

MIL-S-53021
16 February 1983
SUPERSEDING
(See section 6)

MILITARY SPECIFICATION

FOR

STABILIZER ADDITIVE, DIESEL FUEL

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

1. SCOPE

1.1 Scope. This military specification covers a stabilizer additive for use in diesel fuels meeting the requirements of VV-F-800 which are intended for intermediate or long-term storage. Typical applications for this additive are to pre-positioned fuel and equipment, vehicles subject to storage or infrequent use, and to bulk fuel procured for both intermediate and long-term storage (see 6.1).

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications and standards. Unless otherwise specified (see 6.2), the following specifications and standards of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation, form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

TT-S-735	- Standard Test Fluids; Hydrocarbon.
VV-F-800	- Fuel Oil, Diesel.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: US Army Mobility Equipment Research and Development Command, ATTN: DRDME-DS, Fort Belvoir, VA 22060 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

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STANDARDS

FEDERAL

- FED-STD-313 - Material Safety Data Sheets, Preparation and Submission of.
- FED-STD-791 - Lubricants, Liquid Fuels and Related Products; Methods of Testing.

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- MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.
- MIL-STD-290 - Packaging, Packing, and Marking of Petroleum and Related Products.

(Copies of specifications and standards required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. The issues of the documents which are indicated as DoD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS (NACE)

NACE TM-01-72 - Antirust Properties of Petroleum Product Pipeline Cargoes.

(The test method listed above may be ordered from the National Association of Corrosion Engineers, P. O. Box 218340, Houston, TX 77218.)

SOCIETY OF INDUSTRIAL MICROBIOLOGISTS (SIM)

SIM Special Publication No. 2 - Proposed Procedure for the Screening of Microbial Inhibitors in Hydrocarbon/Water Systems.

(This publication is currently out of print. Reproduced copies are available from the U.S. Army Mobility Equipment Research and Development Command, ATTN: DRDME-GL, Ft. Belvoir, VA 22060.)

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

- D93 - Flash Point by Pensky-Martens Closed Tester.
- D270 - Sampling Petroleum and Petroleum Products.
- D445 - Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity).
- D482 - Ash from Petroleum Products.
- D664 - Neutralization Number by Potentiometric Titration.
- D974 - Neutralization Number by Color - Indicator Titration.

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- D1298 - Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method.
D2274 - Oxidation Stability of Distillate Fuel Oil (Accelerated Method).

(The test methods listed above are included in Parts 23, 24, 25 and in other Parts of the Annual Book of ASTM Standards and are available individually. Applications for copies of all ASTM publications should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall take precedence.

3. REQUIREMENTS

3.1 Qualification. The stabilizer additives furnished under this specification are for use in diesel and distillate fuels. The stabilizer additive shall be a product that has passed the applicable qualification requirements of 3.1.1 or has been listed on or approved for listing on the applicable qualified products list. Tentative approval for listing on the qualified products list shall be granted pending successful completion of all requirements of 3.1.1 except for an ongoing 12 month storage stability test (3.13). Full approval shall be granted upon successful completion of the storage stability test. Failure to pass the storage stability test shall be cause for withdrawal of approval.

3.1.1 Qualification requirements. All approved stabilizer additives shall meet the requirements of 3.2 through 3.22 to be qualified for use in fuels conforming to VV-F-800.

3.2 Identification qualification data. The following properties of the finished stabilizer additive shall be determined but not limited during qualification: density at 15°C, viscosity at 0° C and 40° C, neutralization number, pHr, and type of metallic constituent, if present (see 4.6.1). The permissible production variation of individual properties will be established at the time of qualification by mutual agreement between the manufacturer and the qualifying activity. Individual batches of stabilizer additive subsequently subjected to qualification conformance inspections shall conform to the established range of properties. The ranges shall not adversely affect any of the stabilizer additive performance characteristics.

3.3 Materials. The stabilizer additive supplied under this specification shall consist of petroleum-soluble compounds which perform the following functions:

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- a. Antioxidant stabilizer.
- b. Metal deactivator.
- c. Dispersant.
- d. Corrosion inhibitor.
- e. Biocide.

The composition of the finished stabilizer additive is not limited, but is subject to review by the qualifying activity in order to assure service compatibility with previously qualified products.

3.3.1 Recommended effective concentration. Each contractor of stabilizer additive shall designate a recommended effective concentration at which the product should be used in diesel fuel. The recommended effective concentration shall be expressed in both grams per cubic meter (g/m^3) and in quantity per 1000 gallons of diesel fuel. Each container of stabilizer additive shall be labeled with the recommended effective concentration in both units.

3.3.2 Metal deactivator. The stabilizer additive shall contain sufficient metal deactivator so that when the stabilizer additive is mixed with diesel fuel at the recommended effective concentration (see 3.3.1), the concentration of metal deactivator active ingredient shall be not less than 2.5 g/m^3 and not more than 5.7 g/m^3 . The metal deactivator shall be one of the following two types:

- a. N, N' - disalicylidene - 1, 2 - propanediamine.
- b. N, N' - disalicylidene - 1, 2 - cyclohexanediamine.

The use of metal deactivators other than the types and quantities specified shall require prior approval by the qualifying agency. The contractor shall report the type and amount of metal deactivator in the finished stabilizer additive.

3.3.3 Toxic products and formulations. The material 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. A Material Safety Data Sheet in accordance with FED-STD-313 shall be submitted.

3.4 Test Fuel. The test fuel used to evaluate the stabilizer additive shall meet the requirements of Reference 1-H Diesel Fuel, a widely-used reference diesel fuel for qualification acceptance testing of automotive engine oils. The specification for the Reference 1-H fuel is given in FED-STD-791, Method 346.

3.5 Reference formulation. The reference formulation shall consist of the test fuel described in 3.4, prefiltered through a 0.8 micron filter, to which both of the following products shall be added to give the concentrations indicated:

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- a. duPont Fuel Oil additive FOA-15, ^{1/} $71.3 \pm 0.5 \text{ g/m}^3$.
- b. Biobor JF (NSN 6840-01-041-0098), ^{2/} $227 \pm 1 \text{ g/m}^3$.

