AERONAUTICAL ANALYTICAL REWORK PROGRAM: THIXOTROPIC CHEMICAL CONVERSION COATING FOR THE CORROSION PROTECTION OF AIR CRAFT ALUMINUM SURFACES

P. N. Bellavin

Naval Air Development Center
Warminster, Pennsylvania

6 June 1975
AERONAUTICAL ANALYTICAL REWORK PROGRAM

THIXOTROPIC CHEMICAL CONVERSION COATING FOR THE CORROSION PROTECTION OF AIRCRAFT ALUMINUM SURFACES

P. N. Bellavin
Air Vehicle Technology Department
NAVAL AIR DEVELOPMENT CENTER
Warminster, Pennsylvania 18974

6 June 1975

FINIAL REPORT
MAINTENANCE ENGINEERING SUPPORT
Organizational Intermediate and Depot Level
AIRTASK NO. WR4-5134
Work Unit NO. GA801

APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED.

Prepared for
NAVAL AIR SYSTEMS COMMAND
Department of the Navy
Washington, D. C. 20361
NOTICES

REPORT NUMBERING SYSTEM - The numbering of technical project reports issued by the Naval Air Development Center is arranged for specific identification purposes. Each number consists of the Center acronym, the calendar year in which the number was assigned, the sequence number of the report within the specific calendar year, and the official 2-digit correspondence code of the Command Office or the Functional Department responsible for the report. For example: Report No. NADC-73015-40 indicates the fifteenth Center report for the year 1973, and prepared by the Crew Systems Department. The numerical codes are as follows:

<table>
<thead>
<tr>
<th>CODE</th>
<th>OFFICE OR DEPARTMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>00</td>
<td>Commander, Naval Air Development Center</td>
</tr>
<tr>
<td>01</td>
<td>Technical Director, Naval Air Development Center</td>
</tr>
<tr>
<td>02</td>
<td>Program and Financial Management Department</td>
</tr>
<tr>
<td>03</td>
<td>Anti-Submarine Warfare Program Office</td>
</tr>
<tr>
<td>04</td>
<td>Remote Sensors Program Office</td>
</tr>
<tr>
<td>05</td>
<td>Ship and Air Systems Integration Program Office</td>
</tr>
<tr>
<td>06</td>
<td>Tactical Air Warfare Office</td>
</tr>
<tr>
<td>10</td>
<td>Naval Air Facility, Warminster</td>
</tr>
<tr>
<td>20</td>
<td>Aero Electronic Technology Department</td>
</tr>
<tr>
<td>30</td>
<td>Air Vehicle Technology Department</td>
</tr>
<tr>
<td>40</td>
<td>Crew Systems Department</td>
</tr>
<tr>
<td>50</td>
<td>Systems Analysis and Engineering Department</td>
</tr>
<tr>
<td>60</td>
<td>Naval Navigation Laboratory</td>
</tr>
<tr>
<td>81</td>
<td>Administrative and Technical Services Department</td>
</tr>
<tr>
<td>85</td>
<td>Computer Department</td>
</tr>
</tbody>
</table>

PRODUCT ENDORSEMENT - The discussion or instructions concerning commercial products herein do not constitute an endorsement by the Government nor do they convey or imply the license or right to use such products.

APPROVED BY: P. D. STOGES
Commander, USN
Deputy Director, AVTD
DATE: 6 June 1975
This report covers processes for application and the use of a sprayable/brushable thixotropic chemical conversion coating for the corrosion protection of aircraft skin surfaces and components. Results of field evaluations, specification performance and formulations are given. Current application problems encountered during rework operation in the control of rapid run-off from vertical and curved aircraft surfaces are discussed.
INTRODUCTION

The purposes of this investigation, initiated under AIRTASK WR4-5134, Work Unit No. GA 801, and authorized by NAVAIRSYSCOM (AIR 411B4), were to determine the feasibility of use and establish processes for application of a new sprayable or brushable chemical material forming a uniform, non-draining type corrosion protective film. The thixotropic material, NADC-2-72 chemical conversion coating, a development of this command, was designed to eliminate application problems such as rapid run-off from vertical and curved surfaces. It was also intended to eliminate the waste of large quantities of materials during NAVAIREWORKFAC (Naval Air Rework Facility) operations where the standard liquid chemical conversion material procured under reference (a) is used.

