MIL-C-4339D 19 July 1990 Superseding MIL-C-4339C(ASG) 25 February 1965

## MILITARY SPECIFICATION

# CORROSION PREVENTIVE, SOLUBLE OIL FOR WATER INJECTION SYSTEMS, (NATO CODE NUMBER C-630)

This specification has been approved for use by all Departments and Agencies of the Department of Defense.

## 1. SCOPE

l.1 Scope. This specification covers the requirements for one type of corrosion preventive soluble oil used in water-alcohol mixtures. These water-alcohol mixtures are used as thrust augmentation in certain jet aircraft engines and as anti-detonation or internal coolant fluids in certain reciprocating aircraft engines (see 6.1 and 6.3).

#### 2. APPLICABLE DOCUMENTS

- 2.1 Government documents.
- 2.1.1 Specifications, standards, and handbooks. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation (see 6.2).

### SPECIFICATIONS

## FEDERAL

O-M-232 - Methanol (Methyl Alcohol)

QQ-A-250/4 - Aluminum Alloy 2024, Plate and Sheet

QQ-C-502 - Copper Rods and Shapes; and Flat Products with
Finished Edges (Flat Wire, Strips and Bars)

QQ-S-571 - Solder; Tin Alloy, Tin-Lead Alloy, and Lead Alloy

Beneficial Comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Code (68) SA-ALC/SFRM, Kelly AFB TX 78241-5000, by using the self-addressed standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

<u>DISTRIBUTION STATEMENT A</u>. Approved for public release; distribution is unlimited.

MILITARY

MIL-S-7952 - Steel, Sheet and Strip, Uncoated, Carbon (1020 and 1025) (Aircraft Quality)

STANDARDS

FEDERAL

FED-STD-313 - Material Safety Data Sheets, Preparation and the Submission of

MILITARY

MIL-STD-290 - Packaging of Petroleum and Related Products

(Unless otherwise indicated, copies of the federal and military specifications, standards, and handbooks are available from the Standardization Documents Order Desk, Building 4D, 700 Robbins Avenue, Philadelphia PA 19111-5094.)

2.1.2 Other Government documents, drawings, and publications. The following other Government documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues are those cited in the solicitation.

DODISS - Department of Defense Index of Specifications and Standards

2.2. Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of the documents not listed in the DODISS are the issues of the documents cited in the solicitation (see 6.2).

## AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D 95 - Test Method for Water in Petroleum Products and Bituminous Materials by Distillation

ASTM D 97 - Test Methods for Pour Point of Petroleum Oil
ASTM D 130 - Methods for Detection of Copper Corrosion from
Petroleum Products by the Copper Strip Tarnish
Test

ASTM D 482 - Test Method for Ash from Petroleum Products

ASTM D 4057 - Standard Practice for Manual Sampling of Petroleum and Petroleum Products

ASTM E 29 - Recommended Practices for Indicating Which Places of Figures are to be Considered significant in Specified Limiting Values

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia PA 19103-1137.)

(Non-Government standards and other publications are normally available from the organizations that prepare or distribute the documents. These documents also may be available in or through libraries or other informational services.)

2.3 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

## 3. REQUIREMENTS

- 3.1 Material. The soluble oil shall contain not less than 85 percent, by weight, mineral oil and shall contain suitable emulsifying and corrosion-preventive agents. These agents shall have a petroleum metal sulfonate as a principal constituent. The presence of sulfonate shall be determined by an infrared absorption spectrographic analysis. The soluble oil shall contain a minimum of fatty acids and fatty acid soaps. The ratio of sulfonate absorbance to fatty acid absorbance and fatty acid soap absorbance, when determined as specified in 4.5.6.2, shall be not less than 6.0 to 1 and 3.5 to 1, respectively. If no fatty acids or fatty acid soaps are present, then the mere presence of sulfonate will satisfy this requirement.
- 3.2 Pour point. The finished oil shall have a pour point not higher than  $30^{\circ}F$  (-2°C).
- 3.3 <u>Corrosion</u>. The copper strip corrosion rating of the finished oil shall not exceed ASTM strip classification 2, moderate tarnish.
- 3.4 Water content. The water content of the finished oil shall be not more than 3.0 percent.
- 3.5 Emulsion. The following emulsions shall show not more than a trace of froth 15 minutes after preparation, when prepared as specified in 4.5.3.2 and 4.5.3.3:
- (a) l part finished oil, 9 parts synthetic hard water (see 4.5.3.1).
- (b) 1 part finished oil, 9 parts synthetic hard water, and 10 parts grade A methyl alcohol, conforming to 0-M-232.

