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PERFORMANCE SPECIFICATION

LUBRICITY IMPROVER, FUEL SOLUBLE

(NATO S-1747)



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

1. SCOPE

1.1 Scope. This specification covers one type of fuel-soluble lubricity improver additive for use in aviation turbine fuel, motor gasoline, diesel fuel, and related petroleum products.

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3 and 4 of this specification, whether or not they are listed.

2.2 Government documents.

2.2.1 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 cited in the solicitation or contract.

FEDERAL STANDARDS

FED-STD-313	Material Safety Data, Transportation Data, and Disposal Data for Hazardous Materials Furnished to Government Activities
FED-STD-791	Testing Method of Lubricants, Liquid Fuels, and Related Products

COMMERCIAL ITEM DESCRIPTIONS

A-A-52557	Fuel Oil, Diesel; for Posts, Camps, and Stations
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DEPARTMENT OF DEFENSE SPECIFICATIONS

MIL-DTL-5624	Turbine Fuel, Aviation, Grades JP-4 and JP-5
MIL-PRF-7024	Calibrating Fluids, Aircraft Fuel System Components
MIL-PRF-7808	Lubricating Oil, Aircraft Turbine Engine, Synthetic Base
MIL-DTL-25524	Turbine Fuel, Aviation, Thermally Stable
MIL-DTL-83133	Turbine Fuel, Aviation, Kerosene Type, JP-8 (NATO F-34), NATO F-35, and JP-8+100 (NATO F-37)
MIL-DTL-85470	Inhibitor, Icing, Fuel System, High Flash NATO Code Number S-1745

(Copies of the above documents are available online at <https://assist.daps.dla.mil/quicksearch> or <http://assistdocs.com>, or from the Standardization Document Order Desk, 700 Robbins Avenue, Bldg 4D, Philadelphia PA 19111-5094)

2.3 Non-government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

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ASTM INTERNATIONAL

ASTM A108	Standard Specification for Steel Bar, Carbon and Alloy, Cold-Finished (DoD adopted)
ASTM D56	Standard Test Method for Flash Point by Tag Closed Cup Tester (DoD adopted)
ASTM D93	Standard Test Methods for Flash-Point by Pensky-Martens Closed Cup Tester (DoD adopted)
ASTM D97	Standard Test Method for Pour Point of Petroleum Products (DoD adopted)
ASTM D445	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity) (DoD adopted)
ASTM D471	Standard Test Method for Rubber Property – Effect of Liquids (DOD adopted)
ASTM D482	Standard Test Method for Ash From Petroleum Products (DoD adopted)
ASTM D664	Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration (DoD adopted)
ASTM D665	Standard Test Method for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water (DoD adopted)
ASTM D975	Standard Specification for Diesel Fuel Oils (DoD adopted)
ASTM D1298	Standard Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method (DoD adopted)
ASTM D2274	Standard Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method) (DoD adopted)
ASTM D2624	Standard Test Methods for Electrical Conductivity of Aviation and Distillate Fuels (DoD adopted)
ASTM D3948	Standard Test Method for Determining Water Separation Characteristics of Aviation Turbine Fuels by Portable Separometer (DoD adopted)
ASTM D4052	Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter (DoD adopted)
ASTM D4057	Standard Practice for Manual Sampling of Petroleum and Petroleum Products (DoD adopted)
ASTM D4308	Standard Test Method for Electrical Conductivity of Liquid Hydrocarbons by Precision Meter (DoD adopted)
ASTM D4814	Standard Specification for Automotive Spark-Ignition Engine Fuel (DoD adopted)
ASTM D5001	Standard Test Method for Measurement of Lubricity of Aviation Turbine Fuels by the Ball-on-Cylinder Lubricity Evaluator (BOCLE) (DoD adopted)
ASTM D5949	Standard Test Method for Pour Point of Petroleum Products (Automatic Pressure Pulsing Method)
ASTM D5950	Standard Test Method for Pour Point of Petroleum Products (Automatic Tilt Method)

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ASTM D5985	Standard Test Method for Pour Point of Petroleum Products (Rotational Method)
ASTM D6421	Standard Test Method for Evaluating Automotive Spark-Ignition Engine Fuel for Electronic Port Fuel Injector Fouling by Bench Procedure

(Copies of these documents are available at ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. Electronic copies of ASTM standards may be obtained from <http://www.astm.org>.)

