U.S. Army Toxic Metal Reduction Program: Eliminating Cr(VI) and Cd in Army Surface Finishing

For ASETSDefense
6 December 2016

Erik Hangeland
Program Director,
RDECOM Environmental Technology Acquisition Program
<table>
<thead>
<tr>
<th>Process</th>
<th>Specification</th>
<th>Hazardous Chemicals</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum Conversion Coating</td>
<td>MIL-C-5541-E, MIL-DTL-81706B</td>
<td>Sodium Dichromate</td>
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<tr>
<td>Aluminum Anodize</td>
<td>MIL-A-8625F Type I and IB</td>
<td>Chromic Acid, Sodium Dichromate, Chromium Trioxide</td>
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<tr>
<td>Cadmium Brush Plate</td>
<td>MIL-STD-865C</td>
<td>Cadmium Special, Cadmium Alkaline, Cadmium Acid</td>
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<tr>
<td>Cadmium Plating</td>
<td>SAE AMS-QQ-P-416B Type II</td>
<td>Cadmium Oxide, Sodium Cyanide, Cadmium, Nickel Chloride, Iridite</td>
</tr>
<tr>
<td>Hard Chrome Plate</td>
<td>SAE AMS-QQ-C-320</td>
<td>Chromic Acid</td>
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<tr>
<td>Copper Plating</td>
<td>ASTM 2418F</td>
<td>Copper Cyanide, Sodium Cyanide, Sodium Dichromate</td>
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<tr>
<td>Electroless Nickel</td>
<td>AMS2404F</td>
<td>Nickel Chloride</td>
</tr>
<tr>
<td>Magnesium Anodize - Conversion Coating</td>
<td>AMS-M-3171 Type III, IV, VI</td>
<td>Chromic Acid, Sodium Dichromate</td>
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<tr>
<td>Nickel Plating</td>
<td>SAE AMS QQ-N-290</td>
<td>Nickel Chloride, Nickel Sulfate, Nickel Sulfamate</td>
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<tr>
<td>Passivate</td>
<td>SAE AMS 2700B</td>
<td>Sodium Dichromate</td>
</tr>
<tr>
<td>Phosphate</td>
<td>MIL-DTL-16232G TT-C-490, Type I</td>
<td>Chromium Trioxide, Chromic Acid</td>
</tr>
<tr>
<td>Silver Plating</td>
<td>ASTM B700-97</td>
<td>Potassium Cyanide, Silver Cyanide</td>
</tr>
<tr>
<td>Wash Primer</td>
<td>DOD-P-15328 TT-C-490F</td>
<td>Zinc chromate</td>
</tr>
</tbody>
</table>
Metal Plating Projects

Total Army Usage in Plating
Total Cr(VI): 10,000 lbs/yr
Total Cd: 1,270 lbs/yr
Total CN: 80 lbs/yr

- **Cadmium Plating**
  - Cadmium Electroplating
  - Cadmium Brush Plating
  - Cadmium Plate Sealing

- **Chrome Plating**
  - Chrome Plate Stripping
  - Hard Chrome Electroplating

- **Other Metal Plating**
  - Copper Plating
  - Copper Plate Stripping
  - Silver Plating
  - Silver Plate Stripping
  - Nickel Electroplating
  - Electroless Nickel

- **Chemicals Targeted for Replacement**
  - Chromic Acid
  - Sodium Dichromate

- **Surface Finishing Processes Important to the Army**
  - Copper and Sodium Cyanide
  - Chromium Acid
  - Silver Cyanide
  - Potassium Cyanide
  - Nickel Chloride and Sulfate
  - Nickel Chloride

- **Fasteners, Electrical Connectors, Components**
  - Corrosion Resistant Steels
  - Zinc-Nickel or High Purity Aluminum Plating
  - Trivalent Chromium Electroplating
  - Cold Spray with Nickel or Carbide-Nickel-Chrome Powders
  - TBD, Likely Cold Spray or Warm Spray
  - Aqueous Solutions of Copper and Silver Salts