The volume of finished oil separating from emulsion in 72 hours shall not exceed 2 percent of the total volume of the emulsion.

- 3.6 Hydrogen ion concentration. The pH value of an emulsion prepared with 1 part finished oil and 9 parts distilled water, by volume, shall be not less than 8.5 nor more than 10.0 at a temperature of 77°F (25°C).
- 3.7 Corrosion (of the emulsion). Clean, bright, mechanically polished strips of aluminum, copper, steel, and clean strips of tin-lead solder shall show no evidence of etching, pitting, or other corrosion and shall show not more than a slight discoloration when treated with an emulsion prepared with one part oil and four parts distilled water at a temperature of 77° ± 9°F (25° ± 5°C) for 168 hours, when tested as specified in 4.5.5.1, and at a temperature of 100° ± 7°F (38° ± 4°C) for 168 hours, when tested as specified in 4.5.5.2.
- 3.8 Ash content. The finished oil shall not contain more than 2 percent ash by weight (10-g sample).
- 3.9 Workmanship. The finished oil shall be clear and homogeneous. It shall not contain suspended matter, or sediment.

- 3.10 Limiting values. The following applies to all specified limits in this specification: for purposes of determining conformance with these requirements, an observed value or a calculated value shall be rounded off to the nearest unit in the last right-hand place of figures used in expressing the limitation value, in accordance with ASTM Method E 29, 'Recommended Practices for Designating Significant Places in Specified Limiting Values.'
- 3.11 Material Safety Data Sheets. A Material Safety Data Sheet shall be prepared in accordance with the requirements of FED-STD-313 (see 6.4).

## 4. QUALITY ASSURANCE PROVISIONS

- 4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements (examinations and tests) as specified herein. Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in this specification where such inspections are deemed necessary to ensure supplies and services conform to prescribed requirements.
- 4.1.1 Responsibility for compliance. All items shall meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of ensuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling inspection, as part of manufacturing operations, is an acceptable practice to ascertain conformance to requirements, however, this does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to accept defective material.
- 4.2 Quality conformance inspections. Inspections of individual lots which will serve as a basis for acceptance shall consist of all the examinations and tests specified under 4.5.

## 4.3 Lot definitions.

- 4.3.1 <u>Bulk lot of material</u>. A bulk lot of material shall be defined as an indefinite quantity of a homogeneous mixture of material contained in one isolated tank, kettle, or manufactured by a single plant run through the same processing equipment during one continuous operation not exceeding a 24-hour period.
- 4.3.2 Container lot of material. A container lot of material shall be defined as an indefinite number of 55-gallon drums (400-pound drums where applicable) or smaller unit containers of identical size and type, filled with a homogeneous mixture of material manufactured by a single plant run through the same processing equipment during one continuous operation not exceeding a 24-hour period.
- 4.4 <u>Inspection of material</u>. The contractor shall insure that each lot of finished material furnished on the contract be sampled in accordance with 4.4.1, and subjected to all tests and inspection methods specified under 4.5.

- 3.10 Limiting values. The following applies to all specified limits in this specification: for purposes of determining conformance with these requirements, an observed value or a calculated value shall be rounded off 'to the nearest unit' in the last right-hand place of figures used in expressing the limitation value, in accordance with ASTM Method E 29, 'Recommended Practices for Designating Significant Places in Specified Limiting Values.'
- 3.11 Material Safety Data Sheets. A Material Safety Data Sheet shall be prepared in accordance with the requirements of FED-STD-313 (see 6.4).

## 4. QUALITY ASSURANCE PROVISIONS

- 4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements (examinations and tests) as specified herein. Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in this specification where such inspections are deemed necessary to ensure supplies and services conform to prescribed requirements.
- 4.1.1 Responsibility for compliance. All items shall meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of ensuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling inspection, as part of manufacturing operations, is an acceptable practice to ascertain conformance to requirements, however, this does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to accept defective material.
- 4.2 Quality conformance inspections. Inspections of individual lots which will serve as a basis for acceptance shall consist of all the examinations and tests specified under 4.5.