2.4 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 Qualification. The lubricity improvers furnished under this specification shall be products that are authorized by the Qualifying Activity for listing on the applicable Qualified Products List (QPL) before contract award (see 4.2 and 6.6).

3.1.1 Qualification requirements. The qualification requirements for the lubricity improvers are listed for each type of fuel. All approved improvers shall meet the requirements of 3.2 through 3.12, 3.16, and 3.17 to be qualified for use in fuels which conform to MIL-DTL-5624, MIL-PRF-7024, MIL-DTL-25524, and MIL-DTL-83133. To qualify for use in motor gasolines (ASTM D4814) and diesel fuels (ASTM D975), the improver shall meet the requirements of 3.2 through 3.17. Category 2 additives are approved for use in fuels which conform to MIL-DTL-5624, MIL-PRF-7024, MIL-DTL-25524 and MIL-DTL-83133. In addition to the previously mentioned specifications, Category 1 additives are also approved for use in fuels which conform to ASTM D975 and ASTM D4814 (see Table I).

3.2 Materials. The composition of the finished lubricity improver is not limited but is subject to review by the Qualifying Activity to ensure service compatibility with previously qualified products.

3.2.1 Toxic products and formulations. The materials shall have no adverse effect on the health of personnel when used for their intended purpose. Questions pertinent to this effect shall be referred by the Procuring Activity to the appropriate departmental medical service, which will act as an advisor to the Procuring Activity (see 6.4).

3.3 Solubility. The maximum allowable concentration of lubricity improver, as defined in 3.7, shall be readily and completely dissolvable in all fuels for which it is qualified. There shall be no precipitation, cloudiness, or other evidence of insolubility when tested as specified in 4.4.1.

3.4 Compatibility. The lubricity improver shall be compatible with all those currently qualified under this specification, with all the static dissipater additive(s) allowed in MIL-DTL-83133, and with all the thermal stability improver additive(s) allowed in MIL-DTL-83133. There shall be no precipitation, cloudiness, or other evidence of non-compatibility when tested as specified in 4.4.2.

3.5 Relative effective concentration. The relative effective concentration shall be determined in accordance with 4.4.3 and shall be expressed in grams of finished improver per cubic meter of fuel. The relative effective concentration shall not be less than 6 grams of improver per cubic meter of fuel (6 g/m^3), not more than 36 g/m^3 , and shall be approved at concentrations divisible by 3 (i.e.; 6, 9, 12, 15, ... 36 g/m^3). The relative effective concentration shall be identified by the Qualifying Activity and cited on QPL-25017.

3.6 Minimum effective concentration. The minimum effective concentration shall be the larger of the following:

- a. One and one-half (1.5) times the relative effective concentration, as defined in 3.5, rounded to the next whole number of grams per cubic meter (i.e.; 9, 14, 18, 23, ... 54 g/m^3).

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- b. The amount of improver (in whole numbers of grams per cubic meter; e.g.; 9, 10, 11, 12, ... g/m³) 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 4.4.4.1.

The minimum effective concentration shall be identified by the Qualifying Activity and cited on QPL-25017.

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

- a. Fifty-four grams of improver per cubic meter of fuel (54 g/m³).
- b. Four times the relative effective concentration, as defined in 3.5.
- c. The highest concentration that results in a Micro-Separometer Rating (MSEP) of 70 or higher when determined in accordance with 4.4.5.1.
- d. The highest concentration that results in a less-than-40-percent change in electrical conductivity with fuel that contains static dissipater additive (see 4.4.2.2).

The maximum allowable concentration shall be identified by the Qualifying Activity and cited on QPL-25017.

3.8 Ash content. The ash content of the improver shall not be greater than 0.10 percent mass when determined in accordance with 4.4.6.