3.6 Test formulation. The test formulation shall consist of the test fuel described in 3.4, prefiltered through a 0.8 micron filter, after which the contractor's stabilizer additive shall be added to give the recommended effective concentration (see 3.3.1).

3.7 Solubility. The stabilizer additive, at three times the recommended effective concentration, shall be readily and completely soluble in test fuel conforming to 3.4. There shall be no precipitation, cloudiness, or other insolubility when tested as specified in 4.6.2.

3.8 Compatibility. The stabilizer additive shall be compatible with all stabilizer additives currently qualified under this specification. There shall be no precipitation, cloudiness, or other evidence of incompatibility when tested as specified in 4.6.3.

3.9 Flash point. The flash point of the stabilizer additive shall not be less than 40° C when tested in accordance with 4.6.4.

3.10 Ash content. The ash content of the stabilizer additive shall not exceed 0.10 weight percent when determined in accordance with 4.6.5.

3.11 Minimum handling temperature. The minimum handling temperature of the stabilizer additive shall be reported in accordance with 4.6.6.

3.12 Diesel and gas turbine engine operation. The Government reserves the right to perform engine tests to determine the operational acceptability of stabilizer additives in diesel engines and in gas turbine engines authorized to operate on diesel fuel. Engine tests, if conducted, shall be in accordance with 4.6.7. Engine operation shall not be adversely affected and the post-test condition of the engine shall indicate no excessive deposits, wear, corrosion, or other deleterious effects which are attributed to the stabilizer additive.

3.13 Stabilizer additive storage stability. After storage for 12 months in accordance with 4.6.8, the stabilizer additive shall show no precipitation, layering, or other evidence of gross separation or degradation. Stabilizer additive representing the top half of the stored sample shall meet all requirements of this specification.

3.14 Filterability. The test formulation (see 3.6) shall be tested for filterability in accordance with 4.6.9. The filtration ratio shall not exceed 1.0.

^{1/} FOA-15 is available from E.I. du Pont de Nemours and Co., Petroleum Laboratory, Wilmington, DE 19898.

^{2/} Biobor JF is available from U.S. Borax and Chemical Corporation, 3075 Wilshire Blvd., Los Angeles, California 90010.

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3.15 Oxidation stability (accelerated). The test fuel (see 3.4), reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with 4.6.10. The results shall be reported in the certified test report (see 4.3.1).

3.16 High temperature stability. The test fuel (see 3.4), reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with 4.6.11. The results shall be reported in the certified test report (see 4.3.1).

3.17 Fuel storage stability. The test fuel (see 3.4), reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with 4.6.12. The results shall be reported in the certified test report (see 4.3.1).

3.18 Partition coefficient. The contractor shall report the value of the partition coefficient for the biocide component of the stabilizer additive as determined in accordance with 4.6.13.

3.19 Biocidal activity. The stabilizer additive shall be tested in accordance with 4.6.14. The test report shall contain the following data on each sample: test organisms, type of fuel, concentration of stabilizer additive, time since inoculation, and visual changes observed, including presence of granular particles, films on or between layers, mycelial mats, emulsions, flakes, color changes, turbidity, slime, and sludge. The lowest concentration of stabilizer additive which shows 100 percent kill or inhibition of all three test organisms shall be recorded as the effective biocidal concentration. This concentration, when corrected for the partition coefficient (see 4.6.13) and the fuel:water ratio, shall be equal to or lower than the recommended effective concentration (see 3.3.1).

3.20 Antirust properties. The stabilizer additive shall be tested for antirust properties in accordance with 4.6.15. The rating of the test specimen shall be A to pass the test.

3.21 Workmanship. The finished stabilizer additive in bulk or container shall be uniform in appearance and visually free from grit, undissolved water, insoluble matter, or other adulteration.

3.22 Components. The stabilizer additive supplied in accordance with this specification shall consist of no more than two containers per additive package. The two-container packages shall be permitted only when the two components are not mutually soluble in concentrated (undiluted) form.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, 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

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inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Classification of inspections. The inspection and testing of the stabilizer additive shall be classified as follows:

- a. Qualification inspection (see 4.3).
- b. Quality conformance inspection (see 4.4).
- c. Inspection of packaging (see 4.7).

4.3 Qualification inspection. Qualification inspection and testing shall consist of tests specified in 4.6.

4.3.1 Test report. A certified test report shall be forwarded to the activity responsible for qualification (see 6.4) before the qualification sample is supplied. The test report shall contain laboratory data showing the results required by 3.2, 3.3.1, 3.3.2, 3.7, 3.8, 3.9, 3.10, 3.11, 3.13, 3.14, 3.15, 3.16, 3.17, 3.18, 3.19, 3.20, and 3.21. In addition, stabilizer additive formulation data shall be supplied to the qualifying activity. This data shall include one of the following:

- a. Complete chemical name and percentage of each ingredient.
- b. Complete percentage composition of the stabilizer additive by chemical element, plus a general generic description of each ingredient showing type of functional groups.

The contractor shall furnish toxicological data and formulations required to evaluate the safety of the material for the proposed use.

4.3.1.1 Qualification sampling. Unless otherwise specified by the activity responsible for qualification, an initial 1-liter sample of finished stabilizer additive shall be submitted for evaluation in all of the tests except the storage stability and engine operation tests. If the product passes these tests, an additional sample of finished stabilizer additive will be requested for the storage stability, and engine tests. Samples shall be identified and packaged as required and forwarded to the laboratory responsible for testing as designated in the letter of authorization from the activity responsible for qualification (see 6.4).

4.3.1.2 Requalification. Requalification will be required in the event of any change in composition or formulation, source of the stabilizer additive, its ingredients, or manufacturing sites; or if periodic verification determines noncompliance to requirements.

4.3.1.3 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 five years. Unless otherwise specified by the activity responsible for the qualified products list, verification of qualification may be made by certification.

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4.4 Quality conformance inspection. Quality conformance inspection of a bulk lot of stabilizer additive shall consist of tests for conformance to requirements for solubility (see 3.7) flash point (see 3.9), ash (see 3.10), and property limits shown on the Qualified Products List.

4.4.1 Inspection lot.

4.4.1.1 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 as a single isolated batch, or manufactured by a single plant run (not exceeding 5 days) through the same processing equipment with no change in ingredient material.

