermediate Cr^{6+} formulation.

Given that military and aircraft applications are exempt from the EU rules, the

question was asked as to DoD's incentive to change. There are several reasons:

- Carole Leblanc pointed out that being "green" is not just an environmental issue but also a health and safety imperative – the health and safety of our people is mission-critical.
- While we may be exempt from the rules we are not exempt from their effects, when they reduce the availability or increase the cost of processes that are no longer used elsewhere in the industry.

Specs and standards are needed for non-Cr^{6+} treatments. Most of the Cr^{3+} specs were developed for the automotive industry. For DoD use we may need to define different classes of treatments for non-Cr^{6+} processes. DoD and the aerospace industry have additional requirements beyond those in the automotive industry, especially for hydrogen embrittlement and fatigue. In addition DoD will have specific requirements for fasteners that will go beyond the requirements for the automotive industry.

Although most people are concentrating on the EU RoHS rules, there is concern that the China RoHS rules will pose greater difficulties. Partly this is because the rules are in Chinese with no official English translation, and partly because the rules, while based on the EU regulations, are not the same. China RoHS text can be found at

http://english.mofcom.gov.cn/aarticle/policyrelease/domesticpolicy/200605/20060502132549.html

In addition, although enforcement is still some years off, REACH could also have a significant impact on surface treatments. Although Co is not restricted under RoHS it could become an issue under REACH, depending on whether or not Co becomes a material of concern in the EU.

Major issues coming out of discussions:

- **If there is a choice of eliminating either the chromate conversion coating or the chromated primer, the material to eliminate is the primer since it contains 100x as much Cr^{6+} as in the conversion coating.** This is the approach adopted by F-35.
- ESOH issues arise not only when applying the finish, but also with waste disposal when overhauling.
- Non-Cr^{6+} treatments are much more sensitive to surface preparation than Cr^{6+}. If the surface is properly prepared they can work as well as, or in some cases better than chromate processes.
- There are still few treatments that work well for 7075 and 2024 Al.

6. Discussion of needs and COTS products

6.1. Breakout Session – Al and Mg alloys

Mg alloys:

- Current OEM process – Dow 7
- Current MRO process – Dow 19.

Tagnite and Keronite work well, and Tagnite + Rockhard paint finish performs very well on some components. However brush Tagnite is a very difficult process. (Note that in the May 2006 meeting it was pointed out that overhaul procedures require Tagnite to be stripped for NDI.)

Processes in development:

- Cold spray Al – ESTCP project being run by Vic Champage at the Army Research Lab (also a vehicle-related similar program mentioned by Wehyue Zheng, see Section 5.4)
- TCP looks promising for both Al and Mg
- Powder coat
- "Panther grip" (Northrop Grumman?) + powder coat
- Replace Mg by investment cast high strength Al – thinner section allows similar weight.

Al alloys:

- Tank dip processes required for components
- Paint or spray processes required for exteriors of aircraft and vehicles.

Requirements:

- Adhesion
  - Organic coatings, primers to Aluminum
- Corrosion protection
  - Most components are chromate converted, primed and painted
  - For some applications coating has to stand alone without additional paint – Class 3 for low electrical resistance for electrical cabinets, etc.
- Compatible with mixed metal assemblies
  - Minimizes masking and/or removal
  - Increasing need for adhesion of primers on composites.

Options:

- Alternatives
  - TCP (NAVAIR, 4 vendors)
  - NCP/Iridite TCP (not for all alloys)

- Boegel (3 vendors)
- Prekote.

- Application methods
  - All can be sprayed
  - All but Boegel can be used in immersion tanks.
- All require chromated primer when painted.

Needs:

- Need to understand how non-Cr^{6+} materials interact with cleaning and deoxidation
  - Issue of sensitivity of processes to surface prep
  - Interest in all alloys including LiAl and 7000 series Al.
- Interaction between non-Cr^{6+} inhibitors and non-Cr^{6+} coatings (primers). How can we get a satisfactory non-Cr^{6+} system?
- NAVAIR is able to make TCP work well, but other users do not appear to be able to. This appears to be a situation requiring better technology transfer and/or modification of the chemistry to make the process more robust.

