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MIL-PRF-17331H
23 January 1984
SUPERSEDING
MIL-L-17331G(SH)
7 December 1977
(See 6.6)

PERFORMANCE SPECIFICATION
LUBRICATING OIL, STEAM TURBINE
AND GEAR, MODERATE SERVICE

This specification is approved for use by the Naval Sea Systems Command, Department of the Navy, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers steam turbine lubricating oil for use in main turbines and gears, auxiliary turbine installations, certain hydraulic equipment, general mechanical lubrication, and air compressors. The lubricating oil shall be identified as follows:

Military symbol
2190-TEP

NATO symbol
0-250

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications and standards. Unless otherwise specified, 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

- PPP-D-729 - Drums, Shipping and Storage, Steel, 55-Gallon.
(208 Liters).
- PPP-P-420 - Plugs and Flanges (For Drum Closures).

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, Naval Sea Systems Command, SEA 5523, Department of the Navy, Washington, DC 20362 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 9150

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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 and standards required by contractors in connection with specific acquisition functions should be obtained from the contracting 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.

ASTM

- D 92 - Flash and Fire Points by Cleveland Open Cup.
(DoD adopted)
- D 95 - Water in Petroleum Products and Bituminous Materials by Distillation. (DoD adopted)
- D 97 - Pour Point of Petroleum Oils. (DoD adopted)
- D 129 - Sulfur in Petroleum Products (General Bomb Method).
(DoD adopted)
- D 130 - Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test. (DoD adopted)
- D 189 - Conradson Carbon Residue of Petroleum Products.
(DoD adopted)
- D 270 - Sampling Petroleum and Petroleum Products. (DoD adopted)
- D 287 - API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method). (DoD adopted)
- D 445 - Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity). (DoD adopted)
- D 665 - Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water. (DoD adopted)
- D 874 - Sulfated Ash from Lubricating Oils and Additives.
(DoD adopted)
- D 892 - Foaming Characteristics of Lubricating Oils.
(DoD adopted)
- D 943 - Oxidation Characteristics of Inhibited Mineral Oils.
- D 974 - Neutralization Number by Color-Indicator Titration.
(DoD adopted)
- D 1298 - Density, Relative Density (Specific Gravity) or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method. (DoD adopted)

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- D 1401 - Emulsion Characteristics of Petroleum Oils and Synthetic Fluids.
- D 1500 - ASTM Color of Petroleum Products (ASTM Color Scale). (DoD adopted)
- D 1552 - Sulfur in Petroleum Products (High-Temperature Method). (DoD adopted)
- D 1947 - Load-Carrying Capacity of Petroleum Oil and Synthetic Fluid Gear Lubricants. (DoD adopted)
- D 2272 - Oxidation Stability of Steam Turbine Oils by Rotating Bomb.
- D 2622 - Sulfur in Petroleum Products (X-Ray Spectrographic Method). (DoD adopted)
- F 313 - Insoluble Contamination of Hydraulic Fluids by Gravimetric Analysis.

(Application for copies should be addressed to the ASTM, 1916 Race Street, Philadelphia, PA 19103.)

UNIFORM CLASSIFICATION COMMITTEE AGENT

Uniform Freight Classification Ratings, Rules and Regulations

(Application for copies should be addressed to the Uniform Classification Committee Agent, Tariff Publication Officer, Room 1106, 222 South Riverside Plaza, Chicago, IL 60606.)

(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 lubricating oil furnished under this specification shall be products which are qualified for listing on the applicable qualified products list at the time set for opening of bids (see 4.3 and 6.3).

3.2 Material. The lubricating oil shall be a stable homogeneous blend of virgin petroleum lubricating oil stocks plus additive agents, if necessary, to meet the requirements of this specification. However, the use of additives to improve the viscosity-temperature characteristics of the base stocks is not permitted, although additives, in quantities up to 0.5 percent, may be used to meet the pour point requirement. Additives, if used, shall not contain chlorine or zinc materials. Additive components as contained in the sample tested and granted qualification approval shall not be changed without approval of the qualifying activity.

3.3 Bearing compatibility. The lubricating oil shall be noncorrosive to the bearing materials of the equipment as specified in 1.1. Acceptable results from the copper strip corrosion tests and the journal bearing compatibility test as specified in table I will be considered as evidence of noncorrosiveness to bearing materials.

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TABLE I. Chemical and physical requirements and test methods.

Characteristics	Limit	Method no. ^{1/}	ASTM no.
Color	<u>2/</u>	-----	D 1500
Sulphur	<u>2/</u>	-----	D 129 D 1552 or D 2622
Mineral acidity, (neutrality qualitative)	Neutral	5101	-----
Neutralization number, maximum	0.3	-----	D 974
Corrosion (in presence of salt water)	None	-----	D 665, Procedure B ^{3/}
Corrosion test at 100°C (212°F), (Copper strip) appearance, maximum	1	-----	D 130
Bearing compatibility	Pass	<u>4/</u> 3452	-----
Compatibility	Pass	See 3.4	-----
Water, percent	None	-----	D 95
Carbon residue	<u>2/</u>	-----	D 189
Ash (sulphated residue)	<u>2/</u>	-----	D 874
Gravity, API	<u>2/</u>	-----	D 287 or D 1298
Flash point, °C	204 (400)	-----	D 92
(°F), minimum			
Pour point, °C	-6 (20)	-----	D 97
(°F), maximum			
Viscosity:			D 445
Centistokes at 4.4°C (40°F), maximum	870		
Centistokes at 40°C (104°F)	74-97		
Centistokes at 100°C (212°F) minimum	8.0		
Emulsion test, after 30 minutes settling time	-----	-----	<u>5/</u> D 1401
Oil layer, maximum	<u>6/</u> 40 mL	-----	-----
Lacy cuff, maximum	3 mL	-----	-----

See footnotes on next page.