- **NLOS Plating/High Throughput**

- **LOS Plating – Steel/Ground Vehicles**
  - LOS Plating – Non-Ferrous Substrates
  - LOS Plating – Other Niche Applications

- **Gun Barrel Inner Diameters**
  - Trivalent Chromium with Reverse Current Process
  - Cold Spray with Nickel or Carbide-Nickel-Chrome Powders
  - Cold Spray with Tungsten-Tantalum Powders

- **No projects planned**

**Total Army Usage**
- Total Cr(VI): 10,000 lbs/yr
- Total Cd: 1,270 lbs/yr
- Total CN: 80 lbs/yr
Total Army Usage in Pretreatments and Finishes
Total Cr(VI): 140,000+ lbs/yr

Pretreatments
- Wash Priming
- Phosphating
- Conversion Coating

Anodizing
- Magnesium Anodizing
- Aluminum Anodizing
- Anodize Stripping
- Hard Anodize Sealing

Sealing
- Zinc Plate Sealing
- Ion Vapor Deposition Alum. Sealing
- Black Oxide Sealing

Finishes
- Steel Passivating

Chemicals
- Sodium Dichromate, Chromic Acid
- Sodium Dichromate, Trivalent Chromium
- Sodium Dichromate, Various Chromate Salts

Surface Finishing Processes Important to the Army

Chemicals Targeted for Replacement

Manganese/Fluoride, Zirconium or Silane-based Products
- Non-Chemical Pretreatment
- Laser Structuring
- Zirconium Oxide
- Aircraft Parts Spray and Immersion (CCAD)
- Flyable Aircraft Spray (1109th TASMG)

Potential Alternative Technologies
- Trivalent Chromium, Zirconium or Manganese/Fluoride
- Trivalent Chromium
- Trivalent Chromium
- TBD, May Require Reformulation

Laser Structuring
- Zirconium Oxide
- Aircraft Parts Spray and Immersion (CCAD)
- Flyable Aircraft Spray (1109th TASMG)
ETAP supports SERDP/ESTCP objective to reduce Cr(VI) and Cd use and emissions at DoD Depots by 90% in 5 years

Current TMR projects will eliminate 88% of total Cr(VI) and Cd at LEAD
- FY18 cadmium brush plating project to eliminate another 10%

Where to go from here?
- Collect high resolution data at remaining Army depots
- Partner with ESTCP to fill remaining technology gaps

Table 4. Process Cd and Cr\(^{6+}\) Usage (Noblis, 2016)

<table>
<thead>
<tr>
<th>Process</th>
<th>Contains</th>
<th>Ibs Product</th>
<th>Ibs Species</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromated Primers - Wash Primers</td>
<td>Cr(^{6+})</td>
<td>6735</td>
<td>606</td>
</tr>
<tr>
<td>Chromate Conversion Coatings - Tank</td>
<td>Cr(^{6+})</td>
<td>2945</td>
<td>412</td>
</tr>
<tr>
<td>Cadmium Brush Plating</td>
<td>Cd</td>
<td>476</td>
<td>119</td>
</tr>
<tr>
<td>Specialty Coatings - CHOShield</td>
<td>Cr(^{6+})</td>
<td>273</td>
<td>10</td>
</tr>
<tr>
<td>Chromated Primers - Other</td>
<td>Cr(^{6+})</td>
<td>63</td>
<td>9</td>
</tr>
<tr>
<td>Chromate Sealer</td>
<td>Cr(^{6+})</td>
<td>405</td>
<td>8</td>
</tr>
<tr>
<td>Chromate Conversion Coatings - Touch-up Pens</td>
<td>Cr(^{6+})</td>
<td>60</td>
<td>1.8</td>
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<tr>
<td>Specialty Coatings - Silk Screen Red</td>
<td>Cd</td>
<td>0.58</td>
<td>0.01</td>
</tr>
<tr>
<td><strong>Total All</strong></td>
<td></td>
<td>10957</td>
<td>1166</td>
</tr>
<tr>
<td><strong>Total Cd</strong></td>
<td></td>
<td>477</td>
<td>119</td>
</tr>
<tr>
<td><strong>Total Cr</strong></td>
<td></td>
<td>10481</td>
<td>1047</td>
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</tbody>
</table>