To fulfill these objectives, it was considered necessary to evaluate the performance of such a material under the requirements of reference (a), particularly corrosion protection, paint adhesion, low temperature stability, storage stability, flash point and flammability. Upon completion of these tests it would then be submitted to the NAVAIREWORKFACs for their evaluation. Several demonstrations would be made stressing application performance characteristics of the material on aircraft in rework status.

MATERIAL DESCRIPTION AND PROCEDURES

The standard chemical conversion coating material conforming to reference (a) is applied over aluminum alloy surfaces of aircraft as a pre-treatment to obtain corrosion protection and insure optimum adhesion of the paint system. Application of the chemical conversion coating solution to the entire surface insures that all freshly exposed metal is treated. However, during the application, large quantities of material are wasted due to rapid run-off from vertical and curved surfaces. Due to this run-off, the aluminum surfaces of the aircraft are not uniformly coated and, therefore, more susceptible to corrosion attack. Use of large quantities also results in disposal problems since chromates contained in these coatings are toxic.

In order to overcome the above mentioned problems, the flow characteristics of the MIL-C-81706 material were changed from a liquid solution to a material wherein the viscosity was made to be increased until the solution thickened and became a thixotropic fluid. Thus, controlled spray application is possible. Sprayed over vertical surfaces, the material clings to the interface as the conversion coating is being deposited on the metal surface. This avoids "wasting" of large quantities of material and helps decrease chromate pollution.

When the thixotropic NADC-2-72 material is subjected to mechanical shear such as produced while mixing, the viscosity of the material decreases as shear increases. Allowed to stand undisturbed, the material returns to its original viscosity that is always greater than that of the standard material. Figure 1 compares the basic differences in the makeup of the standard material and the thixotropic NADC-2-72 material. The composition and preparation of the material are described in Appendix A.
SPECIFICATION CONFORMANCE

The NADC-2-72 material was evaluated for conformance with the requirements of MIL-C-81706 conventional Class 1A type film for:

1. Corrosion resistance properties, paragraphs 3.6, and 4.5.1 (336 hour 5% salt spray exposure).

2. Paint adhesion properties, paragraphs 3.7, 4.5.2, and 4.5.3.

Corrosion resistance and paint adhesion tests were repeated after three and twelve months storage at room temperature.

Low Temperature Stability

Freeze/thaw stability consisted of exposing quart size samples in plastic containers of the NADC-2-72 and the standard MIL-C-81706 materials at a temperature of 0°F for 8 hours followed by complete thawing at room temperature. This cycle was repeated until a change occurred in the physical/chemical properties of the materials.

Field Application Evaluations

Field evaluations were conducted at NAVAIREFACs Jacksonville and Cherry Point. During reference (b), the application process and performance characteristics of the thixotropic NADC-2-72 chemical conversion coating were demonstrated on a paint stripped A-7 aircraft with fastener areas vacu-blasted for removal of any corrosion deposits. Spray application of the thixotropic NADC-2-72 material was made with production equipment used for spraying the standard chemical conversion material. NADC-2-72 material was also applied by spray gun on a removable skin section. NAVAIREFAC personnel observing the demonstration expressed the opinion that NADC-2-72 coating clung to the skin surfaces and produced a more uniform conversion coating. The demonstration at Jacksonville, depicted by Figures 2 and 3, shows application of the conversion material to a reworked A-7 removable skin section. Figure 2 shows: (1) NADC-2-72 chemical conversion coated areas before water rinsing and drying, and (2) uncoated vacu-blast prepared areas. Figure 3 shows: (1) NADC-2-72 chemical conversion coating after rinsing and drying and (2) uncoated fastener areas.

