MIL-I-25017E <u>15 JUNE 1989</u> SUPERSEDING MIL-I-25017D 27 MAY 1981

MILITARY SPECIFICATION INHIBITOR, CORROSION/LUBRICITY IMPROVER, FUEL SOLUBLE (METRIC)

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

1. SCOPE

1.1 <u>Scope.</u> This specification covers one type of fuel soluble corrosion inhibitor/lubricity improver additive for use in aviation turbine fuel, motor gasoline, diesel fuel, and related petroleum products.

2. APPLICATION DOCUMENTS

2.1 Government documents

2.1.1 <u>Specifications and standards.</u> The following specifications and standards 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

TT-S-735	Standard Test Fluids; Hydrocarbon
VV-F-800	Fuel Oil, Diesel
VV-G-1690	Gasoline, Automotive, Leaded or Unleaded

AMSC N/A

FSC 6850

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to ASD/ENES, Wright-Patterson AFB, OH 45433-6503 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

DISTRIBUTION STATEMENT A: Approved for public release: distribution is unlimited

MILITARY

MIL-G-3056	Gasoline, Automotive, Combat
MIL-T-5624	Turbine Fuel, Aviation grades JP-4 and JP-5
MIL-C-7024	Calibrating Fluid, Aircraft Fuel System Components
MIL-L-7808	Lubricating Oil, Aircraft Turbine Engine, Synthetic Base
MIL-T-25524	Turbine Fuel, Aviation, Thermally Stable
MIL-F-25558	Fuel, Ramjet Engine, Grade RJ-1
MIL-I-27686	Inhibitor, Icing, Fuel System
MIL-G-46015	Gasoline, Automotive, Combat, Referee Grade
MIL-F-46162	Fuel, Diesel, Referee Grade
MIL-T-83133	Turbine Fuel, Aviation, Kerosene Type, Grade JP-8

STANDARDS

FEDERAL

FED_STD_791

Lubricants, Liquid Fuels, and Related Products; Methods of Testing

Military

MIL-STD-290 Packaging of Petroleum and Related Products

(Unless otherwise indicated, copies of federal and military specifications and standards are available from the Naval Publications and Forms Center, (ATTN: NPODS), 5801 Tabor Avenue, Philadelphia, PA 19120-5099.)

2.2 <u>Non-Government publications</u>. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issue 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 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_A_108	Standard-Specification For Steel Bars, Carbon, Cold-Finished.
	Standard Quality
ASTM D 56	Standard Test Method For Flash Point by Tag Closed Tester
ASTM D 97	Standard Test Method For Pour Point of Petroleum
ASTM D 445	Standard Test Method For Kinematic Viscosity of Transparent and
	Opaque Liquids
ASTM D 482	Standard Test Method for Ash From Petroleum Products

ASTM D 664	Standard Test Method For Acid Number of Petroleum Products by Potentiometer Titration
ASTM D 665	Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water, Test Method For
ASTM D 1298	Standard Test Method For Density, Relative Density (Specific Gravity) or API Gravity of Crude Petroleum and Liquid Petroleum Products By Hydrometer
ASTM D 2274	Oxidation Stability of Distillate Fuel Oil (Accelerated Method) Test Method For
ASTM D 2624	Standard Test Methods For Electrical Conductivity of Aviation and Distillate Fuels Containing a Static Dissipator Additive
ASTM D 3948	Test for Water Separation Characteristics of Aviation Turbine Fuels
ASTM D 4057	Standard Practice for Manual Sampling of Petroleum and Petroleum Products
ASTM D 4308	Test Method For Electrical Conductivity of Liquid Hydrocarbons by Precision Meter

(Application for copies should be addressed to the Society for Testing and Materials, 1916 Race Street, Philadelphia PA 19013.)

Coordinating Research Council, Inc.

CRC Report No 560Aviation Fuel Lubricity Evaluation

(Applications for copies should be addressed to the Coordinating Research Council, Inc. 219 Perimeter Center Parkway, Atlanta, GA 30346.)

(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 <u>Order of precedence.</u> 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 <u>Qualification</u>. The inhibitors furnished under this specification shall be products which are authorized by the qualifying activity for listing on the applicable qualified products list at the time of award of contract (see 4.3 and 6.5).

3.1.1 <u>Qualification requirements</u>. The qualification requirements for the inhibitors are listed for each type of fuel. All approved inhibitors shall meet the requirements of 3.2

through 3.12, 3.16 and 3.17 to be qualified for use in fuels conforming to MIL-T-5624, MIL-C-7024, MIL-T-25524, MIL-F-25558, and MIL-T-83133. To qualify for use in motor gasolines (Mil-G-3056 and VV-G-1690) and diesel fuel (VV-F-800), the inhibitors shall also pass the applicable requirements of 3.13 through 3.15.

3.2 <u>Materials.</u> The composition of the finished inhibitor is not limited but is subject to review by the qualifying activity in order to assure service compatibility with previously qualified products.

