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| 14. ABSTRACT Many federal, state and local laws and regulations limit the use, storage and disposal of hydrocarbon-based cleaning solvents. Unfortunately, the Army and other defense agencies rely on these solvents to maintain unique, mission-critical systems and materiel. The uncontrolled replacement of these solvents with environmentally friendly products has resulted in a number of material compatibility issues. Thus, performance validation testing has become a critical component in the process to gain approval to use alternative cleaners in DoD maintenance and repair activities. The preponderance of test methods established in this protocol are in accordance with recognized national and international test methods endorsed by the American Society for Testing and Materials (ASTM) and the Society of Automotive Engineers (SAE). Military unique test specifications and standards are identified where necessary to meet mission-oriented criteria. This protocol is available in electronic format to allow government and private industry stakeholders the opportunity to understand, review and provide input or comment. Final approval of the form and content of this document rest with the commodity commands who have ultimate approval authority for determining critical performance criteria and approving the use of alternative solvents on material items under their purview. | | | | | |
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1. MATERIAL COMPATIBILITY

1.1 CORROSION

1.1.1 TOTAL IMMERSION CORROSION

Objective

The objective of this test is to determine the total immersion characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not show any indication of staining, etching, pitting, or localized attack on the test panels, or cause weight change to an average of three (3) test panels greater than that shown in Table 1. (Test Protocol).

TABLE 1. ALLOY AND MAXIMUM AVERAGE WEIGHT LOSS

| Alloy | Weight Loss, mg/cm ² /168 hr |
|---------------------------------------|---|
| AM-355 CRT | 0.49 |
| PH 13-8 Mo | .49 |
| C-250 | .49 |
| Magnesium 4377 | .70 |
| Al 7075-T6 | .49 |
| Titanium 6AL-4V | .35 |
| Steel 4340 | .49 |
| Cadmium-Plated Steel | ^a .14 |
| Steel 4340 Zinc-Phosphate Coated | ^b .00 |
| Steel 4340 Manganese-Phosphate Coated | ^b .00 |
| Copper Alloy (Brass) UNS C36000 | ^b .00 |
| Copper Alloy UNS C10100 HO2 HH | ^b .00 |

^a mg/cm²/24 hr.

^b no criteria specified

Test Procedures

The total immersion corrosion caused by the manufacturer's suggested working concentration of the cleaning compound is determined using ASTM F-483-90 (app C, ref 17), Standard Test Method for Total Immersion Corrosion Test for Aircraft Maintenance Chemicals.

a. Four test specimens, 50.8 by 25.4 by 1.6 mm (2 by 1 by 0.06 in.), are prepared from the same sheet stock of each of the following materials:

- (1) AM-355 CRT (high-strength steel).
- (2) PH 13-8 Mo (high-strength steel).
- (3) Maraging C-250 (high-strength steel).
- (4) Aluminum 7075-T6.
- (5) Titanium 6AL-4V.
- (6) Steel 4340.
- (7) Magnesium AMS 4377.
- (8) Cadmium-plated steel.
- (9) Copper alloy (brass) UNS C36000.
- (10) Copper alloy UNS C10100 HO2 (half hard).

b. Twelve test specimens, 50.8 by 25.4 by 1.6 mm (2 by 1 by 0.06 in.), are prepared from the same sheet stock of each of the following materials:

- (1) Steel 4340 with zinc-phosphated coating (DOD-P-16232).
- (2) Steel 4340 with manganese-phosphated coating (DOD-P-16232).

c. A mounting hole, 3.2 mm (0.125 in.), is placed at one end of each of the test specimens.

d. The test specimens from each of the materials in paragraph b should be precleaned using the method outlined in ASTM F-483-90.

e. Each test specimen is then weighed to the nearest 0.1 mg.

f. Four test specimens from each material type listed in paragraph b (phosphate coatings) are subjected to a Salt Fog test as outlined in ASTM B-117-95 (app C, ref 29), Standard Practice for Operating Salt Spray (Fog) Apparatus. The test is conducted to failure and that time is recorded.

g. A container of the manufacturer's suggested working concentration of the cleaning compound is prepared for immersing the test specimens. The volume of the cleaner solution is related to the area of the test specimen immersed by 8 mL cleaner per 1-cm² test specimen.

h. Separate containers are used for each of the materials with new manufacturer's suggested working concentrations of the cleaning compound to prevent any contamination.

i. Testing should be conducted at 41 °C (105 °F) for the manufacturer's suggested working concentration of the cleaning compound.

j. Three test specimens of each material type are immersed into the manufacturer's suggested working concentration of the cleaning compound and allowed to soak for 24 hours. The fourth test specimen is used as the control specimen for the test.

k. After 24 hours, the test specimens are removed from the manufacturer's suggested working concentration of the cleaning compound.

l. The test specimens are rinsed under hot tap water (49 to 60 °C (120 to 140 °F)).

m. The test specimens are then rinsed in water conforming to Specification D-1193, Type IV at ambient (room) temperature.

n. The test specimens are then rinsed with a stream of acetone from a wash bottle and oven dried at 120 °C (250 °F).

o. After drying, the test specimens are placed in a container with desiccant until cooled to ambient (room) temperature.

p. The test specimens are then individually weighed to the nearest 0.1 mg.

q. The following visual observations should be conducted on each test specimen in comparison to an unexposed specimen:

- (1) Discoloration and dulling.
- (2) Etching.
- (3) Presence of accretions and relative amounts.
- (4) Pitting.
- (5) Presence of selective or localized attack.

r. The three test specimens are then immersed in the same manufacturer's suggested working concentration of the cleaning compound for an additional 144 hours, except for the cadmium-plated steel. Testing is discontinued on the cadmium-plated steel per the test protocol.

s. The steps in paragraphs k through q are repeated.

t. Four of the test specimens from each listed in paragraph b (phosphate coatings) are subjected to the Salt Fog test outlined in ASTM B-117-95. All test specimens should be photographically documented immediately upon completion of the test.

1. MATERIAL COMPATABILITY

1.1 CORROSION

1.1.2 SANDWICH CORROSION

Objective

The objective of this test is to determine the sandwich corrosion characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause a corrosion rating greater than two (2) on any test panel (Test Protocol).

Test Procedures

The sandwich corrosion caused by the manufacturer's suggested working concentration of the cleaning compound is determined using ASTM F-1110-90 (app C, ref 18), Standard Test Method for Sandwich Corrosion Test.

a. Sixteen test panels, 50 by 100 by 1.5 mm (2 by 4 by 0.06 in.), are prepared from the same sheet stock of each of the materials listed below:

- (1) AM-355 CRT (high-strength steel).
- (2) PH 13-8 Mo (high-strength steel).
- (3) Maraging C-250 (high-strength steel).
- (4) Aluminum 7075-T6.
- (5) Titanium 6AL-4V.
- (6) Steel 4340.
- (7) Magnesium AMS 4377.
- (8) 519 Cadmium-plated AISI/SAE 4340 steel.

(9) Steel 4340, zinc phosphated per DOD-P-16232 (an additional set of eight test coupons of this material should be made for testing against P-D-680 (II)).

b. The test coupons are separated into two sets of eight test coupons from each of the materials. Each of these eight coupons formed four pairs per set from each of the materials.

c. The coupons are individually identified.

d. The nonclad coupons are anodized in accordance with MIL-A-8625C, Type 1.

e. The test panels are cleaned by solvent.

f. A piece of glass fiber filter paper cut to approximately 25 by 75 mm (1 by 3 in.) is draped between each pair of test coupons.

g. The filter paper from one set of test coupon pairs (four pairs for each material) is saturated with the manufacturer's suggested working concentration of the cleaning compound.

h. The filter paper from one set of the test coupon pairs (the other four pairs for each material) is saturated with the reagent water specified in Specification ASTM D-1193-91 (ref 30), Type IV.

i. During each day of the test, the test coupon pairs are exposed to air oven heat and humidity as specified in Table 1.

TABLE 1. SANDWICH CORROSION EXPOSURE CYCLES AT 37.7 °C (100 °F)

| Step No. | Exposure Time, hr (± 0.5) | Relative Humidity, % |
|----------|---------------------------------|----------------------|
| 1 | 8 | Ambient |
| 2 | 16 | 95 to 100 |
| 3 | 8 | Ambient |
| 4 | 16 | 95 to 100 |
| 5 | 8 | Ambient |
| 6 | 16 | 95 to 100 |
| 7 | 8 | Ambient |
| 8 | 16 | 95 to 100 |
| 9 | 8 | Ambient |
| 10 | 64 | 95 to 100 |

j. Each set of test coupon pairs individually, not stacked, is exposed in a horizontal position.

k. After completing the exposure cycles, the test coupon pairs are rinsed in warm tap water and scrubbed lightly with a soft, nonmetallic bristle brush.

l. The test coupon pairs are then dried.

m. Each test coupon is examined under a 10X magnification.

n. The observations are rated using the following convention:

(1) 0 - no visible corrosion (no corrosion).

(2) 1 - very slight corrosion or discoloration (up to 5 percent of the surface area corroded).

(3) 2 - slight corrosion (5 to 10 percent of the surface area corroded).

(4) 3 - moderate corrosion (10 to 25 percent of the surface area corroded).

(5) 4 - extensive corrosion or pitting (25 percent or more of the surface area corroded).

o. The steps in paragraphs b through n are repeated for the additional eight test coupons of steel 4340 zinc phosphated. P-D-680 (II) is substituted for the manufacturer's suggested working concentration of the cleaning compound in the step in paragraph g. The control from the first set is used for this set. All test coupons should be photographically documented immediately upon completion of the test.

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1. MATERIAL COMPATABILITY

1.1 CORROSION

1.1.3 HYDROGEN EMBRITTLEMENT

Objective

The objective of this test is to determine the hydrogen embrittlement characteristics of the manufacturers suggested working concentration of the cleaning compound.

Criterion

The manufacturers suggested working concentration of the cleaning compound shall not cause hydrogen embrittlement of cadmium plated ANSI 4340 steel (Test Protocol).

Test Procedures

The hydrogen embrittlement effect that the manufacturers suggested working concentration of the cleaning compound has on materials is determined using ASTM F-519-93 (app C, ref 16), Standard Test Method for Mechanical Hydrogen Embrittlement Testing of Plating Processes and Aircraft Maintenance Chemicals.

- a. Fifteen cadmium-plated test specimens, type 1d, should be prepared for maintenance chemicals as specified in ASTM F-519-93.
- b. All tests are conducted at 20 to 30 °C (68 to 86 °F) or at the operating temperature of the manufacturers suggested working concentration of the cleaning compound, but not exceeding 66 °C (150 °F).
- c. Ten test specimens are loaded as outlined in ASTM F-519-93, paragraph 8.1.4.
- d. The average diameter of the notched C-ring is determined at fracture with the loading device.
- e. Five test specimens are loaded to 65 percent of the average determined in paragraph d.
- f. The loaded test specimens are then placed into 800 mL of the manufacturers suggested working concentration of the cleaning compound in a clean plastic container. The notched area is immersed in the manufacturers suggested working concentration of the cleaning compound.
- g. The approximate time of the failure is recorded.
- h. All testing is to be discontinued after 150 hours if the test specimen lasts until that point.

1. MATERIAL COMPATABILITY

1.1 CORROSION

1.1.4 EFFECTS ON UNPAINTED SURFACES

Objective

The objective of this test is to determine the effects on unpainted surfaces of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause streaking, stains or other deposits on unpainted surfaces that cannot be easily removed with water (Test Protocol).

Test Procedures

The effects of the manufacturer's suggested working concentration of the cleaning compound on the unpainted surfaces is determined using ASTM F-485-90 (app C, ref 14), Standard Test Method for Effects of Cleaners on Unpainted Aircraft Surfaces, modified by the Test Plan.

a. Two test specimens, 50 by 152 by 0.51 mm (2 by 6 by 0.020 in.), are prepared from each of the following materials:

- (1) AM-355 CRT (high-strength steel).
- (2) PH 13-8 Mo (high-strength steel).
- (3) Maraging C-250 (high-strength steel).
- (4) Aluminum 7075-T6.
- (5) Titanium 6AL-4V.
- (6) Steel 4340.
- (7) Magnesium AMS 4377.

- b. Each test specimen is cleaned with methyl ethyl ketone.
- c. The test specimens are allowed to dry at ambient (room) conditions.
- d. Two test specimens from each material are immersed for 4 minutes in enough of the manufacturer's suggested working concentration of the cleaning compound to cover one-half of the test specimen.
- e. The test specimens are removed from the cleaner.
- f. The test specimens are then placed immediately in a mechanical convection oven at 45° from the horizontal. The oven temperature should be maintained at 65.5 ± 2 °C (150 ± 5 °F).
- g. The test specimens are allowed to remain in the oven for 30 minutes.
- h. The test specimens are removed and allowed to cool to ambient (room) temperature for 15 minutes.
- i. The test specimens are then allowed to air dry for 30 minutes at ambient (room) conditions.
- j. The treated and untreated sections of each test specimen are visually examined for stains or residue.
- k. Each side of each test specimen is rinsed for 1 minute under running tap water.
- l. Each side of each test specimen is rinsed with distilled water from a squeeze bottle for 15 seconds.
- m. The test specimens are then allowed to air dry for 30 minutes at ambient (room) conditions.
- n. The treated and untreated sections of each test specimen are visually examined for stains or residue. All test specimens should be photographically documented immediately upon termination of the test.

1. MATERIAL COMPATABILITY

1.1 CORROSION

1.1.5 STRESS CORROSION

Objective

The objective of this test is to determine the stress corrosion characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause stress corrosion (Test Protocol).

Test Procedures

The stress corrosion caused by the manufacturer's suggested working concentration of the cleaning compound is determined using ASTM G-30-94, type (a) test specimens, Standard Practice for Making and Using U-Bend Stress-Corrosion Test Specimens (App C, ref 23) and the ASTM G-44-94 (app C, ref 24) test method, Standard Practice for Evaluating Stress Corrosion Cracking Resistance of Metals and Alloys by Alternate Immersion in 3.5 percent Sodium Chloride Solution, modified in the following manner: The manufacturer's suggested working concentration of the cleaning compound is substituted for the 3.5-percent sodium chloride solution, a 10-minute soak and 50 minute drying cycle are substituted for a 20-minute soak and 100 minute drying cycle, and the temperature will be 40.6 °C (105 °F) instead of 27 °C (80 °F) at 45 ± 10-percent relative humidity.

(1) Six (6) test specimens should be fabricated in accordance with ASTM G-30-94, type (a) U-bend from each of the following materials:

- (a) AM-355 CRT - high-strength steel.
- (b) PH 13-8 Mo - high-strength steel.
- (c) Maraging C-250 - high-strength steel.
- (d) Aluminum 7076-T6.
- (e) Titanium 6AL-4V.
- (f) Steel 4340.
- (g) Magnesium AMS 4377
- (h) Steel 4340, Manganese Phosphate Coating per DOD-P-16232B

- (2) All of the precautions are followed as outlined in ASTM G-44-94.
- (3) All testing is conducted at 40.6 °C (105 °F).
- (4) The test specimens are immersed in a bath (Figure 1.)of the manufacturer's suggested working concentration of the cleaning compound. The cleaner covered the entire specimen. All of the materials are immersed in separate baths to prevent any cross contamination.
- (5) The test specimens are allowed to soak for 20 minutes.
- (6) The test specimens are then removed from the manufacturer's suggested working concentration of the cleaning compound bath and allowed to air dry for 100 minutes in an area with good air circulation.
- (7) This cycle is continued for 24 hours per day for 90 days.
- (8) The test specimens are cleaned as specified in ASTM-G-1-90.
- (9) The test specimens are then examined for metallographic effects.