6.2. Breakout Session – Steels

Army requirements for vehicles:

- Zn phosphate or chromated wash primer + CARC (Chemical Agent Resistant Coating) epoxy primers and polyurethane topcoats
- A non-Cr^{6+} wash primer is needed for adhesion promotion for small shops, depots, and re-work
  - Must be a robust process with a large process window
  - The whole process must fit together
  - A Cr^{3+} spray or wipe process is needed. Metalast may have such a process
  - Coatings similar to the Zn-flake filled polymers might be an option, but would require a low temperature cure.

6.3. Breakout Session – Coatings

Coatings:

- The various TCP licensed coatings should all work well for Al coatings (IVD Al, AlumiPlate) under the MIL-DTL-81706, Type II spec.
  - Henkel T5900
  - CST SurTec ChromitAl
  - Luster-On Aluminescent
  - Metalast TCP-HF.
- The following also work well on Al coatings

- Iridite NCP (Meets Type II, Class 3)
- Alodine 2000 (TD-3095 Seal)
- Sanchem 3300 + 3400 Permanganate – Silicate Process.

- The following work well for paint adhesion (but not stand-alone corrosion resistance):
  - Boegel and its licensees
    - Alodine 6000
    - AC 130
  - Alodine 5700
  - PreKote.
- Anodize or oxide coatings:
  - Type IC, Boric Sulfuric Acid Anodize (BSAA)
  - Type II, Sulfuric Acid Anodize (SAA)
  - Type IIB, Thin Film Sulfuric Acid Anodize (TSAA)
  - Henkel EC^2 oxide coating (Note: this is a new product with little independent technical data).
- Touch-up coatings
  - There is a need for a version that can be used for touch-up. Luster-On has developed a thixotropic version of their product for touch up, but it is not yet approved.

Issues with MIL-DTL-5541: A major difficulty was brought up that prevents the adoption of chromate alternatives for those alloys for which alternatives work:

- MIL-DTL-5541 still requires a monthly salt spray test on 2024 Al, whether or not the vendor actually treats 2024
- Since most alternatives do not work well on 2024 this prevents the adoption of any alternative for other alloys
- The spec should be changed to read more like MIL-A-8625 for anodize:
  - Corrosion test with 2024 or predominant alloy used that month.

Steve Brown ***(NAVAIR PAX)*** followed up on this immediately and provided the response shown in the link. MIL-DTL-5541 Section 4.2.2 does allow monthly tests of either 2024 or of each alloy and temper treated during the month. Thus if a vendor never treats 2024 he can carry out monthly testing on every other alloy he treats. Of course, this means testing many more specimens.

Zn and Zn alloy coatings:

This is a mature technology with many Cr^{6+}-free COTS alternatives (see Table 3). The various generations of trivalent coatings were described by Mike Kelly (Section 2.4). DoD users may need to work with suppliers to measure missing data or develop alternatives that better match DoD needs.

There was a consensus that DoD should specify non-Co versions of these treatments to avoid any future regulation of Co.

Additional discussions on implementing alternatives are included in Section 7.3.

7. Transitioning to Production in DoD

7.1. Modern surface finishing facilities design

Peter Gallerani*, *Integrated Technologies: Simply eliminating or reducing the use of hazardous materials does not in itself reduce the environmental and worker health impact of a finishing shop. Even where we must continue to use hazardous materials, proper facility design can greatly reduce ESOH problems as well as lowering capital and running costs, whether the facilities are being upgraded or built from scratch.