3.2.1 <u>Toxic products and formulations</u>. The materials shall have no adverse effect on the health of personnel when used for its intended purpose. Questions pertinent to this effect shall be referred by the procuring activity to the appropriate departmental medical service who will act as an advisor to the procuring activity. (see 6.3).

3.3 <u>Solubility.</u> The maximum allowable concentration of inhibitor, as defined in 3.7 shall be readily and completely dissolved in all fuels for which it is qualified. There shall be no precipitation, cloudiness or other evidence of insolubility when tested as specified in 4.6.1.

3.4 <u>Compatibility</u>. The inhibitor shall be compatible with all inhibitors currently qualified under this specification and with the static dissipator additives listed in MIL-T-5624. There shall be no precipitation, cloudiness, or other evidence of noncompatibility when tested as specified in 4.6.2

3.5 <u>Relative effective concentration</u>. The relative effective concentration shall be determined in accordance with 4.6.3 and shall be expressed in grams of finished inhibitor per cubic meter of fuel. The relative effective concentration shall not be less than 6 grams of inhibitor per cubic meter of fuel (g/m³) and shall be approved at concentrations divisible by 3 (e.g., 6, 9, 12, 15, ...33, and 36 g/m³),

3.6 <u>Minimum effective concentration</u>. The minimum effective concentration shall be the larger of the following:

a. One and a half (1.5) times the relative effective concentration, rounded to the next whole number of grams per cubic meter (e.g., 9, 14, 18, 23, ..., 50, 54).

b. The amount of inhibitor (in whole numbers of grams per cubic meter: e.g., 9, 10, 11, 12, ...) that gives a wear scar diameter of 0.65 mm or less when tested for lubricity using the Ball-On-Cylinder Lubricity Evaluator (BOCLE), as specified in 4.6.3.2.

3.7 <u>Maximum allowable concentration</u>. The maximum allowable concentration shall be the lowest of the following (all expressed in grams of inhibitor per cubic meter of fuel):

a. Fifty-four grams of inhibitor per cubic meter of fuel.

b. Four times the relative effective concentration.

c. The highest concentration giving a Micro-Separometer Rating (MSEP) of 70 or higher when determined in accordance with 4.6.4.

d. The highest concentration giving less than 40% change in electrical conductivity with fuel containing static dissipator additive (see 4.6.2.2).

3.8 <u>Ash content.</u> The ash content of the inhibitor shall not exceed 0.10 percent when determined in accordance with 4.6.5.

3.9 <u>Pour point</u>. The maximum allowable pour point of the finished inhibitor shall be -18° when determined as specified in 4.6.6.

3.10 <u>Aircraft turbine engine operation</u>. Grade JP-4 fuel (MIL-T-5624) or JP-8 (MIL-T-83133) fuel containing twice the maximum allowable concentration (see 3.7) of the inhibitor shall be tested in accordance with 4.6.7 to determine its acceptability for turbine engine use. Engine operation shall not be adversely affected and the post-test condition of the engine shall indicate no excessive deposits, water, corrosion, et cetera, which are attributed to the inhibitor.

3.11 <u>Specification requirements.</u> A blend of the inhibitor at its maximum allowable concentration in a representative fuel shall meet all of the requirements of each applicable specification when tested in accordance with 4.6.8. For example, to be qualified for use in a motor gasoline, a gasoline conforming to MIL-G-3056 shall continue to meet all applicable requirements of MIL-G-3056 after the maximum allowable concentration of the inhibitor is added.

3.12 <u>Storage stability.</u> After storage for 12 months in accordance with 4.6.9, the inhibitor shall show no precipitation, layering, or other evidence of gross separation or degradation. Inhibitor representing the top half of the stored sample shall meet all requirements of this specification except 3.10.

3.13 <u>Induction system deposit</u>. For use in motor gasolines conforming to VV-G-1690 and MIL-G-3056, the inhibitor shall pass the induction system deposit test performed in accordance with 4.6.10

3.14 <u>Emulsification tendency</u>. To obtain approval for use in motor gasolines conforming to VV-G-1690 and MIL-G-3056 and diesel fuel conforming to VV-F-800, the inhibitor shall pass the emulsification tendency test performed in accordance with 4.6.11.

3.15 <u>Accelerated stability.</u> To obtain approval for use in diesel fuels conforming to VV-F-800 the inhibitor shall pass the accelerated stability test performed in accordance with 4.6.12.

3.16 <u>Identification qualification data.</u> The following properties of the finished inhibitor shall be determined but not limited during qualification: density 15°C, viscosity at 40°C, flash point, neutralization number, pH, and type of metallic constituent, if present (see 4.6.13). The permissible production variation of individual properties shall be established at the time of qualification by mutual agreement between the manufacturer and the qualifying activity. Individual batches of inhibitor subsequently subjected to qualification conformance inspections shall conform to the established range of properties. The ranges shall not adversely affect any of the inhibitor performance characteristics such as relative effective concentration and Micro Separometer Rating (MSEP).

