Solvent Replacement for Super Corr-A
Corrosion Preventive Compound (CPC)

2011 Air Force Corrosion Conference

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Battelle

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Hill AFB
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Overview

• Project Team
• Background & Objectives
• Technical Approach
• Test Matrix
• Laboratory and Field Testing – Results
• Conclusions
• Recommendations
Project Team

• Primary Stakeholder – F-16 SPO, 388th Fighter Wing
• COTR – Paul Hoth 501 ACSS/GFLB
• Program Manager – John Stropki
• Task Leader – Jim Tankersley
• Support Staff
  – Bill Abbott (Consultant)
  – Annie Lane (Research Scientist)
  – Jill Gregory (Researcher)
• Subcontractor Support
  – Lektro-Tech, Inc., Tampa, FL (Ron Knight and Robert Kay)
    - Assistance w/ solvent down-selection and formulation
  – SMI, Inc., Miami, FL
    - Perform first article testing on new formulations
Background

• The Super Corr-A corrosion preventative compound (CPC) is qualified as a MIL-L-87177A, Type I, Grade B material for electrical connector applications
  – The Super Corr-A lubricant has had two solvent-related formulation modifications since 1994 (CFC-113 and HCFC-141B)
  – Super Corr-A has met or exceeded performance requirements in extensive evaluations by Hill AFB
• The current Super Corr-A formulation contains an HCFC AK225T solvent
  – Considered Class II Ozone Depleting Substances (ODS)
  – Banned in the European Union (EU) and Canada on 1 January 2009
• All maintenance and manufacturing operations in the EU requiring use of MIL-L-87177A are currently shutdown with no alternative replacement identified
• Unless a replacement solvent can be implemented, use of these ODSs will also be prohibited in the United States beginning in 2015
Objective & Approach

Objective:
Identify a more environmentally friendly and COTS alternative to the HCFC AK225T solvent currently in the Super Corr-A lubricant.

Program Approach:
• Research US and EU compliant solvents with chemistry compatible with Super Corr-A CPC
• Define material and performance requirements based on previous assessments of lubricants
• Conduct laboratory and field testing for comparative evaluation of the lubricant performance containing the alternative solvents
• As required, update MIL-L-87177A specification and associated process order
Test Matrix

• Test plan includes nine CPC formulations and one control
  1. Existing Super Corr-A formulation with AK225T solvent
  2. Previous Super Corr-A formulation with 141B solvent
  3-6. Super Corr-A formulated with 4 solvent candidates
     a. DuPont Vertrel® SDG w/ current concentration of CPCs
     b. DuPont Vertrel® SDG w/ higher concentration of CPCs
     c. Kyzen Cybersolv® 141R w/ higher concentration of CPCs
     d. Kyzen Cybersolv® 141R w/ current concentration of CPCs
  7. ILFC 1006 CON-TAC
  8. Zip-Chem D-5026NS
  9. Zip-Chem D-5026NS with alternative propellant (Noxit-86)
MIL-L-87177A Assessments

• SMI Laboratories conducted first article testing specified in MIL-SPEC to validate performance characteristic requirements of experimental lubricant formulations

• **Results:** New and old formulations of Super Corr-A do not meet first article requirements of MIL-L-87177A
  
  – Original formulations were never tested
  
  – Both formulations perform appropriately for intended application

• **Recommendation:** Update first article requirements and revise MIL-SPEC
  
  – Stakeholders include; Hill AFB, DLA-Richmond, AFRL/CTIO, and AFCPCO
## First Article Testing Results