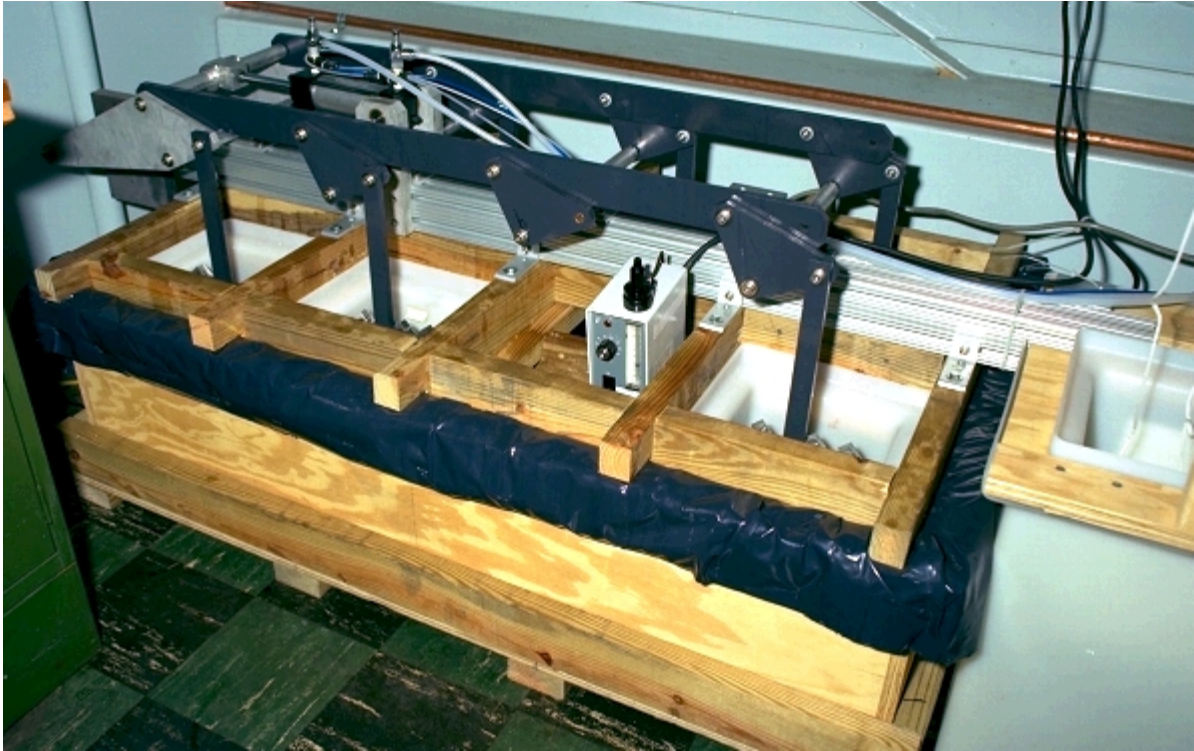


Figure 1. Stress Corrosion Test Fixture.

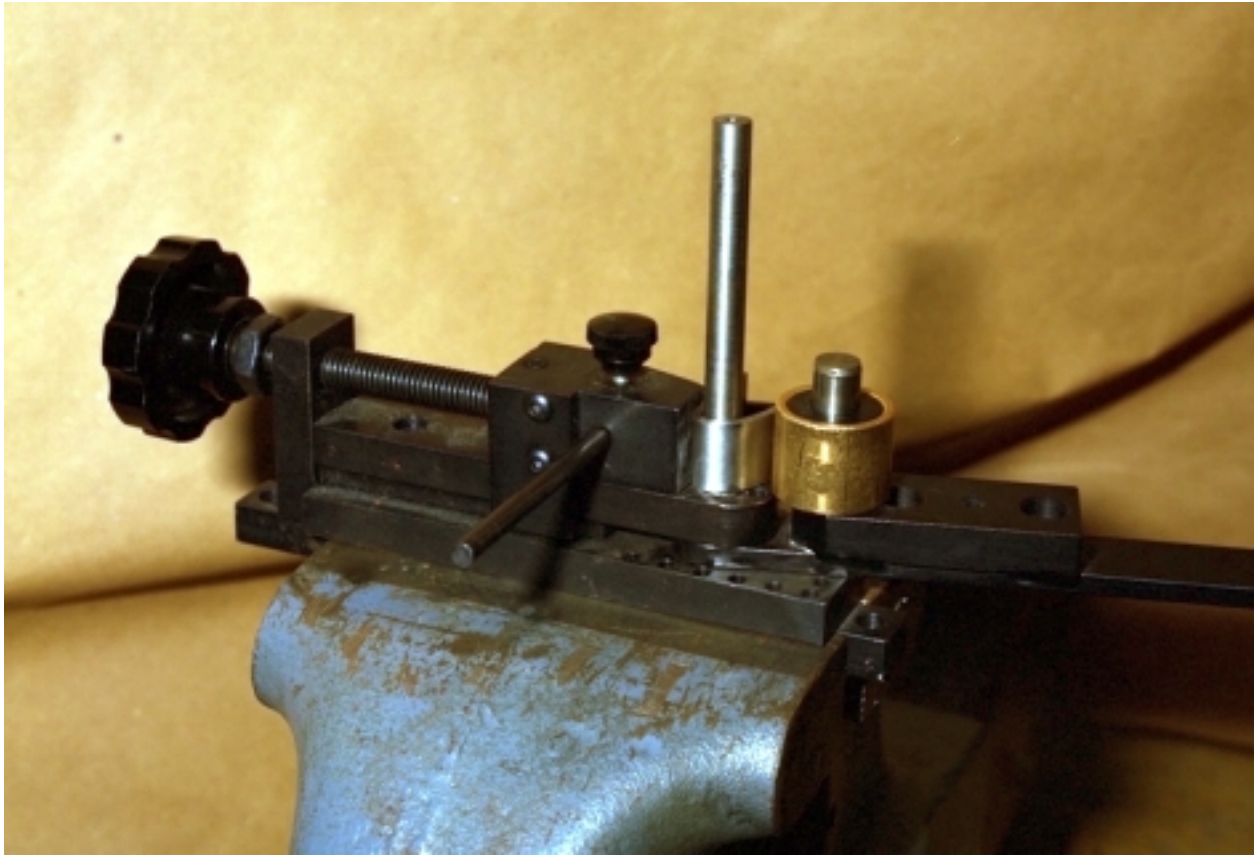


Figure 2. Bending Device for Stress Corrosion Samples.



Figure 3. Stress Corrosion Test Samples.

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1. MATERIAL COMPATABILITY

1.1. CORROSION

1.1.6 COPPER CORROSION

Objective

The objective of this test is to determine the copper corrosion characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause a copper corrosion rating of higher than 1a (Test Protocol).

Test Procedures

The copper corrosion characteristics of the manufacturer's suggested working concentration of the cleaning compound are determined using ASTM-D-130-94 (app C, ref 25) Standard Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test.

Two test specimens and one control specimen (3 by 0.5 by 0.125 in. of smooth-surfaced, hard-tempered, cold-finished copper of 99.9-percent purity) are used. The surfaces of the specimens are prepared using a fine-grade sandpaper to remove any imperfections or contaminants. Thirty mL of the manufacturer's suggested working concentration of the cleaning compound is placed in each of two 25- by 150-mm test tubes. Within 1 minute of cleaning the test specimens, they are to be placed into the test tubes with the manufacturer's suggested working concentration of the cleaning compound. The test tubes are then placed into the stainless steel copper corrosion bombs, the lids are screwed on tight, and they should be placed into a water bath maintained at 105 °F for 3 hours. After 3 hours, the corrosion bombs are removed from the bath and the contents are emptied into a clean beaker. Immediately, the test specimens are to be removed with stainless steel forceps and immersed in wash solvent, then removed and blotted dry. The specimens are then inspected for evidence of tarnishing, pitting, and corrosion and compared with the ASTM copper strip corrosion standard.

The above procedure in the preceding paragraph should then be repeated with the bath temperature set at 212 °F using two new specimens.

a. Two test specimens, 7.5 by 1.25 by 0.15 cm (3 by 0.5 by 0.125 in.), are prepared from smooth-surfaced, hard-tempered, cold-finished copper of at least 99.9-percent purity.

b. The 30 mL of manufacturer's suggested working concentration of the cleaning compound is placed into a 25- by 150-mm test tube.

c. The surfaces of the test specimens are prepared as outlined in ASTM-D-130-94, ensuring that the surfaces were free of imperfections and contaminants.

d. Within 1 minute of sample surface preparation, the test specimen should be placed into the test tube with the manufacturer's suggested working concentration of the cleaning compound and the test tube should be stoppered with a vented cork.

e. The test tube is placed into a water bath maintained at 100 °C (212 °F).

f. The test tube is to remain in the bath for 3 hours.

g. The test tube is removed from the bath and the contents are emptied into a clean, tall form beaker.

h. The test specimen is immediately removed with stainless steel forceps, immersed in a wash solvent (P-D-680 (II)), and removed immediately.

i. The test specimen is then blotted dry with quantitative filter paper.

j. The test specimen is inspected for evidence of tarnishing, pitting, and corrosion and compared with the ASTM copper strip corrosion standard.

k. The steps in paragraphs b through j are repeated with the second test specimen.

l. The steps in paragraphs a through k are repeated except that the bath temperature is changed to 40.6 °C (105 °F) to mimic the operating temperature of the manufacturer's suggested working concentration of the cleaning compound. All test specimens should be photographically documented immediately upon completion of the test.

1. MATERIAL COMPATIBILITY

1.1. CORROSION

1.1.7 STEEL CORROSION

Objective

The objective of this test is to determine the steel corrosion characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause any steel corrosion when tested by ASTM D-130 modified (Test Protocol).

Test Procedures

The steel corrosion characteristics of the manufacturer's suggested working concentration of the cleaning compound are determined using ASTM D-130-94, Standard Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test.

Two test specimens and one control specimen (3 by 0.5 by 0.125 in. of SAE standard steel) are used. The surfaces of the specimens are prepared using 00 steel wool to remove any imperfections or contaminants. Thirty mL of the manufacturer's suggested working concentration of the cleaning compound is placed in each of two 25- by 150-mm test tubes. Within 1 minute of cleaning the test specimens, they should be placed into the test tubes with the manufacturer's suggested working concentration of the cleaning compound. The test tubes are then placed into the stainless steel copper corrosion bombs, the lids are screwed on tight, and they are placed into a water bath maintained at 105 °F for 24 hours. After the 24 hours, the corrosion bombs are removed from the bath and the contents are emptied into a clean beaker. Immediately, the test specimens are removed with stainless steel forceps and immersed in wash solvent then removed and blotted dry. The specimens are then inspected for evidence of tarnishing, pitting, and corrosion.

The above procedure in the preceding paragraph is then repeated with the bath temperature set at 212 °F using two new specimens.

a. Two test specimens, 7.5 by 1.25 by 0.15 cm (3 by 0.5 by 0.125 in.), from SAE Standard Steel are prepared as outlined in SAE J1703-95 (ref 28).

b. The 30 mL of the manufacturer's suggested working concentration of the cleaning compound is placed into a 25- by 150-mm test tube.

c. The surfaces of the test specimens are prepared by polishing with 00 steel wool, ensuring that the surfaces are free of imperfections and contaminants.

d. Within 1 minute of the sample surface preparation step, the test specimen is placed into the test tube with the manufacturer's suggested working concentration of the cleaning compound and stoppered with a vented cork.

e. The test tube is placed into a water bath maintained at 100 °C (212 °F).

f. The test tube is to remain in the bath for 24 hours.

g. The test tube is removed from the bath and the contents are emptied into a clean, tall form beaker.

h. The test specimen is immediately removed with stainless steel forceps, immersed in a wash solvent (P-D-680 (II)), and removed immediately.

i. The test specimen is blotted dry with quantitative filter paper.

j. The test specimen is immediately inspected for evidence of pitting, corrosion, permanent discoloration of the surface, or the formation of a layer of reaction products.

k. The steps in paragraphs b through j are then repeated with the second test specimen.

l. The process in the steps in paragraphs a through k is repeated with a second set of test samples except that the temperature for the step in paragraph e is changed to 40.6 °C (105 °F) to mimic the operating temperature of the manufacturer's suggested working concentration of the cleaning compound. All test specimens should be photographically documented immediately upon completion of the test.

1. MATERIAL COMPATIBILITY

1.1 CORROSION

1.1.8 BIMETAL CORROSION

Objective

The objective of this test is to determine the bimetallic corrosion characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause corrosion on a bimetallic couple (Test Protocol).

Test Procedures

The bimetallic couple corrosion characteristics of the manufacturer's suggested working concentration of the cleaning compound are determined using FED-STD-791C (app C, ref 26), Corrosiveness of Oil on a Bimetallic Couple, Test Method 5322.2.

a. Three steel disks (ASTM-A-322-91, composition G52986) are prepared as indicated in FED-STD-791C.

b. Three brass clips (QQ-B-613, composition 2) are prepared as indicated in FED-STD-791C, Test Method 5322.2.

c. Enough of the manufacturer's suggested working concentration of the cleaning compound is dripped over the polished surface of the steel disks so that the entire surface was covered.

d. The brass clips are then placed on the steel disks as indicated in Figure 1.

e. The test items are then to be placed into a humidity cabinet for ten days. The temperature of the cabinet is 27 °C (79 °F) and the relative humidity is 50 percent.

f. The test items are removed from the cabinet in ten days.

g. Marks are made around the area where the brass clips are positioned using a hard pencil.

h. The brass clips are then removed from the steel disks and the surface is wiped clean with a dry, lint-free cloth.

i. The marked-off area of the disk is inspected visually for signs of discoloration, etching, and pitting. All test specimens should be photographically documented immediately upon completion of the test.



Figure 1. Bimetal Couple Corrosion Test Specimen Pair.

1. MATERIAL COMPATABILITY

1.2 EFFECTS ON SURFACES

1.2.1 COATING ADHESION

Objective

The objective of this test is to determine the effects on coating adhesion of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause the primer coating to peel away from the substrate from any test panels after immersion in distilled water for 24 hours (Test Protocol).

Test Procedures

The effect of the manufacturer's suggested working concentration of the cleaning compound on coating adhesion is determined using the Federal Test Method Standard for Paint, Varnish, Lacquer and Related Materials: Methods of Inspection, Sampling and Testing, Method 6301.2, Adhesion (Wet) Tape Test (app C, ref 13), modified for this test.