# 4.3 Lot definitions.

- 4.3.1 <u>Bulk lot of material</u>. A bulk lot of material shall be defined as an indefinite quantity of a homogeneous mixture of material contained in one isolated tank, kettle, or manufactured by a single plant run through the same processing equipment during one continuous operation not exceeding a 24-hour period.
- 4.3.2 Container lot of material. A container lot of material shall be defined as an indefinite number of 55-gallon drums (400-pound drums where applicable) or smaller unit containers of identical size and type, filled with a homogeneous mixture of material manufactured by a single plant run through the same processing equipment during one continuous operation not exceeding a 24-hour period.
- 4.4 <u>Inspection of material</u>. The contractor shall insure that each lot of finished material furnished on the contract be sampled in accordance with 4.4.1, and subjected to all tests and inspection methods specified under 4.5.

- 4.4.1 Sampling. Sampling shall be in accordance with ASTM D 4057.
- 4.4.1.1 Sample submission. When required (see 6.2), a one gallon sample shall be forwarded to the laboratory designated by the procuring activity for subjection to the tests specified herein.
  - 4.5 Inspection methods.
- 4.5.1 Examination of product. The finished oil shall be inspected visually for clearness, homogeneity, suspended matter, sediment, or free water.
- 4.5.2 Standard tests. The following tests shall be conducted in accordance with the ASTM method specified:

Test	ASTM No.		
Water in Petroleum Products and Bituminous Materials by Distillation	D	95	
Pour Point of Petroleum Oils	D	97	
Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test	D	130	*
Ash from Petroleum Products	D	482	

- \* Use 212°F (100°C) test bath temperature on less volatile materials.
  - 4.5.3 Procedure for testing the emulsion.
- 4.5.3.1 Preparation of synthetic hard water. Synthetic hard water shall be prepared by adding the following chemicals, of chemically pure grade, to 10 liters of distilled water and bubbling carbon dioxide through the solution until the chemicals are completely dissolved:

Calcium carbonate, anhydrous, CaCO <sub>3</sub>	1.000 g
Calcium chloride, anhydrous, CaCl <sub>2</sub>	0.250 g
Calcium sulfate, anhydrous, CaSO <sub>4</sub>	2.500 g
Magnesium carbonate, anhydrous, MgCO <sub>3</sub>	1.000 g
Sodium sulfate, anhydrous, Na <sub>2</sub> SO <sub>4</sub>	1.000 g

If anhydrous salts are not available, proportionately larger weights determined by the chemical formula of the corresponding hydrates may be used.

4.5.3.2 Water emulsion. Five mL of the finished oil shall be added to 45 mL of the synthetic hard water contained in a 100-mL graduated cylinder. The temperature of the liquid shall be adjusted to 77° ± 5°F (25° ± 3°C). The mixture shall then be stirred vigorously for 5 minutes by means of an apparatus, similar to that shown in Figure 1. The graduated cylinder should be approximately 1-1/8 inch (28.6 mm) ID. The heating bath should be deep enough to cover the 85 mL mark on the graduated cylinder. The stirring mechanism consists of the following:

- (a) Paddle, copper strip, 12.1 cm by 1.9 cm by 0.16 cm.
- (b) Shaft, stirring, with means for attaching paddle long enough to immerse paddle to approximately 0.64 cm from bottom of graduated cylinder.
- (c) Motor, stirring, approximately 1500 rpm, for rotating shaft and paddle.

As soon as the stirring is completed, the paddle shall be withdrawn from the emulsion and held for approximately 3 minutes in such position that the greater part of the emulsion clinging to it will drain into the cylinder. The cylinder containing the emulsion shall be allowed to stand without any agitation, at a temperature of 77° ± 5°F (25°C ± 3°C) for 15 minutes, measured from the time of withdrawal of the paddle, and shall then be inspected for the presence of froth. The cylinder and its contents shall then be allowed to stand undisturbed for 72 hours at the same temperature, and shall then be

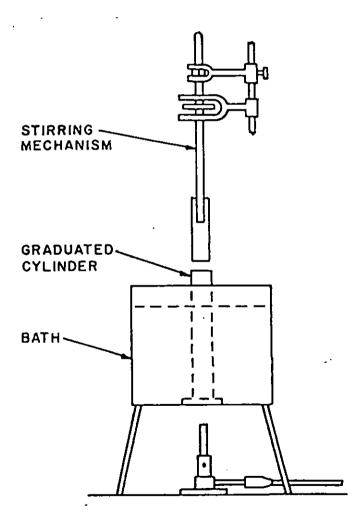


Figure 1. Stirring Mechanism

inspected for evidence of separation. Creaming (formation of a creamy portion of emulsion which tends to rise to the top without actually separating as a layer of free oil) shall not be considered separation.