3.9 Pour point. The pour point of the finished improver shall not be greater than -18°C when determined as specified in 4.4.7.

3.10 Aircraft turbine engine operation. Grade JP-8 (MIL-DTL-83133) or JP-5 (MIL-DTL-5624) fuel that contains twice the maximum allowable concentration (see 3.7) of the improver shall be tested in accordance with 4.4.8 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, or other adulteration, attributable to the improver.

3.11 Specification requirements. A blend of the improver at its maximum allowable concentration in a representative fuel shall meet all of the requirements of each applicable specification when tested in accordance with 4.4.9. For example, to be qualified for use in a motor gasoline, a gasoline that conforms to ASTM D4814 shall continue to meet all applicable requirements of ASTM D4814 after the maximum allowable concentration of the improver is added.

3.12 Storage stability. After stored for 12 months in accordance with 4.4.10, the improver shall show no precipitation, layering, or other evidence of gross separation or degradation. Improver that represents the top-half of the stored sample shall meet all requirements of this specification except 3.10.

3.13 Fuel Injection fouling. Improver for use in motor gasolines that conform to ASTM D4814 shall pass the fuel injection fouling bench procedure performed in accordance with 4.4.11.

3.14 Emulsification tendency. To obtain approval for use in motor gasolines that conform to ASTM D4814 and diesel fuels that conform to ASTM D975, the improver shall pass the emulsification tendency test performed in accordance with 4.4.12.

3.15 Accelerated stability. The improver shall pass the accelerated stability test performed in accordance with 4.4.13 before approved for use in diesel fuels that conform to ASTM D975.

3.16 Identification qualification data. The following properties of the finished improver shall be determined but not limited during qualification: density at 15°C, viscosity at 40°C, flash point, neutralization number, pH, and type of metallic constituent, if present (see 4.4.14). 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 improver subsequently subjected to qualification conformance inspections shall conform to the established range of properties. The ranges shall not adversely affect any of the improver performance characteristics such as relative effective concentration and MSEP.

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3.17 Workmanship. The finished product in bulk or container shall be uniform in appearance and visually free from grit, un-dissolved water, insoluble components, or other adulteration.

4. VERIFICATION

4.1 Classification of inspections. The inspection requirements specified herein are classified as follows:

- a. Qualification inspection (see 4.2).
- b. Conformance inspection (see 4.3).

4.2 Qualification inspection conditions. Unless otherwise specified, all inspections shall be performed in accordance with the test conditions specified in 4.4.

4.2.1 Qualification sampling. Unless otherwise specified by the activity responsible for qualification, an initial 1-liter sample of finished improver shall be submitted for evaluation by all the tests required in 3.1.1, with the exception of the storage stability (3.12) and aircraft turbine engine test (3.10), to an independent laboratory approved by the Qualifying Activity. If the product passes all the tests, an additional sample of finished improver will be required for the storage stability and aircraft turbine engine tests. The additional samples shall be identified as required and forwarded to the Qualifying Activity (see 6.6).

4.2.2 Requalification. Requalification will be required in the event any change in composition or formulation, source of improver or its ingredients, or manufacturing practice or site occurs.

4.2.3 Retention of qualification. 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.

4.3 Conformance inspection. Quality conformance inspection of a bulk lot, prior to becoming a packaged lot, of improver shall consist of tests for conformance requirements for ash content (3.8), pour point (3.9), MSEP (3.7), BOCLE (3.6.), and the property limits (3.16) shown on the Qualified Products List. (see Table I)

4.3.1 Inspection lots.

4.3.1.1 Bulk lot. A bulk lot is defined as an indefinite quantity of a homogeneous mixture of material offered for acceptance in a single, isolated container manufactured through the same processing equipment with no change in ingredient material.

4.3.1.2 Packaged lot. A packaged lot is defined as an indefinite number of 208-liter (55-gallon) drums or smaller unit packages of identical size and type, or an indefinite number of 2080-liter (550-gallon) or less returnable containers, such as totes, offered for acceptance and filled with a homogenous mixture of material from a bulk lot.