- a. A set of six test panels, 10 by 10 by 0.635 cm (4 by 4 by 0.25 in.), are prepared from aluminum (2024-T3 bare) and anodized per MIL-A-8625, Type I.
- b. A second set of six test panels, 10 by 10 by 0.635 cm, are prepared from aluminum (2024-T3 clad) with a conversion coat per MIL-C-5541, Class 3.
- c. Both sets of test panels are dried for 24 hours at ambient (room) conditions.
- d. Both sets of panels are cleaned with the manufacturer's suggested working concentration of the cleaning compound.
- e. The test panels are allowed to dry at ambient (room) conditions.
- f. Three panels from each set are then painted with MIL-P-23377 primer and MIL-C-85285 topcoat.
- g. The other three panels from each set are painted with MIL-P-85582 primer and MIL-C-85285 topcoat.

h. The painted panels are allowed to dry for seven days at ambient (room) conditions
Figure 1.

i. All of the test panels are immersed in distilled water for 24 hours.

j. The test specimen should then be removed from the water and wiped dry with a clean cloth.

k. Within 1 minute of removal from the water, two parallel scratches, 2.54 cm (1 in.) apart, are made through the coatings to the metal with a stylus.

l. A 2.54-cm wide strip of masking tape is applied across the scratches.

m. The tape should be pressed into the surface by rolling the tape with a 2.05-kg (4.5-lb) rubber-covered roller eight times.

n. The tape is then removed with one quick motion.

o. The damage to the intercoat or surface adhesion is examined. All test specimens should be photographically documented immediately upon termination of the test.

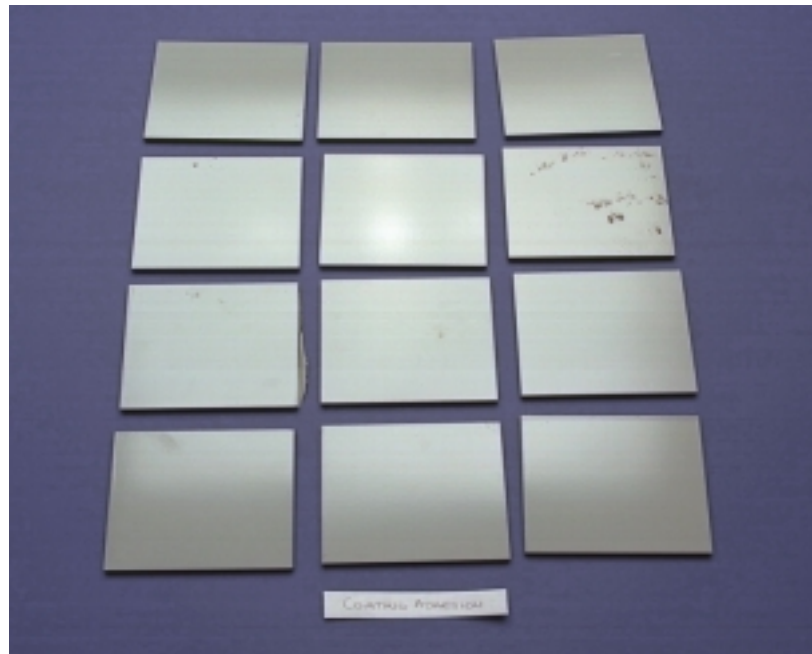


Figure 1. Coating Adhesion Test Panels

1. MATERIAL COMPATIBILITY

1.2 EFFECTS ON SURFACES

1.2.2 EFFECTS ON PAINTED SURFACES

Objective

The objective of this test is to determine the effects on painted surfaces of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause streaking, discoloration, blistering or a permanent decrease in film hardness of more than one (1) pencil hardness level on any painted surfaces. This criterion does not apply to MIL-L-46159 (Test Protocol).

Test Procedures

The effect of the manufacturer's suggested working concentration of the cleaning compound on the painted surfaces is determined using ASTM F-502-93 (app C, ref 15), Standard Test Method for Effects of Cleaning and Chemical Maintenance Materials on Painted Aircraft Surfaces, modified by the Test Protocol.

- a. The 36 test panels of aluminum Alclad 7075-T6, 76 by 152 by 0.5 mm (3 by 6 by 0.02 in.) are prepared.
- b. All of the test panels are cleaned with acetone, dried, and abraded lightly with a fine aluminum oxide mat. The surface is then rinsed with distilled water.
- c. All of the test panels are coated with a conversion coat conforming to MIL-C-81706, Class 1A, and allowed to dry.
- d. One coat of MIL-P-23377 epoxy primer, 0.6 to 0.8 mil (0.010 to 0.015 mm), is applied to the test panels.
- e. The test panels are allowed to dry for 1 hour at ambient (room) conditions.
- f. Each of the following coatings is to be applied to on six test panels:
 - (1) MIL-C-22750 epoxy topcoat.
 - (2) MIL-C-85285 polyurethane, high-solids topcoat.
 - (3) MIL-C-46168 aliphatic polyurethane, single-component topcoat.

- (4) MIL-L-46159 lacquer, acrylic, low reflective.
- (5) MIL-P-14105 heat-resistant paint.
- (6) MIL-E-52891B enamel, lusterless, zinc phosphate, styrenated alkyd type was not tested because the material was not available. No substitute paint could be found.
- g. The coatings in paragraph f are to be applied as a mist coat and allowed to dry for 30 minutes in ambient (room) conditions.
- h. An additional three coats of the coatings should be applied in 0.010 to 0.015mm applications and allowed a 1-hour drying time at ambient (room) conditions between each coat.
- i. The test panels are allowed to dry for four days at ambient (room) conditions.
- j. The test panels are then baked for 24 hours in a mechanical convection oven at 82 °C (180 °F).
- k. A set of drawing pencils is prepared as described in the Test Plan.
- l. The test panels are tested for hardness in the following manner:
 - (1) Pencils are held in decreasing hardness by hand at a 45° angle (fig. 2.2.3-1).
 - (2) The pencil is pushed across the paint film with firm, uniform pressure.
 - (3) The step in paragraph l(2) is repeated with decreased pencil hardnesses until a pencil was found not to cut the film but to leave a black mark on the surface.
 - (4) The hardness of the pencil that cut the film should be recorded as the hardness of the paint.
 - (5) The steps in paragraphs l(1) through (4) are repeated three times.
- m. The paint hardness is not allowed to differ by more than one pencil hardness unit for each of the same type of paint test panels.
- n. Three of the test panels with each of the different types of coating are then placed into an oven (horizontally) at 38 ± 2 °C (100 ± 5 °F).
- o. The manufacturer's suggested working concentration of the cleaning compound is applied to one-half of each of the test panels.
- p. The cleaner is allowed to remain on the test panels for 30 minutes.

q. The test panels are then rinsed with distilled water and allowed to air dry for 24 hours under ambient (room) conditions.



Figure 2.2.3-1. Test for hardness of the painted surface.

- r. The test panels are visually examined for streaking, discoloration, or blistering.
- s. The steps in paragraphs l(1) through (4) are repeated for both the exposed and the unexposed halves of each panel.

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1. MATERIAL COMPATABILITY

1.2 EFFECTS ON SURFACES

1.2.3 EFFECTS ON BONDING

This procedure is currently under review and may be revised by ARL and AMCOM who are the original proponents of the procedure.

Objective

The objective of this test is to determine the effects on bonding of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause lower percentage of bondline cohesive failure compared to the baseline surface cleaner or solvent (Test Protocol).

Test Procedures

The effect that the manufacturer's suggested working concentration of the cleaning compound has on the peel resistance of adhesives is determined using ASTM D-3167-93 (app C, ref 21), Standard Test Method for Floating Roller Peel Resistance of Adhesives, modified for different temperatures and includes surface preparation methods outlined in ASTM D-3933-93 (app C, ref 22), Standard Guide for Preparation of Aluminum Surfaces for Structural Adhesives Bonding (Phosphoric Acid Anodizing), modified by the Test Plan.

a. The following materials are used to fabricate a test panel: aluminum 7075-T6 bare, stainless steel AM-355, titanium 6AL-4V, and nickel (electroformed) 4340 steel. A minimum of six test specimens should be prepared for each material type for both the manufacturing and repair simulations. The test is conducted for both the baseline solvent (what is presently used for each of the scenarios) and the cleaning compound.

b. The test panels are fabricated from two pieces of the same material with the following dimensions:

- (1) Flexible adhered 3.62- by 25.4-cm by 0.63-mm (3- by 10- by 0.025-in.).
- (2) Rigid adhered 3.62- by 20.32-cm by 1.63-mm (3- by 8- by 0.064-in.).

c. A standardized contaminant mixture is made from two parts (by weight) of hydraulic fluid (MIL-H-83282) and one part (by weight) of lubricating grease (MIL-G-81322).

d. The standardized contaminant mixture is applied to the test panels, baked for 2 hours at 54 °C (130 °F), and allowed to cool to ambient (room) temperature.

e. The test panels are then cleaned with the manufacturer's suggested working concentration of the cleaning compound, and allowed to air dry at ambient (room) temperature for 1 hour.

f. For simulating the manufacturing process, the steps in paragraphs g through l should be followed. For simulating the repair process, the steps in paragraphs m through s should be followed.

g. To activate the test panel surfaces for bonding for the manufacturing simulation, the following methods are used for the respective materials:

(1) Aluminum 7075-T6. The surface is prepared in accordance with ASTM D-3933-93.

(2) Electroplated nickel.

(a) The test materials are immersed in a solution of Turco (HTC) and deionized water (10 oz of Turco HTC/1 gal. of solution) for 10 minutes at 82 to 93 °C (180 to 200 °F) with an agitation system engaged.

(b) Then the materials are immediately immersed (being careful to not allow any Turco HTC solution to dry on the metal) in a deionized water rinse tank for 4 minutes at 25 °C with an agitation system engaged.

(c) The materials are rinsed in running deionized water at 25 °C.

(d) A Water-Break test per ASTM F-22-65 is then performed to verify the cleanliness of the surface.

(e) The materials are immersed in a solution of 50-percent by volume ferric chloride (40-percent solution) and 50-percent by volume hydrochloric acid (37-percent solution) for 5 minutes at 25 °C.

(f) The materials are then immersed in an agitated deionized water bath for 4 minutes at 25 °C.

(g) The materials are rinsed in running deionized water for 4 minutes at 25°C.

(h) The materials are then immersed in a de-smut solution (35-percent by volume nitric acid, 15-percent by volume Turco 4104, 50-percent by volume deionized water) for 6 to 30 seconds as required at 25 °C. The panels are not allowed to exhibit any shiny areas after de-smutting. All areas must to exhibit a uniform, dull matte finish.

(i) The materials are immersed in agitated deionized water for 4 minutes at 25 °C.

(j) The materials are rinsed in running deionized water at 25 °C.

(k) The materials are then immersed in deionized water for 2 minutes at 80 to 90 °C.

(l) The materials are dried for 30 minutes at 65°C.

(m) The primer in the next section of the test procedure is applied within 2 hours of completing the drying cycle.

(3) Steel, AM-355.

(a) The test materials are immersed in a solution of Turco HTC and deionized water (10 oz of Turco HTC/1 gal. of solution) for 10 minutes at 82 to 93 °C (180 to 200 °F) with an agitation system engaged.

(b) The materials are immediately immersed (being careful not to allow any Turco HTC solution to dry on the material) in a deionized water rinse tank for 4 minutes at 50 to 65 °C (120 to 150 °F) with an agitation system engaged.

(c) The materials are rinsed in running deionized water at 25 °C.

(d) A Water-Break test per ASTM F-22-65 is performed to ensure the cleanliness of the materials.

(e) The materials are then immersed in a solution of 50-percent by volume ferric chloride (40-percent solution) and 50-percent by volume hydrochloric acid (37-percent solution) for 5 minutes at 25 °C.

(f) The materials are immersed in agitated deionized water for 4 minutes at 25 °C.

(g) The materials are rinsed in running deionized water at 25 °C.

(h) The materials are then immersed in agitated deionized water for 4 minutes at 65 °C (150 °F).

(i) The materials are allowed to dry for 30 minutes at 79 °C (175 °F).

(j) The primer applied in the next section of the test procedures is done within 2 hours of completing the drying cycle.

(4) Titanium 6AL-4V.

(a) The test materials are immersed in a solution of Oakite 90 alkaline cleaner and deionizing water (12 oz of Oakite 90/1 gal. of water) for 10 minutes at 65 to 75 °C with an agitation system engaged.

(b) The materials are then immediately immersed (being careful not to allow any Oakite 90 solution to dry on the material) in a deionized water rinse tank for 4 minutes at 25 °C with an agitation system engaged.

(c) The materials are rinsed in running deionized water at 25 °C.

(d) A Water-Break test is performed per ASTM F-22-65.

(e) The materials are immersed in an aqueous pickle solution (2 to 3 oz of hydrofluoric acid/1 gal. of solution, 1.5 to 2.5 oz of sodium sulfate/1 gal. of solution, 40.0 to 50.0 oz of nitric acid (70 percent)/1 gal. of solution, balance deionized water) for 2.5 minutes at 24 °C.

(f) The materials are then immersed in agitated deionized water for 4 minutes at 24 °C (75 °F).

(g) The materials are rinsed in running deionized water at 25 °C.

(h) The materials are then immersed in a conversion-coating aqueous solution (6.5 oz of trisodium phosphate/1 gal. of solution, 2.5 to 3.0 oz of potassium fluoride/1 gal. of solution, 2.2 to 2.5 oz of hydrofluoric acid (70 percent)/1 gal. of solution, balance deionized water) for 1.5 to 2.5 minutes at 25 °C.

(i) The materials are then immersed in agitated deionized water for 4 minutes at 25 °C.

(j) The materials are rinsed in running deionized water at 25 °C.

(k) The materials are immersed in agitated deionized water for 15 minutes at 65 to 70 °C.

(l) The materials are rinsed in running deionized water at 25 °C.

(m) The materials are allowed to dry for 30 minutes at 70 °C.

(n) The application of the primer in the next section of the test procedure is done within 2 hours of completing the drying cycle.

h. The primer EA 9210H is applied to each of the test panels to a dry film thickness of between 0.25 and to 0.35 mil.

i. The test panels are then air dried for 30 minutes, and then oven cured for 30 minutes at a temperature of 149 °C (300 °F).

j. The flexible test panel is adhered to the rigid test panel using the film adhesive EA 9628H and allowed to cure to the manufacturer's specifications.

k. The test panels are then cut lengthwise in 1.27-cm (5-in.) sections, as shown in Figure 1, in a method that is not deleterious to the bond.



Figure 1. Test panel and test specimen.

l. The floating roller peel tests are performed in accordance with ASTM D-3167-93, using a crosshead speed of 12.7 cm/minute (5.0 in./min) for the peel test instead of 15.24 cm/minute (6.0 in./min). Six test specimens are conditioned and tested for each substrate preparation as follows:

(1) One set is conditioned at -55 °C (-67 °F)/dry for 24 hours minimum. Testing is performed at -55 °C (-67 °F).

(2) One set is conditioned at 25 °C (75 °F)/ambient humidity for 24 hours minimum. Testing was performed at 25 °C (75 °F).

(3) One set is conditioned at 80 °C (180 °F)/condensing humidity environment for 30 days. Testing is performed at 80 °C (180 °F).

m. For the repair simulation process, the test panels are prepared using the steps in paragraphs a through e.

n. The surface of all the test panels is abraded with 220 grit paper.

o. The test panels are hand wiped with a clean cloth soaked with the manufacturer's suggested working concentration of the cleaning compound until no visible residue remained on the cloth.

p. Immediately, the test panels are wiped with deionized water. The excess water is wiped off of the test panels with a clean cloth and allowed to air dry at ambient (room) temperature for 1 hour.

q. The rigid adherend is adhered to the flexible adherend from the same material using EA 9309 paste adhesive.

r. The test panels are allowed to cure to the manufacturer's specifications.

s. The floating roller peel tests are performed in accordance with ASTM-D-3167-93, using a crosshead speed of 12.7 cm/minute (5.0 in./min) for the peel test instead of 15.24 cm/minute (6.0 in./min) (see Figure 2). Six test specimens are prepared and conditioned for each substrate preparation as follows:

(1) One set is conditioned at -55 °C (-67 °F)/dry for 24 hours minimum. Testing is performed at -55 °C.