- 4.5.3.3 Water-alcohol emulsion. Five mL of the finished oil shall be added to 45 mL of the synthetic hard water contained in a 100-mL graduated cylinder. The temperature of the liquid shall be adjusted to 77° ± 5°F (25° ± 3°C). The mixture shall then be stirred for 5 minutes by means of an apparatus similar to that described in section 4.5.3.2 above. As soon as stirring is completed, 50 mL of methyl alcohol shall be added conforming to grade A of 0-M-232. The emulsion shall be stirred again for 5 minutes in the same manner. As soon as the stirring is completed, the paddle shall be withdrawn from the emulsion and held for approximately 3 minutes in such position that the greater part of the emulsion clinging to it will drain into the cylinder. The cylinder containing the emulsion shall be allowed to stand without any agitation, at a temperature of 77° ± 5°F (25° ± 5°C) for 15 minutes, measured from the time of withdrawal of the paddle, and shall then be inspected for the presence of froth. The cylinder and its contents shall then be allowed to stand undisturbed for 72 hours at the same temperature, and shall then be inspected for evidence of separation. Creaming (formation of a creamy portion of emulsion which tends to rise to the top without actually separating as a layer of free oil) shall not be considered separation.
- 4.5.4 Hydrogen ion concentration test. An emulsion shall be prepared by vigorously shaking 5 mL of the finished oil and 45 mL of distilled water, having a pH value of 6.5 to 7.0, in a glass-stoppered flask. An immediate determination of the pH value shall be made electrometrically, using a glass electrode.

## 4.5.5 Corrosion tests (of the emulsion).

- 4.5.5.1 Spot test. Three test panels (one each) approximately 2 inches square (13 cm<sup>2</sup>) shall be made of: Aluminum alloy conforming to QQ-A-250/4. copper conforming to QQ-C-502, and low-carbon steel conforming to MIL-S-7952. These panels shall be polished to a finish of 6 to 8 microinches (0.15 - 0.20 micrometres) roughness height rating (RHR) by suitable means. Just before use, the panel shall be finish cleaned with a paste of magnesium oxide (heavy powder magnesium oxide, Fisher Scientific part \* M68-3 or equivalent) and water until free from water break. The test panels shall be rinsed in distilled water, then by 95 percent ethyl alcohol, dried and placed polished side up on a glass or other noncorroding surface. One-half mL of emulsion, prepared with 20 parts of finished oil and 80 parts of distilled water, by volume, shall be placed on each test panel. In addition, a specimen of solid tin-lead solder, conforming to QQ-S-571, having a surface area of not less than 1 square inch (6.54 cm2), shall be rinsed with distilled water, then 95 percent alcohol, dried, dipped into the emulsion and then placed immediately with the other test specimens. The test specimens shall then be allowed to stand undisturbed and exposed in an atmosphere of 50 ± 5 percent relative humidity at a temperature of 77° ± 9°F (25° ± 5°C), for 168 hours. The test specimens shall then be washed successively with distilled water, ethyl alcohol, and ASTM precipitation naphtha, and blown dry with compressed air. The test specimens shall then be examined visually for evidence of discoloration and under a magnification of 20 to 30 diameters for evidence of etching, pitting, or other corrosion.
- 4.5.5.2 Immersion test. Three test strips (one each) approximately 1/2 inch (1.27 cm) by 3 inches (7.62 cm) shall be made of: Aluminum alloy conforming to QQ-A-250/4, copper conforming to QQ-C-502, and low-carbon steel

conforming to MIL-S-7952. These panels shall be polished to a finish of 6 to 8 microinches (0.15 - 0.20 micrometres) RHR by suitable means. Just before use, the panel shall be finish cleaned with a paste of magnesium oxide (heavy powder magnesium oxide, Fisher Scientific part \* M68-3 or equivalent) and water until free from water break. The test strips shall be rinsed in distilled water, then by 95 percent ethyl alcohol, dried, and placed in individual test tubes. In addition, a specimen of solid tin-lead solder conforming to QQ-S-571, approximately 3 inches (7.62 cm) in length and not less than 1/16 inch (0.16 cm) in diameter, shall be rinsed in distilled water, 95 percent ethyl alcohol, dried, and placed in an individual test tube. Sufficient emulsion, prepared with 20 parts of oil and 80 parts of distilled water, by volume, shall be added to the test tubes to completely immerse the metal strips. The test tube shall be tightly stoppered and allowed to stand vertically at a temperature of 100° ± 7°F (38° ± 4°C) for 168 hours. The metal strips shall be examined visually for evidence of discoloration, and shall be examined under a magnification of 20 to 30 diameters for evidence of etching, pitting, or other corrosion.