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

4.4.2 Sampling. Each bulk or package lot of material shall be sampled for verification of product quality and compliance in accordance with ASTM D270.

4.5 Inspection. Inspection shall be in accordance with Method 9601 of FED-STD-791.

4.6 Test methods.

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

Density at 15° C	ASTM D1298
Viscosity at 0° and 40° C	ASTM D445
Neutralization number	ASTM D664
pHr	Add 0.1 to 0.11 gram of stabilizer additive to 125 mL of D974 titration solvent. Standardize apparatus and read pHr in accordance with Appendix X2 of D974.
Metallic constituents	Emission or atomic absorption spectrograph.

4.6.2 Solubility test. Filter 1.0 liter of test fuel (see 3.4) through a 0.8 micron filter into a clean, round, flat-bottomed, transparent bottle or beaker. Add an amount of stabilizer additive sufficient to give not less than three times the recommended effective concentration (see 3.3.1). Stir or swirl the test fuel for a period not to exceed three minutes to dissolve the stabilizer additive. Check for insoluble materials by swirling the sample so a vortex is formed. All sediment and insoluble matter will accumulate on the bottom of the bottle directly beneath the vortex. The presence of visible sediment, water, haze, cloudiness, or other insolubility shall constitute failure of the test. If no insoluble matter is present, cover the sample and check again for insoluble matter after the sample has stood for 24 hours. The presence of visible sediment, water, haze, cloudiness, or other insolubility shall constitute failure of the test.

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4.6.3 Compatibility. The test formulation (see 3.6) shall be mixed in equal proportions with samples of test fuel (see 3.4) containing the recommended effective concentrations of each stabilizer additive previously approved under this specification. After the mixtures have stood for 24 hours or more, they shall be visually inspected for the presence of haze, cloudiness, precipitate, or other evidence of noncompatibility.

4.6.4 Flash point. The flash point of the stabilizer additive shall be determined in accordance with ASTM D93.

4.6.5 Ash content. The ash content of the stabilizer additive shall be determined in accordance with ASTM D482.

4.6.6 Minimum handling temperature. The minimum handling temperature of the stabilizer additive shall be recorded as the temperature below which the stabilizer additive crystallizes or becomes too viscous for pouring, pumping, mixing, and blending operations as normally practiced in the petroleum industry.

4.6.7 Diesel and gas turbine engine operational test. The extent of engine testing shall be determined by the activity responsible for qualification based on the chemical composition of the stabilizer additive, evaluation of previous engine test data, if any, available from Government or commercial sources, and consideration of the number and type of military engines operating on diesel fuel. Fuel for engine testing shall conform to 3.4 or VV-F-800 and shall contain stabilizer additive at twice the recommended effective concentration. The engine test cycle shall be not less than 100 hours and shall conform to an established test method for which baseline data are available on the engine and lubricating oil used in the test. After completion of the engine test, the engine shall be disassembled and inspected for evidence of excessive wear, deposits, corrosion, or other deleterious effects attributed to the stabilizer additive.

4.6.8 Stabilizer additive storage stability test. Two 1-liter amber glass bottles shall each be filled with 850 mL of the stabilizer additive and shall be tightly capped by means of a screw cap having a conical polyethylene liner. Each bottle shall be wrapped in a minimum amount of opaque packing material sufficient for protection against physical 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. The crated samples shall be kept off the ground and stored in an upright position under a canopy, open shed roof, or similar ventilated shelter for protection from direct sunlight and precipitation. The crated samples shall remain undisturbed for the specified period. One of the samples shall be stored for exactly 12 months and then removed for examination and testing; the other samples shall be stored for 12 months or less and may be removed for examination and testing 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

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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 3.1.1. The bottom-half sample, in the original storage bottle, shall be retained for examination and possible additional testing to detect changes caused by storage.

4.6.9 Filterability test. The test formulation (see 3.6) shall be tested for filterability in accordance with Appendix, Test Method No. 1.

4.6.10 Oxidation stability (accelerated). The test fuel (see 3.4), the reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with ASTM D2274. In addition to the standard 16 hour test, each sample shall be tested for 32 hours and 48 hours.

4.6.11 High temperature stability test. The test fuel (see 3.4), the reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with Appendix Test Method No. 2 for 1.5, 3.0, and 4.5 hour aging periods.

4.6.12 Fuel storage stability test. Duplicate samples of the test fuel (see 3.4), the reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with Appendix, Test Method No. 3 for 8 and 16 week aging periods.

4.6.13 Partition coefficient. Equal volumes of distilled water and test formulation (see 3.6) shall be shaken vigorously in a separatory funnel and allowed to equilibrate for not less than 24 hours at $25 \pm 1^\circ \text{C}$. By means of a suitable analytical method, the concentration of the biocide component of the stabilizer additive shall be determined in the water layer and the fuel layer. The partition coefficient shall be calculated by means of the equation:

$$P = \frac{[B]_w}{[B]_f}$$

where P = partition coefficient.

$[B]_w$ = concentration of biocide in the water layer.

$[B]_f$ = concentration of biocide in the fuel layer.

Report the value of the partition coefficient and the analytical method used to determine it.

4.6.14 Biocidal activity test. The stabilizer additive shall be tested for biocidal activity in accordance with Part I of SIM Special Publication No. 2. The test inocula shall include the following strains identified by American Type Culture Collection number: *Cladosporium resinae* 20495, *Pseudomonas aeruginosa* 33988, and *Candida tropicalis* 48138. Organisms are available from

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the American Type Culture Collection, 12301 Parklawn Drive, Rockville, MD 20852, and may require an adaptation period of up to 30 days to demonstrate viable growth in the hydrocarbon/nutrient test medium. The concentration range of stabilizer additive to be used in the tests shall be calculated taking into account the recommended effective concentration (see 3.3.1), the partition coefficient (see 3.18), and an assumed 500:1 ratio of fuel to water in a typical fuel tank or storage tank. A minimum of three concentrations of stabilizer additive shall be tested, using a hydrocarbon fuel known to be susceptible to microbial growth. Each flask shall be examined at 30, 60, and 90 days after inoculation for evidence of growth or physical changes resulting from microbial growth.