4.3.2 Sampling. Each bulk or packaged lot of material shall be sampled for verification of product quality and compliance in accordance with ASTM D4057.

4.4 Methods of inspection.

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TABLE I. Testing.

Characteristic	Qualification Test	Conformance Test	Requirement Paragraph	Test Method Paragraph
Material Compatibility	Required		3.2	
Solubility in Fuel	Required		3.3	4.4.1
Compatibility with other Fuel Additives	Required		3.4	4.4.2
Rust Test	Required		3.5	4.4.3.1
Lubricity Test	Required	Required	3.6	4.4.4.1, ASTM D5001
Water Separation	Required	Required	3.7	4.4.5.1, ASTM D3948
Electrical Conductivity	Required		3.7	4.4.2.2, ASTM D2624 or ASTM D4308
Ash Content	Required	Required	3.8	4.4.6, ASTM D482
Pour Point	Required	Required	3.9	4.4.7, ASTM D97 or ASTM D5949 or ASTM D5950 or ASTM D5985
Engine Operation	Required		3.10	4.4.8
Fuel Specification Properties	Required		3.11	4.4.9
Storage Stability	Required		3.12	4.4.10
Fuel Injection Fouling (Bench Procedure)	Required for Category 1 Qualification		3.13	4.4.11, ASTM D6421
Emulsification Tendency	Required for Category 1 Qualification		3.14	4.4.12, Method 550 of FED-STD-791
Accelerated Stability	Required for Category 1 Qualification		3.15	4.4.13, ASTM D2274
Density	Required	Required	3.16	4.4.14, ASTM D1298 or pycnometer or ASTM D4052
Viscosity	Required	Required	3.16	4.4.14, ASTM D445
Flash Point	Required	Required	3.16	4.4.14, ASTM D56 or ASTM D93
Neutralization Number	Required	Required	3.16	4.4.14, ASTM D664
pH	Required		3.16	4.4.14, ASTM D664
Metallic Constituent	Required		3.16	4.4.14
Workmanship	Required		3.17	

4.4.1 Solubility. The maximum allowable concentration of improver shall be mixed with each of the following fuels. The fuel shall contain no other improvers. 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-5 fuel that conforms to MIL-DTL-5624 or JP-8 fuel that conforms to MIL-DTL-83133 and contains the maximum allowable concentration of specified fuel system icing inhibitor.

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- b. JP-4 fuel that conforms to MIL-DTL-5624 and contains the maximum allowable concentration of specified fuel system icing inhibitor.
- c. A motor gasoline that conforms to ASTM D4814 (required only if additive is to be qualified for use in motor gasolines).
- d. A diesel fuel that conforms to ASTM D975 (required only if additive is to be qualified for use in diesel fuel).

4.4.2 Compatibility.

4.4.2.1 Other improvers. Grade JP-8 fuel (MIL-DTL-83133) or grade JP-5 fuel (MIL-DTL-5624) that contains the maximum allowable concentration of the lubricity improver under test and no other lubricity improvers shall be mixed in equal proportions with samples of grade JP-8 fuel (MIL-DTL-83133) or grade JP-5 fuel (MIL-DTL-5624), respectively, which contain the maximum allowable concentration of each improver currently on the Qualified Products List for this specification. The grade JP-8 fuel (MIL-DTL-83133) or grade JP-5 fuel (MIL-DTL-5624) used shall contain the maximum allowable amount of fuel system icing inhibitor that conforms to MIL-DTL-85470. At the end of a 24-hour period, the samples shall be visually inspected for precipitation, cloudiness, or other evidence of non-compatibility.

4.4.2.2 Static dissipater additive. Grade JP-8 fuel (MIL-DTL-83133) filtered through clay as described in appendix X.1 of ASTM D3948 shall be blended with each static dissipater additive approved in MIL-DTL-83133 to provide test fuels which have a conductivity of 400 picosiemens per meter (pS/m) \pm 100 pS/m . After a 24-hour period, to ensure equilibrium fuel conductivity has been established, the lubricity improver additive under test shall be added and mixed. At the end of another 24-hour period, no more than a \pm 40 percent change in the electrical conductivity of the fuel shall have occurred as a result of the test improver. The fuel electrical conductivity shall be measured using either ASTM D2624 or ASTM D4308 test methods. The post-test visual inspection of the sample shall reveal no precipitation, cloudiness, or other evidence of incompatibility. (NOTE: Some loss in fuel conductivity may occur over time when bare glass bottles or bare metal cans are used with fuels which contain static dissipater 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).