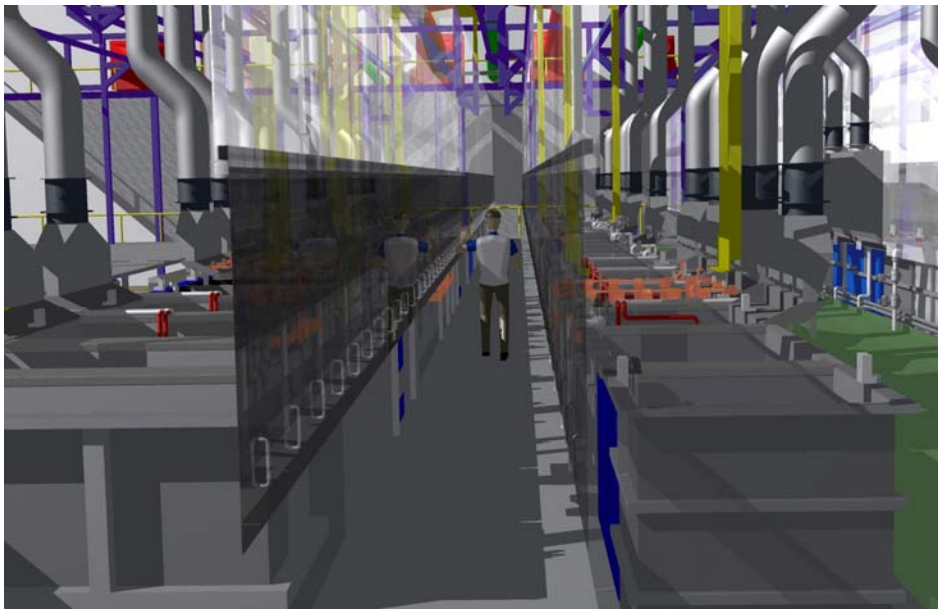


Figure 14. Robins AFB plating shop design showing integrated hazard and energy management including front and back shields and improved push-pull ventilation.

The newly designed Robins AFB plating shop was taken as an example (see Figure 14). The design incorporates many features to reduce labor and energy cost while improving worker safety and reducing emissions:

- Process automation for process control and minimum operator handling
- Bulkhead with automatic doors between the setup area and the process area. This allows the two areas to be air conditioned separately and minimizes operator time in the process area
- Front and back sliding shields on the tanks to shield the operators from hazardous chemical fumes and splashes, and minimal tank obstructions to make the push-pull ventilation more efficient

- Energy efficient ductwork
- Automated tank covers where needed
- Lean maintenance with alternating operator and maintenance aisles
- Use of corrosion resistant materials for long plant life
- Under shop spill prevention and capture to protect the soil and groundwater
- Recycling of treated wastewater.

7.2. ESTCP hydrogen embrittlement testing

***Steve Gaydos*, Boeing:** It takes a very small amount of hydrogen incorporated into high strength steels or other high strength alloys to cause them to become brittle and fail catastrophically. One of the primary issues with changing coatings and surface treatments is therefore ensuring that the alternatives do not cause hydrogen embrittlement of high strength alloys (due to processing) or environmental embrittlement (re-embrittlement, due to attack by salt water or fluids).

While standard tests have been in use for many years there is no agreement on specimen design, test methods, or even at what strength level embrittlement occurs. Therefore there is no agreement on what is safe. The same material that is accepted by one agency may be rejected by another on the basis of different test designs. ESTCP is funding a project involving Boeing, ARL and others to put embrittlement testing onto a sound engineering basis.

The standard test is ASTM F519, which requires the specimen to survive 200 hours at 75% of notch tensile strength. An alternative step load procedure is also permitted, but not widely accepted. The 200 hour requirement is a big problem for vendors since the test is used for QA but getting the results takes a week. There are a great many different specimen shapes and sizes, some more sensitive than others.

We need a simplified and standard test method to re-qualify all maintenance chemicals, plating and surface treating, and test new ones. The potential for cost savings is significant.

Round robin testing is being carried out between Boeing St Louis, Boeing Mesa and the Army Research Lab. Organizations wishing to become involved should contact Steve Gaydos.

7.3. Panel Discussion – Running a non-Cr^{6+} line

Issues with putting new lines into DoD depots:

- Although some DoD depots are constructing new processing lines, they are still having to invest in old lines to keep legacy systems maintained. This means that we are in the position of having to invest in technologies that we advocate replacing.
- Putting new technologies into DoD depots is a major operation with a steep learning curve, especially because the process control and cleanliness requirements are a great deal tighter.
- The cycle time for investments in new equipment is 5 years – this is far too long.

- We need to spend money on instituting best practices. Emerging Contaminants is interested in doing this. Their aim is **risk management** not necessarily process elimination.
- Jeff Hannapel pointed out that companies that participated in the ill-fated EPA Common Sense Initiative found it improved their bottom line; green processing can be a significant cost saving.
- Vendors are driven by customer demand and will not make major investments in cleaner technologies until there is a customer base. On the other hand, some companies will identify processes with good long term potential and then invest in small scale units to learn how to run them so as to be ready when the demand materializes.