4.6.15 Test for antirust properties. The test for antirust properties shall be conducted in accordance with NACE TM-01-72. The fuel used in this test shall be isooctane conforming to 4.6.15.1.

4.6.15.1 Test fuel for antirust properties. Isooctane conforming to TT-S-735, shall be 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). Approximately 3.8 liters of isooctane are 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 isooctane should be used within 1 week after treatment. The stabilizer additive to be tested shall be blended with the depolarized isooctane at the recommended effective concentration (see 3.3.1) to form the test fuel for 4.6.15.

4.7 Inspection of packaging.

4.7.1 Quality conformance inspection of pack.

4.7.1.1 Unit of product. For the purpose of inspection, a completed pack prepared for shipment shall be considered a unit of product.

4.7.1.2 Examination. Each container shall be examined to see how full the container is. Each container that is not full will be considered a defect. Each shipping container shall be examined to determine if the shipping container will transport the small containers from the contractor to destination without damage or leakage. Sampling shall be in accordance with MIL-STD-105, inspection level S-2. AQL shall be 4.0 percent defective for all defects.

5. PACKAGING

5.1 Packing and marking. The stabilizer additive contained in the size and type container specified (see 6.2) shall be packed and marked in accordance with MIL-STD-290. The degree of packing shall be as specified (see 6.2).

6. NOTES

6.1 Intended use. The stabilizer additive is intended to be added into diesel fuel to retard or prevent the formation of fuel deterioration products (i.e., gums, sludge, particulates) resulting from auto-oxidation processes, to

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reduce the potential for microbiological growth, and to provide for corrosion protection of fuel-wetted surfaces. Primarily, this product is for the treatment of fuel in (1) depot facilities where vehicles/equipment are in re-build or storage, (2) pre-positioned materiel at locations involving storage of equipment partially or fully fueled, and (3) fuel stocks intended for intermediate or long-term storage.

6.2 Ordering data.

- a. Title, number and date of this specification.
- b. Date of issue of DoDISS applicable to this contract and exceptions thereto (see 2.1.1).
- c. Size and type of container required (see 5.1).
- d. Degree of packing required (see 5.1).
- e. Quantity of stabilizer additive required. The unit of purchase is one US gallon (3.785 liters) at 60° F (15.6° C).

6.3 Stabilizer additive for addition to fuels. When a fuel contractor or the government purchases the stabilizer additive for addition to fuels to be used by the Government, the manufacturer of the stabilizer additive must certify to the purchaser that the product 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.

6.4 Qualification. With respect to products requiring qualification, awards will be made only for product which are at the time set for opening of bids, qualified for inclusion in the applicable qualified products list, 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 that they propose to offer the Federal Government tested for qualification in order that they may be eligible to be awarded contracts or orders for the products covered by this specification. The activity responsible for the qualified products list is the US Army Mobility Equipment Research and Development Command, ATTN: DRDME-GL, Ft. Belvoir, VA 22060.

6.5 Supersession. This specification supersedes US Army Mobility Equipment Research and Development Command Purchase Description PD ME-103, 19 December 1980.

Custodians:

Army - ME
Navy - YD
Air Force - 68

Preparing activity:

Army - ME

Project 6850-0685

Review activities:

Army - AT, GL, SM
DLA - PS, GS

User activity:

Navy - SH

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APPENDIX

STABILIZER ADDITIVE, DIESEL FUEL

10. SCOPE

10.1 Scope. The test methods contained within this appendix are to determine whether the stabilizer additive procured under this specification conform to the requirements set forth.

20. APPLICABLE DOCUMENTS

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)^{1/}

- D381 - Existent Gum in Fuels by Jet Evaporation.
- D1500 - ASTM Color of Petroleum Products (ASTM Color Scale).
- D2276 - Particulate Contaminant in Aviation Turbine Fuels.

(Application for copies of all ASTM publications should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

^{1/} The documents listed herein are applicable to the Appendix only and are not listed in Section 2 of the specification.

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APPENDIX

TEST METHOD NO. 1

DETERMINING THE FILTERABILITY OF DIESEL FUEL

1. SCOPE

1.1 This method is for determining the filterability characteristics of diesel fuel. It is intended to provide an indication as to whether the fuel will exhibit any potential filter plugging problem.

2. SUMMARY OF METHOD

2.1 In this method, 500 mL samples of clean and test fuel are filtered through a 0.8 micron filter disc. Fuel filterability is evaluated by the ratio of the filtering time for the test fuel to the filtering time for the clean fuel. As both fuels originate from the same source, clean refers to prefiltered whereas test refers to unfiltered prior to testing.

3. APPARATUS

3.1 The following equipment or its equivalent is used:

(SUGGESTED SUPPLIER)

a. 01-092-25 Pressure/Vacuum Pump	Fisher Scientific.
b. 1000-mL Filter Flask	Fisher Scientific.
c. 500-mL Graduated Cylinder	Fisher Scientific.
d. Stop Watch	Fisher Scientific.
e. XX10-047-00 Pyrex Filter Folder	Millipore Corporation.
f. 0.8 Micron Filter Disc#AAWP-04700	Millipore Corporation.

4. PROCEDURE

4.1 Store the fuel samples and glassware at $25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 4 hours or until temperature equilibrium is attained.

4.2 Pour approximately 510 mL of fuel to be tested in a 500-mL graduated cylinder. Maintain the sample at $25^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

4.3 Install a 0.8 micron filter disc in the holder. Turn on vacuum (allow to run 10 or 15 seconds to assure full vacuum) then pour the fuel into the filter at such a rate that the fuel filter bowl is full and kept full until all fuel has been poured from the graduated cylinder.

4.4 When all the fuel has been pulled through the filter and the filter sucked dry, remove the disc. Pour exactly 500 mL of filtrate into the 500-mL graduated cylinder.

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4.5 Install a clean 0.8 micron disc in the filter holder. Then pour 500 mL of filtered fuel into the filter exactly as before, but start the stop watch when the fuel hits the filter disc and stop it when all the fuel has been pulled through. (Starting and stopping the watch at the exact time is very important). The sample is to be maintained within $\pm 1^{\circ}$ C of the test temperature during this step.