3.17 <u>Workmanship</u>. The finished product in bulk or container shall be uniform in appearance and visually free from grit, undissolved water, insoluble components, or other adulteration.

4. QUALITY ASSURANCE PROVISIONS

4.1 <u>Responsibility for inspection.</u> 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 <u>Responsibility for compliance.</u> All items shall meet all requirements of sections 3 and 5. The inspections 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 <u>Classification of inspections</u>. The inspection requirements specified herein are classified as follows:

a. Qualification inspection (see 4.3)

b. Quality conformance inspection (see 4.4).

4.3 <u>Oualification inspection conditions</u>. Unless otherwise specified, all inspections shall be performed in accordance with test conditions specified in 4.6.

4.3.1 <u>Test report.</u> A certified test report shall be forwarded to the activity responsible for qualification before the qualification sample is supplied. The test report shall contain laboratory data showing the results required by 3.3, 3.4, 3.5, 3.6, 3.7, 3.8, 3.9, 3.11, 3.12, and 3.16. The test report shall also contain laboratory data on any of the special tests conducted to qualify the inhibitor for use in motor gasoline and diesel fuel (e.g., 3.13, 3.14, and 3.15). In addition, complete formulation data shall be supplied to the qualifying activity. This data shall include chemical composition (I.U.P.A.C. nomenclature and structural diagrams of each ingredient), the percentages of each ingredient, the manufacturer and trade names of each ingredient, and where available, the purity of each ingredient. The contractor shall furnish toxicological data and formulations required to evaluate the safety of the material for the proposed use.

4.3.1.1 <u>Qualification sampling</u>. Unless otherwise specified by the activity responsible for qualification, an initial 1-liter sample of finished inhibitor shall be submitted for evaluation by all of the tests with the exception of the storage stability and aircraft turbine engine tests. If the product passes these tests, an additional sample of finished inhibitor will be required for the storage stability and aircraft turbine engine tests. Samples shall be identified as required and forwarded to the laboratory responsible for testing as designated in the letter of authorization from the activity responsible for qualification (see 6.5).

4.3.1.2 <u>Requalification</u>. Requalification will be required in the event any change in composition or formulation, source of inhibitor or its ingredients, or manufacturing sites is made.

4.3.1.3 <u>Retention of qualification</u>. The retention of qualification of products approved for listing on the qualified products list (QPL) shall be accomplished by a periodic verification to determine continued compliance of a supplier's product with the requirements of this specification. The verification intervals shall not exceed two years. Unless otherwise specified by the activity responsible for the qualified products list, verification of qualification may be made by certification.

4.4 <u>Ouality conformance inspection</u>. Quality conformance inspection of a bulk lot of inhibitor shall consist of tests for conformance to requirements for solubility (3.3) ash (3.8), pour point (3.9) and property limits shown on the Qualified Products List. In addition, the product shall be required to pass a rusting test when blended in depolarized iso-octane at the relative effective concentration and tested in accordance with 4.6.3.1 and shall also be required to give a Micro Separometer Rating (MSEP) of 70 or higher (average of three tests) when tested at the maximum allowable concentration in accordance with 4.6.4).

4.4.1 Inspection lot

4.4.1.1 <u>Bulk lot.</u> A bulk lot is defined as an indefinite quantity of a homogeneous mixture of material offered for acceptance in a single isolated container, manufactured as a single isolated batch, or manufactured by a single plant run (not exceeding 24 hours) through the same processing equipment with no change in ingredient material.

4.4.1.2 <u>Packaged lot.</u> A packaged lot is defined as a indefinite number of 55-gallon drums or smaller unit packages of identical size and type, offered for acceptance, and filled with a homogenous mixture of material from a bulk lot.

4.4.2. <u>Sampling</u>. Each bulk or package lot of material shall be sampled for verification of product quality and compliance in accordance with ASTM D 4057.

4.5 <u>Inspection</u>. Inspection shall be in accordance with Method 9601 of FED-STD-791.

4.6 Test methods.

4.6.1 <u>Solubility</u>. The maximum allowable concentration of inhibitor shall be mixed with each of the following fuels. The fuel shall contain no other inhibitors. Immediately after mixing and at the end of 24 hours, the samples shall be visually inspected for precipitation, cloudiness, or other evidence of insolubility.

a. JP-4 and JP-5 fuel conforming to MIL-T-5624 containing the maximum allowable concentration of specified fuel system icing inhibitor.

b. A motor gasoline conforming to MIL-G-3056, MIL-G-46015, or VV-G-1690. (Required only if inhibitor is to be qualified for use in motor gasolines).

c. A diesel fuel conforming to VV-F-800 or MIL-F-46162. (Required only if inhibitor is to be qualified for use in diesel fuel).