4.4.2.3 Thermal stability improver additive. Grade JP-8 fuel (MIL-DTL-83133) filtered through clay as described in appendix X.1 of ASTM D3948 shall be blended with each thermal stability improver additive approved in MIL-DTL-83133 at the specified concentration. The lubricity improver additive under test shall be added at its maximum allowable concentration and mixed. At the end of a 24-hour period, the samples shall be visually inspected for precipitation, cloudiness, or other evidence of non-compatibility.

4.4.3 Relative effective concentration. The relative effective concentration of the improver shall be determined by testing the improver at various concentrations in depolarized *iso*-octane in accordance with 4.4.3.1. The improver shall be tested at concentrations divisible by 3 (i.e.; 6, 9, 12, 15, ... 36 g/m^3). No intermediate concentrations shall be tested. The relative effective concentration shall be defined as the lowest concentration that provides a passing result in accordance with 4.4.3.1.6.

4.4.3.1 Rusting test method.

4.4.3.1.1 Test apparatus. The test apparatus shall conform to the following requirements:

- a. Oil bath that conforms to ASTM D665, with the additional requirement that it must be able to maintain the test sample at a temperature of $38.0^\circ\text{C} \pm 0.5^\circ\text{C}$.
- b. Beaker, beaker cover, stirrer, stirring apparatus, and chuck and motors to hold and rotate specimens while polished shall conform to ASTM D665.
- c. Infrared heat lamp, 250 watts.
- d. Hypodermic syringe, glass, 3-mL, with 15.24-cm (6-inch) stainless steel needles.
- e. Disposable microliter pipettes which consist of calibrated capillary tubes that contain 1, 2, 3, 4, 5, 10, 15, and 20 microliters.

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- f. Column, chromatographic, glass, 40 mm ID x 600 mm with nonmetallic stopcock. A separatory funnel, Squibb, 1-liter, with nonmetallic stopcock may be substituted for the chromatographic column.
- g. Specimen holder, nonmetallic, dimensions as specified in ASTM D665 for type 2 holder.
- h. Specimen, dimensions as specified in ASTM D665, made of grade 1018 steel in accordance with ASTM A108. 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) or less.

4.4.3.1.2 Test materials. Test materials shall conform to the following requirements:

- a. Silica gel, 28-200 mesh, heated to 107 °C (225 °F) for 2 hours and cooled in a desiccator before use.
- b. Test solvent, *iso*-octane that conforms to ASTM D471, 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; the silica gel is retained by a glass wool plug. (NOTE: Do not use stopcock grease.) One gallon (3.79 liters) of *iso*-octane is passed through the silica-gel bed by gravity, the first 50 mL discarded, and the remainder collected 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: Prepare 3 stock solutions using ACS reagent-grade chemicals in distilled water. Each one of the solutions shall contain one of these chemicals: 16.4 g/L NaHCO₃, 13.2 g/L CaCl₂ · 2H₂O, and 8.2 g/L MgSO₄ · 7H₂O. Pipette 10 mL of the NaHCO₃ stock solution into 800 mL of distilled water in a 1 liter volumetric flask, and shake vigorously. While swirling the contents of the flask, pipette 10 mL of the CaCl₂ stock solution and then 10 mL of the MgSO₄ stock solution into the flask, add distilled water to bring the volume to 1 liter, and mix thoroughly. The final blend shall be clear and free of precipitation.
- d. Isopropanol, ACS reagent grade.
- e. Glassware cleaning solution.
- f. Lintless paper tissues such as Kimtech Science Kimwipes Delicate Task Wipers.
- g. Abrasive cloth, 150-, 240-, and 400-grit metalworking aluminum oxide abrasive cloth, closed coat on jeans backing. The abrasive cloth is available in rolls of 1 inch (2.54-cm) tape, the most convenient form for use in this test.
- h. Disposable vinyl gloves.