4.6 Do not remove the filter disc, but pour out the fuel in the flask. Then filter and time 500 mL of test fuel through the same filter disc that was used in step 5. The sample is to be maintained within $\pm 1^{\circ}$ C of the test temperature for 4 hours prior to completing this step.

5. CALCULATION

5.1 Divide time obtained in 4.6 by time obtained in 4.5, and round to the nearest 0.1. If the result is 1.0 the fuel was perfectly filterable. Values greater than 1.0 denote a potential problem which is due to additive-fuel incompatibilities. The higher the ratio value, the greater the problem in filterability.

6. PRECAUTIONARY STATEMENT

6.1 It is very important to make all filtrations of any one sample at the same temperature. It is also important to keep the filter base clean. Clean it with petroleum solvents only. This should be done by pouring solvent into the outlet of the filter base and then forcing it through the base with air; then suck all the solvent from the base before using it to filter fuel.

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TEST METHOD NO. 2

HIGH TEMPERATURE (150° C) STABILITY

OF DISTILLATE FUELS

1. SCOPE

1.1 This method determines relative stability of distillate fuels under high temperature aging conditions (150° C) with limited air exposure.

2. SUMMARY OF METHOD

2.1 Measured volumes of distillate fuel are aged for 1.5, 3.0, and 4.5 hours at 150° C in an open tube with air exposure. After aging and cooling, fuel is filtered and the amount of insoluble residue formed is estimated by determining the light reflectance of the filter pad.

3. APPARATUS

3.1 Aging tubes, 2.5 x 20 cm, heavy wall test tubes made of borosilicate glass.

3.2 Filter paper, glass fiber, 4.25 cm, 1.2 micron retention.

3.3 Membrane filter holder, to fit 47 mm membrane filters, vacuum source, and filtration flasks.

3.4 Heating bath, with liquid heating medium, thermostatically controlled and stirred to maintain the oil sample in the aging tube within 1.5° C of 150° C. It shall be large enough to hold aging tubes immersed in the heating liquid to a depth above the level of samples in the tubes. The bath and its location shall enable shielding of the samples from direct light during aging. The volume of oil in the bath and its heat recovery rate shall be such that the temperature of the heating medium does not drop more than 5° C when the maximum number of aging tubes are inserted, and recovery to 150° C does not require more than 15 minutes.

3.5 Paper reflectance meter^{1/}, black glass surface for calibration, and opaque white surface at least 10 x 20 cm in size.

^{1/} A Photovolt Paper Brightness Meter 670 complete with search unit W and black glass calibration standard, Catalog No. 00-572-32 is suitable and is available from Photovolt, 1115 Broadway, New York, NY 10010. Other meters may be satisfactory, provided the measured area on a filter pad is greater than 1 cm and less than 3 cm in diameter, and the following reflectance readings are obtained on the G-scale of an ASTM D2276 Color Standard for Rating Filter Membranes, after the meter is calibrated as in parts 7.7 to 7.9 of this method.

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4. REAGENTS

4.1 Hydrocarbon solvent, isooctane, ASTM knock test reference fuel grade. Adherent insolubles solvent, equal parts of reagent grade toluene, methanol, and acetone.

5. PREPARATION OF SAMPLE

5.1 Samples for stability testing should be all-level samples obtained according to methods outlined in D270, Sampling Petroleum and Petroleum Products. Samples should be stored in metal cans, preferably with epoxy lining. Clear glass bottles are not acceptable as sample containers. Undue exposure to sunlight may produce erratic results. If a sample cannot be tested immediately, it should be stored under nitrogen at a temperature not higher than 10° C. If samples are stored longer than one week, the date of sampling and date of testing shall be reported.

6. PREPARATION OF APPARATUS

6.1 Cleaning aging tubes - Clean new tubes by filling with cleaning solution^{2/} and allow to soak at least 2 hours. Rinse each scrupulously with tap water to remove all traces of acid, then with distilled water, followed by acetone, then dry. If compressed air is used it must be oil-free. Used tubes should be rinsed with adherent insolubles solvent, then with detergent, tap water, distilled water followed by acetone, then air dried.

6.2 Cleaning filter assembly - Membrane filter holders for which the filter rests on a sintered glass surface must be periodically cleaned with adherent insolubles solvent or cleaning solution followed by rinses as above.^{3/}

7. PROCEDURE

7.1 Adjust the heating bath to a temperature high enough to maintain the oil in the aging tubes at 150 \pm 1.5° C.

7.2 Filter a 175-mL sample through a new filter paper into a clean vacuum flask using the membrane filter assembly.^{4/}

^{2/} A chromium-free cleaning solution, "Nochromix," is available from Codex Labs, 6 Varick Street, New York NY 10013.

^{3/} Partial blockage of the sintered glass surface can lead to uneven deposition of insoluble residues on the filter surface and reduce reliability of results. Contaminants may also be introduced during pre-filtration of fuel to give erratic results.

^{4/} When testing fuel with additives which may be adsorbed on the filter paper, the procedure may be modified so that the neat fuel is filtered first, then the additive is mixed with the filtered fuel.

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7.3 Measure three 50-mL portions of the filtered oil and decant into the aging tubes.

7.4 Place the uncapped sample tube in the heating bath for 1.5 hours +3 minutes, 3 hours +5 minutes, and 4.5 hours +7 minutes.

7.5 Remove each sample from the bath at the end of its aging time and allow to cool gradually to 21 to 26° C over a period of 2 to 4 hours in a location shielded from light.

7.6 Prepare a filtration assembly for each sample with a new filter paper and filter the fuel. Wash the aging tube with 3 small portions of isooctane and filter. Wash the filter assembly with isooctane and remove the funnel portion of the assembly. Wash the filter pad with several very small portions of isooctane and air dry.

7.7 Turn on the reflection meter and allow at least 30 minutes for warm-up. The "suppression" knob should be in the "off" position.

7.8 Place a new filter paper on the opaque white surface, place the search unit in the center of the filter and adjust the meter reading to 100 percent using the "sensitivity" knob.

7.9 Place the search unit in the center of the black glass standard and adjust the meter reading to 0 percent using the "zero" knob. Recheck the 100 percent adjustment against the new filter pad resting on the white surface and readjust if necessary.