The following notes are from the Breakout Session 3 – Coatings:

Issues with non-Cr^{6+} finishing systems:

- These processes are less forgiving than Cr^{6+}
  - Pay attention to the details
  - Tighter process control, including chemistry, temperature and pH
  - Rinse water quality is extremely critical for rinsing prior to application.
- Contamination can be a very serious problem
  - Replace old tanks, liners, pumps and piping.
- Chemicals costs are higher
  - Need methods for extending bath life (done for Zn alloys).
- At present non-Cr^{6+} pretreats do not work well with non-Cr^{6+} primers. We need to understand these systems to make them work together. Note: SERDP has solicited proposals in this area for FY2008, so a solution to this problem may be developed in the next 3 years.
- Reliability of no-Cr^{6+} treatments:
  - Zn treatments are fully commercial and reliable
  - Al treatments are reliable except for 2024
  - Touch up of Zn alloys can be done with brush plating
  - Touch up of Al coatings (IVD, AlumiPlate) is less certain
    - Metalast brushable treatment
    - Sur-Tec pen applicators for ChromitAl
    - Henkel is qualifying non-Cr^{6+} Alodine 871 Touch-N-Prep pens.

Replacement of chromated Cd:

- AlumiPlate is a drop-in under MIL-DTL-81706 Type II. Non-Cr^{6+} primers appear to work very well on this. Note: Cannot be done in standard tank; requires fully enclosed non-aqueous system with inert gas fill.
- ZnNi coatings have COTS products available but still require all the detailed testing needed for military and aerospace applications – fatigue, embrittlement, galvanic corrosion, paintability, etc.

7.4. Introducing products to DoD using OSD's on-line tool

***Robert Herron*, US Army RDE Command:** DoD has a new on-line tool for easing the introduction of corrosion prevention products. The tool can be found at www.dodcorrosionexchange.org (see Figure 15).