Current TMR projects will eliminate 98% of Cr(VI) at LEAD
### CCAD Plating Shop Tanks Currently Containing Cr6+

<table>
<thead>
<tr>
<th>Line</th>
<th>Process</th>
<th>Num Tanks</th>
<th>% Tanks</th>
</tr>
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<tbody>
<tr>
<td>A</td>
<td>Black Oxide Seal</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>B</td>
<td>Manganese Phosphate</td>
<td>1</td>
<td>2</td>
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<td>G</td>
<td>Al Conversion Coat</td>
<td>3</td>
<td>8</td>
</tr>
<tr>
<td>H</td>
<td>Copper Strip</td>
<td>2</td>
<td>5</td>
</tr>
<tr>
<td>L</td>
<td>Stainless Steel Passivation</td>
<td>6</td>
<td>15</td>
</tr>
<tr>
<td>N-P</td>
<td>Chrome Plating</td>
<td>11</td>
<td>28</td>
</tr>
<tr>
<td>Q</td>
<td>Magnesium Conversion Coat</td>
<td>5</td>
<td>13</td>
</tr>
<tr>
<td>R</td>
<td>Cadmium Sealer</td>
<td>3</td>
<td>8</td>
</tr>
<tr>
<td>T</td>
<td>Chromic Acid Anodize</td>
<td>4</td>
<td>10</td>
</tr>
<tr>
<td>T</td>
<td>Anodize Strip</td>
<td>2</td>
<td>5</td>
</tr>
<tr>
<td>T</td>
<td>Hard Coat Anodize Seal</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td></td>
<td><strong>39</strong></td>
<td><strong>100</strong></td>
</tr>
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### CCAD Plating Shop Tanks Containing Cr6+ if Existing Projects are 100% Successful

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<td></td>
<td><strong>39</strong></td>
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</tr>
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</table>
Objective: Eliminate Cr(VI) in multi-metal spray-on pretreatment applications (alternative to wash primer)

Magnitude of impact:
- Reduce Cr(VI) by 24K lbs/year, volatile organic compounds (VOCs) by 2.4M lbs/year
- Violation of VOC emission limits could restrict maintenance
- Cancellation of DOD-P-15328 planned for September 2017

Intended end product: Validated Cr(VI) spray applied chemical pretreatments for multi-metal applications per TT-C-490F

Technology:
- Commercially available metal pretreatment technologies on multiple substrates and mixed metal assemblies added to QPD
  1. Bonderite 7400 (phosphoric acid, hexafluorotitanic acid, Mn)
  2. Zircobond 4200 (zirconium immersion chemistry)
  3. Oxsilan 9810/2 (organo-silane polymers)

Weapon systems impacted: All systems currently using DOD-P-15328 chromated wash primer (ground vehicles, combat service support equipment and aviation/missile systems)

Transition Path: TT-C-490F Qualified Product Database

POC: Jack Kelley, ARL, john.v.kelley8.civ@mail.mil

IPT: ARL, Letterkenny Army Depot (LEAD), Anniston Army Depot (ANAD), Red River Army Depot (RRAD), Henkel, PPG

- FY12: Laboratory testing
- FY13: Down-selection/Outdoor testing
- FY15: Demo at LEAD, implementation through TT-C-490 QPD
- FY17: Demo at ANAD
Objective: Eliminate Cr(VI) in conversion coatings (CC)

Magnitude of impact:
- Eliminate 100K lbs/yr of Cr(VI) in Al CC, 6M pounds of CARC waste
- Consolidated ferrous and non-ferrous pretreatment line

Intended end product: Multiple approved Cr(VI)-free CCs for aircraft and Ground Support Equipment (GSE) (multi-metal and composites), application by spray and immersion