(2) One set is conditioned at 25 °C (75 °F)/ambient humidity for 24 hours minimum. Testing is performed at 25 °C (75 °F).

(3) One set is conditioned at 80 °C/condensing humidity environment for 30 days. Testing is performed at 80 °C (180 °F).

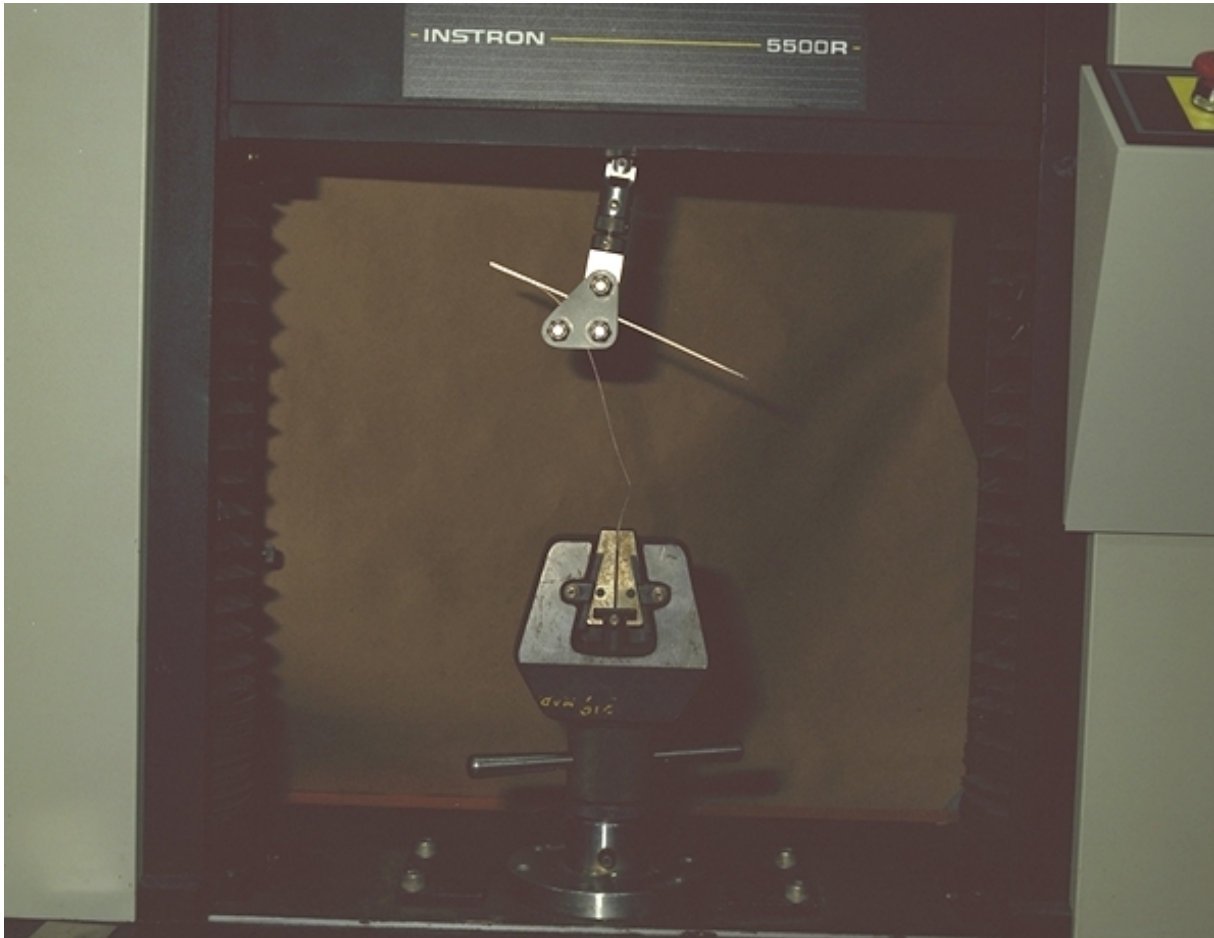


Figure 2.28.3-2. Float Roller Peel Test Fixture.

1. MATERIAL COMPATIBILITY

1.3 PLASTICS/SEALANTS

1.3.1 EFFECTS ON POLYIMIDE WIRE

Objective

The objective of this test is to determine the effects on polyimide wire of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause cracking, discoloration, dissolution, dielectric breakdown of the polyimide insulated wire in excess of that produced by distilled water (Test Protocol).

Test Procedures

The effect that the manufacturer's suggested working concentration of the cleaning compound has on the polyimide-insulated wire is determined using MIL-C-87937B, paragraph 4.6.26.

- a. Two lengths of approximately 61 cm (24 in.) of MIL-W-81481/11-20 wire are cut.
- b. Each segment is then coiled into separate 118-mL (4-oz) widemouth jars.
- c. Enough of the manufacturer's suggested working concentration of the cleaning compound is added to one jar to completely cover the wire coil.
- d. Enough distilled water is added to the other jar to completely cover the wire coil.
- e. Both jars are capped and stored at ambient (room) temperature 20 to 25 °C (68 to 77 °F) for 14 days.
- f. After 14 days, both coils are removed and rinsed thoroughly with distilled water.
- g. Both coils are then suspended and allowed to dry completely.
- h. When dry, the coils are examined for dissolution.
- i. Both coils are then double-reverse wrapped on a 0.3-cm (0.125-in.) mandrel and observed for cracking.

j. If the test wire cracks, the test is ended. If the control wire cracks, the test is invalidated and must be rerun.

k. The wires are then subjected to a 1-minute dielectric test of 2500 volts (rms).

l. The wires are then examined for breakdown and/or leakage. All wires should be photographically documented immediately upon completion of the test

1. MATERIAL COMPATABILITY

1.3 PLASTICS/SEALANTS

1.3.2 EFFECTS ON ACRYLIC PLASTICS

Objective

The objective of this test is to determine the effects on acrylic plastic of the manufacturer's suggested working concentration of the cleaning compound.

Criteria

The manufacturer's suggested working concentration of the cleaning compound shall not cause stress crazing or staining of acrylic plastics (Test Protocol).

Test Procedures

The stress-crazing effect that the manufacturer's suggested working concentration of the cleaning compound has on acrylic plastics is determined using ASTM F-484-83 (app C, ref 19), Standard Test Method for Stress Crazing of Acrylic Plastics in Contact with Liquid or Semi-Liquid Compounds.

a. Four test specimens are prepared and conditioned in accordance with ASTM F-484-83, paragraphs 5 and 6, for each of the following materials:

(1) Type A - MIL-P-5425, finish A.

(2) Type B - MIL-P-8184, finish B.

(3) Type C - MIL-P-25690.

b. Testing is conducted at 23 ± 5 °C (75 ± 10 °F).

c. Each test specimen is loaded as a cantilever beam as shown in Figure 1.

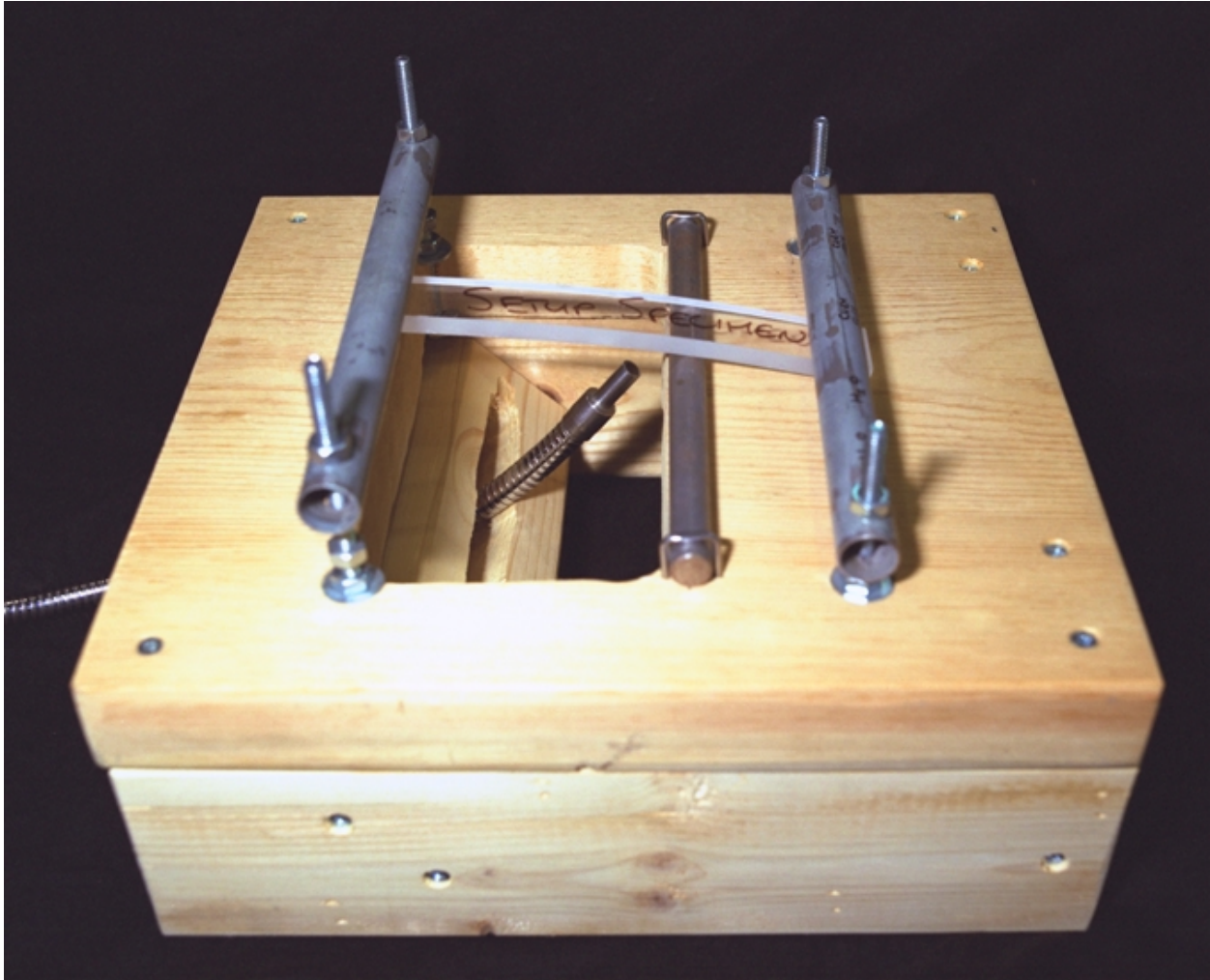
d. The following loads are to be applied:

(1) Type A - 3000 psi (20685 kPa) outer fiber stress.

(2) Type B - 3500 psi (24132 kPa) outer fiber stress.

(3) Type C - 4500 psi (31027 kPa) outer fiber stress.

Figure 1. Plastic Loading Test Fixture.



- e. The test specimens are stressed for 10 minutes.
- f. The test specimens are examined for crazing as prescribed in ASTM F-484-83.
- g. If no crazing occurs, testing proceeds. If crazing occurs, a new test specimen is used and the loading test is repeated.
- h. The test specimen is loaded again to the proper load.

- i. An absorbent cotton swatch, 12.7-mm (0.5-in.) square, is placed directly over the fulcrum on the tension surface in the middle of the test specimen's width.
- j. The cotton swatch is completely soaked with the manufacturer's suggested working concentration of the cleaning compound.
- k. At no time is the manufacturer's suggested working concentration of the cleaning compound allowed to migrate to within 3.2 mm (0.125 in.) of the edge of the test specimen.
- l. The test specimen is inspected for crazing as in paragraph f, after loading of 0.5, 1, 2, 4, and 8 hours. Inspections are conducted by sliding the swatch 5.08 cm (2 in.) towards the loaded end of the test specimen.
- m. Testing is terminated at any point when crazing or degradation is observed.
- n. The steps in paragraphs c through m are repeated for each type of acrylic.
- o. The steps in paragraphs c through n, are repeated with the substitution of distilled water conforming to Specification D-1193, Type IV, for the manufacturer's suggested working concentration of the cleaning compound in paragraph j. All test specimens should be photographically documented immediately upon completion of the test.

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1. MATERIAL COMPATABILITY

1.3 PLASTICS/SEALANTS

1.3.3 EFFECTS ON POLYCARBONATE PLASTICS

Objective

The objective of this test is to determine the effects on polycarbonate plastic of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause stress crazing or staining of polycarbonate plastics (Test Protocol).

Test Procedures

The stress-crazing effect that the manufacturer's suggested working concentration of the cleaning compound has on polycarbonate plastics is determined using ASTM F-484-83, Standard Test Method for Stress Crazing of Acrylic Plastics in Contact with Liquid or Semi-Liquid Compounds.

- a. Four test specimens should be prepared and conditioned in accordance with ASTM F-484-83, paragraphs 5 and 6, using polycarbonate plastic conforming to MIL-P-83310.
- b. Testing is conducted at 23 ± 5 °C (75 ± 10 °F).
- c. Each test specimen is loaded as a cantilever beam, as shown in Figure 1.
- d. The following load is applied: Type B - 3500 psi (24132 kPa) outer fiber stress.
- e. The test specimen is stressed for 10 minutes at the above load.
- f. The test specimen is then examined for crazing.
- g. If no crazing occurs, the test should proceed. If crazing occurs, then a new test specimen should be made and the preceding steps are repeated.
- h. The test specimen is again loaded to the stated load.
- i. An absorbent cotton swatch, 12.7 mm (0.5 in.) square, is placed directly over the fulcrum on the tension surface in the middle of the test specimen's width.
- j. The swatch is then completely soaked with the manufacturer's suggested working concentration of the cleaning compound.

k. The manufacturer's suggested working concentration of the cleaning compound is not allowed to migrate to within 3.2 mm (0.125 in.) of the edge of the test specimen.

l. The test specimen is then inspected for crazing as in paragraph f, after loading of 0.5, 1, 2, 4, and 8 hours. Inspections are conducted by sliding the swatch 5.08 cm (2 in.) towards the loaded end of the test specimen.

m. Testing is terminated when crazing is observed.

n. The steps in paragraphs c through m are repeated once more with the manufacturer's suggested working concentration of the cleaning compound, and then twice more with distilled water conforming to Specification D-1193, Type IV. All test specimens should be photographically documented immediately upon termination of the test.

1. MATERIAL COMPATABILITY

1.3 PLASTICS/SEALANTS

1.3.4 EFFECTS ON SEALANT PEEL STRENGTH

Objective

The objective of this test is to determine the effects on sealant peel strength of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall cause the sealing compound to have a minimum peel strength of 20 pounds force per inch (lbf/in.) after a seven (7) day exposure in jet reference fluid. There shall also be 100 percent cohesive failure (Test Protocol).

Test Procedures

The effect the manufacturer's suggested working concentration of the cleaning compound has on sealant peel strength is determined by using a method presented in the Test Plan.

