MIL-F-46162C(ME)
12 November 1985
SUPERSEDING
MIL-F-46162B(ME)
14 August 1981

MILITARY SPECIFICATION

FUEL, DIESEL, REFEREE GRADE

This specification is approved for use within the USA Belvoir Research and Development Center, Department of the Army, and is available for use by all Departments and Agencies of the Department of Defense.

- 1. SCOPE
- 1.1 Scope. This specification covers one grade of referee diesel fuel (see 6.2) for use in research, development and proof testing of all compressionignition engines, diesel-powered auxiliary units, gas turbine engine driven ground vehicles and mobile electric power generators, and other fuel handling supply items designed to operate with tactical grades of diesel fuel conforming to VV-F-800 (see 6.1).
 - 2. APPLICABLE DOCUMENTS
 - 2.1 Government documents.
- 2.1.1 Specifications, standards, and handbooks. The following specifications, standards, and handbooks form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of these documents shall be those listed in the issue of the Department of Defense Index of Specifications and Standards (DoDISS) and supplement thereto, cited in the solicitation.

SPECIFICATIONS

FEDERAL

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: USA Belvoir Research and Development Center, ATTN: STRBE-TSE, Fort Belvoir, VA 22060-5606 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 9140

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MILITARY

MIL-T-5624	- Turbine Fuel, Aviation, Grades JP-4 and JP-5.
MIL-S-53021	- Stabilizer Additive, Diesel Fuel.
MIL-T-83133	- Turbine Fuel, Aviation, Kerosine Type, Grade JP-8.

STANDARDS

FEDERAL

FED-STD-791	- Lubricants, Liquid Fuels, and Related
	Products; Methods of Testing.

MILITARY

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

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

2.2 Other publications. The following document(s) form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted shall be those listed in the issue of the DoDISS specified in the solicitation. Unless otherwise specified, the issues of documents not listed in the DoDISS shall be the issue of the nongovernment documents which is current on the date of the solicitation.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

- D 86 Distillation of Petroleum Products.
- D 93 Flash Point by Pensky-Martens Closed Tester.
- D 97 Pour Point of Petroleum Oils.
- D 129 Sulfur in Petroleum Products by the Bomb Method.
- D 130 Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test.
- D 240 Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Method.
- D 445 Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity).
- D 482 Ash from Petroleum Products.
- D 524 Ramsbottom Carbon Residue of Petroleum Products.
- D 613 Ignition Quality of Diesel Fuels by the Cetane Method.
- D 974 Neutralization Number by Color-Indicator Titration.
- D 976 Calculated Cetane Index of Distillate Fuels.

- D 1298 Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method.
- D 1319 Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption.
- D 1552 Sulfur in Petroleum Products (High Temperature Method).
- D 2274 Oxidation Stability of Distillate Fuel Oil (Accelerated Method).
- D 2276 Particulate Contaminant in Aviation Turbine Fuels.
- D 2500 Cloud Point of Petroleum Oils.
- D 2622 Sulfur in Petroleum Products (X-Ray Spectrographic Method).
- D 4057 Manual Sampling of Petroleum and Petroleum Products.
- D 4176 Free Water and Particulate Contamination in Distillate Fuels (Clear and Bright Pass/Fail Procedures.)

(The test methods listed above are included in Volumes 05.01, 05.02, 05.03, and 05.04 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.)

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

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, (except for associated detail specifications, specifications sheets or MS standards), the text of this specification shall take precedence. Nothing in this specification, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

- 3.1 <u>Material</u>. The diesel fuels shall be refined petroleum distillates. Those fuels meeting the requirements shall contain catalytically or thermally processed blending fractions with additives as specified in 3.2.
- 3.2 Additives. Additive use shall be as specified in 3.2.1 through 3.2.3. Addition of stabilizer additive (see 3.2.1) is mandatory. Addition of cetane improvers, pour-point depressants, or flow improvers is permissible, if necessary to meet requirements of this specification.
- 3.2.1 Stabilizer additive. The finished fuel shall contain one of the following stabilizer additives at the concentration indicated:
 - a. Any product qualified under the provisions of MIL-S-53021 at the recommended effective concentration for that product.