4.6.2 <u>Compatibility</u>

4.6.2.1 <u>Other inhibitors.</u> Grade JP-4 fuel (MIL-T-5624) or JP-8 fuel (MIL-T-83133) containing the maximum allowable concentration of the inhibitor under test and no other corrosion inhibitors shall be mixed in equal proportions with samples of MIL-T-5624, grade JP-4 fuel or MIL-T-83133, grade JP-8 fuel containing the maximum allowable amount of each inhibitor previously qualified under this specification. The MIL-T-5624, grade JP-4 fuel or MIL-T-83133, grade JP-8 fuel used shall contain the maximum allowable amount of fuel system icing inhibitor conforming to MIL-I-27686. At the end of a 24-hour period, the samples shall be visually inspected for precipitation, cloudiness or other evidence of noncompatibility.

4.6.2.2 <u>Static dissipator additive</u>. Grade JP-4 fuel (MIL-T-5624), filtered through clay as described in appendix A.4 of ASTM D 3948, shall be blended with each static

dissipator additive approved in MIL-T-5624 to provide test fuels having a conductivity of 400 picosiemens per meter $(pS/m)\pm100 pS/m$. After a 24-hour period, to insure that equilibrium fuel conductivity has been established, the inhibitor under test shall be added and mixed. At the end of another 24-hour period, no more than ±40 percent change in the electrical conductivity of the fuel shall have occurred as a result of the test inhibitor. The fuel electrical conductivity shall be measured using either ASTM D 2624 or ASTM D 4308 test methods. The post-test visual inspection of the sample shall reveal no precipitation, cloudiness or other evidence of noncompatibility. (Note: Some loss in fuel conductivity with time may occur when bare glass bottles or bare metal cans are used with fuels containing static dissipator additives. The use of an epoxy-coated container is suggested. Also, fuel conductivity is temperature sensitive; no significant change in temperature should be allowed during the test.)

4.6.3 <u>Relative effective concentration</u>. The relative effective concentration of the inhibitor shall be determined by testing the inhibitor at various concentrations in depolarized iso-octane in accordance with 4.6.3.1. The inhibitor shall be tested at concentrations divisible by 3 (e.g., 6, 9, 12, 15, ... 33, and 35 grams inhibitor per cubic meter of fuel). No intermediate concentrations shall be tested. The relative effective concentration shall be defined as the lowest concentration giving a passing result in accordance with 4.6.3.1.6

4.6.3.1 Rusting test method

4.6.3.1.1 <u>Test apparatus.</u> The test apparatus shall conform to the following requirements:

a. Oil bath, conforming to ASTM D 665, with the additional requirement that it must be capable of maintaining the test sample at a temperature of $38^{\circ}C \pm 0.5^{\circ}C$

b. Beaker, beaker cover, stirrer, stirring apparatus, and chuck and motors for holding and rotating specimens while polishing shall conform to ASTM D 665.

c. Infrared heat lamp, 250 watts.

d. Hypodermic syringe, glass, 3-ml, with 15.24 cm (6 inch) stainless steel needles.

e. Disposable microliter pipets, consisting of calibrated capillary tubes containing 1,2,3,4,5,10,15 and 20 microliters.

f. Column, chromatographic, glass, 40 mm ID x 600 mm with poly (tetrafluoroethylene) stopcock. A separatory funnel, Squibb, 1-liter, with poly (tetrafluoroethylene) stopcock may be substituted for the chromatographic column.

g. Specimen holder, poly (tetrafluoroethylene) dimensions as specified in ASTM D 665 for type 2 holder.

h. Specimen, dimensions as specified in ASTM D 665, made of grade 1018 steel in accordance with ASTM A 108. The specimen shall be fabricated from 1.58 cm (0.625 inch) diameter round stock by machining or grinding to the final diameter of 1.27 cm. (0.50 inch). The specimen may be reused from test to test but shall be discarded when the diameter is reduced to 0.953 cm (0.375 inch).

4.6.3.1.2 <u>Test materials</u>. Test materials shall conform to the following requirements:

a. Silica gel, 28–200 mesh, heated to 107.2°C for 2 hours and cooled in a dessicator before use.

b. Test solvent, iso-octane conforming to TT-S-735, which has been freshly depolarized as follows: A glass chromatographic column or 1 liter separatory (Squibb) funnel is filled with silica gel to a height 20 cm above the stopcock, retaining the silica gel by means of a glass wool plug. (NOTE: Do not use stopcock grease). One gallon of iso-octane is passed through the silica-gel bed by gravity, discarding the first 50 ml and collecting the remainder in a chemically clean glass container. The depolarized iso-octane should be used within one week after treatment.

c. Test water, type B medium hard, prepared as follows: Make up three stock solutions using ACS reagent-grade chemicals in distilled water. Each solution shall contain one of these chemical 16.4 g/liter NaHCO₃, 13.2 g/liter CaC1₂ \cdot 2H₂O, or 8.2 g/liter MgSO₄ \cdot 7H₂O. Pipet 10 ml of the NaHCO₃ stock solution into 800 ml of distilled water in a one liter volumetric flask, and shake vigorously. While swirling the contents of the flask, pipet 10 ml of the Cacl₂ stock solution and then 10 ml of the MgSO₃ stock solution into the flask, add distilled water to bring the volume to one liter, and mix thoroughly. The final blend shall be clear and free of precipitation.

d. Isopropanol, ACS reagent grade.

e. Glass ware cleaning solution.

f. Lintless paper tissue. (NOTE: Cel-Fibe 1710 Wipes, available from Cel-Fibe, Milltown, New Jersey are satisfactory.)

g. Abrasive cloth, 150-, 240-, and 400- grit metal working aluminum oxide abrasive cloth, closed coat on jeans backing. The abrasive cloth is available in rolls of 1-inch tape, the most convenient form for use in this test.

h. Disposable vinyl gloves.