7.10 Place the test filter pad on the white surface, center the unit on the filter, and record the meter value as "percent reflectance after test".^{5/}

8. REPORT

8.1 Report the sample identification and aging time at 150° C.

8.2 Report the "percent reflectance after test".

8.3 If the test was not run within one week of fuel sampling, report sampling date and date of test.

9. PRECISION

9.1 The repeatability and reproducibility of this test have not been determined. The following factors have been reported to affect results in some laboratories.

^{5/} Dark fuels may stain the filter pads, resulting in lower reflectance ratings than due to insoluble residue alone.

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9.2 Sample storage time. Some fuels form degradation precursors during storage which markedly affect results, even when fuel appearance has not changed.

9.3 Filter paper porosity. The sensitivity of the method is changed when filters with different porosity or surface roughness are used.

9.4 Additive adsorption. Some additives are tenaciously adsorbed on the aging tubes and may affect results.

9.5 Heating bath design and location. Exposure to light during the aging step may affect results. High air flow rates across the open tubes during aging may affect the severity of the test.

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TEST METHOD NO. 3

DISTILLATE FUEL STORAGE STABILITY AT 43.3° C

1. SCOPE

1.1 This method covers the measurement of the inherent stability (as defined in paragraph 3.1 below) of a distillate fuel. The fuel is stored at a slightly elevated temperature over an extended period to give an indication of the fuel's potential long-term storage stability when stored at lower temperatures.

Note 1: This method is intended for use with high flashpoint ($<38.7^{\circ}\text{C}$) distillate-type fuels.

2. SUMMARY OF METHOD

2.1 A measured volume of filtered fuel in several glass containers is stored at 43.3°C for up to 24 weeks. After aging for various time intervals, one set of samples is removed from storage, cooled to room temperature, and analyzed for particulates by filtration. Adherent gum on the wall of the aging container is also measured. Color and steam jet gum tests are also performed on the fuel.

3. SIGNIFICANCE

3.1 Past research has indicated that storage at 43.3°C for 1 week is approximately equivalent to storage at average ambient (21°C) conditions for 4 weeks, assuming all other factors are equivalent. This test does not account for any environmental effects or container effects that may be encountered under a given field storage condition.

4. APPARATUS

4.1 Sample containers are borosilicate glass bottles. The containers should preferably have a lid or cover with a Teflon insert and a hole for a borosilicate glass vent. The total capacity of the container is 500 mL.

4.2 Storage oven should be large enough to contain all sample bottles. It should be thermostatically controlled to maintain a temperature of $43.3^{\circ} \pm 1^{\circ}\text{C}$. It shall be as dark as possible to prevent degradation due to photolytic reactions and shall also be "explosion proof."

4.3 Drying oven, maintained at $99^{\circ} \pm 1^{\circ}\text{C}$.

Warning! There are exposed hot surfaces on the drying oven. Avoid contact with exposed skin by use of protective equipment as required.

4.4 Gooch crucible, porcelain, No. 4

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4.5 Filter flask assembly, as shown in figure 1.

5. REAGENTS AND MATERIALS

5.1 Glass fiber filter, 2.4-cm circle 1/.

5.2 Hydrocarbon solvent, isooctane, ASTM knock test reference fuel grade, filtered through two glass-fiber filter papers.

Warning! Flammable. Harmful if inhaled (see 13).

5.3 Adherent insolubles solvent--Mix equal parts of reagent grade acetone, methanol, and toluene.

Danger! Extremely flammable. Vapors harmful. Vapors may cause flash fire. (see 13).

5.4 All equipment required to determine color by ASTM Method D1500.

5.5 All equipment required to perform steam jet method described in ASTM Method D381.

6. SAMPLING PROCEDURE

6.1 Samples for testing shall be procured by the method outlined in ASTM Method D270. Sample containers should preferably be 3.785 liter or larger epoxy-lined cans. These containers should be filled almost to the top to avoid a significant air space, and then the ullage should be purged with nitrogen. Where possible, samples should be stored at reduced temperature, -7° to 4° C, prior to use.

7. PREPARATION OF APPARATUS

7.1 Scrub each sample storage bottle with a detergent solution and rinse it with water. Soak the bottles overnight in an alkaline laboratory glassware cleaning solution.^{2/} Rinse the bottles with tapwater, then invert and flush with a stream of distilled water. Allow the bottles to dry, and rinse the bottles with 50 mL of the fuel sample. Bottles are vented during storage preferably by a Teflon insert and a borosilicate glass bend to prevent contamination of the sample from airborne particulates (see figure 2).

1/ The glass fiber filter paper available from H. Reeve Angel & Co., New York, NY, as Catalog No. X-934-AH, has been found satisfactory for this purpose.

2/ A 2-percent solution of RBS-35, manufactured by Pierce Chemical Company, has been found suitable. An alternate procedure is to use a chromium-free cleaning solution, "Nochromix," available from Godax Labs, 6 Varick Street, New York, NY 10013.

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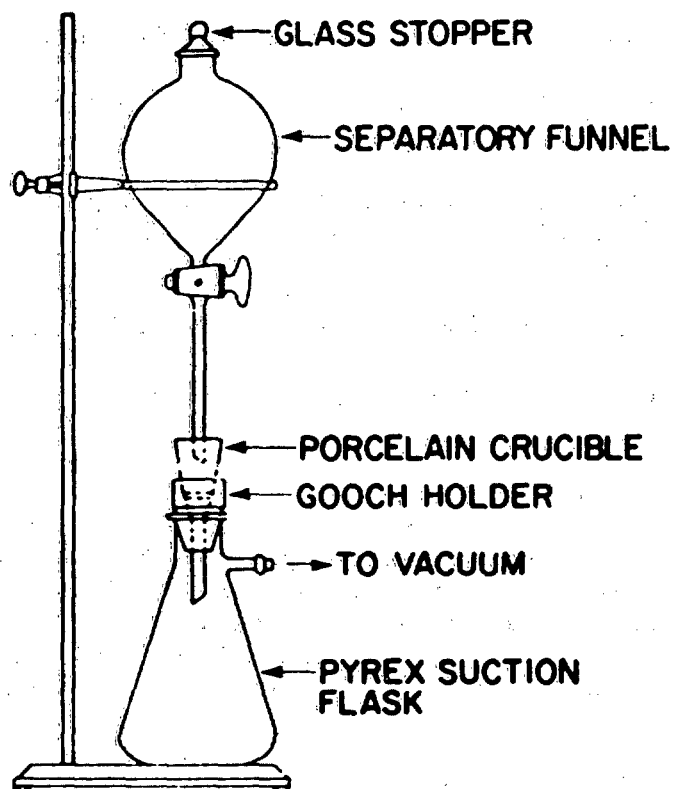


FIGURE 1. Self-feeding filtering assembly.