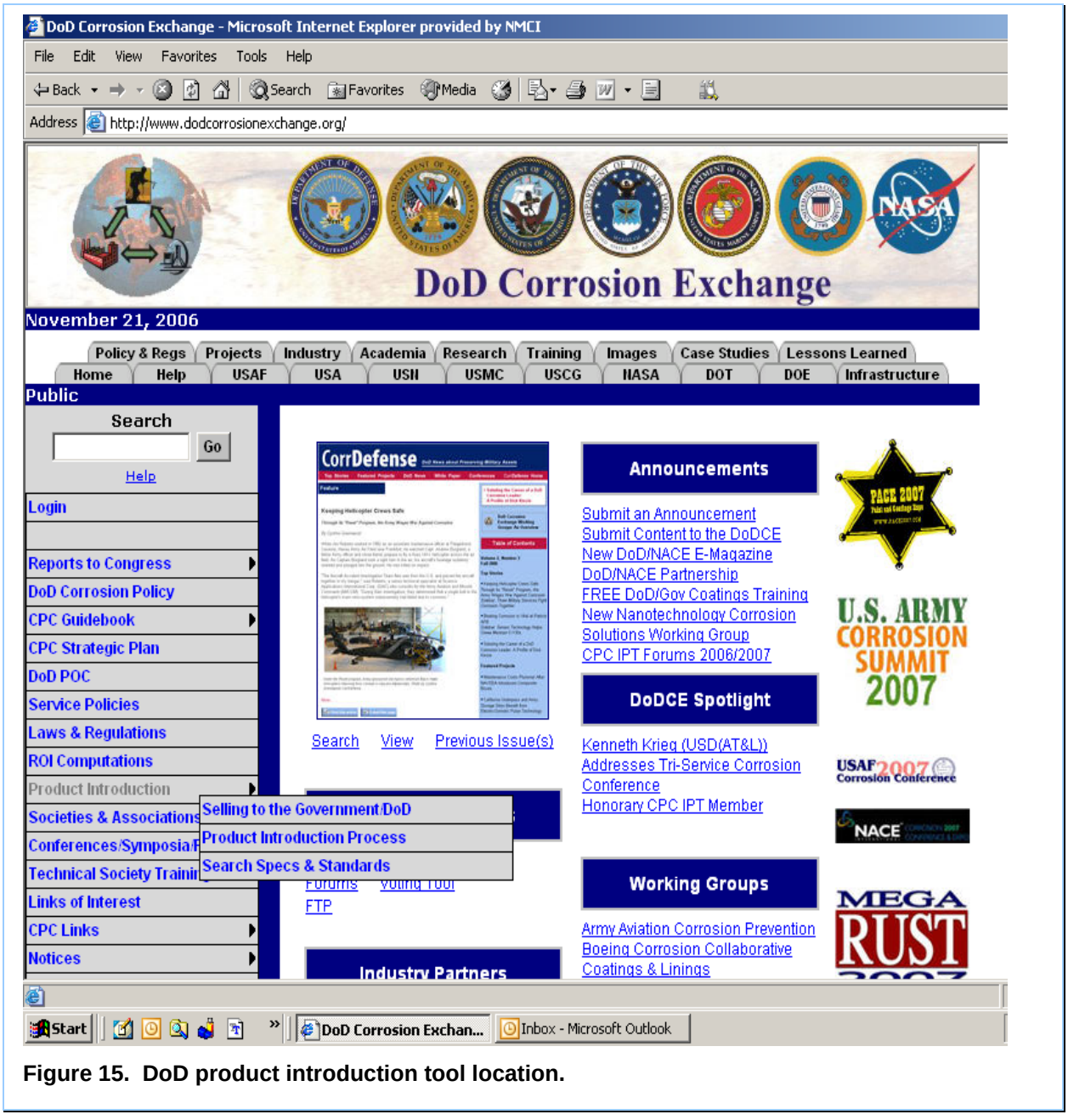


Figure 15. DoD product introduction tool location.

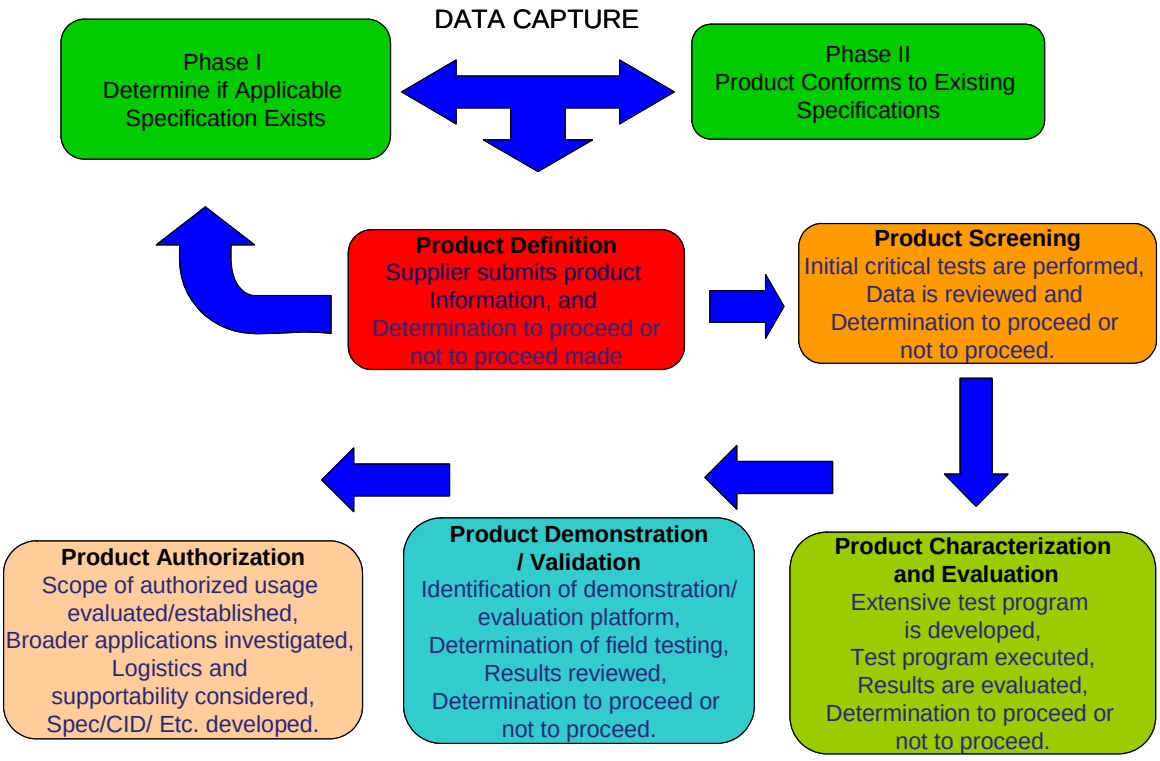


Figure 16. The product introduction process.

A flow diagram of the product introduction process is shown in Figure 16. The tool takes vendors through the product introduction process, allowing them to input information on their product and laying out the course that must be taken to gain approval.