<table>
<thead>
<tr>
<th>Requirement</th>
<th>Test Method Specification</th>
<th>Limit</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dryness</td>
<td>MIL-SPEC 4.6.1</td>
<td>0.0100 gram (max)</td>
<td>Failed</td>
</tr>
<tr>
<td>Flash Point</td>
<td>ASTM D1310</td>
<td>243°C/470°F (min)</td>
<td>Inconclusive</td>
</tr>
<tr>
<td>Dielectric Breakdown</td>
<td>ASTM D877</td>
<td>24,000 volts (min)</td>
<td>Failed</td>
</tr>
<tr>
<td>Lubricity</td>
<td>ASTM D226</td>
<td>1.20 mm wear scar diameter (max)</td>
<td>Failed</td>
</tr>
<tr>
<td>Residue Solubility</td>
<td>MIL-SPEC 4.6.3</td>
<td>No visible residue</td>
<td>Failed</td>
</tr>
<tr>
<td>Leakage</td>
<td>MIL-SPEC 4.6.4</td>
<td>No leakage or distortion</td>
<td>Passed</td>
</tr>
<tr>
<td>Content</td>
<td>MIL-SPEC 4.6.5</td>
<td>16 ounces (min)</td>
<td>Failed (container content 12 oz.)</td>
</tr>
<tr>
<td>Performance of pressurized containers</td>
<td>MIL-SPEC 4.6.6</td>
<td>Uniform spray, panel adherence, no sagging</td>
<td>Passed</td>
</tr>
<tr>
<td>Oxidation Stability</td>
<td>ASTM D942</td>
<td>&lt;5 pounds/100 hours</td>
<td>Failed</td>
</tr>
<tr>
<td>Grade B Corrosion</td>
<td>ASTM B117</td>
<td>No corrosion after 168 hours</td>
<td>Passed</td>
</tr>
<tr>
<td>Sprayability</td>
<td>MIL-SPEC 4.6.9</td>
<td>Sprayable w/ no clogs</td>
<td>Passed</td>
</tr>
</tbody>
</table>
Battelle Laboratory Results

• Grade B Corrosion Testing
  – Alternative Super Corr-A formulations showed improved corrosion resistance in salt fog exposure testing
  – Most extensive pitting damage noted with the control and CON-TAC
  – “Streaked” pitting noted on Noxit-86, D5026NS; may have been caused by formation and collection of water droplets along top edge
Battelle Laboratory Results - Connector Card Testing

Conditions:
- 1000 hours
- 80°C (176°F)

Requirements:
- $\Delta R < 10$ milliohms

Results:
- All passed

Change in Contact Resistance Resulting from Thermal Aging Exposure Testing of Coated Electrical Connectors
Battelle Laboratory Results – Low Temperature Testing

Conditions:
- 15 minutes @ each temperature

Requirements:
- $\Delta R < 10$ milliohms

Results:
- Only CPC No. 1 failed
Battelle Laboratory Results – Disturbance Cycle Testing

**Conditions:**
- 100 demate/remate cycles

**Requirements:**
- \( \Delta R < 10 \) milliohms

**Results:**
- All passed

Change in Contact Resistance Resulting from 100 Disturbance Cycles Completed on Coated Coupons attached to Connector Card
Battelle Laboratory Results – Class II Flowing Mixed Gas Testing

**Conditions:**
- 10 day exposure

**Requirements:**
- \( \Delta R < 10 \) milliohms

**Results:**
- CPCs No. 1 & 3, CON-TAC, and Noxit-86 failed

---

Change in Contact Resistance After Exposure of Coated Coupons to Class II Flowing Mixed Gas Test

**.business sensitive**
# Battelle Laboratory Results – Grade B Corrosion Testing

## Photographs Documenting Placement of Coated Panels in ASTM B117 Salt Fog Cabinet and Corrosion Pitting Noted on Coupons Coated with CON-TAC CPC

<table>
<thead>
<tr>
<th>CPC</th>
<th>Panel 1</th>
<th>Panel 2</th>
<th>Panel 3</th>
<th>Average Score (Max: 5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td><strong>5.0</strong></td>
</tr>
<tr>
<td>CPC No. 1</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2.0</td>
</tr>
<tr>
<td>CPC No. 2</td>
<td>1</td>
<td>2</td>
<td>1</td>
<td>1.3</td>
</tr>
<tr>
<td>CPC No. 3</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td><strong>1.0</strong></td>
</tr>
<tr>
<td>CPC No. 4</td>
<td>1</td>
<td>1</td>
<td>2</td>
<td>1.3</td>
</tr>
<tr>
<td>Super Corr A</td>
<td>3</td>
<td>2</td>
<td>1</td>
<td>2.0</td>
</tr>
<tr>
<td>Super Corr B</td>
<td>1</td>
<td>1</td>
<td>2</td>
<td>1.3</td>
</tr>
<tr>
<td>CON-TAC</td>
<td>5</td>
<td>5</td>
<td>4</td>
<td>4.7</td>
</tr>
<tr>
<td>Noxit-86</td>
<td>3</td>
<td>2</td>
<td>3</td>
<td>2.7</td>
</tr>
<tr>
<td>D-5026NS</td>
<td>3</td>
<td>2</td>
<td>3</td>
<td>2.7</td>
</tr>
</tbody>
</table>

Salt Fog CPC Ratings Calculated from Pit Density Evaluation Referenced in ASTM G46-94 and ASTM D610-08
Battelle Laboratory Results – Polycarbonate Compatibility (canopies)