Technology: Assess commercially available Al pretreatments
- Aviation: CCAD, TASMG, Corrosion Repair Facility
  - Spray/immersion: Henkel Bonderite MN-T-5700, PPG Industries 11-TGL-27
- GSE (immersion): ANAD, LEAD, Tobyhanna Army Depot
  - PPG X-Bond 4000 (Zr), Henkel Bonderite MN-T-5200
- Leverage: ESTCP (LEAD) and USMC - Albany demos

Weapon systems impacted: All tactical equipment that requires CARC

Transition Path: TT-C-490, MIL-DTL-53072, MIL-DTL-5541, MIL-DTL-81706

POC: Fred Lafferman, ARL, fred.lafferman.civ@mail.mil

IPT: AMCOM, AMRDEC, AED, TACOM, LEAD, RRAD, CCAD, TASMG, PPG Ind., Henkel

FY14
- Select candidates
- Laboratory testing

FY15
- Downselect products

FY16
- Demo at CCAD (spray)
- Demo at CCAD, ANAD (immersion)

FY17
- Demo at TASMG (spray)
- Observe demo on end items

FY19
- Implementation
**Objective:** Eliminate Cr(VI) in aluminum anodizing, stripping and sealing

**Magnitude of impact:**
- CCAD anodize and anodize stripping baths use:
  - Anodize: 2300 gallon tank with 1500 lbs. chromic acid, added as needed (500 lbs. added in 2010-2011)
  - Stripping: 1 process line, 2050 lbs of dry chromic acid
- International regulation impact on supply chain (REACH)

**Intended end product:** 1) Validated Cr(VI)-free anodizing process in production environment, 2) validated Cr(VI) free chemical stripper for all forms of anodized aluminum

**Technology:** Two anodize technologies, Cr(VI)-free strippers
- 1. Sikorsky: Tartaric Sulfuric Acid Anodizing
- 2. NAVAIR: Thin Film Sulfuric Acid Anodizing process
- 3. Cr(VI)-free strippers for legacy, alternative anodize (ARL)

**Weapon systems impacted:** All aircraft maintained at CCAD, including other Service aircraft

**Transition Path:** CCAD process standard, MIL-A-8625, MEO added to DMWRs

**POC:** Scott Howison, AMCOM, stephen.s.howison.civ@mail.mil
- **IPT:** AMCOM, ARL, Sikorsky, AMRDEC-AED, CCAD, UH-60 Project Office (PO), AH-64E Apache PO, CH-47 PO

**Schedule:**
- **FY15**
  - Initiate laboratory testing with Sikorsky
- **FY16**
  - Evaluation of TSAA and anodic coating stripper
- **FY18**
  - Demo at CCAD
- **FY19**
  - Implementation through MEO
Objective: Eliminate chromic acid (Cr(VI)) used in stripping anodized coatings from aluminum

Magnitude of impact:
- Eliminate 1,400 lbs/yr of chromic acid at Corpus Christi Army Depot (CCAD) in anodize stripping processes

Intended end product: Validated Cr(VI) free chemical stripper for anodized coating on aluminum (Type I, Type III and alternative processes)

Technology: Commercially available chemical strippers
- NaOH Stripper/Deoxidizer
- LNC Deoxidizer (ferric sulfate, nitric acid, HF)
- Sikorsky (proprietary)
- Stripol ANO
- Metalast ADS 1000 (sulfuric acid)

Weapon systems impacted: All systems that use anodized aluminum, including ground tactical and support equipment and aviation systems

Transition Path: Revision to MIL-A-8625

POC: Jack Kelley, ARL, john.v.kelley8.civ@mail.mil

IPT: ARL, AMCOM, AMRDEC, ANAD, CCAD, PEO-Stryker Brigade Combat Team, Hubbard Hall, Henkel, Chemetall, AMZ Manufacturing, PPI Aerospace

FY14:
- Develop testing protocol
- Laboratory testing

FY16:
- Down-select

FY17:
- Demonstration at ANAD/CCAD

FY18:
- Specification revision and implementation
**Objective:** Eliminate Cr(VI) from electroplated hard chrome (EHC) processes

**Magnitude of impact:**
- Eliminate 5 tons of chromic acid used in EHC in Army depot operations (ANAD, CCAD, Rock Island Arsenal (RIA))