7.5. Panel Discussion – Getting alternatives into production

***Robert Herron*, US Army RDE Command:** Vendors are often frustrated by the slowness of growth in demand for products even after they have been qualified. However, there is a big difference between Qualification, Authorization and Implementation (see Table 4).

Table 4. Requirements for qualification, authorization and implementation.

Qualification	Authorization	Implementation
Meets Minimum Specified Requirements (QPL)	Approves Product's use for a specific application	Gets product into actual use in DoD organizations
Acceptable for where specification is approved for use in Tech Orders, Manuals, Drawings, etc.	Used for new products or different applications of existing products	Applicable Engineering Authority Sign-off ❑ E.g. Materials, Structures, Subsystems, Systems, Aeromechanics, Depot ❑ Program: Engineering/ Logistics/Original Equipment Manufacturer
Does not insure/promise procurement ❑ Must follow standard GSA, DLA, etc. procurement processes	Path to Authorization ❑ Lab testing ❑ Field prototype ❑ Verification/Validation ❑ Data analysis and use envelope definition	Create or Revise specs and other documentation ❑ Local drawings/Tech orders/ MIL/AMS/etc.)
Qualification does not automatically mean Product Authorization	Authorization letter, Local Process Spec, Tech Order or Manual, etc. Issued	Revise Relevant Control Manuals Supply, NSN, Order Quantities, etc.

There is a Defense Standardization Program (DSP), for qualification and approval, although the details for implementation of new products vary from one DoD agency to another and one application to another. The first major step for products such as chromate conversion coating alternative chemistries is to get onto the Qualified Products List (QPL)[4]. However, all this means is that the product meets minimum DoD specifications. This does not imply that anyone will actually authorize its use. Authorization is done by the cognizant engineers and program managers, who may not be sure that it meets the performance requirements for their particular weapons system. Therefore, authorization may require a great deal of testing. Authorization means that the product can be used on a weapons system, but it does not require that it will be used. implementation requires that a specific depot, OEM or other organization change their overhaul manuals, drawings or contracts (often very expensive and time-consuming) and become able to use the product. They must obtain the necessary equipment, chemicals and training, and it is in the vendor's interest to ensure that the product is used correctly and for the right applications.

Qualification is a legal process, involving legal requirements for the supplier and the government. There are several types of qualifications under the DSP:

- **Formal Qualification (Qualified Products List) (SD-20, SD-6)**
  - Performance (PRF) or Detail (DTL) Specification
  - In General, Most Specs do not have Qualified Products Lists (QPL).
- **Non Government Standard (NGS) (SD-9)**
  - Can be Adopted if Technically Adequate for DoD Needs
  - In General, Most NGS do not have an associated QPL
- **Commercial Item Description(CID) (SD-2, SD-5)**
  - For Existing Suitable Commercial Products where an appropriate NGS Does Not Exist

- **Non Developmental Item (NDI) (FAR, SD-2)**
- **First Article Inspection (Acceptance Testing) (FAR)**
- **Purchase Description (SD-2, SD-5)**
    - When preparation and maintenance of a specification is not cost effective
    - For small dollar purchases under $25,000
    - For one-time buys.

Many of the chromate alternatives are (or could be) included in a QPL. In fact, most of the COTS TCP products fall into this category and have been submitted for inclusion in QPL 81706, which is the QPL for MIL DTL 5541. At the present time there are no Cr^{3+} products in this QPL. This is because there have been a number of applications and the QPL will not be updated until the testing is complete. It will then be updated with all the new products at once. Most of these products will ultimately be available through the Defense Logistics Agency (DLA).

Note: Even having a product on the QPL does not guarantee that DLA will purchase it. DLA only purchases products for which there is a demand. Under these circumstances the product must be bought by each individual organization. Since many organizations cannot easily purchase these types of products except through the DLA this can create a Catch-22.

What is a QPL and how do you get onto it?

- A QPL is a list of products (and their manufacturing site) which have been tested and certified to meet the minimum specification requirements
- A QPL allows the manufacturer to provide and the purchaser to obtain satisfactory pre-contractual evidence that a product meets specification requirements
- QPLs simplify the procurement process and reduce acquisition/ procurement lead time
- QPLs reduce test costs by minimizing redundant, long, and expensive tests
- QPLs provide a source for continuous availability of reliable products.

What does a QPL **not** do?