Consistent with previous testing, crazing noted with CON-TAC, AK225T (slight), 141-B (dramatic)

Polycarbonate Stressed Coupons: CON-TAC (left), Control (right)
<table>
<thead>
<tr>
<th>Ranking of EIS Data</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Noxit86</td>
<td>1</td>
</tr>
<tr>
<td>D-5026NS</td>
<td>2</td>
</tr>
<tr>
<td>Super Corr-A</td>
<td>3</td>
</tr>
<tr>
<td>CPC-4</td>
<td>4</td>
</tr>
<tr>
<td>CPC-3</td>
<td>5</td>
</tr>
<tr>
<td>Super Corr-B</td>
<td>6</td>
</tr>
<tr>
<td>CPC-2</td>
<td>7</td>
</tr>
<tr>
<td>CON-TAC</td>
<td>8</td>
</tr>
<tr>
<td>CPC-1</td>
<td>9</td>
</tr>
<tr>
<td>Control (uncoated)</td>
<td>10</td>
</tr>
</tbody>
</table>
# Battelle Field Testing

<table>
<thead>
<tr>
<th>Test Type</th>
<th>Tests</th>
<th>Test Reference</th>
<th>Sample Size</th>
<th>Time Periods</th>
<th>Replicates</th>
<th>Sample Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>Field Exposure Testing</td>
<td>Connector Field Testing</td>
<td>Abbott 1996 report</td>
<td>10 CPCs</td>
<td>3 (1 mo, 3 mo, 6 mo)</td>
<td>50 (pin count)</td>
<td>Test connectors with gold-plated bars (2 to a PC board)</td>
</tr>
<tr>
<td></td>
<td>Corrosion Coupons</td>
<td>Abbott 1996 report</td>
<td>10 CPCs</td>
<td>3 (1 mo, 3 mo, 6 mo)</td>
<td>1</td>
<td>Rack with 5 steel coupons</td>
</tr>
<tr>
<td></td>
<td>Lap Splice Testing</td>
<td>Rice 2004 report</td>
<td>10 CPCs</td>
<td>3 (1 mo, 3 mo, 6 mo)</td>
<td>1</td>
<td>Lap splice fixture with steel coupon fastened to 2024 T3 Al coupon</td>
</tr>
<tr>
<td></td>
<td>Steel Sensors</td>
<td>Recent Abbott work</td>
<td>10 CPCs</td>
<td>Measurements in place at 1 mo, 3 mo, 6 mo</td>
<td>1</td>
<td>Steel sensors</td>
</tr>
</tbody>
</table>

**Corrosion Coupons**

- Test connectors with gold-plated bars (2 to a PC board)
- Rack with 5 steel coupons

**Lap Splice Fixtures**

- Lap splice fixture with steel coupon fastened to 2024 T3 Al coupon

**Steel Sensor**

- Steel sensors
## Battelle Field Testing Results - Corrosion Testing Summary

<table>
<thead>
<tr>
<th>CPC</th>
<th>Average Weight Loss (g)</th>
<th>Average Corrosion Rate (mpy)</th>
</tr>
</thead>
<tbody>
<tr>
<td>D-5026NS</td>
<td>0.14590</td>
<td>2.22</td>
</tr>
<tr>
<td>CPC 2</td>
<td>0.21215</td>
<td>3.23</td>
</tr>
<tr>
<td>CPC 4</td>
<td>0.21465</td>
<td>3.27</td>
</tr>
<tr>
<td>Noxit 86</td>
<td>0.23494</td>
<td>3.58</td>
</tr>
<tr>
<td>CPC 1</td>
<td>0.32854</td>
<td>5.01</td>
</tr>
<tr>
<td>CPC 3</td>
<td>0.33280</td>
<td>5.07</td>
</tr>
<tr>
<td>Super Corr-A</td>
<td>0.33346</td>
<td>5.08</td>
</tr>
<tr>
<td>Super Corr-B</td>
<td>0.35096</td>
<td>5.35</td>
</tr>
<tr>
<td>CON-TAC</td>
<td>0.43267</td>
<td>6.59</td>
</tr>
<tr>
<td>Control</td>
<td>0.51872</td>
<td>7.91</td>
</tr>
</tbody>
</table>

*Average for each CPC over the 4 month period with the three location sets combined

CPC Lubricant Ranking of Coated Corrosion Coupons Based on Weight Loss
**Battelle Field Testing Results - Summary**