**Intended end product:** Cr(VI)-free Non-Line of Sight (NLOS) plating process that results in a hard chrome plate that meets AMS 2460 performance requirements

**Technology:** Faraday Technologies developed process
  - Trivalent chromium (Cr(III)) bath chemistry
  - Pulsed, reverse waveform rectifiers/power supply
  - Non-lead anodes
  - Leverage: SBIR for stripping chrome plating

**Weapon systems impacted:** All aircraft maintained at CCAD; ground vehicles at ANAD and specific processes at RIA

**Transition Path:** Individual MEOs, CCAD process standard

**POC:** Michael Johnson, AMCOM, michael.l.johnson17.ctr@mail.mil

**IPT:** AMCOM, TACOM, AED, ARL, PEO Aviation, CCAD, Faraday Technologies

**FY14**
- Laboratory testing (130 gallon)

**FY16**
- Process validation and characterization

**FY18**
- Establish Pilot Process (400 gallon)
- Demonstration at CCAD

**FY20**
- Implementation through MEOs
Objective: Eliminate Cr(VI) in electroplated hard chrome

Magnitude of impact:
- Potential to eliminate Cr(VI) in all Line-of-Sight (LOS) hard chrome applications (and Ni plating)
- Increase throughput for dimensional restoration
- Mobile repair processes

Intended end product: Cr(VI)-free portable CS system for field repair, production process for inner diameter applications at ANAD, LEAD and CCAD.

Technology:
- Portable CS equipment with optimized ID nozzle with amorphous iron, Cr, Ni, and CrC-NiCr powders
- Transition powders developed by SERDP WP-2607
- Dimensional restoration of hard (HRC 45+) surface
- Coordinated path forward for LOS applications

Weapon systems impacted: All LOS hard chrome surfaces at ANAD, LEAD and CCAD.

POC: Vic Champagne, ARL, victor.k.champagne.civ@mail.mil
Objective: Eliminate cyanide from copper and silver electroplating at CCAD

Magnitude of impact:
- Cyanide alarm requirement: Up to 1 hr evacuation per alarm
- 80 lbs/yr of cyanide used at CCAD

Intended end product:
- Non-cyanide products and processes for copper and silver plating/strike demonstrated at CCAD
- Non-chromic acid and non-cyanide stripping methods to remove copper and silver plating/strike demonstrated at CCAD

Technology:
- Leverage DoD, commercially available plating chemistry
  - E-Brite 30/30 and E-Brite Ultra Cu (Copper)
  - E-Brite 50/50 (Silver), Silver Cyless II
  - Cold spray for LOS Cu or Ag deposition
  - Cyanide, Cr(VI)-free stripping process for copper and silver

Transition: MEOs at CCAD

Weapon systems impacted: All aircraft maintained at CCAD

POC: Sheree York, AMCOM, sheree.t.york.civ@mail.mil

FY15: • Establish Pilot Process at CCAD
      • Evaluate CS
FY16: • Demonstrate Plating/Strike
      • Laboratory testing
FY17: • Implement Plating/Strike
      • Demonstrate Stripping
FY18: • Implement Cr(VI)-Free Stripping
- **Objective:** Eliminate Cr(VI) in sealers for MIL-DTL-13924

- **Magnitude of impact:**
  - Reduce the estimated 1.4M gallons chromic acid solution used in 2011 at ANAD

- **Intended end product:** Cr(VI)-free technologies for sealing MIL-DTL-13924 on ferrous substrates on all classes within specification and chromic acid sealers mandated in MIL-DTL-16232

- **Technology:**
  - TCP (SurTec 580) already demonstrated as TRL-8 on Zn Phosphate, but considered TRL-6 until demonstrated on black oxide
  - Provide the non-Cr(VI) options via a revision of MIL-DTL-13924 and MIL-DTL-16232
  - Perform demonstration at ANAD on misc and small arms parts

- **Weapon systems impacted:** Small arms and related accessories at ANAD

- **POC:** Jack Kelley, ARL, [john.v.kelley8.civ@mail.mil](mailto:john.v.kelley8.civ@mail.mil)