## 4.5.6 Finished oil composition tests.

- 4.5.6.1 Infrared sulfonate determination. Infrared spectra shall be obtained on a double-beam recording spectrophotometer operating from 2 to 16 micrometres. The presence of petroleum sulfonates shall be determined by the presence of two strong absorption bands located at 8.40 and at 9.45 micrometres. Cell thickness shall be chosen so that the sulfonate absorption at 8.4 micrometres lies between 0.5 and 0.6.
- 4.5.6.2 Infrared fatty acid and fatty acid soap determination. The concentration of fatty acids and fatty acid soaps shall be expressed as a ratio of sulfonate absorbance to the acid and soap absorbances. The fatty acid absorbance shall be calculated from the carbonyl absorption band at approximately 5.8 micrometres. The absence of a distinct peak at this wavelength shall be considered as no fatty acids present. If a peak is present, a line to be known as the I. baseline for fatty acids shall be drawn from the curve at 5.50 micrometres to the curve at 6.50 micrometres. The fatty acid soap absorbance shall be calculated at the 6.40-micrometre absorption band. A line to be known as the I. baseline for fatty acid soaps shall be drawn from the curve at 5.50 micrometres to the curve at 7.80 micrometres. The petroleum sulfonate absorbance shall be calculated at the 8.40-micrometre absorption band. Its I, baseline shall be drawn from the curve at 7.80 micrometres to the curve at 10.0 micrometres. The ratio of sulfonate absorbance to fatty acid absorbance shall be no less than 6.0 to 1; the ratio of sulfonate to fatty acid soap absorbance shall be no less than 3.5 to 1.
- 4.5.7 Rejection and retest. Materials not conforming to the requirements of this specification shall be rejected. Rejected material shall not be submitted without furnishing full particulars concerning previous rejection and measures taken to overcome defects.
- 4.5.8 Packaging, packing, and marking. Preparation for delivery shall be inspected for conformance to section 5.

## 5. PREPARATION FOR DELIVERY

5.1 Packaging, packing, and marking. Packaging, packing, and marking shall be in accordance with MIL-STD-290 as specified (see 6.2).

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

- 6.1 Intended use. The finished oil is intended only to be used to prevent corrosion caused by water-alcohol mixtures contained in ground handling equipment and used in aircraft water-alcohol injection systems. The aircraft water-alcohol injection systems include the thrust augmentation system used in certain jet aircraft engines and the anti-detonation injection system used in certain reciprocating aircraft engines.
- 6.2 Acquisition requirements. Acquisition documents should specify the following:
  - (a) Title, number, and date of this specification.
  - (b) Size of containers (see 4.3.2).
  - .(c) Where the test sample should be sent (see 4.4.1.1).
  - (d) Applicable levels of packaging and packing (see 5.1).
- (e) Issue of DODISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.1.1 and 2.2).
- 6.3 International standardization agreements. Certain provisions of this specification (see 1.1) are the subject of an international standardization agreement. Air Standardization Co-ordinating Committee (ASCC) Air Standard 15/1 (STANAG 1135, Annex C, Appendix 3). When amendment, revision, or cancellation of this specification is proposed which will modify the international agreement concerned, the preparing activity will take appropriate action through international standardization channels including departmental standardization offices to change the agreement or make other appropriate accommodations.
- 6.4 <u>Material Safety Data Sheets</u>. Contracting officers will identify those activities requiring copies of completed Material Safety Data Sheets prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.
- 6.5 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.
  - 6.6 Subject term (key word) listing.

Corrosion preventive Soluble oil Thrust augmentation Anti-detonation

Custodian:

Air Force - 68

Preparing activity: Air Force - 68

PROJECT NO. 6850-1057

Review activities:

Navy - SH

Air Force - 11

International interest (see 6.3)