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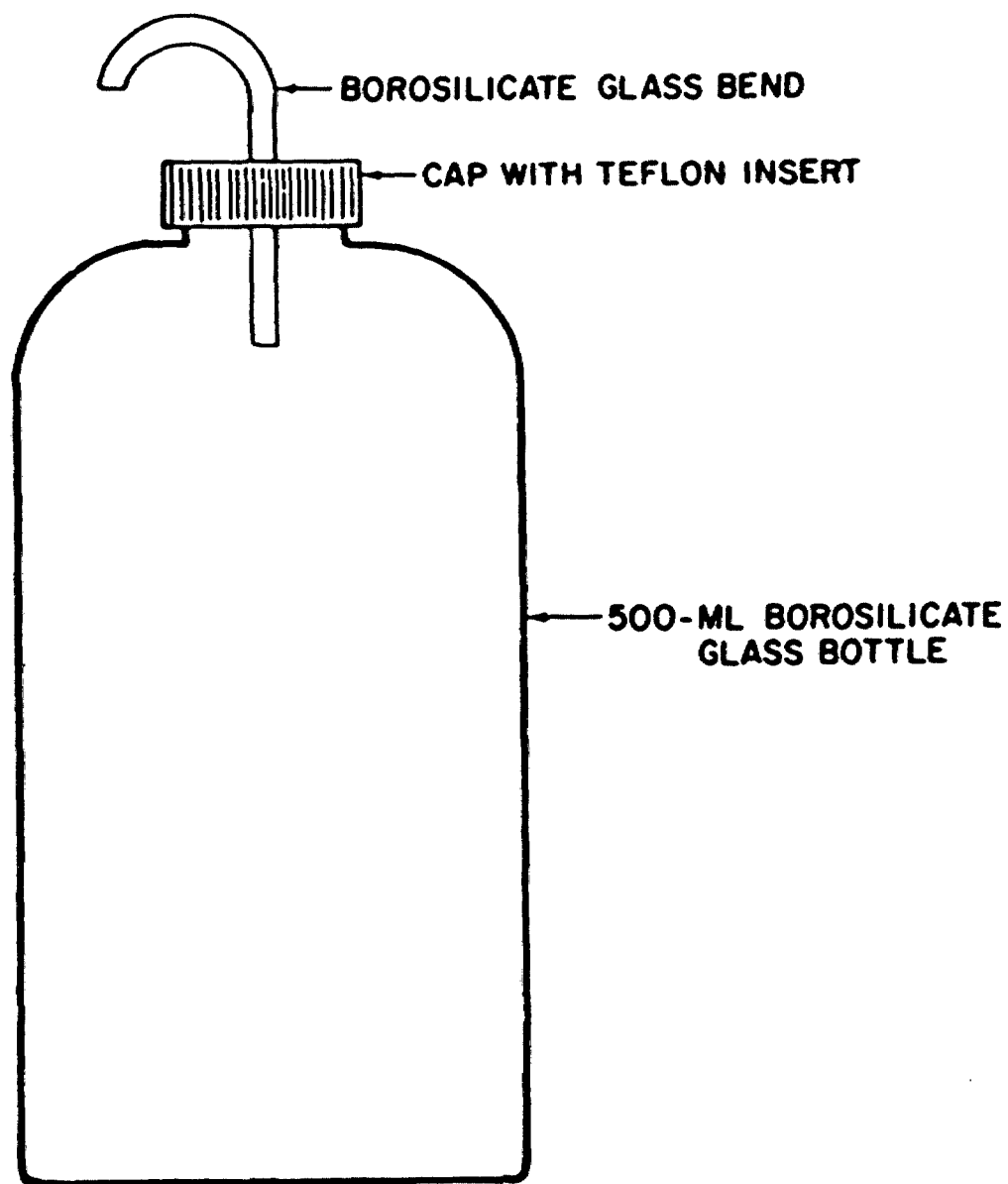


FIGURE 2. Sample storage container.

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7.2 Insert two glass-fiber filter disks into each of three clean Gooch crucibles (Note 2). Wash the filters by pouring 200 mL of isooctane through the Gooch crucible. Dry the crucible and filters for 1 hour in an oven maintained at $99^{\circ} \pm 1^{\circ}$ C. After drying, place the crucible and filters in a dessicator (without dessicant) for at least 30 minutes (Note 3). Weigh to the nearest 0.1 mg and retain for sample analysis. This procedure must be followed for the original sample and for each test sample interval. Prepare one crucible with filters for each bottle to be analyzed at each test sample interval and one crucible with filters to be used as a moisture correction. When a set of bottles is being analyzed, only one "moisture blank" is required. The "moisture blank" crucible should be weighed whenever the other crucibles are weighed and its change in weight subtracted from the final sample weight to give a sample weight that is corrected for atmospheric moisture.

Note 2: A single paper will not prove satisfactory since it may rupture under strong vacuum.

Note 3: A dessicator is used to protect the crucible and filters from airborne particulates. The dessicant is removed from the dessicator to allow the crucible to cool in an environment similar to the surrounding environment and thereby prevent possible errors due to sudden absorption of moisture from the atmosphere when the crucible is removed from the dessicator. The crucible used as a "moisture blank" will correct for the atmospheric moisture.

8. PREPARATION OF SAMPLE

8.1 Prior to storage, the fuel oil shall be filtered through two glass fiber filters. It is suggested that a filtration system as shown in figure 1 be used to filter the fuel (Note 4).