4.4.3.1.3 Specimen preparation. 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. 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 irregularities, but grinding shall be completed with new cloth. Stop the motor and scratch the static specimen longitudinally with one pass of new 150-grit cloth. Use light pressure so visible scratches appear.
- b. Grind with 240-grit cloth, remove all marks from the 150-grit cloth, and finish with new 240-grit cloth. Stop the motor and scratch the static specimen longitudinally with one pass of new 240-grit cloth; use light pressure so visible scratches appear.

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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. Move 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 marks from the previous 240-grit operation have been removed and the surface presents a uniform appearance, free of longitudinal or spiral scratches, where all polishing marks appear 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 that contains silica gel or other noncorrosive desiccant until ready for use. The storage period in the *iso*-octane shall not exceed 7 days.

4.4.3.1.4 Preparation of test blend. The test blend shall be prepared in the test beaker, not more than 2 hours before 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 naphtha 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 air- or oven-dry. 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 improver in the test beaker with direct addition of the improver. 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 pipette or pipettes to add integral numbers of μL of the improver to the beaker to increase measurement accuracy. Add the calculated volume of depolarized *iso*-octane to the test beaker. Fill the appropriate microliter pipette or pipettes with improver, wipe off excess, and force the improver into the *iso*-octane. Allow the pipette to fill with *iso*-octane by capillary attraction and force this rinse into the test beaker. Repeat the rinse 4 times. Calculate the amount of *iso*-octane and improver to be added to the test beaker using the instructions given in 4.4.3.1.4.c.

c. Calculate the volume of *iso*-octane required for each concentration desired using the following equation, where density (ρ) is in g/mL at 15°C:

$$\text{mL of } iso - octane = \frac{(\rho \text{ of improver}) \times (\mu\text{L of improver}) \times (1000)}{\text{desired improver concentration, g/m}^3}$$

For example, assume the improver 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 μL of improver:

$$\text{mL of } iso - octane = \frac{(0.95) \times (2) \times (1000)}{6} = 316.7 \text{ mL}$$

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For improvers less dense 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 improver required for 300 mL of *iso*-octane.
- (2) Increase the volume of improver to the next integral microliter and add to 300 mL of *iso*-octane in the test beaker. Mix well.
- (3) Calculate the amount of improver/*iso*-octane blend to be removed from the test beaker to leave the desired amount of improver.
- (4) Remove the calculated amount of improver/*iso*-octane blend and replace with an equivalent volume of depolarized *iso*-octane. Mix well. This approach is illustrated for the preceding example:

- (a) Using this equation, calculate the desired volume of improver for 300 mL of *iso*-octane for an improver with a density of 0.85 and for a desired concentration of 6 g/m³.

$$300 \text{ mL of } iso - octane = \frac{(0.85)x (Z \mu\text{L of improver})x (1000)}{6}$$

$$Z = 2.12 \mu\text{L of improver}$$

- (b) Add the next integral volume of improver (i.e., 3 μL) to 300 mL of *iso*-octane and mix well. This gives an improver concentration of 3 μL improver/300 mL *iso*-octane or 1 μL /100 mL.

- (c) The desired amount of improver is 2.12 μL . Thus, $(2.12) \times (100) = 212$ mL of improver/*iso*-octane blend is needed.

- (d) Remove 88 mL of the improver/*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 improver (i.e., 2.12 μL) in 300 mL of *iso*-octane.

d. Place the beaker in the oil bath which has been regulated previously to maintain a sample temperature of $38.0^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$. 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 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 5 minutes. Turn off the stirrer. Use a clean pipette or syringe to withdraw enough test blend to leave exactly 300 mL in the beaker. Allow the test blend to come to $38.0^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$. Replace the thermometer with a cork or plastic plug.