- b. FOA- $15\frac{1}{}$ at a concentration of 71 ± 3 g/m³ plus Biobor JF $\frac{2}{}$ at a concentration of 227 +10 g/m³.
- 3.2.2 Cetane improver. The cetane number requirements specified in table I may be obtained by use of any one or any combination of the approved cetane improvers (isopropyl nitrate, amyl nitrate, hexyl nitrate, cyclohexyl nitrate, and octyl nitrate), including the structural isomers of amyl, hexyl, and octyl nitrates. The total concentration of cetane improvers in the finished fuel shall not exceed 0.50 percent by weight.
- 3.2.3 <u>Pour-point depressant or flow improver</u>. A pour-point depressant or flow-improver additive may be used to meet the pour point requirement specified in table I.
- 3.3 Physical and chemical requirements. The finished diesel fuel shall conform to the requirements specified in table I.

TABLE I. Physical and chemical requirements.

Property	Value	
Density, Kg/L @ 15° C	Report	
Flash point, °C, min	52	
Cloud point, °C max	-13	
Pour point, °C max	-18	
Kinematic viscosity, cSt at 40° C	1.9 to 4.1	
Distillation, °C:		
Initial boiling point	Report	
10% recovered, min	220	
50% recovered	255 to 305	
90% recovered	310 to 360	
95% recovered	315 to 365	
End point, max	385	
Residue, volume %, max	3	
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^{1/} FOA-15 is available from E.I. du Pont de Nemours and Co., Wilmington, DE 19898.

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

TABLE I. Physical and chemical requirements. (Continued)

Property	Value	
Carbon residue on 10% bottoms, % wt, max1/	0.20	
Sulfur, % wt ² /	0.95 to 1.05	
Copper strip corrosion, 3 hrs @ 50° C, ASTM classification, max	1	
Ash, % wt, max	0.02	
Accelerated stability, total insolubles, mg/100 mL, max	1.5	
Neutralization No., TAN, max	0.2	
Aromatics, volume %	Report	
Net heat of combustion, MJ/kg	Report	
Particulate contamination, mg/L, max	10	
Cetane number	37 to 43	
Free water and particlulate contamination	Pass	

^{1/} See Appendix. If the fuel contains cetane improver additives, this limit shall apply only to the base fuel.

4. QUALITY ASSURANCE PROVISIONS

- 4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.
- 4.1.1 Responsibility for compliance. All items must meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of assuring that all products or supplies

^{2/} Naturally-occurring sulfur is preferred. If additional sulfur is required, non-naturally-occurring sulfur compounds may be blended into the fuel. Not less than half of the total sulfur in the finished fuel shall be naturally-occurring.

submitted to the Government for acceptance comply with all requirements of the contract. Sampling in quality conformance does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to acceptance of defective material.

4.2 Lot.

- 4.2.1 <u>Bulk lot</u>. An indefinite quantity of a homogeneous mixture of one grade of fuel oil offered for acceptance in a single, isolated container; or manufactured in a single plant run (not exceeding 24 hours), through the same processing equipment, with no change in the ingredient materials.
- 4.2.2 Packaged lot. An indefinite number of 55-gallon drums or smaller unit containers of identical size and type, offered for acceptance, and filled with a homogeneous mixture of one grade of fuel oil from a single, isolated container; or filled with a homogeneous mixture of one grade of fuel oil, manufactured in a single plant run (not exceeding 24 hours), through the same processing equipment, with no change in the ingredient materials.

4.3 Sampling.

- 4.3.1 Sampling for examination of filled containers. Take a random sample of filled containers from each packaged lot in accordance with MIL-STD-105 at inspection level II and acceptable quality level (AQL) = 2.5 percent defective.
- 4.3.2 Sampling for tests. Take samples for tests in accordance with ASTM Method D $\overline{4057}$.
- 4.4 <u>Inspection</u>. Perform inspection in accordance with Method 9601 of FED-STD-791.
- 4.4.1 Examination of filled containers. Examine samples taken in accordance with 4.3.1 for compliance with MIL-STD-290 with regard to fill, closure, sealing, leakage, packaging, packing, and marking requirements. Reject any container having one or more defects or under the required fill. If the number of defective or underfilled containers exceeds the acceptance number for the appropriate plan of MIL-STD-105, reject the lot represented by the sample.
 - 4.5 Classification of tests. All tests are quality conformance tests.
- 4.6 <u>Test methods</u>. Perform tests in accordance with the applicable methods listed in table II and appendix.