FY16:
- Laboratory evaluation of black oxide and heavy zinc phosphate sealer alternatives

FY17:
- Demonstrate at ANAD on current process line

FY18:
- Implement alternatives through changes to MIL-DTL-13924 and MIL-DTL-16232
Objective: Eliminate Cr(VI) in post-treatment sealers

Magnitude of impact:
- Reduce the overall usage of chromic acid solution at ANAD, CCAD, RRAD, RIA

Intended end product: Qualified Cr(VI)-free post-treatment sealers for zinc plating, IVD aluminum, and hard coat aluminum anodize processes

Technology:
- Zinc plating: Corrosil 501 (BG)
- Hard coat aluminum anodize: Potassium permanganate
- Surveying other services and vendors for alternatives
- Demonstrate the new materials in a production environment (CCAD and TACOM depot)

Weapon systems impacted:
- ANAD: Ground vehicles and towed artillery
- CCAD: All aircraft
- RRAD: Ground vehicles and other heavy equipment
- RIA: Wide variety of equipment

POC: Lisa Maddox, AMCOM, lisa.j.maddox.civ@mail.mil

FY16 • Develop test protocol
FY17 • Evaluate sealers in laboratory
FY18 • Demonstrate in production environment (3 demos)
FY19 • Implement through new process standards and specifications
ETAP established Cadmium IPT in 2016
Surveyed depots to collect annual Cd plating and brush plating usage
- CCAD: Internal active plating shop plates 550+ parts, does not include connectors and fasteners (purchased through DLA)
- LEAD: 476 lbs/yr in cadmium brush plating, <1 lb used in Silk Screen Red paint
- Tobyhanna Army Depot: Usage quantity TBD
- ANAD, RRAD, RIA: No cadmium plating reported at facility
- Watervliet Arsenal: Eliminated Cd from most applications eliminated - estimated 3 systems continue to use Cd plating for gun barrels (e.g., bore evacuator)

Group parts into categories based on similar performance requirements
- Gears, bushings, miscellaneous parts plated in-house at Army depots
- Brush plating repair
- Common fasteners, electrical connectors purchased through DLA
Identify Army-specific performance requirements from users, if applicable
Collect performance data for alternatives from the Air Force, Navy (ES3)
Evaluate maturity of alternative technologies (e.g., ZnNi, high purity Al) for all applications
- May require independent study to compile data and compare to requirements
Develop demonstration project plans for FY18-19 start
U.S. Army Public Health Center evaluates all proposed alternatives using Toxicology Assessment (TA) Process
- Literature review
- Computational modeling
- Data collection
- Toxicity Testing, if necessary

TA review concurrent with project maturity

Data will inform acquisition documentation and occupational exposure requirements
- Toxicity Clearance, Health Hazard Assessment
- Occupational Exposure Limits, Industrial Hygiene Plan
- Programmatic Environment, Safety and Occupational Health Evaluation
- Life Cycle Environmental Assessment
- DESHE is a process and not a report or document
- Purpose: Develop and document a baseline level of ESOH performance data for each level of research in order to support risk-based decisions
  - Should include human toxicity (mammalian), fate and transport, eco-toxicity and safety data
- Phased approach to gather, develop and document ESOH performance data for materials, processes and technologies during all phases of RDT&E
  - Data requirements determined by Budget Activity (BA) level or TRL
  - Early stages – qualitative data
  - Higher maturity technologies – more robust, quantitative data
  - Data should be collected using regulatory-approved methods (ASTM, OECD) and consistent with Good Laboratory Practices
- Army EQT P2 TMR Program will conduct demonstrations of more sustainable surface finishing processes at Army depots and installations from FY15-23

- P2 Technology Team will support transition through document changes, maintenance orders and updates to QPD

- Goal: Eliminate 100% of Cr(VI), Cd or toxic constituents in select processes (with Army-wide goals consistent with SERDP/ESTCP 90% reduction)

- Seeking leveraging opportunities, data sharing, support for specification changes and promising technologies for future demonstrations