4.4.3.1.5 Test procedure. After preparing a test specimen as described in 4.4.3.1.3 and a test blend as described in 4.4.3.1.4, 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; handle the specimen 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 gentle 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 fresh abrasive is exposed to the specimen. Inspect the specimen to ensure the surface presents a uniform appearance, free of longitudinal or spiral scratches, where all polishing marks appear to be circumferential. Additional polishing is required if the specimen appearance is other than described. After polishing is completed, remove the specimen 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 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, place the specimen and holder 6 inches

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from a 250-watt infrared heat lamp and rotate for 1 minute before the rinsing operation. 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. Carefully add 30 mL of test water to the bottom of the test 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 5 hours; hold the bath temperature at the same setting so the test samples will be maintained at $38.0^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$.
- e. At the end of 5 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 646 lux or 60 foot-candles. Scan the surface very carefully to detect any small pits. Record observations of visible rust, pits, stains, or deposits.

4.4.3.1.6 Interpretation of test results. A test shall be reported as failed if the center 48 mm section of the specimen shows 6 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 to a given improver at a given concentration, two tests shall be performed. The improver 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 improver shall be reported as failing at the given concentration. If both of the additional tests give passing ratings, the improver shall be reported as passing the given concentration.

4.4.4 Minimum effective concentration.

4.4.4.1 Lubricity test. The improver shall be tested in accordance with the BOCLE as described in ASTM D5001, using ISOPAR M as the solvent and a 1000 gram load on the ball.

4.4.5 Maximum allowable concentration.

4.4.5.1 Micro-Separometer Rating. The improver shall be blended into the reference fluid base as described in ASTM D3948 and tested in accordance with ASTM D3948. For any given concentration of improver, the average of three test results shall be used to determine the conformance to the requirements of 3.7 or 4.3.

4.4.6 Ash content determinations. The ash content of the improver shall be determined in accordance with ASTM D482, using a platinum or porcelain crucible.

4.4.7 Pour point determination. Pour point shall be determined in accordance with ASTM D97, ASTM D5949, ASTM D5950, or ASTM D5985.

4.4.8 Aircraft engine test. The engine shall be operated for 100 hours in accordance with the engine operating requirements of MIL-PRF-7808. The fuel, grade JP-8 fuel that conforms to MIL-DTL-83133 or grade JP-5 fuel that conforms to MIL-DTL-5624, shall contain twice the maximum allowable concentration of the improver. 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 exchangers, 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 6.6).

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

4.4.10 Storage stability test. Two 1 liter or 1 quart amber glass bottles shall each be filled with 850 mL of the improver and shall be tightly capped by means of a screw cap that has a conical, 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 examination and testing; the other sample 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.

4.4.11 Fuel injection fouling test. The improver, at its maximum concentration, shall be blended into an ASTM D4814 motor gasoline. The test fuel shall then be tested for the formation of deposits in accordance with ASTM D6421. The ASTM D4814 gasoline without improver shall also be tested in accordance with ASTM D6421. The resultant difference between the gasoline with improver versus without improver shall not exceed one percent of the fuel injector fouling.

4.4.12 Emulsification tendency test. The improver, at maximum allowable concentration, shall be blended into an ASTM D4814 motor gasoline and an A-A-52557 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 3 are evidence of unsatisfactory emulsification tendencies and shall not be allowed. The ASTM D4814 motor gasoline and the A-A-52557 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 improver.

4.4.13 Accelerated stability test. The test improver, at its maximum allowable concentration, shall be blended into a A-A-52557 diesel fuel that contains no additives. Each test fuel shall be tested for the formation of total insolubles in accordance with ASTM D2274. The total insolubles shall not exceed 1.5 mg/100 mL. The diesel fuel without the test improver shall also be tested in accordance with ASTM D2274 concurrently to define the level of insolubles occurring without the presence of the improver.

4.4.14 Identification tests. Identification tests shall be conducted in accordance with the following methods:

Density at 15°C	ASTM D1298 or pycnometer or ASTM D4052
Viscosity at 40°C	ASTM D445
Flash Point	ASTM D56 or ASTM D93
Neutralization Number	ASTM D664, Total Acid Number
pH	On a 0.10 - 0.11 gram sample in 125 mL of ASTM D664 titration solvent. Use the same apparatus as used in ASTM D664 to perform pH measurement.