4.6.3.1.3 <u>Specimen preparation</u>. The specimen, whether new or reused from a previous test, shall be cleaned by solvent rinsing or brushing as needed to remove oily residues, loose rust, or foreign material. After this preliminary cleaning, the specimen

shall be handled only with vinyl gloves. (NOTE: It is essential to avoid contamination of the specimen, particularly by perspiration residues, and care should be taken to avoid transfer of such contaminants to the specimen via the abrasive cloth or the lintless paper tissues). The specimen shall then be ground successively with 150-, 240-, and 400-grit abrasive cloth while mounted in the chuck of the grinding and polishing apparatus and turned at 1700 to 1800 rpm, in accordance with the following procedures.

a. Grind with 150-grit cloth to remove all defects, irregularities, pits, and scratches as determined by visual inspection. Old 150-grit cloth may be used to remove rust or major iregularities, but the grinding shall be completed with new cloth. Stop the motor and scratch the static specimen longitudinally with one pass of new 150-grit cloth, using light pressure so that visible scratches appear.

b. Grind with 240-grit cloth, removing all marks from the 150-grit cloth, and finishing with new 240-grit cloth. Stop motor and scratch the static specimen longitudinally with one pass of new 240-grit cloth, using light pressure, so that visible scratches appear.

c. Polish with 400-grit cloth by wrapping a strip of cloth halfway around the specimen and applying a firm but gentle downward pull to the ends of the strip and moving the strip slowly along the specimen. Shift the position of the abrasive cloth frequently to expose fresh abrasive to the specimen. Continue this procedure, using new strips of abrasive cloth as required, until all makes from the previous 240-grit operation have been removed and the surface presents a uniform appearance, free of longitudinal or spiral scratches, with all polishing marks appearing to be circumferential. The final passes along the specimen shall be made with fresh abrasive cloth.

d. Remove the specimen from the chuck, wipe with lintless tissue, and store in a beaker of depolarized iso-octane in a desiccator containing silica gel or other noncorrosive desiccant until ready for use. The storage period in the iso-octane shall not exceed seven days.

4.6.3.1.4 <u>Preparation of test blend</u>. The test blend shall be prepared in the test beaker, not more than two hours before the immersion of the specimen in the test blend. The test blend shall be prepared in accordance with the following procedure:

a. Clean the test beaker with a suitable cleaning solution (see note below). Clean the stainless steel stirrer and methyl methacrylate beaker cover by rinsing in any suitable aliphatic hydrocarbon solvent such as a light naptha or iso-octane, washing thoroughly with hot distilled water, and oven drying (not over 65.6°C for cover). NOTE: If a glass stirrer or beaker cover is used, it should be cleaned in the same manner as the

test beaker. Any suitable cleaning method that provides cleaning quality comparable to the use of chromic acid may be used. The use of a detergent cleaning solution is suggested. Use stainless steel forceps to handle the glassware. Wash with tap water and then with distilled water. Rinse with reagent grade isopropyl alcohol and dry in the air or oven. Detergent cleaning avoids the potential hazards and inconvenience associated with handling corrosive chromic acid solution. The latter remains as the reference cleaning practice and as such may function as an alternative to the preferred use of detergent solutions.

b. Prepare the blend of iso-octane and inhibitor in the test beaker with direct addition of the inhibitor. No intermediate blends, concentrates, or stock solutions are permitted. Prepare each test blend using between 300 and 400 ml of iso-octane in the test beaker. Use pipet or pipets to add integral numbers of microliters of the inhibitor to the beaker to increase measurement accuracy. Add the calculated volume of depolarized iso-octane to the test beaker. Fill the appropriate microliter pipet or pipets with inhibitor, wipe off excess, and force the inhibitor into the iso-octane. Allow the pipet to fill with iso-octane by capillary attraction and force this rinse into the test beaker. Repeat the rinse four times. Calculate the amount of iso-octane and inhibitor to be added to the test beaker using the instructions given in 4.6.3.1.4.c.

c. Calculate the volume of iso-octane required for each concentration desired using the following equation. where density is in g/ml at 15°C.

ml of iso-octane = <u>density of inhibitor</u>) (microliters of inhibitors) (1000) (desired inhibitor concentration, grams/cubic meter)

For example, assume the inhibitor has a density of 0.95 and the desired concentration is 6 grams/cubic meter of fuel. Calculate the volume of iso-octane required when using 2 microliters of inhibitor:

ml iso-octane = (0.95) (2) (1000)/(6) = 316.7

For inhibitor having a density less than 0.9 g/ml, the volume of iso-octane for many concentrations of interest will be less than 300 ml or more than 400 ml. Use the following procedure:

(1) Calculate the volume of inhibitor required for 300 ml of iso-octane.