- a. Two sets of eight test panels, 15.24- by 7- by 0.1-cm (6- by 2.75- by 0.04-in.) aluminum 2024-T3, are prepared.
- b. The primer MIL-P-23377 is applied to one set of panels.
- c. The primer MIL-P-85582 is applied to the other set.
- d. A standardized contaminant mixture of two parts (by weight) of hydraulic fluid (MIL-H-83282) and one part (by weight) of lubricating grease (MIL-G-81322) is prepared.
- e. The contaminant mixture is applied to all the test panels and baked for 2 hours at 54 °C (130 °F).
- f. The test panels are cooled to ambient (room) temperature.
- g. The gross contaminant is removed from the test panels with a clean dry cloth.
- h. Four of each set of test panels are wiped five times with a clean cloth soaked with the manufacturer's suggested working concentration of the cleaning compound.

- i. The test panels are then cleaned with distilled water.
- j. All of the test panels primed with MIL-P-85582 should be scuff sanded with abrasive mats (MIL-A-9962, Type I, Class I, Grade A).
- k. The steps in paragraphs g through j are then repeated.
- l. Each test panel is coated with MIL-S-8802, Type I, Class B sealing compound. The coating must be at least 12.7 cm (5 in.) of only one side and only 0.3175 ± 0.0396875 cm (0.125 ± 0.015625 in.) thick.
- m. The 16 strips, 30.5- by 7-cm (12- by 2.75-in.), are cut of 20 to 40 mesh aluminum, monel wire fabric, or cotton duck (CCC-C-419, Type III).
- n. The fabric is impregnated with the sealant. The sealant should cover 12.7 cm at one end on both sides.
- o. The sealant-impregnated end of the fabric is placed on the sealant-coated test panels. The sealant is smoothed down so that no air was trapped under the fabric.
- p. An additional 0.79375 mm (0.03125 in.) of sealant is applied over the fabric end attached to the test panel.
- q. The sealant is cured in accordance with the manufacturer's suggested methods.
- r. A jet reference fluid is made using the following formulation:
 - (1) Toluene (TT-T-548), 30 volumes.
 - (2) Cyclohexane (technical grade), 60 volumes.
 - (3) Iso-octane (TT-S-735, Type I), 10 volumes.
 - (4) Tertiary dibutyl disulfide (Doctor Sweet), 1 volume.
 - (5) Tertiary butyl mercaptan, 0.015 ± 0.0015 weight percentage of the other four components.
- s. The test panels are immersed into the jet reference fluid for seven days at 60 ± 1.1 °C (140 ± 2 °F). The test panels are completely immersed in the jet reference fluid.
- t. The test panels are cooled in the jet reference fluid for 24 hours.

u. The peel strength is measured within 10 minutes of removal from the jet reference fluid.

v. A 2.5-cm (1-in.) wide section is cut into two places on the sealant sections of each test panel.

w. The test panel is then tested by stripping back at an angle of 180° to the test panel in a tensile testing machine with a jaw separation rate of 5 cm (2 in.) per minute (Figure 1).

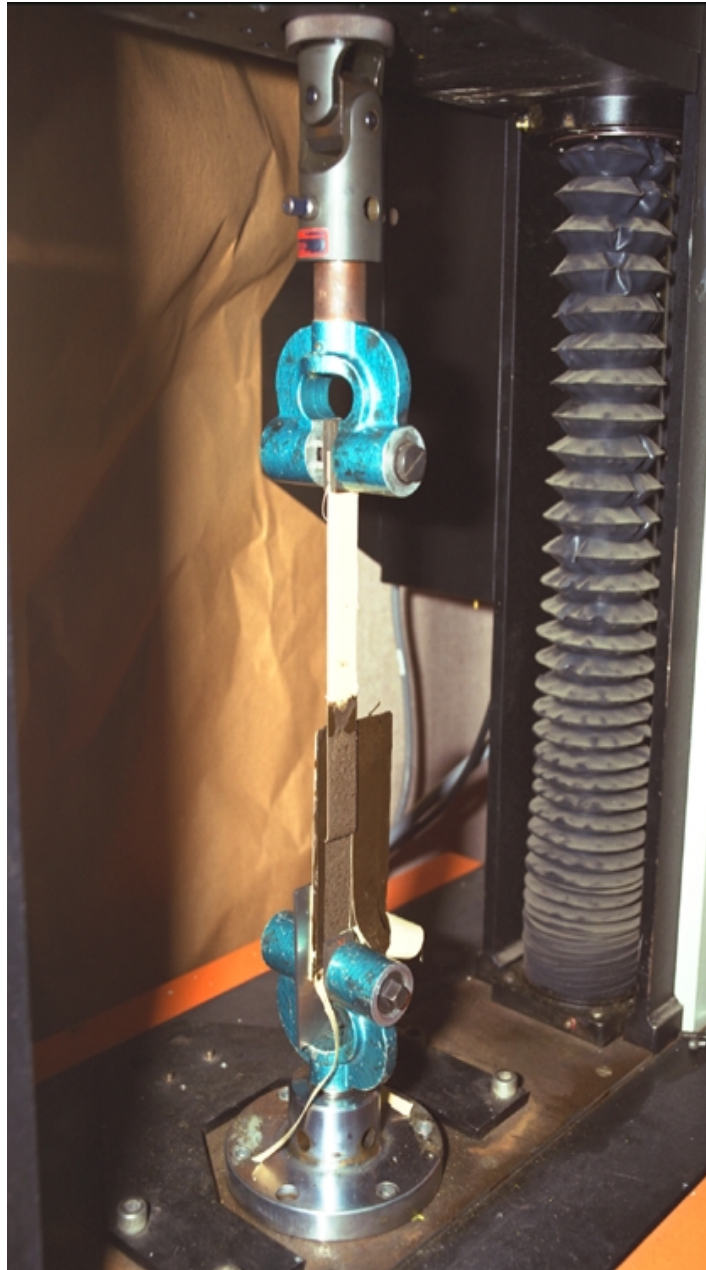


Figure 1. Sealant Peel Testing.

x. During the peel strength testing, three cuts are made through the sealant compound to the panel at approximately 2.5-cm (1-in.) separations. This is done to promote adhesive failure.

y. The peak load values are then recorded.

1. MATERIAL COMPATABILITY

1.3 PLASTICS/SEALANTS

1.3.5 EFFECTS ON POLYSULFIDE SEALANTS

Objective

The objective of this test is to determine the effect on polysulfide sealant of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not change the durometer hardness of polysulfide sealants more than 5 units (Test Protocol).

Test Procedures

The effect that the manufacturer's suggested working concentration of the cleaning compound has on polysulfide sealants is determined using MIL-C-87937B, paragraph 4.6.19.

- a. Four test specimens should be prepared from MIL-S-81733, Type 1, and MIL-S-8802, Type 1 sealants sheet stock.
- b. It is assumed that these materials do not have hardnesses that depend on humidity.
- c. The sheet stock is made by mixing the sealants as specified by the manufacturer and pressing each into a 0.3175-cm (0.125-in.) thick sheet mold (Figures 1 through 3).
- d. The sealant is cured for seven days at 49 °C (120 °F).
- e. All testing is conducted at ambient (room) temperature.
- f. Two test specimens from each sealant are immersed into the manufacturer's suggested working concentration of the cleaning compound.
- g. The test specimens are allowed to soak for 30 minutes.
- h. The test specimens are removed from the manufacturer's suggested working concentration of the cleaning compound and rinsed with cool tap water.
- i. The test specimens are then tested for a Shore A hardness in accordance with ASTM D-2240-95 within 30 minutes of removal from the cleaning compound.

- j. The two remaining test specimens (unexposed) are tested for Shore A hardness in accordance with ASTM D-2240-95.



Figure 1. MIL-S-81733, Type I Sealant and the Molds.

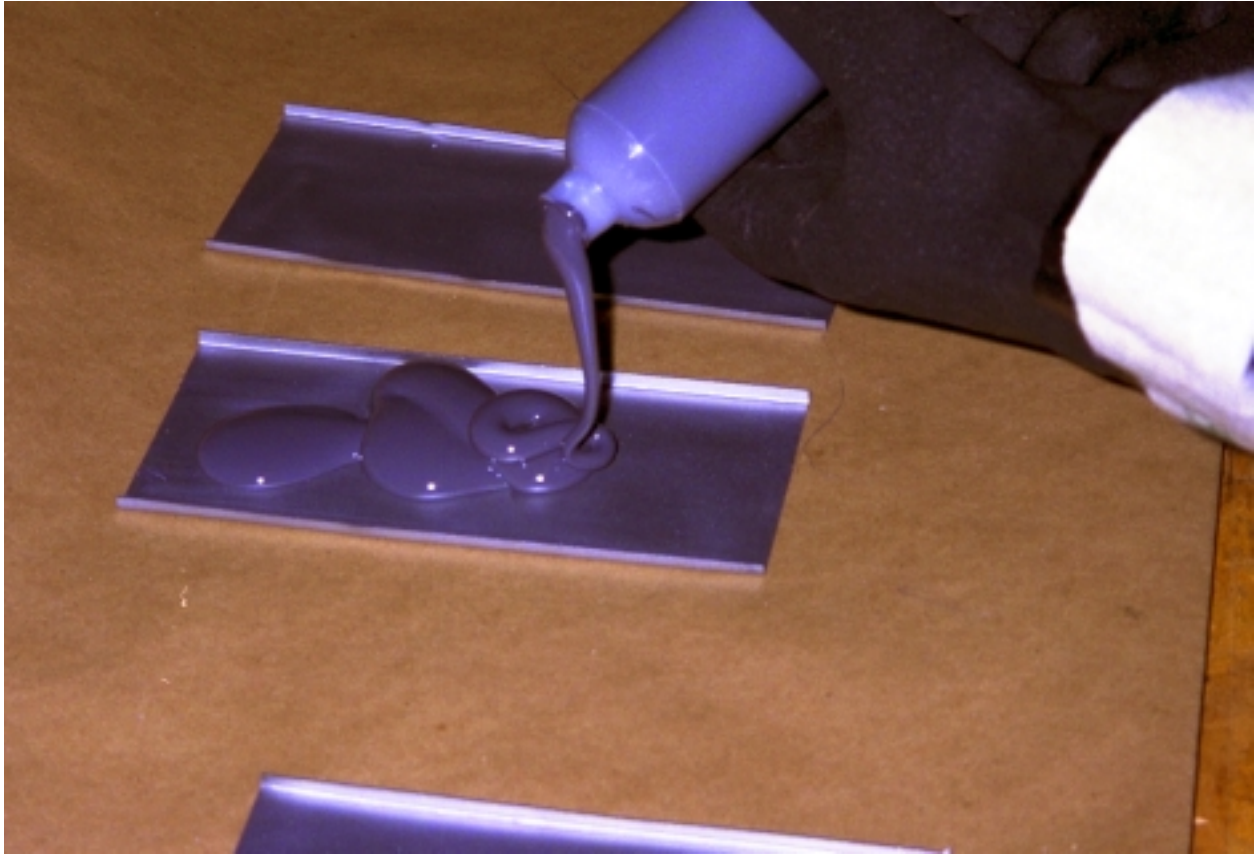


Figure 2. MIL-S-81733, Type I Sealant Being Formed in Molds.



Figure 3. MIL-S-81733, Type I Sealant Cured Sheet.

1. MATERIAL COMPATABILITY

1.3 PLASTICS/SEALANTS

1.3.6 RUBBER COMPATIBILITY

Objective

The objective of this test is to determine the rubber compatibility characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not change the durometer hardness of rubber materials more than 5 units (Test Protocol).

Test Procedures

The manufacturer's suggested working concentration of the compatibility of the cleaning compound and rubber compounds is determined using ASTM D-2240-95 (app C, ref 20), Standard Test Method for Rubber Property - Durometer Hardness.

- a. Three test specimens should be prepared from AMS 3204 and AMS 3209 rubbers in accordance with ASTM D-2240-95.
- b. It is assumed that these materials do not have hardnesses that depend on humidity.
- c. All testing is conducted at 23 ± 2 °C (73.4 ± 3.6 °F).
- d. A cotton swatch is placed on the test specimen.
- e. The cotton swatch is saturated with the manufacturer's suggested working concentration of the cleaning compound.
- f. The swatch is allowed to remain on the test specimen for the following exposure times: 30 minutes and 1, 2, 4, and 8 hours.
- g. The swatch is removed and the durometer hardness is measured as outlined in ASTM D-2240-95 after each of the exposure times (figure 1). The same test specimen should be used for all of the exposure times. The final durometer hardness measurement is an average of five readings.

h. The same cotton swatch is replaced on the test specimen after each hardness measurement.



Figure 1. Durometer measurement of rubber specimen.

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2. CLEANER EVALUATION

2.1 CHARACTERISTICS

2.1.1 FLASH POINT

Objective

The objective of this test is to determine the flash point characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The flash point of the manufacturer's suggested working concentration of the cleaning compound shall be greater than 212 °F (100 °C) (Test Protocol) (app C, ref 1).

Test Procedures

a. The flash point of the manufacturer's suggested working concentration of the cleaning compound is determined using the method outlined in American Society for Testing and Materials (ASTM) D-92-90 (app C, ref 3), Standard Test Method for Flash and Fire Points by Cleveland Open Cup.

b. The following procedures are to be used:

(1) The test apparatus is prepared, as seen in Figure 1, the samples are taken, and all cautionary statements in ASTM D-92-90 should be applied.

(2) The test cup is filled with the manufacturer's suggested working concentration of the cleaning compound.

(3) The test flame on the Cleveland test apparatus is lit and adjusted to 3.2 to 4.8 mm (0.125 to 0.1875 in.).

(4) The sample should initially be heated at a rate of 14 to 17 °C (25 to 30 °F) per minute.

(5) During the last 28 °C (50 °F), the sample should be heated at a rate of 5 to 6 °C (9 to 11 °F) per minute.

(6) During the last 28 °C, every 2 °C (5 °F), the test flame is applied across the center of the test solution cup.

(7) The test flame applications are continued until the test solution is raised to 100 °C.