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Metallic Constituent

Emission spectrograph, not applicable for materials with ash contents of 0.05 percent mass or lower.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of materiel is to be performed by DoD or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activities within the Military Service or Defense Agency, or within the Military Service's system commands. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

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 improvers covered by this specification are used, when specifically authorized, in military jet engine fuels to improve the lubricating qualities of military jet fuels. Certain of the improvers are also used in automotive gasoline, diesel fuel, and related petroleum products for military applications. These additives were previously used as pipeline corrosion inhibitors.

6.2 Acquisition requirements. Acquisition documents must specify the following:

- a. Title, number, and date of this specification.
- b. Quantity required.
- c. Packaging requirements (see 5.1).

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

6.3 Material Safety Data Sheets. Contracting officers will identify those activities that require copies of completed Material Safety Data Sheets (MSDS) prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.

6.4 Toxicity. Questions pertaining to toxicity should be directed to the medical service department of the procuring activity. The U.S. Army Center for Health Promotion and Preventive Medicine (USACHPPM) will act as advisor to Army procuring activities. The Department of the Army Pamphlet (DA) 70-3 requires a Toxicity Clearance (approval) prior to use of a new material or chemical. Army toxicity questions and/or a Toxicity Clearance request should be addressed to: Commander, US Army Center for Promotion and Preventative Medicine (MCHB-TS-T), 5158 Blackhawk Road, Aberdeen Proving Ground, MD 21010-5403.

6.5 Improver for addition to fuels. When a fuel contractor or the Government purchases the lubricity improver for addition to fuels to be used by the Government, the manufacturer of the improver must certify to the purchaser that the product is a lubricity improver that has been qualified under this specification. In addition, a test report that shows compliance of the product with the requirements of 4.3 must be supplied to the purchaser. Additional data may be required by the Purchasing Activity to establish compliance with this specification.

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6.6 Qualification. With respect to products that require qualification, awards will be made only for products that are, at the time of award of contract, qualified for inclusion in Qualified Products List (QPL) No. 25017 whether or not such products have actually been so listed by that date. The attention of the contractors is called to these requirements, and manufacturers are urged to arrange to have the products 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 covered by this specification. Information pertaining to qualification of products may be obtained from AFPA/PTPT, 2430 C Street, Bldg 70, Area B, Wright-Patterson AFB, OH 45433-7632. An online listing of products qualified to this specification may be found in the Qualified Products Database (QPD) at <https://assist.daps.dla.mil>.

6.6.1 Qualification test report. The Qualifying Activity will request a certified test report from the independent laboratory. The test report will contain laboratory data which detail 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 will also contain laboratory data on any of the special tests conducted to qualify the lubricity improver for use in motor gasolines and diesel fuels (i.e.; 3.13, 3.14, and 3.15). In addition, complete formulation data will be supplied to the Qualifying Activity. This data will 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 will be required to furnish toxicological data and formulations necessary to evaluate the safety of the material for the proposed use.

6.7 Conversion of metric units. Units of measure have been converted to the International System of Units (Metric) in accordance with ASTM E380. If test results are obtained in units other than Metric or there is a requirement to report dual units, ASTM E380 or ASTM D1250 Volume XI/XII should be used to convert the units.

6.8 Subject term (key word) listing.

- Diesel
- Fuel additive
- Fuel lubricity additive
- Gasoline
- Isopropanol
- Petroleum
- Turbine fuel

6.9 International standardization agreements. Certain provisions of this specification are the subjects of International Standardization Agreements STANAG 3390 and STANAG 1135. 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.

6.10 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

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CONCLUDING MATERIAL

Custodians:

Army – AT
Navy – AS
Air Force – 68

Preparing activity:

Air Force – 68
(Project 6850-2010-004)

Review activities:

Army – AV, MD1
Navy – SH
Air Force – 11
DESC – PS
DLA – GS

Note: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information using the ASSIST Online database at <https://assist.daps.dla.mil>.