During reference (c), an application/performance demonstration was made on a paint-stripped F-4-B auxiliary aluminum fuel tank. In this demonstration, application of thixotropic NADC-2-72 material was compared with that of the standard chemical conversion coating. Sprayed standard material produced rapid run-off from the skin surface as shown in Figure 4, whereas the NADC-2-72 material did not run-off, but clung to the surface and produced a continuous film as shown in Figure 5. NAVAIREFAC personnel expressed the opinion that it showed several advantages over the standard material; namely, that it clings and produces a more complete coverage of the surface. It was also indicated that less of the NADC-2-72 material is required in
Figure 2. A-7 removable skin section showing: (1) NADC 2-72 chemical conversion coated areas before water rinsing and drying, and (2) uncoated vacu-blasted areas.
Figure 3. A-7 removable skin section showing: (1) fastener areas coated with NADC 2-72 chemical conversion coating after water rinsing and drying, and (2) uncoated fastener areas.
Figure 4. F-4B auxiliary aluminum fuel tank showing skin area coated with standard MIL-C-8776 chemical conversion coating before water rinsing and drying.
rework operations. Therefore use of NADC-2-72 chemical conversion coating would reduce chromate pollution.

NAVAIREWORKFAC, Cherry Point (Code 340 personnel) requested an amendment to the military specification for inclusion of the NADC-2-72 chemical conversion coating.

Several NAVAIREWORKFACs expressed interest in the NADC material; viz., Alameda, Norfolk, North Island and Pensacola. NAS (Naval Air Station), Miramar has also evaluated it. Reference (d) reports that the NADC-2-72 material used in rework operations is (1) easy to apply and (2) the adherence to vertical skin surfaces of aircraft produces improved film coatings. Reference (e) states that the NADC-2-72 material is considered suitable for use as a surface preparation for touch-up by organizational level.

RESULTS

A summary of results is presented in Table 1.

1. The results of tests conducted on the material for conformance with the requirements of reference (a) show that the material satisfactorily passed corrosion resistance and paint adhesion requirements after three and twelve months of storage stability. At the end of the one-year storage, the material still remained in the thickened state without settling or separation. It returned readily to the sprayable condition when the thixotropic state of material was changed by mixing.

2. Results of the freeze/thaw cycling demonstrated that the NADC-2-72 showed no change during six 8-hour freeze/thaw cycles. After the 6th cycle, a two-phase separation was formed that consisted of more than 75% of a finely divided suspension of the thickener in the liquid phase. The freeze/thaw cycling had no effect on the chemical properties of the material. The cycled material was sprayable, still giving less run-off from vertical surfaces than the standard material initially as well as after 3 months shelf storage. A normal corrosion resisting coating was deposited. The standard MIL-C-81706 material under the same freeze/thaw conditions produced a very coarse sediment after two 8-hour cycles. Because of the presence of this coarse sediment no spray tests were performed since the spray apparatus would become clogged. The heavy sediment would impede spray operations. Chemical properties of the material also did not appear to change.