- QPL listing does NOT imply or grant authorization for implementation
- Even after QPL listing, authorization to implement may be required by the cognizant engineering authority for specific DoD applications. This means that even if a product is on the QPL, approval authorities at a depot or DoD agency are not required to use it, and they may still require additional testing.
- Does not allow vendor to change manufacturing methods or materials. Changes will require requalification.
- Does not relieve the OEM or supplier of contractual obligation to deliver items meeting all specification requirements.
- Does not guarantee acceptability under contract
- Does not waive any requirements for inspections

- Does not waive any requirements for maintaining quality control measures. Products can be removed from the QPL if they cease to meet the requirements or if the requirements change and the product cannot continue to meet them.

US Coast Guard approach to approvals:

The USCG follows a similar process to the above, (see the two documents at the left).

Cognizant engineering authorities:

- Naval Aircraft – NAVAIR, Putaxent River, MD
- Naval Vessels – NAVSEA
- Army
  - Aircraft and helicopters – AMCOM, Huntville, AL
  - Vehicles – Tank Automotive Command (TACOM), Warren, MI.
- Air Force – Air Logistics Centers
  - Landing gear – Ogden ALC, Hill AFB, UT
  - Airframe, skin – Warner Robins ALC, Robins AFB, GA
  - Engines – Oklahoma City ALC, Tinker AFB, OK.
- US Coast Guard
  - Aircraft – Product line managers.

Information sources:

- DoD Specifications and Standards can be found on ASSIST (http://assist.daps.dla.mil/online/start/)
- Documents of various kinds can be searched for at http://www.assistdocs.com/search/search_basic.cfm.
- DoD Corrosion Exchange and DoD introduction tool www.dodcorrosionexchange.org.
- Standardization documents (DSP, SD-6, SD-20, etc.) http://www.dsp.dla.mil/APP_UIL/content/documents/sds.htm#SD-6
- SERDP and ESTCP program offices http://www.serdp.org/ ; http://www.estcp.org/
- DoD Office of Emerging Contaminants https://www.denix.osd.mil/denix/Public/Library/MERIT/merit.html
- HCAT engineering data and reports http://www.materialoptions.com ; http://www.hazmat-alternatives.com

8. Action items

There was one major action item for completion after the meeting, and several smaller items:

1. **Users agreed to supply their requirements for various applications, and vendors to identify COTS products that meet those requirements, match against commercial specs, and provide supporting data if it can be made available.**
2. Information needed from suppliers on wipe processes that might replace wash primers. To be sent to John Beatty, ARL.
3. NAVAIR needs to transfer information to vendors on how to make TCP work well in production. Several commercial users noted that COTS versions do not seem to work as well as the NAVAIR lab process does.
4. Specify requirements for Zn alloy coatings beyond the 96 hr B117 requirement. This will be done in completing Item 1.
5. Change MIL-DTL-5541 to eliminate requirement for monthly testing on 2024 Al. Steve Brown followed up on this, see Section 6.3.

To complete Action Item 1, below (blue paper clip) is an Excel spreadsheet to be loaded with user requirements and COTS products. The data will then be put into a form in which users can readily see which COTS products best match their applications, what performance data is available, and what additional testing may be needed. This database matching COTS products with requirements will be issued as a document or web database.

APPENDIX 1. ATTENDEES

Attendees and their contact information are provided in the file below. Double click on the blue paper clip.

APPENDIX 2. VENDOR LITERATURE

1. **Atotech information**
2. **Enthone information**
3. **Luster-On information**
4. **MacDermid information**
5. **Pantheon Chemical information**
6. **Taskem information**

APPENDIX 3. MIL SPECS

1. MIL-A-8625F – Anodization of Al
2. MIL-DTL-5541 - Chromate conversion of Al
3. MIL-DTL-81706 – Chromate conversion of Al
4. MIL-DTL-83488B – Al coatings
5. MIL-STD-870B – Cd plating

REFERENCES

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[2] MERIT URL: https://www.denix.osd.mil/denix/Public/Library/MERIT/merit.html

[3] G. Shaw, "The Nuts and Bolts of Cadmium Plating Alternatives: A Study on the Long Term Performance Characteristics by the US Army", Automotive Finishing Online, http://www.afonline.com/articles/99sp01.html

[4] "DoD Corrosion Prevention and Mitigation Strategic Plan", http://www.dodcorrosionexchange.org/References/Files/DoD%20Strategic%20Plan%20for%20Corrosion%20Prevention%20and%20Mitigation.pdf