Note 4: The receiving flask, separatory funnel, and glass funnel should be as clean as the storage bottles (see 7.1). These items should also be rinsed with filtered isooctane.

9. PROCEDURE

9.1 Adjust the storage oven for sample storage to a temperature of $43.3^{\circ} \pm 1^{\circ}$ C.

Warning! Avoid contact with exposed hot surfaces on the drying oven by use of protective equipment as required.

9.2 Prepare the fuel for storage (see 8.1).

9.3 Place 400 ml of filtered fuel into each bottle. Use two bottles for each sampling period. The required sampling periods are 8 and 16 weeks. Also, fill two bottles with fuel to be used as extras. These extra bottles

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may be used in the case of accidents, if extra fuel is needed for further tests, or if desired to extend the overall storage test duration. Also filter enough fuel to be analyzed for zero-week data. These analyses should be performed on the same day that the other samples are placed in storage. Label each storage bottle completely.

9.4 At the end of each prescribed period, remove two bottles and allow them to cool to 21°-27°C for 4-24 hours in a dark environment.

9.5 Analysis of the fuel, after it has cooled, should be performed in the following manner. Pour the sample from one bottle into a clean separatory funnel, using a glass funnel (Note 4), and filter through a tared Gooch crucible containing two glass-fiber filter papers (Note 5). Use a self-feeding system as illustrated in figure 1. Repeat this procedure with the other bottle of fuel using a different crucible and filters (Note 6). Retain the filtrate for ASTM color and steam jet gum determinations. Rinse the bottles used during aging with filtered hydrocarbon solvent (use three rinsings of approximately 50 mL each) to remove all traces of fuel from the bottle.

Warning! Flammable. Harmful if inhaled (see 13).

Filter these rinsings through the Gooch crucible. After the crucible is washed oil-free on the inside, turn off the suction and remove the crucible. Then rinse the outside of the crucible (using a wash bottle) with additional hydrocarbon solvent until its surface is oil free. The filtrate can be discarded.

Note 5: If severe filter plugging is encountered so that filtration is not complete in 3 hours, discontinue the test with the notation that filter plugging occurred.

Note 6: ASTM D1500 color and D381 steam jet gum must also be determined on the filtered fuel. Do not add rinsings or contaminate the filtered fuel in any manner. Protect the sample from light until these tests are completed.

9.6 Place the crucibles, including the "moisture blank" in the drying oven for 4 to 6 hours. Then place the crucibles in a dessicator without dessicant and allow to cool to room temperature [requires at least 1 hour (Note 3)]. Weigh to nearest 0.1 mg.

9.7 After the final washing with the hydrocarbon solvent, dissolve any adherent gum on the sample container walls with two washings of 30 to 35 mL of the adherent insolubles solvent.

Danger! Extremely flammable. Vapors harmful. Vapors may cause flash fire (see 13).

Determine the adherent insolubles content by evaporating the solvent at 160° C by the air jet method described in ASTM Method D381, using 1 or 2 gum beakers as required for the amount of solvent used.

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Warning! There are exposed hot surfaces on the evaporation bath. Avoid contact with exposed skin by use of protective equipment as required.

Warning! The sample beakers and conical jets are extremely hot after completion of evaporation of solvent. Handle with care and use protective equipment or forceps as required.

Danger! The solvent vapors evaporated during this procedure may be extremely flammable or combustible and hazardous to inhale. The evaporation bath must be provided with an effective exhaust hood to control such vapors.

10. CALCULATION

10.1 Calculate the total insolubles after aging, in milligrams per 100 mL, as follows:

$$A = (B+C)/4$$

where:

A = Total insolubles, mg/100 mL,
B = Weight of filterable insolubles (Note 7), mg, and
C = Weight of adherent insolubles (Note 7), mg, as determined in 9.7.

Note 7: In this calculation, B and C must be corrected for moisture using the moisture blank.

11. COLOR AND STEAM JET GUM

11.1 Perform color and steam jet gum tests in accordance with ASTM D1500 and D381 on the filtrate retained in 9.5.

12. REPORT

12.1 For unaged, filtered fuel, report results from 10.1 including A and B in appropriate units, and color and steam jet gum from 11.

12.2 For the aged fuel, report A, B, and C from 10.1 for both bottles to show repeatability of tests at each aging period. Also report color and steam jet gum from 11.

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13. PRECAUTIONARY STATEMENTS

Adherent Insolubles Solvent

Danger! Extremely flammable. May cause flash fire.
Inhalation of vapors harmful.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Vapors may spread long distances and ignite explosively.
Avoid build-up of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.
Avoid prolonged breathing of vapor or spray mist.
Avoid contact with eyes and skin.
In case of spillage, absorb in clay or diatomaceous earth and flush with large volumes of water.
In case of fire, use water, dry chemical, CO₂, or foam.

Distillate Fuel

Caution! Combustible! Vapor harmful.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid breathing vapor or spray mist.
Avoid prolonged or repeated contact with skin.
In case of spillage, soak up with clay, diatomaceous earth, or similar materials.
In case of fire, use foam, dry chemical, or CO₂.

Isooctane

Danger! Extremely flammable.
Harmful if inhaled. Vapors may cause flash fire.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid build-up of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.
In case of spillage, soak up with sand or diatomaceous earth.
In case of fire, use water spray, foam, dry chemical, or CO₂.

Chronic Acid (Cleaning Solution)

Danger! Causes severe burns. A recognized carcinogen.
Strong oxidizer--contact with other material may cause fire. Hygroscopic.

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Do not get in eyes, on skin, or on clothing.

Avoid breathing vapor or mist.

Keep container closed.

Use with adequate ventilation.

Do not take internally.

Wash thoroughly after handling.

In case of spillage, soak up with excess sodium carbonate and flush with plenty of water.

Acetone

Danger! Extremely flammable. Vapors may cause flash fire.

Keep away from heat, sparks, and open flame.

Keep container closed.

Use with adequate ventilation.

Vapors may spread long distances and ignite explosively.

Avoid build-up of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.

Avoid prolonged breathing of vapor or spray mist.

Avoid contact with eyes and skin.

In case of spillage, absorb and flush with large volumes of water.

In case of fire, use water, dry chemical, CO₂, or foam.

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