3. Results of field demonstrations conducted at the NAVAIREWORKFACs are shown in Figures 2, 3, 4 and 5. The application performance characteristics of the new material compared to the standard material are depicted in Figures 6, 7, 8 and 9. The NADC-2-72 material showed uniform adherence to vertical and curved surfaces without rapid run-off, whereas the standard MIL-C-81706 material exhibited rapid run-off.
<table>
<thead>
<tr>
<th>Specification</th>
<th>Test</th>
<th>Requirement</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>MIL-C-81706</td>
<td>Corrosion Resistance</td>
<td>No corrosion after 336 hours in 5% salt spray</td>
<td>Satisfactory</td>
</tr>
<tr>
<td>Para 3.6</td>
<td>(Test performed initially</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>and at three-month intervals up to 1 year inclusive)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>MIL-C-81706</td>
<td>Paint Adhesion</td>
<td>No loss of adhesion</td>
<td>Satisfactory</td>
</tr>
<tr>
<td>Para 3.7</td>
<td>(Test performed at same time interval as corrosion resistance)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>MIL-C-81706</td>
<td>Storage Stability</td>
<td>No change after one year's storage at room temperature</td>
<td>Met requirements and satisfactorily retained its thixotropic properties</td>
</tr>
<tr>
<td>Para 3.10</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>---</td>
<td>Low Temperature Stability</td>
<td>---</td>
<td>Standard formula - Course sediment after 2 cycles. Thixotropic formula - Two phase separation after 6 cycles</td>
</tr>
</tbody>
</table>
Figure 6. Application of NADC-2-72 thixotropic chemical conversion coating to aluminum alloy substrate showing uniform adherence to vertical surfaces.
Figure 7. Application of standard MIL-C-81706 chemical conversion coating showing rapid run-off of the material.
Figure 8. Completely coated panels before rinsing and drying showing:
Panel (a) - coated with standard material; Panel (b) -
coated with NADC-2-72 material.
Figure 9. Panel (a) - consisting of untreated aluminum alloy. Panel (b) - coated with standard chemical conversion material showing incomplete coverage of substrate. Panel (c) - coated with NADC-2-72 thixotropic material showing uniform coverage of substrate.
CONCLUSIONS

1. The thixotropic NADC-2-72 chemical conversion coating is in conformance with the requirements of specification MIL-C-81706.

2. The material is considered to be useful at all NAVAIR1 WORKFAC operations mainly by spray application and as a surface preparation for touch-up by organizational level.

3. The material is a significant improvement over the standard MIL-C-81706 unthickened material in that it is easier to apply and control when sprayed over vertical surfaces.

4. As with MIL-C-81706, the exposure of the NADC-2-72 material to freezing temperatures should be avoided.

RECOMMENDATIONS

It is recommended that:

1. The thixotropic NADC-2-72 chemical conversion coating material be used as a surface preparation for touch-up at organizational and intermediate levels, and at all Naval Air Rework Facilities for aircraft rework operations where the material would be spray-applied to aluminum and aluminum alloy skin surfaces and components of naval aircraft, and brush applied to treat small and difficult to reach surfaces.

   a. Appendices A, B and C be used to form the basis for local process specifications.

   b. Specification MIL-C-81706 be amended to reflect the changes given in Appendix D.

   c. The new material, prepared in accordance with Appendix E, be assigned a National Stock Item number.

   d. The material be stored and used at temperatures listed in specification MIL-C-81706.
REFERENCES

(a) Military Specification MIL-C-81706, Chemical Conversion Materials for Coating Aluminum and Aluminum Alloys


APPENDIX A

PREPARATION OF NADC 2-72 THIXOTROPIC CHEMICAL CONVERSION COATING MATERIAL

The material consists of three component parts:

Part A - the aqueous chemically active portion
Part B - inert thickener
Part C - wetting/thixotropy-promoting surfactant for rendering adherence of the material to the substrate surface.

The composition of the recommended chemical conversion coating material used in this investigation was as follows:

<table>
<thead>
<tr>
<th>Part</th>
<th>Ingredient</th>
<th>Parts by Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>A *</td>
<td>Chemical Conversion Coating Material</td>
<td>(1) 2.5</td>
</tr>
<tr>
<td>A-1</td>
<td>Water</td>
<td>100.0</td>
</tr>
<tr>
<td>B</td>
<td>Cab-O-Sil (Grade M-5)</td>
<td>(2) 4.0</td>
</tr>
<tr>
<td>C</td>
<td>Brij-35 20% by Wt. Aqueous Solution</td>
<td>(3) 0.1 - 0.4 **</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Source of Supply</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1) Chemical Conversion Coating Material</td>
<td>Qualified Product listed in QPL-81706-3</td>
</tr>
<tr>
<td>(2) Cab-O-Sil</td>
<td>Cabot Corporation Boston, Massachusetts 02110</td>
</tr>
<tr>
<td>(3) Brij-35 (Solid)</td>
<td>Atlas Chemical Co. Wilmington, Delaware</td>
</tr>
</tbody>
</table>

* Although only one product was used in this investigation, the similarity among the various products on QPL-81706-3 makes it reasonable to assume that any qualified product would perform satisfactorily in the thickened form.