TABLE II. Test methods.

Test	ASTM Test Method No.
Density	D 1298
Flash point	D 93
Cloud point	D 2500
Pour point	D 97
Kinematic viscosity	D 445
Distillation	D 86
Carbon residue 1/	D 524
Sulfur2/	D 1552, D 129, or D 2622
Copper strip corrosion	D 130
Ash	D 482
Accelerated stability	D 2274
Neutralization number	D 974
Aromatics	D 1319
Net heat of combustion	D 240
Particulate contamination	D 2276 (Appendix A2)
Cetane number 3/	D 613 or D 976
Free water and particulate	D 4176
contamination	

- $\frac{1}{2}$ See Appendix. If the fuel contains cetane improver additives, the carbon residue test shall be performed on the base fuel.
- $\frac{2}{D}$ D 1552 is the preferred method but either D 129 or D 2622 may be used as an alternate.
- 3/ D 613 is the preferred method for determining cetane quality and D 976 is permitted as an alternate. If cetane improvers (as determined by the method in the appendix) or non-naturally-occurring sulfur compounds have been added to the fuel, only method D 613 shall be permitted.
- 4.7 Report of analysis. The contractor shall provide copies of analysis reports, giving the results of these tests and a statement of the types and concentrations of the additives used in the diesel fuel.

PACKAGING

5.1 Packing and marking. Diesel fuel contained in the size and type container specified (see 6.2), shall be packed and marked in accordance with MIL-STD-290. Packing shall be level B, level C, or commercial as specified (see 6.2).

NOTES

6.1 Intended use. This diesel fuel is intended for use in research, development and proof-testing of all compression-ignition engines, diesel-powered auxiliary units, gas turbine engine driven ground vehicles and mobile electric

power generators, and other fuel handling supply items. The use of referee fuel in conjunction with Development Testing (DTI through DTIII) is considered necessary to uncover potential operational problems with respect to performance and to enable data correlation of all testing sites regardless of geographical location. Referee fuels should not be used in engines or other equipment in the field as a substitute for VV-F-800 diesel fuel without approval of the engine manufacturer or from the US Army Belvoir Research and Development Center, ATTN: STRBE-VF, Fort Belvoir, VA 22060-5606.

6.1.1 Temperature range. This referee grade diesel fuel is intended for use in the same temperature range as OCONUS DF-2 and has the same cloud point (-13°C) and pour point (-18°C) as DF-2 procured for Europe and South Korea. The actual low temperature operability limit depends on the vehicle fuel system design (filter and pump locations, filter size and porosity, proximity to engine heat, etc.) as well as a fuel properties. Most vehicles will operate satisfactorily down to approximately the cloud point of the fuel, but some vehicles may be equipped with fuel heaters which enable them to operate well below the cloud point. If fuel waxing causes operability problems at or near the cloud point, JP-8 fuel conforming to MIL-T-83133 or JP-5 fuel conforming to MIL-T-5624 should be used.

6.2 Ordering data.

- a. Title, number, and date of this specification.
- b. Date of issue of DoDISS applicable 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 diesel fuel required. The unit of purchase is the US gallon [231 cubic inches at 60° F (15.6° C)]
- 6.3 <u>Definition</u>. Referee grade diesel fuel is defined as diesel fuel representing the minimal or marginal quality level which can be procured under specification VV-F-800 while meeting all of its requirements. It is designed to be equivalent to the quality of OCONUS distillate production or that production available in times of national emergency. It is used for research, development, and proof-testing to assure that all diesel-consuming equipment will perform adequately with all diesel fuels procured under VV-F-800.

Custodian:

Army - ME

Preparing activity: Army - ME

Review activities:

Army - AT, CE

DLA - PS

Project 9140-A104

User activities:

Army - TE

APPENDIX

DETECTION OF NITRATE-TYPE IGNITION IMPROVERS IN DIESEL FUEL

10. SCOPE

10.1 Scope. This method of test covers the determination of organic nitrate ester-type cetane improver additives used in diesel fuel. It is intended as a screening test for those diesel fuel inspection test procedures that are affected by the presence of cetane improvers; namely, ASTM D 524, Ramsbottom Carbon Residue of Petroleum Products and ASTM D 976, Calculated Cetane Index of Distillate Fuels.