- The worst corrosion resistance was measured for the control or uncoated coupon sets,
- The best corrosion resistance was measured for the coupon sets coated with the D-5026N lubricant,
- The corrosion resistance of the CPC-2 lubricant was only slightly lower than the performance measured for the D-5026N material,
Battelle Field Testing Results – Lap Splice Testing

Area of CPC Application Along Upper Edge of Lap Splice Coupons

Lap Splice Coupon Sets Mounted on Chain Link Fence at FMRF
# Battelle Field Testing Results – Lap Splice Testing Summary

## Ranking Scores for CPC Coated Lap Splice Coupons
(ref. ASTM D610-08)

<table>
<thead>
<tr>
<th>CPC</th>
<th>West Jefferson</th>
<th>FMRF</th>
<th>Total (Max: 60)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1 mo.</td>
<td>3 mo.</td>
<td>4 mo.</td>
</tr>
<tr>
<td>Control</td>
<td>1</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>CPC No. 1</td>
<td>3</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>CPC No. 2</td>
<td>3</td>
<td>3</td>
<td>1</td>
</tr>
<tr>
<td>CPC No. 3</td>
<td>2</td>
<td>5</td>
<td>9</td>
</tr>
<tr>
<td>CPC No. 4</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Super Corr A</td>
<td>3</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>Super Corr B</td>
<td>3</td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td>CON-TAC</td>
<td>3</td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td>Noxit-86</td>
<td>10</td>
<td>5</td>
<td>10</td>
</tr>
<tr>
<td>D-5026NS</td>
<td>10</td>
<td>9</td>
<td>8</td>
</tr>
</tbody>
</table>
• Horizontally mounted sensors had increased corrosion

• Visual corrosion on controls, CON-TAC, and D5026NS variants

• CPC No. 2 consistently showed the least change in resistance
Conclusions

• No tested lubricants met all first article testing requirements
• DuPont Vertrel SDG and Kyzen Cybersolv C141R performed well
• Independent testing conducted by SMI Laboratories confirm solvent alternatives are not corrosive or embrittling to high strength aerospace alloys
• Performance of formulations blended with compliant solvents and higher concentrations of proprietary CPC was equal to, or greater than lubricants approved per MIL-L-87177A and MIL-PRF-81309F
• Demonstrated superior performance of the D-5026NS, CPC No. 3 and CPC No. 4 lubricants
• Compliant solvent alternatives can replace the 225T solvent in the current Super Corr-A formulation without compromising the performance of the lubricant
**Recommendations**

- Work closely with representatives at Hill AFB, DLA, AFRL, and AFCPCO to revise or update the chemical, physical and performance requirements currently referenced in the MIL-L-87177A specification.

- A preliminary set of deletions, modifications or additions include:
  - Update flash point requirement based on lubricant chemistry.
  - Update or delete the dielectric breakdown requirement based on lubricant chemistry and intended use applications.
  - Assess and update oxidation stability requirements.
  - Input compatibility requirement with MIL-PRF-32033 and MIL-PRF-81309F lubricants.
  - Input Electronics Lubricant Effectiveness tests referenced in MIL-PRF-81309F
    - Initial contact resistance (fixed and disturbed).
    - Low temperature exposures.
    - Thermal aging.
    - Durability cycling.
    - Corrosive gas exposures.
    - Compatibility with electrical insulating compounds.
Questions & Discussion

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Back-up Slides
UC Laboratory Testing

- Testing by University of Cincinnati (UC) supplemented Battelle’s CPC performance testing

<table>
<thead>
<tr>
<th>Test Type</th>
<th>Tests</th>
<th>Test Reference</th>
<th>Sample Size</th>
<th>Time Periods</th>
<th>Replicates</th>
<th>Sample Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>UC Laboratory Testing</td>
<td>Grade B Corrosion</td>
<td>MIL-L-877177A, ASTM B117 - 168 hrs salt fog exposure</td>
<td>9 CPCs</td>
<td>1 (168 hrs)</td>
<td>3</td>
<td>2024 T3 Al coupons</td>
</tr>
<tr>
<td></td>
<td>DC Polarization Resistance</td>
<td>ASTM G59, ASTM G96, ASTM G102</td>
<td>9 CPCs</td>
<td>1 (record resistance for each sample)</td>
<td>2</td>
<td>2024 T3 Al coupons</td>
</tr>
<tr>
<td></td>
<td>Electrochemical Impedance Spectroscopy</td>
<td>Battelle April 2005 study</td>
<td>9 CPCs</td>
<td>7 (at 8, 24, 48, 96, 168, 336, and 504 hrs)</td>
<td>2</td>
<td>2024 T3 Al coupons</td>
</tr>
</tbody>
</table>
UC Laboratory Testing Results