** Approximately the upper limit for control of thickening.
Preparation

1. Dissolve Part A in water

2. Weigh Part B and add slowly to Part A while mixing at slow rate. Continue mixing at rapid rate until free of lumps.

3. Add Part C. Continue mixing at medium rate for 15 minutes.

Allow above mixture to stand undisturbed for 24 hours before using. When the material is needed for immediate use, further additions in increments of 0.05 part by weight of Part C followed by mixing is made until the thixotropic material does not appreciably flow off from a steel spatula vertically inserted into the material and withdrawn. Caution: Do not overmix. Over-mixing may create excessive foaming which may interfere with deposition of the coating. Mixing at high speed causes excessive thinning.
Power Mixing

1. Weigh out the specified amount or multiple thereof of Part A component listed in Appendix A. Add required amount of water. Mix until Part A is dissolved.

   NOTE: A power or air-driven mixer may be used. Mix in a plastic-lined or stainless steel container.

2. Add Part B material at a slow rate. Continue mixing at low speed.

3. Increase mixing to a medium rate and continue mixing until mixture is homogeneous.

4. Add Part C material, continue mixing at medium speed until mixture is thoroughly mixed.

   NOTE: Addition of 0.1 part by weight of Part C component produces thickening. When further thickening is required, such as for immediate use, add Part C material in increments of 0.1 part by weight followed with mixing after each addition.

   CAUTION: Do not overmix. Overmixing may create foaming which interferes with deposition of the coating. Mixing at high speed may cause excessive thinning of the thickened material. Follow procedure for thickening as indicated in the above "Note."
APPENDIX C

PROCESS FOR SPRAY AND BRUSH APPLICATION

1. Surface of paint-stripped aircraft should be prepared for chemical conversion coating treatment as specified in NAVAIR 01-1A-509.

2. NADC 2-72 thixotropic chemical conversion coating material is applied by spraying using production equipment (30 psi air pressure) or standard paint spray gun (acid resistant) at 30 psi air pressure to the wet surface of A/C after a minimum of 5 minutes drainage.

3. Conversion coating is allowed to form for 2 to 5 minutes.

4. Unreacted material is rinsed off thoroughly with clean water.

5. NADC 2-72 material can be brush-applied over small and hard to reach areas.

6. Allow coating to air dry. Do not wipe while wet. Wiping will remove the film.
APPENDIX D

PROPOSED CHANGES FOR MIL-C-21706

SCOPE

1. Paragraph 1.2.2.1 - Material Forms - Delete Form III and substitute Form IIIA - Thixotropic Form - Premixed powder (ready for use in spray or brush application after addition of water).

2. Add a new paragraph to include the formulation and preparation of the thixotropic chemical conversion coating material described in Appendix A.

3. Add a new paragraph to include provisions for procurement of material listed in Appendix A. The materials listed are to be packed pre-weighted in dry form into single package units. Formulation of the dry form is given in Appendix E.
PREWEIGHED POWDER FORM OF NADC-2-72 FOR MIXING IMMEDIATELY PRIOR TO USE

The weight of each ingredient required to make one gallon of NADC-2-72 is as follows:

<table>
<thead>
<tr>
<th>Part</th>
<th>Ingredient</th>
<th>Weight</th>
</tr>
</thead>
</table>
| A    | Conversion Coating Material | 3.35 oz.
| B    | Cab-O-Sil                   | 5.36 oz.
| C    | BRIJ-35                     | 0.11 oz.

Wide mouth quart and gallon sized plastic jars, which could be used for both storage and mixing, would be suitable for Fleet touch-up use. Larger pre-weighed packages would be required for use by NAVAIREWORKFACS.