(2) Increase the volume of inhibitor to the next integral microliter and add to 300 ml of iso-octane in the test beaker. Mix well.

(3) Calculate the amount of inhibitor/iso-octane blend to be removed from the test beaker to leave the desired amount of inhibitor.

(4) Remove the calculated amount of inhibitor/iso-octane blend and replace with an equivalent volume of depolarized iso-octane. Mix well. This approach is illustrated for the above example:

(a) Using this equation, calculate the desired volume of inhibitor for 300 ml of iso-octane for an inhibitor with a density of 0.85 and for a desired concentration of 6 g/m^3 .

3000 ml iso-octane = (0.85) (X microliters inhibitor) (1000)/(6).

x = 2.12 microliter of inhibitor

(b) Add the next integral volume of inhibitor (i.e., 3 microliters) to 300 ml iso-octane and mix well. This gives an inhibitor concentration of 3 microliters inhibitor/300 ml iso-octane or 1 microliter/100 ml.

(c) The desired amount of inhibitor is 2.12 microliters. Thus, we need (2.12) (100) = 212 ml of inhibitor/iso-octane blend.

(d) Remove 88 ml of the inhibitor/iso-octane blend (i.e., 300 - 212 = 88 ml). Replace with 88 ml of the depolarized iso-octane. Mix well. This results in the correct volume of inhibitor (i.e., 2.12 microliters) in 300 ml of iso-octane.

d. Place the beaker in the oil bath which as been regulated previously to maintain a sample temperature of $38^{\circ}C \pm 0.5^{\circ}C$. The beaker is inserted in a hole of the bath cover and suspended at a level such that the oil level in the bath is not below the sample level in the beaker. Cover the beaker with the beaker cover and the stirrer in position. Adjust the stirrer so that the shaft is 6 mm off-center in the beaker, and the blade is within 2 mm of the bottom of the beaker. Then suspend a thermometer through the hole in the cover intended for that purpose, so that it is immersed to a depth of 57 mm. Stir for at least five minutes. Turn off the stirrer. Using a clean pipet or syringe, withdraw enough test blend to leave exactly 300 ml in the beaker. Allow the test blend to come to $38^{\circ}C \pm 0.5^{\circ}C$. Replace the thermometer with a cork or plastic plug.

4.6.3.1.5 <u>Test procedure</u>. After preparing a test specimen as described in 4.6.3.1.3 and a test blend as described in 4.6.3.1.4, the test shall be performed in accordance with the following procedure:

a. Remove a test specimen from the iso-octane in the desiccator and wipe dry with a lintless paper tissue, handling with vinyl gloves throughout this step and the following operations. Repolish with 400-grit abrasive cloth by wrapping a strip of the cloth halfway around the specimen and applying a firm but gently downward pull to the ends of the strip. Move the strip slowly along the specimen, twice in each direction,

shifting the strip after the first back-and-forth pass so that fresh abrasive is exposed to the specimen. Inspect the specimen to insure that the surface presents a uniform appearance, free of longitudinal or spiral scratches, with all polishing marks appearing to be circumferential. Additional polishing is required if the specimen appearance is other than described. After polishing is completed, remove the from the chuck, wipe lightly with lintless paper tissue, and screw the specimen into the specimen holder. Rinse the specimen with a stream of isopropanol from a wash bottle. Wipe dry immediately, wiping twice with fresh lintless paper tissues, using firm pressure and rotating the specimen while drawing through the the tissue. NOTE: Under conditions of high ambient humidity, it is necessary to heat the specimen to prevent condensation of moisture and premature rusting. Under such conditions before the rinsing operation, place the specimen and holder six inches from a 250-watt infrared heat lamp and rotate for one minute. Keep the specimen under the lamp while proceeding with the rinsing and wiping operations.

b. Immediately after rinsing and wiping, insert the specimen and holder through the specimen hole in the beaker cover and suspend the specimen so that its lower end is 13 to 15 mm from the bottom of the beaker. Leave the specimen in the test blend for a 10-minute static soak, then turn on the stirrer and soak dynamically for 20 minutes. NOTE: When multiple tests are run simultaneously, it is permissible to extend the static soak period to not more than 40 minutes in the case of the "first-in" specimen, giving the "last-in" specimen a 10-minute soak.

c. Turn off the stirrer. Remove the cork or plug from the beaker cover, and add 30 ml of test water to the test beaker, adding it very carefully to the bottom of the beaker by means of a hypodermic syringe. Change to a clean needle for each test beaker. Replace the cork or plug in the beaker cover.

d. Start the stirrer immediately and run for five hours, holding the bath temperature at the same setting so that the test samples will be maintained at $38^{\circ}C \pm 0.5^{\circ}C$.

e. At the end of five hours, stop the stirrer, remove the specimen and holder, rinse immediately with isopropanol, and allow to air-dry. Examine at once without magnification under normal indoor illumination, approximately 60-foot candles, scanning the surface very carefully, to detect any small pits. Record observations of visible rust, pits, stains, or deposits.