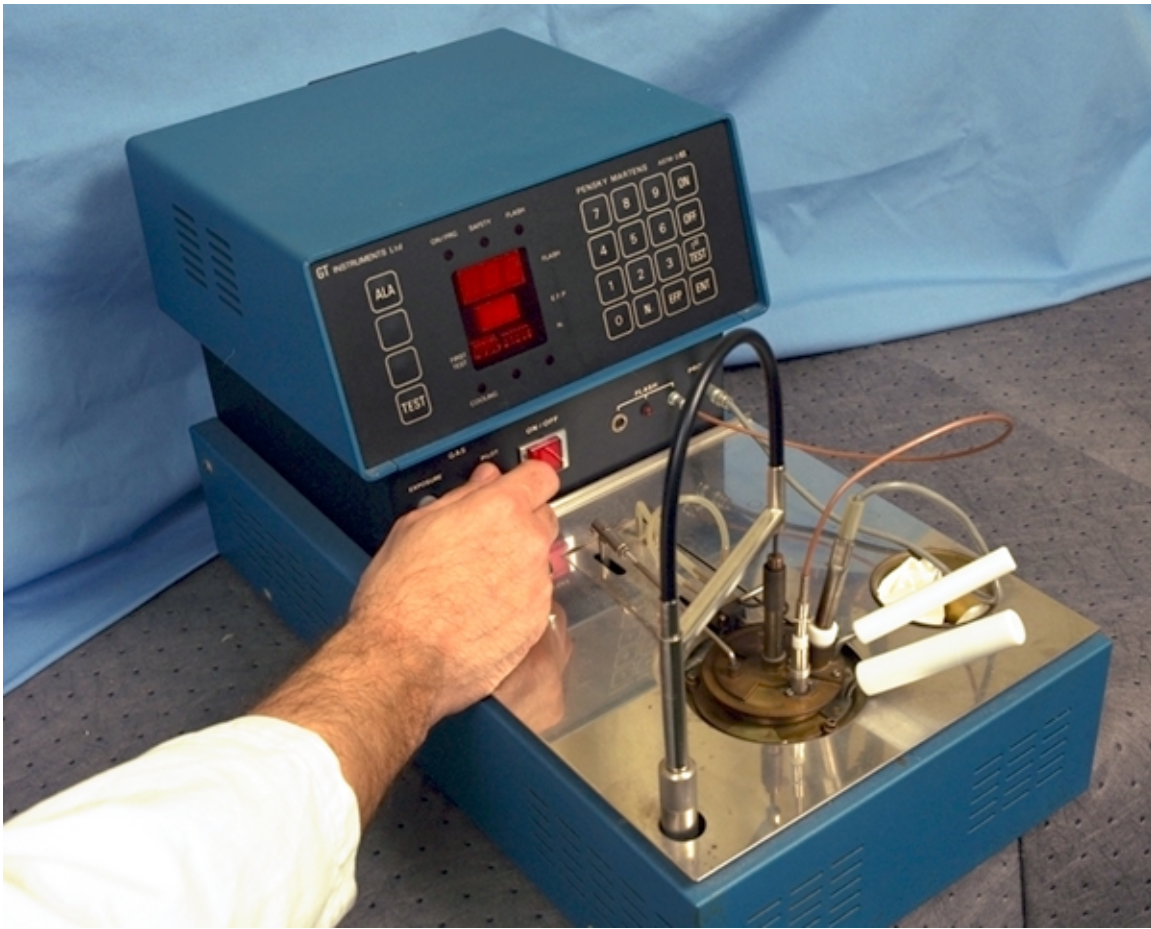


Figure 1. Cleveland Open Cup Flash Point Setup.

2. CLEANER EVALUATION

2.1 CHARACTERISTICS

2.1.2 pH

Objective

The objective of this test is to determine the pH of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The pH value of the manufacturer's suggested working concentration of the cleaning compound shall be no less than 5 and no more than 10 when in its most concentrated form (Test Protocol).

Test Procedures

The pH value of the manufacturer's suggested working concentration of the cleaning compound is to be determined using the method outlined in ASTM E-70-90 (app C, ref 4), Standard Test Method for pH of Aqueous Solutions with the Glass Electrode:

- a. The pH meter is standardized using the methods outlined in ASTM E-70-90.
- b. A clean test cup should be filled with a portion of the manufacturer's suggested working concentration of the cleaning compound and a preliminary value for pH should then be obtained Figure 1.
- c. The pH of water samples is measured, as is that of slightly buffered solutions that are in equilibrium with the air, as described in ASTM E-70-90, paragraphs b(1) and (2), except the pH is measured of successive portions of water, with vigorous agitation, until the observed results for two successive portions agree within 0.1 unit.



Figure 1. pH Measurement.

2. CLEANER EVALUATION

2.1 CHARACTERISTICS

2.1.3 CONSTITUENTS

Objective

The objective of this test is to determine the constituents of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not contain the following: a) Constituents that are cited in the Clean Air Act, 1990 Amendments for reduction or elimination (Ozone Depleting Chemicals (ODCs), Hazardous Air Pollutants (HAPs) and Volatile Organic Chemicals (VOCs)), b) Constituents that cause the product to be disposed of as a characteristic or listed waste under the Resource Conservation and Recovery Act (RCRA), c) Constituents that are listed as known or suspected carcinogens by the National Toxicology Program, d) Constituents that are listed as Total Toxic Organic (TTO) in 40 CFR Part 122, Appendix D (app C, ref 27) (Test Protocol).

Test Procedures

- a. The MSDS for the manufacturer's suggested working concentration of the cleaning compound should be reviewed for basic information about the cleaner.
- b. The manufacturer's suggested working concentration of the cleaning compound is then submitted for Fourier Transform Infrared Spectroscopy (FTIR) analysis. The analysis should include the comparison against an extensive library of materials that met the criteria requirements (ODCs, HAPs, VOCs, RCRA, etc.).
- c. The FTIR data is then compared to the MSDS and the Toxicity Certification for discrepancies.

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2. CLEANER EVALUATION

2.1 CHARACTERISTICS

2.1.4 APPEARANCE

Objective

The objective of this test is to determine the appearance of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The appearance of the manufacturer's suggested working concentration of the cleaning compound shall be free of separation or colloidal dispersion (Test Protocol).

Test Procedures

The appearance of the manufacturer's suggested working concentration of the cleaning compound is to be determined using a modified version of MIL-C-29602, paragraph 4.5.2.1 (app C, ref 9).

- a. The package in which the manufacturer's suggested working concentration of the cleaning compound is delivered should remain undisturbed for a minimum of 48 hours at ambient conditions.
- b. With minimal disturbance, the package is then opened.
- c. The manufacturer's suggested working concentration of the cleaning compound is observed for separations or colloidal dispersions.
- d. A uniform 1-liter sample is then taken (using calowasa) and placed into a clean glass jar with a screw-type lid.
- e. The closed sample is allowed to sit undisturbed for 48 hours at ambient conditions.
- f. With minimal disturbance, the sample is then observed for separations or colloidal dispersions. Appearance at this time should be photographically documented.

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2. CLEANER EVALUATION

2.1 CHARACTERISTICS

2.1.5 DRYING POINT

Objective

The objective of this test is to determine the drying point of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not exceed 206.7 °C (404 °F) drying point (Test Protocol).

Test Procedures

The drying point of the manufacturer's suggested working concentration of the cleaning compound is determined using ASTM D-86-96 (app C, ref 12), Standard Test Method for Distillation of Petroleum Products.

- a. The test setup should be assembled in accordance with the instructions for Group IV.
- b. Heat is applied to the distillation flask and contents. The heating at this stage is regulated to reach the initial boiling point in 5 to 15 minutes from initial heating.
- c. The heating rate is adjusted after reaching the initial boiling point to a uniform average rate of condensation from 5 percent recovered to 5 mL residue in the flask of 4 to 5 mL/minute.
- d. The final boiling point (dry point) is recorded. The dry point is the thermometer reading that is observed at the instant the last drop of liquid evaporates from the lowest point in the flask. Any drops or film of liquid on the side of the flask or on the thermometer are disregarded.

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2. CLEANER EVALUATION

2.2 SAFETY/ENVIRONMENTAL

2.2.1 TOXICITY

Objective

The objective of this test is to determine the toxicity of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The toxicity of the manufacturer's suggested working concentration of the cleaning compound shall conform to AR 40-5 (app C, ref 6) and shall have no adverse effects on the health of personnel or the environment when used properly and with the appropriate personal protection equipment (PPE) (Test Protocol).

Test Procedures

Toxicity certification for any product to be used by U.S. Army military personnel is investigated and granted or denied by the CHPPM, using the process in AR 40-5. Additional information should be forwarded to CHPPM for investigation and inclusion in the toxicity certification of the cleaning product. The following is a list of microbes that could potentially propagate in an enzyme-based cleaning solution:

- a. *Bordetella pertussis* (whooping cough).
- b. *Francisella tularensis* (tularemia).
- c. *Pseudomonas aeruginosa* (wound infection).
- d. *E. coli*.
- e. *Salmonella typhi* (typhoid fever).
- f. *Vibrio cholerae* (cholera, Asiatic).
- g. *Yersinia pestis* (plague).
- h. *N. meningitidis* (one type of bacteria meningitis).
- i. *Staphylococcus aureus*.

- j. *Streptococcus pyogenes* (strep throat, scarlet fever, rheumatic fever).
- k. *Bacillus anthracis* (anthrax).
- l. *Clostridium botulinum* (botulism).
- m. *C. tetani* (tetanus).
- n. *Listeria monocytogenes* (encephalitis).
- o. *Corynebacterium diphtheriae* (diphtheria).
- p. *Mycobacterium tuberculosis*.
- q. *M. leprae* (leprosy).
- r. Hepatitis (all types).
- s. Poliomyelitis.
- t. Rabies (both types).
- u. Yellow fever.
- v. HIV.

2. CLEANER EVALUATION

2.2 SAFETY/ENVIRONMENTAL

2.2.2 BIODEGRADABILITY

Objective

The objective of this test is to determine the biodegradability characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The surfactants of the manufacturer's suggested working concentration of the cleaning compound shall be readily biodegradable in accordance with 40 CFR Part 796, Subpart D 796.3100 (app C, ref 7). The minimum requirement is 90 percent at the end of the 28 day period (Test Protocol).

Test Procedure

Biodegradability. Biodegradability of the manufacturer's suggested working concentration of the cleaning compound is determined using the methods outlined in 40 CFR Part 796, Subpart D 796.3100, Aerobic Aquatic Biodegradation and using the 28-day test period.

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2. CLEANER EVALUATION

2.2 SAFETY/ENVIRONMENTAL

2.2.3 VOLATILE ORGANIC CHEMICALS (VOCs)

Objective

The objective of this test is to determine the VOC content of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall contain less than 0.4164 lb/gal. (50 gm/L) of Volatile Organic Chemicals (VOCs) (Test Protocol).

Test Procedures

VOC Content. The VOC content of the cleaner shall be determined using Environmental Protection Agency (EPA) Method 8260A, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique (app C, ref 10). Since the method is lengthy, only a summary is presented in the following paragraphs. This method and all precautions will be followed as outlined in EPA Method 8260A.

a. The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection (in limited applications). Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb trapped sample components. The analytes are desorbed directly to a large-bore capillary or cryofocused on a capillary precolumn before being flash-evaporated to a narrow-bore capillary for analysis. The column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer interfaced to the gas chromatograph. Wide-bore capillary columns require a jet separator, whereas narrow-bore capillary columns can be directly interfaced to the ion source.

b. If the sample introduction techniques in paragraph (a) are not applicable, a portion of the sample is dispersed in solvent to dissolve the volatile organic constituents. A portion of the solution is combined with organic-free reagent water in the purge chamber. It is then analyzed by purge-and-trap GC/MS, following the normal water method.

c. Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra or

authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard with a five-point calibration curve.

d. The method includes specific calibration and quality control steps that replace the general requirements in EPA Method 8000.

2. CLEANER EVALUATION

2.3 STORAGE

2.3.1 COLD STABILITY

Objective

The objective of this test is to determine the cold stability of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The cold stability of the manufacturer's suggested working concentration of the cleaning compound shall be such that the cleaning compound returns to its original homogeneous condition after exposure to cold environments (Test Protocol).

Test Procedures

The cold stability of the manufacturer's suggested working concentration of the cleaning compound is determined using MIL-C-87937B, paragraph 4.6.6.

- a. A 50-mL sample of the manufacturer's suggested working concentration of the cleaning compound is placed in a suitable, clean test tube.
- b. The test tube is then cooled to 0 °C (32 °F).
- c. The test tube is held at this temperature for 1 hour.
- d. The test tube is then allowed to return to the ambient (room) temperature.
- e. The steps in paragraphs c through e are repeated four more times (a total of five cycles).
- f. The manufacturer's suggested working concentration of the cleaning compound should then be observed for homogeneity or any changes noted from the original solutions. Appearance at this time should be photographically documented.

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2. CLEANER EVALUATION

2.3 STORAGE

2.3.2 HEAT STABILITY

Objective

The objective of this test is to determine the heat stability of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The heat stability of the manufacturer's suggested working concentration of the cleaning compound shall be such that the cleaner shows no marked color changes or precipitation, and shall not corrode or stain 1020 steel strip (a slight darkening shall not be objectionable). Layering or separation will constitute failure if it does not return to its original homogenous state upon cooling (Test Protocol).

Test Procedures

The heat stability of the manufacturer's suggested working concentration of the cleaning compound is to be determined using MIL-C-87937B, paragraph 4.6.5 (app C, ref 5).

- a. 141.75 grams (4.97 oz) of the manufacturer's suggested working concentration of the cleaning compound should be placed into each of two clean 255-ml (12-oz) clear glass bottles approximately 24 cm (9.5 in.) high with a 6.35-cm (2.5-in.) diameter.
- b. One bottle containing the manufacturer's suggested working concentration of the cleaning compound is sealed with a screw-type cap and stored in a dark place at standard conditions for six days (144 hrs) for reference purposes.
- c. A strip of steel (15.24 by 1.27 by 0.05 cm (6.0 by 0.5 by 0.02 in.)), conforming to MIL-S-7952, is then cleaned by abrasively polishing it to remove the surface scale and corrosion. The strip should then be immersed in P-D-680 for 1 minute followed by immersion in isopropyl alcohol (TT-I-735, grade A) for 1 minute.
- d. The test strip should be placed into the second bottle of the manufacturer's suggested working concentration of the cleaning compound at standard conditions, and the bottle should be sealed.
- e. The bottle is then shaken thoroughly for 1 minute and placed into a bath maintained at 46 ± 2 °C (115 ± 3 °F) for 5 hours. Upon removal, the container is allowed to cool to ambient conditions for 19 hours.

- f. The steps in paragraphs a through e are repeated five times.

- g. The steel strip is removed and the portion of the strip which was immersed in the manufacturer's suggested working concentration of the cleaning compound and the portion that was exposed to the vapor (the cleaning compound/air mixture above the liquid level) are inspected.

- h. The bottle is then resealed without the steel strip.

- i. The control bottle and the test bottle are thoroughly shaken for 1 minute and then allowed to remain undisturbed for 1 hour at room temperature.

- j. The bottles are then examined for any marked change in color, precipitation, layering, or separation. All test coupons should be photographically documented immediately upon termination of the test.

2. CLEANER EVALUATION

2.4 SURFACE CLEANING

2.4.1 NONVOLATILE RESIDUE

Objective

The objective of this test is to determine the nonvolatile residue (NVR) characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The nonvolatile residue of the manufacturer's suggested working concentration of the cleaning compound shall be less than 10 mg of residue per 100 mL of solution (Test Protocol).

Test Procedure

Five grams \pm 0.001 grams of the manufacturer's suggested working concentration of the cleaning compound is placed in a porcelain dish about 6 to 8 cm in diameter and about 2 to 4 cm in depth. The sample is then placed in a circulating air oven at a temperature of 105 ± 2 °C for 1 hour. The dish is then cooled and weighed, and the weight is compared to its previous weight. These steps are repeated until the weight loss or gain is not more than 0.1 percent.

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2. CLEANER EVALUATION

2.4 SURFACE CLEANING

2.4.2 NONVOLATILE RESIDUES PER P-D-680 SPECIFICATION

Objective

The objective of this test is to determine the nonvolatile residue of the manufacturer's suggested working concentration of the cleaning compound based on the P-D-680 specification.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not exceed 0.07 percent Non-volatile residue when tested per modified ASTM E-1131-93 (app C, ref 11), Standard Test Method for Compositional Analysis by Thermogravimetry (Test Protocol).

Test Procedures

The nonvolatile residue of the of the manufacturer's suggested working concentration of the cleaning compound is determined by using a modified version of ASTM E-1131-93, developed by the U.S. Army Tank-automotive and Armaments Command (TACOM).