20. SUMMARY

20.1 Summary. A diesel fuel sample is saponified in a potassium hydroxide 1-butanol mixture and then filtered through a glass fiber filter disc. The
material remaining on the disc is treated with diphenylamine reagent after
drying. The presence of a nitrate ester cetane improver is revealed by the
formation of a blue ring or blue-black spot due to oxidation of diphenylamine to
intense blue quinoidal compounds by the nitrate salt. No color change confirms
the absence of a cetane improver.

30. APPARATUS

- 30.1 Reaction bottle. Screw-cap bottle, 29.6 mL (1 fl oz) capacity, wide mouth, flint glass, with screw-cap lined with tin or tetrafluorethylene (TFE) resin.
- 30.2 Glass fiber filter paper. 37-mm diameter, Grade 934 AH (Whatman, Ltd., or equivalent).
- 30.3 <u>Pipette</u>. 10-mL capacity, fitted with a pipetting bulb. Several types and makes of pipetting bulbs and assemblies are available. One of the following is suggested: Fisher Cat No. 13-681, Safety Pipet Filler, or equal; Fisher Cat No. 13-681-50, Pipet Filler, or equal.
 - 30.4 Graduated cylinders. 10-mL and 25-mL capacity.
- 30.5 Suction flask with a suitable holder to accomodate a 60-mL glass-fritted crucible.
 - 30.6 Crucible. 60-mL capacity, glass-fritted, medium porosity.
 - 30.7 Oven. Suitable for drying filter discs at 110° C.

MIL-F-46162C(ME) APPENDIX

40. REAGENTS

- 40.1 Saponification mixture (1N). Prepare by mixing 6.5 g of potassium hydroxide (ACS reagent grade) with 100 ml of absolute 1-butanol (ACS reagent grade) and heat to dissolve the KOH. After the solution cools, filter the mixture through the glass-fiber filter paper.
- 40.2 Diphenylamine (1 percent solution). Prepare by dissolving 0.250 g of diphenylamine (ACS indicator grade in 25 mL of sulfuric acid (sp gr 1.834).
 - 40.3 Toluene (ACS reagent grade).

NOTE: Toluene is flammable and toxic. Avoid breathing vapors or contact with skin.

50. PROCEDURE

50.1 Pipette 10 mL of the sample into the reaction bottle and add 5 mL of toluene followed by 10 mL of the saponification mixture.

NOTE: Oral pipetting techniques should not be used because of the toxicity of the substances involved. A pipetting bulb or assembly similar to one of those described in 30.3 should be used.

- 50.2 Affix cap to the reaction bottle tightly and, after mixing the contents, place it in an oven maintained at 110°C for four hours.
- 50.3 Remove the reaction bottle from the oven and allow it to cool to 25° C plus or minus 3° C.
- 50.4 Filter the contents of the reaction bottle through the 60-mL glass-fritted crucible fitted with the glass fiber filter disc.
- 50.5 Wash the reaction bottle with 25 mL of toluene and transfer it to the glass-fritted crucible.
- 50.6 Carefully remove the glass fiber filter disc and dry it in oven at 110° C for 15 minutes.
 - 50.7 Remove the filter disc and cool it to 25° plus or minus 3° C.
- 50.8 Add three drops of diphenylamine solution to the center of the disc and observe whether a blue or blue-black color forms.

MIL-F-46162C(ME) APPENDIX

60. REPORT

60.1 The presence of organic nitrate ester-type cetane improvers will be reported if the formation of a blue color occurs. Reference samples of diesel fuels containing 0.5 percent by volume of any one of the approved cetane improvers (see 3.2.2) give an intense blue to blue-black color throughout the reagent spot whereas those samples containing only 0.1 percent by volume produce a blue ring at the outer boundary of the reagent. If a positive reaction occurs (i.e., a blue or blue-black coloration), the carbon residue determination (D 524) must be performed on a neat or base-fuel blend.

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34 NAME OF SUBMITTING ORGANIZATION		4. TYPE OF ORGANIZATION (Mark one) VENDOR USER
b. ADDRESS (Street, City, State, ZIP)	Code)	1
		MANUFACTURER
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