4.6.3.1.6 <u>Interpretation of test results.</u> A test shall be reported as failing if the center 48 mm section of the specimen shows six or more rust spots of any size, or if it shows any rust spot 1 mm in diameter or larger. (NOTE: The ends of the specimen, outside the center section, are ignored in rating the specimen.) Visible deposits or stains other than rust shall not constitute failure; deposits or stains may be examined microscopically to determine their classification. In order to assign a pass-fail rating

14 -

to a given inhibitor at a given concentration, two tests shall be performed. The inhibitor shall be reported as passing at the given concentration if both tests give passing ratings, or failure at the given concentration if both tests give failing ratings. If the two tests give one passing rating and one failing, two additional tests shall be performed. If either or both of these additional tests give a failing rating, the inhibitor shall be reported as failing at the given concentration. If both of the additional tests give passing ratings, the inhibitor shall be reported as passing the given concentration.

4.6.3.2 <u>Lubricity test.</u> The inhibitor shall be tested in accordance with the Ball-On-Cylinder Lubricity Evaluator (BOCLE), as described in Appendix Y of the CRC Report No. 560, using Isopar M as the solvent and a 1000 gram load on the ball.

4.6.4 <u>Micro separometer rating</u>. The inhibitor shall be blended into the reference fluid base, as described in ASTM D 3948, and tested in accordance with ASTM D 3948. For any given concentration of inhibitor, the average of three test results shall be used to determine the conformance to the requirements of 3.7 or 4.4.

4.6.5 <u>Ash content determinations.</u> The ash content of the inhibitor shall be determined in accordance with ASTM D 482, using a platinum crucible.

4.6.6 <u>Pour point determination</u>. Pour point shall be determined in accordance with ASTM D 97.

4.6.7 <u>Aircraft engine test.</u> The engine shall be operated for 100 hours in accordance with the engine operating requirements of MIL-L-7808. Grade JP-4 fuel, conforming to MIL-T-5624, or JP-8 fuel, conforming to MIL-T-83133, shall contain twice the maximum allowable concentration of the inhibitor. Upon completion of the test. components of the engine exposed to the fuel such as fuel controls, fuel nozzles, combustion section, turbine blades, exhaust section, elastomers, fuel/oil heat exchanges; and fuel pumps shall be examined for evidence of excess wear, deposits, corrosion or other deleterious effects. This test shall be performed by the activity responsible for qualification (see 6.5).

4.6.8 <u>Specification tests.</u> The inhibitor shall be added at its maximum concentration to a base fuel that contains no inhibitor but is otherwise representative of each grade of fuel for which the additive is to be qualified. The blend of fuel and inhibitor shall be subjected to all of the tests of each applicable specification.

4.6.9 <u>Storage stability test.</u> Two 1-liter or 1-quart amber glass bottles shall each be filled with 850 ml of the inhibitor and shall be tightly capped by means of a screw cap having a concical polyethylene liner. Each bottle shall be wrapped in a minimum amount of opaque packing materials sufficient for protection against mechanical damage, but minimal in thermal insulation qualities. The wrapped bottles shall be

enclosed in a tight wooden or metal box for further protection against breakage and sunlight. The crated samples shall be stored at ambient, outdoor conditions in a temperate climate. The box shall be kept off the ground and protected from direct sunlight and precipitation under a canopy, open shed roof, or similar ventilated shelter. The crated samples shall be stored undisturbed in an upright position for the specified period. One of the samples shall be stored for exactly 12 months and then removed for examinations and testing; the other samples shall be stored for 12 months or less and may be removed for examination and testing at any time at the option of the qualifying activity. Whenever a sample is removed for examination and testing, it shall be uncrated with minimum disturbance; the bottle shall not be shaken, inverted, or otherwise agitated. The contents of the bottle shall be inspected visually for precipitation, separation into layers, or other evidence of gross separation. The presence or absence and the nature of such separation shall be recorded. The top half of the liquid sample shall be carefully removed by suction or siphoning into another bottle, without disturbing the bottom half of the original sample. The top-half sample, after transfer to the second bottle, shall be shaken thoroughly and then used in laboratory testing, performed in accordance with 3.12. The bottom-half sample, in the original storage bottle, shall be retained for examination and possible additional testing to detect changes caused by storage.

4.6.10 <u>Induction system deposit test.</u> The inhibitor, at its maximum concentration, shall be blended into a MIL-G-3056 motor gasoline. The test fuel shall then be tested for the formation of induction system deposits in accordance with Method 500 of FED-STD-791. The naptha-washed deposits shall not exceed 2 mg/100 mg of fuel. The MIL-G-3056 gasoline without inhibitor shall also be tested in accordance with Method 500 of FED-STD-791 concurrently to define the level of deposition occurring as a result of the inhibitor.