- **ASTM B117 Neutral Salt Spray Corrosion Testing Results**
  - 168 hour exposure period
  - Extensive corrosion pitting observed on control coupons
  - Good corrosion resistance for all CPCs tested
  - Visual scoring ranked CPCs:
    - CON-TAC (best)
    - D-5026NS, CPCs No. 2, 3, 4
    - Noxit-86, SC-A, SC-B, and CPC No. 1 (worst)
**UC Laboratory Testing Results**

- **Polarization Resistance**
  - Electrochemical technique that assesses corrosion rates using direct applied current

<table>
<thead>
<tr>
<th>Sample</th>
<th>OCP (mV)</th>
<th>Polarization Resistance, $R_p$ (kOhm)</th>
<th>Ranked by Highest Corrosion Resistance</th>
</tr>
</thead>
<tbody>
<tr>
<td>CPC-4</td>
<td>-476.3</td>
<td>22530</td>
<td>1</td>
</tr>
<tr>
<td>CPC-3</td>
<td>-404.7</td>
<td>4487</td>
<td>2</td>
</tr>
<tr>
<td>Super Corr-A</td>
<td>-414.8</td>
<td>2465</td>
<td>3</td>
</tr>
<tr>
<td>D-5026NS</td>
<td>-381</td>
<td>782.2</td>
<td>4</td>
</tr>
<tr>
<td>Super Corr-B</td>
<td>-394.9</td>
<td>388.8</td>
<td>5</td>
</tr>
<tr>
<td>CPC-1</td>
<td>-385.8</td>
<td>337.5</td>
<td>6</td>
</tr>
<tr>
<td>NOXIT86</td>
<td>-385.9</td>
<td>295.3</td>
<td>7</td>
</tr>
<tr>
<td>CON-TAC</td>
<td>-374.5</td>
<td>145.7</td>
<td>8</td>
</tr>
<tr>
<td>Control - uncoated</td>
<td>-383.2</td>
<td>7.7</td>
<td>9</td>
</tr>
<tr>
<td>CPC-2</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
</tbody>
</table>

1 = highest corrosion resistance
UC Laboratory Testing Results

- Electrochemical Impedance Spectroscopy (EIS)
  - Estimates corrosion rate
  - Rapid evaluation of thin coatings
  - Results plotted in a **Bode** plot (Impedance vs. Frequency)
Battelle Field Testing Results

• Connector field testing – West Jefferson, OH
  – All CPC lubricants passed the five month exposure with a change in the initial contact resistance of $<10 \text{ m}\Omega$

Change in Contact Resistance After 5 Months Field Exposure at West Jefferson Test Location
Battelle Field Testing Results

- Connector field testing – FMRF Daytona Beach, FL
  - All passed the five month exposure with a change in the initial contact resistance of <10 mΩ
Battelle Field Testing Results – Corrosion Coupons – West Jefferson, OH

– All lubricants showed improved corrosion resistance over the control
– CON-TAC showed the least corrosion resistance
– CPCs No. 3 and 4 performance comparable to SC-A and SC-B
Corrosion Rates Calculated for Corrosion Coupons Exposed Vertically at West Jefferson Test Location
Battelle Field Testing Results - Corrosion Coupons @ FMRF Location

• Similar CPC performance was observed at FMRF with increased overall corrosion on all coupons due to the harsher environmental conditions.

• CON-TAC showed the greatest overall corrosion following the control.

CPC Coated Corrosion Coupon Sets Mounted at FMRF Test Location:

Horizontal Mount (left) Vertical Mount (right)
Battelle Field Testing Results – Corrosion Coupons @ FMRF Location

Corrosion Rates Calculated for Corrosion Coupons Exposed Horizontally at FMRF Test Location
Battelle Field Testing Results – Corrosion Coupons @ FMRF Location

Corrosion Rates Calculated for Corrosion Coupons Exposed Vertically at FMRF Test Location
Battelle Field Testing Results – Steel Sensors at FMRF and West Jefferson

Steel Sensors Exposed Vertically at West Jefferson Test Location
Battelle Field Testing Results – Steel Sensors at FMRF and West Jefferson

Steel Sensors Exposed Vertically at FMRF Test Location