- a. 20 mg of the manufacturer's suggested working concentration of the cleaning compound should be measured, making sure to get a sample as uniform as possible.
- b. The test apparatus (thermogravimetric analyzer, furnace, etc.) should be set up with the reagents as specified in ASTM E-1131-93. The inert gas flow rate should be 50 mL/minute (this was also the reactive environment flow rate), and the initial temperature should be set at 50 °C.
- c. The apparatus is opened and the specimen holder is exposed.
- d. The specimen temperature sensor is then positioned to the same location used in calibration steps outlined in ASTM E-1131-93.
- e. The specimen holder is then enclosed.
- f. The initial mass is recorded. The mass loss recorder is set up with the sensitivity of 25 µg mass change per 1 cm of chart deflection. The mass loss is measured with respect to time.
- g. The heating program is then initiated within the desired temperature range. For this test, the temperature range is 50 to 600 °C at a heating rate of 10 °C per minute.

h. Once a mass loss profile has been established at 600 °C, the unit is switched from the inert environment to the reactive environment.

i. The analysis is completed when the mass loss plateau is established after switching to the reactive environment.

j. The final mass is recorded after the unit is switched to the inert purge gas.

2. CLEANER EVALUATION

2.4 SURFACE CLEANING

2.4.3 CLEANING EFFICIENCY

Objective

The objective of this test is to determine the cleaning efficiency of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The cleaning efficiency of the manufacturer's suggested working concentration of the cleaning compound shall leave no visible residue or stains (Test Protocol).

Test Procedures

Cleaning efficiency of the manufacturer's suggested working concentration of the cleaning compound is determined using a modified version of ASTM F-22-65 (app C, ref 8), Standard Test Method for Hydrophobic Surface Films by the Water-Break Test.

- a. A 10- by 10- by 1.0-cm (4- by 4- by 0.25-in.) test coupon of aluminum 7075-T6 should be abraded and cleaned with a Scotch-Brite pad.
- b. The test coupon is placed into a container of deionized or distilled water.
- c. The test coupon is then removed vertically from the water.
- d. The time it takes for the draining water layer to become a discontinuous film is determined.
- e. The steps in paragraphs a through d are repeated until the time exceeds 1 minute.
- f. The test coupon is then dried.
- g. A standard contaminant mixture is made by combining two parts (by weight) of hydraulic fluid (MIL-H-83282) and one part (by weight) of lubricating grease (MIL-G-81322).
- h. The standardized mixture is applied to the test coupon and baked for 2 hours in an air-circulating oven at 54 °C (130 °F). The test coupon is allowed to cool to ambient (room) temperature.

i. The test coupon is then cleaned with the manufacturer's suggested working concentration of the cleaning compound and allowed to air dry.

j. The test panel is visually examined for residue, stains, and discoloration. All test coupons should be photographically documented immediately upon termination of the test

2. CLEANER EVALUATION

2.4 SURFACE CLEANING

2.4.4 WATER BREAK FREE

Objective

The objective of this test is to determine the water break free of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The water break free for any surface cleaned with the manufacturer's suggested working concentration of the cleaning compound shall be greater than one (1) minute (Test Protocol).

Test Procedures

The water break of the manufacturer's suggested working concentration of the cleaning compound is to be determined using the method outlined in ASTM F-22-65.

- a. A 10- by 10- by 1.0-cm (4- by 4- by 0.25-in.) test coupon of aluminum 7075-T6 should be abraded and cleaned with a Scotch-Brite pad.
- b. The test coupon is placed into a container of distilled water.
- c. The test coupon is then removed vertically from the water.
- d. The time it takes for the draining water layer to become a discontinuous film is determined.
- e. The steps in paragraphs a through d are repeated until the time exceeds 1 minute.
- f. The test coupon is then dried.
- g. A standard contaminant mixture should be made by combining two parts (by weight) of hydraulic fluid (MIL-H-83282) and one part (by weight) of lubricating grease (MIL-G-81322).
- h. The standardized mixture is applied to the test coupon and baked for 2 hours in an air-circulating oven at 54 °C (130 °F). The test coupon is allowed to cool to ambient (room) temperature.
- i. The test coupon is then cleaned with the manufacturer's suggested working concentration of the cleaning compound and allow to air dry.

j. The test coupon is then tested again for water break using the steps in paragraphs b through d.

2. CLEANER EVALUATION

2.4 SURFACE CLEANING

2.4.5 FLUORESCENT PENETRATION INSPECTION

Objective

The objective of this test is to determine the fluoride penetration inspection characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not impede the detection of known cracks using Fluorescent Penetration Inspection (Test Protocol).

Test Procedures

Fluorescent Penetration Inspection. The fluorescent penetration inspection characteristics of the manufacturer's suggested working concentration of the cleaning compound are determined by using the Level 4 inspection method presented in the Test Plan.

a. A standardized contaminant mixture is made by mixing two parts (by weight) of hydraulic fluid (MIL-H-83282) and one part (by weight) of lubricating grease (MIL-G-81322) in a clean container.

b. The test coupon specimens (table 1) are precleaned by first hand-washing with soapy water and clean water rinsing. The coupons are then immersed in a methanol dip, followed by immersion in a 1:1:1 ultrasonic bath and allowed to dry. Inspect coupons under black light to ensure penetrant removal.

TABLE 1. TEST COUPON SPECIMENS

Penetrant: FP-4, Type I, Level 4, Method D, Magnaflux ZL-37.

Developer: D-1, Type I, Form A, Dry Powder, Magnaflux ZP-4B.

Emulsifier: 20-percent FE-2, Type I, Method D, Magnaflux ZR-10B.

| Test Coupon Specimen | Measurements | | Crack Measurement | |
|-------------------------|------------------------|----------------|----------------------|-------|
| | cm | in. | cm | in. |
| Inconel 718- Bar | 2.54 by 15.24 by 0.635 | 1 by 6 by 0.25 | 0.0533 | 0.021 |
| Inconel 718-Bar | 2.54 by 15.24 by 0.635 | 1 by 6 by 0.25 | .133 | .052 |
| Ti-641-4V-Bar | 2.54 by 15.24 by 0.635 | 1 by 6 by 0.25 | .137 | .054 |
| Ti-641-4V-Bar | 2.54 by 15.24 by 0.635 | 1 by 6 by 0.25 | .945 | .372 |

- c. Two drops of the contaminant mixture are applied to the test specimens, utilizing a clean glass rod.
- d. The contaminant is spread evenly over the test specimens, using a wooden applicator.
- e. The test specimens are placed in a dry air-circulating oven for 2 hours at 130 °F.
- f. The test specimens are allowed to cool to ambient (room) temperature.
- g. The excess contaminant is wiped from the test specimens with a clean cloth (no cleaners).
- h. The test specimens are cleaned with the manufacturer's suggested working concentration of the cleaning compound using any of the following methods: manual wiping, ultrasonic cleaning, vapor degreasing, spraying, or immersion cleaning.
- i. The cleaned test specimens are then placed in an air-circulating oven at 130 °F for 5 minutes.
- j. The test specimens are removed from the oven and allow to cool to ambient (room) temperature for 3 minutes.
- k. The test specimens are then dipped in liquid penetrant (table 1) and allowed to dwell for 5 minutes.
- l. With the black light on, the test specimens should be rinsed using clean water by manually spraying at a maximum of 40 psi and temperature between 50-100 °F). A coarse spray nozzle should be set at an angle of 45° and a distance of 30.5 cm (12 in.).
- m. The emulsifier (table 1) should be applied to the test specimens by dipping them for 5 seconds and then standing them upright for the remainder of the dwell time. The dwell time is optimally 1 minute 55 seconds, but shall not exceed 2 minutes.
- n. With the black light turned on, the test specimens are rinsed using clean water by manually spraying at a maximum of 40 psi with the temperature between 50 and 100 °F. The coarse spray nozzle is set at an angle of 45° and a distance of approximately 12 inches.
- o. The cleaned test specimens are then placed in an air-circulating oven at 130 °F for 5 minutes. The test specimens are removed from the oven and allowed to cool to ambient (room) temperature for 3 minutes.
- p. The developer (table 1) is applied to the test specimens and allowed to glide off and dwell for 5 minutes.

q. The spotmeter is zeroed, calibrated, and focused before taking data. All of the data measurements on the test specimens should be in the same sequence and the time between measurements should be minimized to provide similar dwell times.

r. The value of the crack indication and the background value on the surface near the crack are then measured and recorded. Repeat three times.

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2. CLEANER EVALUATION

2.4 SURFACE CLEANING

2.4.6 RELATIVE SOLVENCY

Objective

The objective of this test is to determine the relative solvency characteristics of the manufacturer's suggested working concentration of the cleaning compound.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not have a relative solvency of less than 94.4 percent (Test Protocol).

Test Procedures

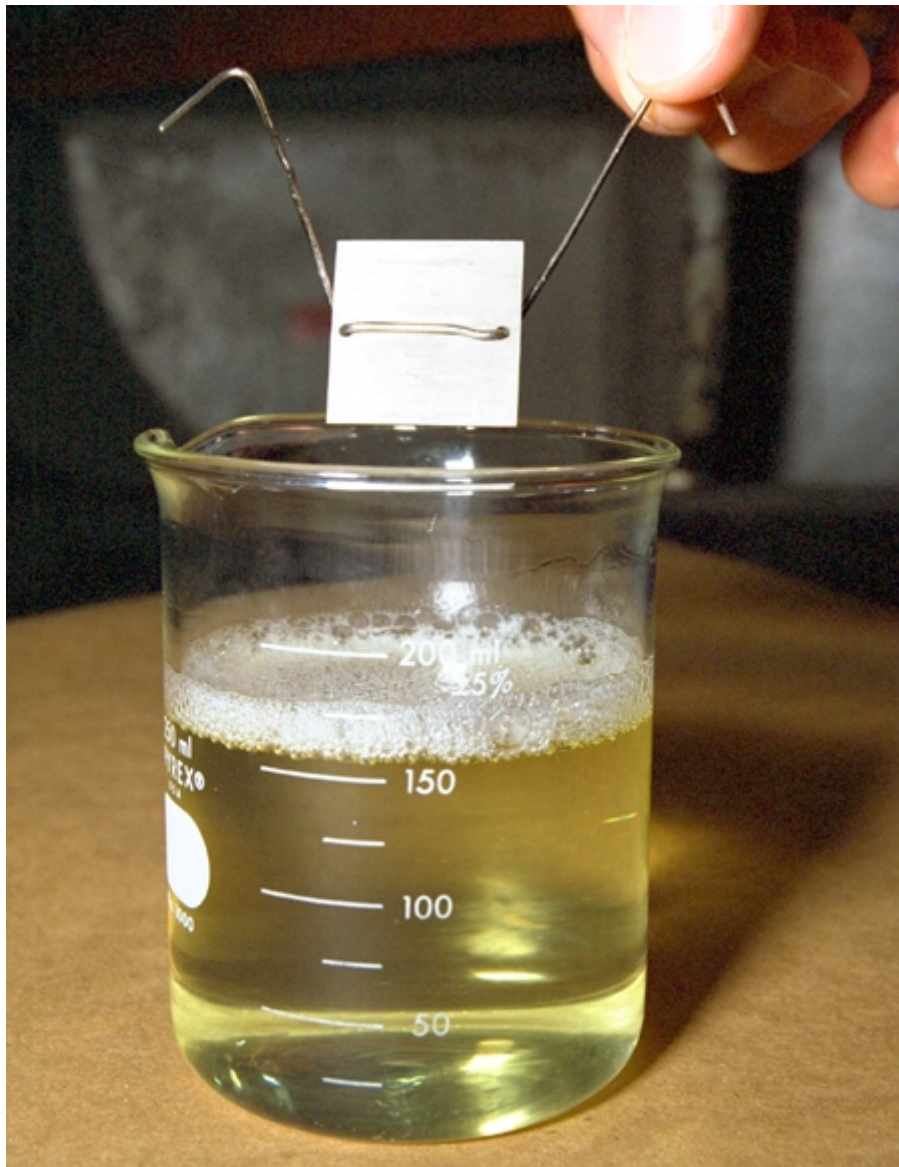
The solvency of the manufacturer's suggested working concentration of the cleaning compound is to be determined using a test method developed by TACOM.

- a. Three test specimens 1 by 1 by .04 inches, are made from steel, carbon, mild (ASTM-A-366, class 1, commercial bright finish).
- b. The metal specimens are washed in toluene (CP, 99 percent) until free of any soils and greases.
- c. The test specimens are dried with dry, clean air.
- d. The test specimens are then weighed to the nearest 0.1 gram.
- e. Approximately 0.4 gram of MIL-G-10924F grease, automotive, artillery is applied to the test specimen, covering both sides uniformly.
- f. The test specimen is then placed into a beaker using a holder to prevent contact with the sides or bottom of the beaker (figure 1).
- g. The manufacturer's suggested working concentration of the cleaning compound is added to the beaker until the test coupon is completely immersed.
- h. The beaker is placed into an ultrasonic cleaner that is at a maintained water temperature of 50 °C (122 °F).

i. The test specimen is observed until all of the grease is removed and the time is recorded.

- j. If grease still remains on the test specimen after 100 minutes, testing is terminated and the cleaning time is recorded as 100 minutes.
- k. The test specimen is dried using dry, clean air.
- l. The test specimen is weighed to the nearest 0.1 gram.
- m. The steps in paragraphs a through l are repeated with the other two remaining specimens.

Figure 1. Relative Solvency Test Specimen



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3. RELIABILITY

3.1 RELIABILITY

3.1.1 RELIABILITY

Objective

The objective of this test is to determine the effect the manufacturer's suggested working concentration of the cleaning compound had on the reliability of actual materiel.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause a degradation of reliability or performance of any system or component (Test Protocol).

Test Procedures

a. This test has a limited scope both in materiel tested and in statistical significance. Only one type of materiel is tested, the M16A2 rifle. The test is conducted using M16A2 rifles currently in the ATC weapons inventory. The guns are fitted with new bolts and barrels prior to initiating the test.

b. Testing is conducted in accordance with the procedures listed in TOP 3-2-045 (ref 32) and MIL-R-63997B (ref 33). The testing is conducted under the prevailing environmental conditions existing at ATC during the time of the test.

c. Three M16A2 rifles (serial No. 6002933, 7067801, and 7075589) each fire 6000 rounds of 5.56-mm, M855 Ball ammunition, lot No. LC-91J086-130. Firing is conducted in 120-round cycles following the scenario listed in Table 1. The weapons are forced air-cooled to range ambient temperature between cycles. Each M16A2 rifle is wiped and lubricated with CLP every 600 rounds (five cycles). Each rifle is thoroughly cleaned, inspected, and lubricated every 1200 rounds (ten cycles). Fifty cycles are fired for a total of 6000 rounds per weapon.

TABLE 1. FIRING SCHEDULE

| Magazine No. | No. of Rounds | Type of Fire | Firing Rate, spm |
|--------------|---------------|--------------|------------------|
| 1 | 30 | Burst | 22 to 36 |
| 2 | 30 | Quick burst | 36 to 90 |
| 3 | 30 | Single shot | 10 to 30 |
| 4 | 30 | Single shot | 10 to 30 |

spm = Shots per minute.

d. The cyclic rate-of-fire of each weapon is recorded during the second magazine of cycles No. 1, 11, 21, 31, and 41.

e. Magnetic-particle inspections are conducted on the following components at 3000 rounds:

- (1) Barrel with upper receiver.
- (2) Bolt.
- (3) Bolt carrier.
- (4) Bolt cam pin.

4. STORAGE

4.1 STORAGE

4.1.1 STORAGE

Objective

The objective of this test is to determine the effect of the manufacturer's suggested working concentration of the cleaning compound on stored materiel.