4.6.11 <u>Emulsification tendency test.</u> The inhibitor, at maximum allowable concentration, shall be blended into a MIL-G-3056 motor gasoline and a MIL-F-46162 diesel fuel. Each test fuel shall then be examined for emulsification tendencies in accordance with Method 550 of FED-STD-791. Interface ratings in excess of three are evidence of unsatisfactory emulsification tendencies and shall not be allowed. The MIL-G-3056 motor gasoline and the MIL-F-46162 diesel fuel shall also be tested in accordance with Method 550 of FED-STD-791 to identify the quality of the fuels before the addition of the inhibitor.

4.6.12 <u>Accelerated stability test.</u> The test inhibitor, at its maximum allowable concentration, shall be blended into a diesel fuel (VV-F-800) that contains no additives. Each test fuel shall be tested for the formation of total insolubles in accordance with ASTM D 2274. The total insolubles shall not exceed 1.5 mg/100 ml. The diesel fuel without the test inhibitor shall also be tested in accordance with ASTM

D 2274 concurrently to define the level of insolubles occurring without the presence of the inhibitor. (NOTE: A suitable reference diesel fuel for this evaluation is described in Method 341.4 of FED-STD-791.)

4.6.13 <u>Identification tests</u>. Identification tests shall be conducted in accordance with the following methods:

Density at 15°C	ASTM D 1298 or pyonomreter
Viscosity at 37.8°C	ASTM D 445
Flash Point	ASTM D 56
Neutralization Number	ASTM D 664, total acid number

pH

On 0.10 - 0.11 g sample in 125 ml of ASTM D 664 titration solvent. Read the constant pH as defined in note 9 of ASTM D 664.

Metallic constituent

Emission spectrograph not applicable for mate rials with ash contents of 0.05 percent or lower.

4.7 <u>Inspection of packaging</u>. The packing and marking shall be examined for compliance with the requirements of section 5, specified herein.

5. PACKAGING

5.1 <u>Packing and marking</u>. The packing and marking shall be in accordance with level B or C requirements of MIL-STD-290.

6. NOTES

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

6.1 <u>Intended use.</u> The inhibitors covered by this specification are used, when specifically authorized, in jet engine fuels for the prevention of corrosion in fuel handling, transportation, and storage equipment and to improve the lubricating qualities of jet fuels. Certain of the inhibitors are also used in automotive gasoline, diesel fuel, and related petroleum products.

6.2 <u>Acquisition requirements</u>. Acquisition documents must specify the following:

a. Title, number, and date of the specification.

b. Issue of DODISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.).

c. Quantity required.

d. Levels of packing required (see 5.1).

6.2.1 <u>Amount of use of inhibitor</u>. When Government procurement documents specify the use of inhibitors in fuels and related petroleum products, the concentration of inhibitor shall be specified in grams of inhibitor per cubic meter of fuel and shall not be less than the minimum effective concentration nor more than the maximum allowable concentration as listed on the Qualified Products List. Since the inhibitor is intended for use under many different environments, it is not possible to establish a single optimum concentration for all uses. Therefore, when a specific concentration is not required by the Government, the quantity of inhibitor used may vary to meet specific conditions.

6.3 <u>Toxicity.</u> Questions pertinent to toxicity should be referred by the procuring activity to the appropriate department medical service who will act as an advisor to the procuring activity. In case of army procurement, the Surgeon General will act as advisor to the procuring activity.

6.4 <u>Inhibitor for addition to fuels.</u> When a fuel contractor or the Government purchases the inhibitor for addition to fuels to be used by the Government, the manufacturer of the inhibitor must certify to the purchaser that the product is an inhibitor that has been qualified under this specification. In addition, a test report showing compliance of the product with the requirements of 4.4 must be supplied to the purchaser. Additional data may be required by the purchasing activity to establish compliance with this specification.

6.5 <u>Qualification</u>. With respect to products requiring qualification, awards will be made only for products which are, at the time of award of contract, qualified for inclusion in Qualified Products list QPL 25017 whether or not such products have actually been so listed by that date. The attention of the contractors is called to these reqirements and manufacturers are urged to arrange to have the products that they propose to offer to the Federal Government tested for qualification in order that they may be eligible to be awarded contracts or purchase orders for the products List is Wright Research and Development Center, ATTN: POSF, Wright-Patterson AFB OH 45433-6563, and information pertaining to qualification of products may be obtained from that activity.

6.6 Key words

Corrosion inhibitor

Fuel additive

Fuel lubricity additive

Diesel

Gasoline

Petroleum

Turbine fuel

6.7 <u>Changes from previous issue</u>. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

Custodians: Army – ME

Navy – AS Air Force – 11

Review activities: Army – MD, AV Navy – SH Air Force – 68

User activity: Army – AT Preparing activity: Air Force - 11

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