Criterion

The manufacturer's suggested working concentration of the cleaning compound shall not cause any problems related to long term storage of any system or component (Test Protocol).

Test Procedures

a. This test has a limited scope both in materiel tested and in statistical significance. Only one type of materiel is tested, the M16 rifle. The test is conducted using three M16A1 rifles currently in the weapons inventory at ATC (serial No. 5266546, 5267854, and 5212736). All three rifles are inspected prior to the Storage test to ensure that all are in serviceable condition.

b. The test consisted of preparing the three rifles for storage following the current storage procedures for the M16 rifles. The three M16A1 rifles are disassembled and thoroughly cleaned with the manufacturer's suggested working concentration of the cleaning compound. The weapon components are wiped and blown dry; lubricated with cleaner, lubricant, preservative (CLP) (MIL-L-63460A); and reassembled. The test support weapons are stored in an arms storage facility for a minimum of 60 days. The storage facility is climate controlled to a temperature of 75 °F and a moderate humidity level.

c. After the 60-day storage period is observed, the weapons are removed from the storage facility and inspected. Each weapon is disassembled and each component part is examined for damage. Any resulting damages should be noted. Each component part should be photographically documented immediately after completion of the visual inspection.

d. Following the inspection, the M16A1 rifles are reassembled and function fired. Two 30-round magazines of 5.56-mm, M855 Ball ammunition are to be fired from each rifle. The first 30-round magazine is fired in short three- to five-round bursts. The second 30-round magazine is fired in single-shot mode. Any weapon stoppages should be recorded.



Figure 1.1.3-1. M16A2 test item used for storage test.

APPENDIX A. CRITERION

TEST 1.1.1 The manufacturer’s suggested working concentration of the cleaning compound shall not show any indication of staining, etching, pitting, or localized attack on the test panels, or cause weight change to an average of three (3) test panels greater than that shown in Table 1 (Test Protocol).

TABLE 1. ALLOY AND MAXIMUM AVERAGE WEIGHT LOSS

| Alloy | Weight Loss, mg/cm ² /168 hr |
|---------------------------------------|---|
| AM-355 CRT | 0.49 |
| PH 13-8 Mo | .49 |
| C-250 | .49 |
| Magnesium 4377 | .70 |
| Al 7075-T6 | .49 |
| Titanium 6AL-4V | .35 |
| Steel 4340 | .49 |
| Cadmium-Plated Steel | ^a .14 |
| Steel 4340 Zinc-Phosphate Coated | ^b .00 |
| Steel 4340 Manganese-Phosphate Coated | ^b .00 |
| Copper Alloy (Brass) UNS C36000 | ^b .00 |
| Copper Alloy UNS C10100 HO2 HH | ^b .00 |

^amg/cm²/24 hr.

^b no criteria specified

TEST 1.1.2 The manufacturer’s suggested working concentration of the cleaning compound shall not cause a corrosion rating greater than two (2) on any test panel (Test Protocol).

TEST 1.1.3 The manufacturers suggested working concentration of the cleaning compound shall not cause hydrogen embrittlement of cadmium plated ANSI 4340 steel (Test Protocol).

TEST 1.1.4 The manufacturer’s suggested working concentration of the cleaning compound shall not cause streaking, stains or other deposits on unpainted surfaces that cannot be easily removed with water (Test Protocol).

TEST 1.1.5 The manufacturer's suggested working concentration of the cleaning compound shall not cause stress corrosion (Test Protocol.)

TEST 1.1.6 The manufacturer's suggested working concentration of the cleaning compound shall not cause a copper corrosion rating of higher than 1a (Test Protocol).

TEST 1.1.7 The manufacturer's suggested working concentration of the cleaning compound shall not cause any steel corrosion when tested by ASTM D-130 modified (Test Protocol).

TEST 1.1.8 The manufacturer's suggested working concentration of the cleaning compound shall not cause corrosion on a bimetallic couple (Test Protocol).

TEST 1.2.1 The manufacturer's suggested working concentration of the cleaning compound shall not cause the primer coating to peel away from the substrate from any test panels after immersion in distilled water for 24 hours (Test Protocol).

TEST 1.2.2 The manufacturer's suggested working concentration of the cleaning compound shall not cause streaking, discoloration, blistering or a permanent decrease in film hardness of more than one (1) pencil hardness level on any painted surfaces. This criterion does not apply to MIL-L-46159 (Test Protocol).

TEST 1.2.3 The manufacturer's suggested working concentration of the cleaning compound shall not cause lower percentage of bondline cohesive failure compared to the baseline surface cleaner or solvent (Test Protocol).

TEST 1.3.1 The manufacturer's suggested working concentration of the cleaning compound shall not cause cracking, discoloration, dissolution, dielectric breakdown of the polyimide insulated wire in excess of that produced by distilled water (Test Protocol).

TEST 1.3.2 The manufacturer's suggested working concentration of the cleaning compound shall not cause stress crazing or staining of acrylic plastics (Test Protocol).

TEST 1.3.3 The manufacturer's suggested working concentration of the cleaning compound shall not cause stress crazing or staining of polycarbonate plastics (Test Protocol).

TEST 1.3.4 The manufacturer's suggested working concentration of the cleaning compound shall cause the sealing compound to have a minimum peel strength of 20 pounds force per inch (lbf/in.) after a seven (7) day exposure in jet reference fluid. There shall also be 100 percent cohesive failure (Test Protocol).

TEST 1.3.5 The manufacturer's suggested working concentration of the cleaning compound shall not change the durometer hardness of polysulfide sealants more than 5 units (Test Protocol).

TEST 1.3.6 The manufacturer's suggested working concentration of the cleaning compound shall not change the durometer hardness of rubber materials more than 5 units (Test Protocol).

TEST 2.1.1 The flash point of the manufacturer's suggested working concentration of the cleaning compound shall be greater than 212 °F (100 °C) (Test Protocol) (app C, ref 1).

TEST 2.1.2 The pH value of the manufacturer's suggested working concentration of the cleaning compound shall be no less than 5 and no more than 10 when in its most concentrated form (Test Protocol).

TEST 2.1.3 The manufacturer's suggested working concentration of the cleaning compound shall not contain the following: a) Constituents that are cited in the Clean Air Act, 1990 Amendments for reduction or elimination (Ozone Depleting Chemicals (ODCs), Hazardous Air Pollutants (HAPs) and Volatile Organic Chemicals (VOCs)), b) Constituents that cause the product to be disposed of as a characteristic or listed waste under the Resource Conservation and Recovery Act (RCRA), c) Constituents that are listed as known or suspected carcinogens by the National Toxicology Program, d) Constituents that are listed as Total Toxic Organic (TTO) in 40 CFR Part 122, Appendix D (app C, ref 27) (Test Protocol).

TEST 2.1.4 The appearance of the manufacturer's suggested working concentration of the cleaning compound shall be free of separation or colloidal dispersion (Test Protocol).

TEST 2.1.5 The manufacturer's suggested working concentration of the cleaning compound shall not exceed 206.7 °C (404 °F) drying point (Test Protocol).

TEST 2.2.1 The toxicity of the manufacturer's suggested working concentration of the cleaning compound shall conform to AR 40-5 (app C, ref 6) and shall have no adverse effects on the health of personnel or the environment when used properly and with the appropriate personal protection equipment (PPE) (Test Protocol).

TEST 2.2.2 The surfactants of the manufacturer's suggested working concentration of the cleaning compound shall be readily biodegradable in accordance with 40 CFR Part 796, Subpart D 796.3100 (app C, ref 7). The minimum requirement is 90 percent at the end of the 28 day period (Test Protocol).

TEST 2.2.3 The manufacturer's suggested working concentration of the cleaning compound shall contain less than 0.4164 lb/gal. (50 gm/L) of Volatile Organic Chemicals (VOCs) (Test Protocol).

TEST 2.3.1 The cold stability of the manufacturer's suggested working concentration of the cleaning compound shall be such that the cleaning compound returns

to its original homogeneous condition after exposure to cold environments (Test Protocol).

TEST 2.3.2 The heat stability of the manufacturer's suggested working concentration of the cleaning compound shall be such that the cleaner shows no marked color changes or precipitation, and shall not corrode or stain 1020 steel strip (a slight darkening shall not be objectionable). Layering or separation will constitute failure if it does not return to its original homogenous state upon cooling (Test Protocol).

TEST 2.4.1 The nonvolatile residue of the manufacturer's suggested working concentration of the cleaning compound shall be less than 10 mg of residue per 100 mL of solution (Test Protocol).

TEST 2.4.2 The manufacturer's suggested working concentration of the cleaning compound shall not exceed 0.07 percent Non-volatile residue when tested per modified ASTM E-1131-93 (app C, ref 11), Standard Test Method for Compositional Analysis by Thermogravimetry (Test Protocol).

TEST 2.4.3 The cleaning efficiency of the manufacturer's suggested working concentration of the cleaning compound shall leave no visible residue or stains (Test Protocol).

TEST 2.4.4 The water break free for any surface cleaned with the manufacturer's suggested working concentration of the cleaning compound shall be greater than one (1) minute (Test Protocol).

TEST 2.4.5 The manufacturer's suggested working concentration of the cleaning compound shall not impede the detection of known cracks using Fluorescent Penetration Inspection (Test Protocol).

TEST 2.4.6 The manufacturer's suggested working concentration of the cleaning compound shall not have a relative solvency of less than 94.4 percent (Test Protocol).

TEST 3.1.1 The manufacturer's suggested working concentration of the cleaning compound shall not cause a degradation of reliability or performance of any system or component (Test Protocol).

TEST 4.1.1 The manufacturer's suggested working concentration of the cleaning compound shall not cause any problems related to long term storage of any system or component (Test Protocol).

APPENDIX B. MATERIALS

Acrylic Plastic: listed under “Plastic, acrylic”

Aluminum (2024-T3) with primers (MIL-P-23377) and (MIL-P-85582): Test 1.3.4

Aluminum (2024-T3 bare) anodized in conforming with (MIL-8625, Type 1), with primers (MIL-P-23377) and (MIL-P-85582), and topcoat (MIL-C-85285): Test 1.2.1

Aluminum (2024-T3 clad) conversion coated in conforming with (MIL-C-5541, Class 3), with primers (MIL-P-85582) and (MIL-P-23377), and topcoat (MIL-C-85285): Test 1.2.1

Aluminum Alclad (7075-T6): Tests 1.1.1, 1.1.2, 1.1.4, 1.1.5, 2.4.3, 2.4.4

Aluminum (7075-T6) bare: Test 1.2.3

Aluminum Alclad (7075-T6) with conversion coating material conforming to (MIL-C-81706, class 1a), Epoxy primer (MIL-P-23377), Epoxy Topcoat (MIL-C-22750), Polyurethane high solids topcoat (MIL-C-85285), Aliphatic Polyurethane single component topcoat (MIL-C-46168), Lacquer acrylic low reflective (MIL-L-46159), Heat resistant paint (MIL-P-14105): Test 1.2.2

Brass (QQ-B-613, composition 2) conforming to FED-STD-791C, test method 5322.2: Test 1.1.8

Cadmium plated steel: listed under “Steel, cadmium plated”

Copper alloy (BRASS) (UNS-C36000): Test 1.1.1

Copper alloy (BRASS) (UNS-C10100 HO2) half hard: Test 1.1.1

Copper, smooth-surfaced, hard-tempered, cold-finished of 99.9% purity: Test 1.1.6

Inconel 18-Bar: Test 2.4.5

M16A1 rifle with 5.56mm M855 ball ammunition: Test 4.1.1

M16A2 rifle with 5.56mm M855 ball ammunition: Test 3.1.1

Magnesium (AMS-4377): Tests 1.1.1, 1.1.2, 1.1.4, 1.1.5

Nickel, electroplated: Test 1.2.3

Plastic, acrylic, conforming to (MIL-P-5425, finish A): Test 1.3.2

Plastic, acrylic, conforming to (MIL-P-8184, finish B): Test 1.3.2

Plastic, acrylic, conforming to (MIL-P-25690): Test 1.3.2

Plastic, polycarbonate conforming to (MIL-P-83310): Test 1.3.3

Polymide wire: listed under “Wire, polymide”

Polysulfide synthetic rubber: listed under “Rubber, polysulfide synthetic”

Rubber conforming to AMS 3204: Test 1.3.6

Rubber conforming to AMS 3209: Test 1.3.6

Sealant, polysulfide synthetic with separate curing agent added to base compound conforming to (MIL-S-81733, Type 1): Test 1.3.5

Sealant, polysulfide synthetic, dichromate cured and conforming to (MIL-S-8802, Type 1): Test 1.3.5

Steel (AISI 4340): Tests 1.1.1, 1.1.2, 1.1.4, 1.1.5

Steel (AISI 4340), cadmium plated according to (ASTM F 519-93): Tests 1.1.1, 1.1.2, 1.1.3

Steel (AISI 4340) with zinc phosphated coating (DOD-P-16232): Tests: 1.1.1, 1.1.2

Steel (AISI 4340) with manganese phosphate coating (DOD-P-16232B): Tests 1.1.1, 1.1.5

Steel, composition G52986, conforming to FED-STD-791C: Test 1.1.8

Steel, 1020 conforming to (MIL-S-7952): Test 2.3.2

Steel, high strength (AM-355 CRT): Tests 1.1.1, 1.1.2, 1.2.3, 1.1.4, 1.1.5

Steel, high strength (PH 13-8 Mo): Tests 1.1.1, 1.1.2, 1.1.4, 1.1.5

Steel, high strength, maraging (C-250): Tests 1.1.1, 1.1.2, 1.1.4, 1.1.5

Steel, SAE standard conforming to (SAE J1703-95, ref 28): Test 1.1.7

Steel Carbon, mild, class 1, commercial bright finish: Test 2.4.6

Titanium (641-4V-Bar): Test 2.4.5

Titanium (6AL-4V): Tests 1.1.1, 1.1.2, 1.2.3, 1.1.4, 1.1.5

Wire, polyimide insulated conforming to (MIL-W-81481 11/20): Test 1.3.1

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APPENDIX D. ABBREVIATIONS

| | |
|---------|---|
| AEC | = U.S. Army Environmental Center |
| ANSI | = American National Standards Institute |
| ARL | = U.S. Army Research Laboratory |
| ASTM | = American Society for Testing and Materials |
| ATC | = U.S. Army Aberdeen Test Center |
| AMCOM | = U.S. Army Aviation and Missile Command |
| CFR | = Code of Federal Regulations |
| CHPPM | = U.S. Army Center for Health Promotion and Preventive Medicine |
| CLP | = cleaner, lubricant, preservative |
| DOD | = Department of Defense |
| EPA | = Environmental Protection Agency |
| FEJ | = failure-to-eject |
| FEX | = failure-to-extract |
| FFR | = failure-to-fire |
| FORSCOM | = U.S. Army Forces Command |
| GC/MS | = gas chromatography/mass spectrometer |
| HAP | = hazardous air pollutant |
| MSDS | = Material Safety Data Sheet |
| NSN | = national stock number |
| ODC | = ozone depleting chemical |
| PPE | = personnel protection equipment |
| RCRA | = Resource Conservation and Recovery Act |
| SAE | = Society of Automotive Engineers |
| SEM | = scanning electron microscope |
| TACOM | = U.S. Army Tank-automotive and Armament Command |
| TECOM | = U.S. Army Test and Evaluation Command |
| TOP | = Test Operating Procedure |
| TTO | = total toxic organic |
| VOC | = volatile organic chemical |

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