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TABLE I. Chemical and physical requirements and test methods. - Continued

Characteristics	Limit	Method no. ^{1/}	ASTM no.
Oxidation test, time required in hours to reach neutralization value of 2.0 mg KOH, minimum	1000	-----	D 943
After 1000 hours:			
Total sludge, mg maximum	100	See appendix A	-----
Total iron, mg maximum	100		-----
Total copper, mg maximum	100		-----
Oxidation by rotating bomb	2/		D 2272
Load-carrying ability, (lb/in) minimum	2200	-----	7/D 1947
Wear test, scar diameter mm, maximum	.33	8/6503	-----
Homogeneity	No separation	See 4.7	-----
Foam characteristics:			10/D 892
Tendency/stability			
Sequence 1, mL maximum	11/65/0		
Sequence 2, mL maximum	11/65/0		
Sequence 3, mL maximum	11/65/0		
Cleanliness, mg/100 mL maximum	9/4.0	-----	F 313
Valve sticking characteristics	Pass	See appendix C	-----

1/ FED-STD-791.

2/ No limiting requirement. In each case determination is made as a matter of record.

3/ Prior to the test, the oil shall be water washed as follows: 300 grams of the oil to be tested shall be stirred with 50 grams of water for 30 minutes at 90 degrees Celsius (°C) (194 degrees Fahrenheit (°F)) using the beaker and stirrer as specified in ASTM D 665. After 30 minutes stirring, the mixture shall be transferred to a separatory funnel and left to separate; then the water layer shall be drawn off and the oil submitted to the salt water corrosion test. The test shall be run for 48 hours.

4/ Evidence of significant corrosion or deposit will be disqualifying. A new bearing is required for each test. The work factor portion of method 3452 is not required.

5/ Distilled water and a stirring temperature of 54°C (130°F) shall be used.

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- 6/ Discrete water droplets entrained in the oil layer and clinging to the wall of the cylinder may be added to the water layer volume at the rate of 0.1 mL per droplet.
- 7/ Only those Ryder gear machines having a Navy reference oil number 1 average rating of 2200-2700 pounds per inch (lb/in) are acceptable.
- 8/ (a) Load - 15 kilograms.
(b) Oil temperature - 80°C (176°F).
(c) Speed - 600 revolutions per minute (r/min).
(d) Duration of test - 120 minutes.
- 9/ In addition to the samples taken under 4.4 for compliance with the cleanliness requirement in table I, a sample shall be taken at the filling pipe immediately prior to entering a container (drums, tank truck). Only one sample for one continuous drum filling operation is required and shall be taken after approximately 10 percent of the volume of product has been drummed. Test sample in accordance with ASTM F 313 using 0.8 micrometer pore diameter membrane filters.
- 10/ Option A shall not be used.
- 11/ A ring of bubbles around the edge of the graduate shall be considered complete collapse or 0 mL.

3.4 Compatibility. The lubricating oil shall be compatible with reference oils furnished by the Government and meeting all the requirements of the specification. The oil shall be considered compatible if 1:1 mixtures of a test oil with all reference oils give passing values on the oxidation, corrosion (salt water), foam and emulsion tests.

3.5 Homogeneity. Additive agents, when used, shall remain uniformly distributed throughout the oil at all temperatures above the pour point and up to 121°C (250°F). If the oil is cooled below its pour point, it shall regain its homogeneity on standing at temperatures of 6°C (10°F) above its pour point and shall retain that homogeneity up to 121°C (250°F).

3.6 The lubricating oil shall conform to the chemical and physical requirements as specified in table I.

3.7 Lot acceptance requirements. Each sample selected as specified in 4.4, when tested as specified in table I, shall not vary from the analysis of the sample given qualification approval to an extent of the tolerances listed below:

Gravity, degrees API - plus or minus 1.0

Sulphur, percent - plus or minus 20 1/

Viscosity

Centistokes kinematic at 40°C (104°F) - plus or minus 8 2/

Centistokes kinematic at 100°C (212°F) - plus or minus 1 2/

Centistokes at 4.4°C (40°F), maximum - 870

Pour point, °C - plus 3 2/

Oxidation by rotating bomb - minus 15 percent maximum.

1/ For oils qualified below 0.15 percent sulfur: No minimum restriction.
Plus .08 percent allowable.

2/ Shall meet requirements of this specification (see table I).

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If base oil materials have changed from the sample tested at qualification (see 6.3.2(a)), the above sulfur tolerance does not apply and the load-carrying value shall be determined in addition to those quality conformance tests specified in 4.5.

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 supplies and services conform to prescribed requirements.

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

- (a) Qualification inspection (see 4.3).
- (b) Quality conformance inspection (see 4.5).

4.3 Qualification inspection. Qualification inspection shall be conducted at a laboratory satisfactory to NAVSEA. Qualification inspection shall consist of the tests specified in table I. A minimum sample size of 30 gallons is required for this testing.

4.4 Sampling.

4.4.1 Inspection lot.

4.4.1.1 At place of manufacture. For the purpose of sampling, a lot of lubricating oil shall consist of a manufacturer's batch. When material cannot be identified by batch, a lot shall consist of not more than 10,000 gallons.

4.4.1.2 At place of delivery. For the purpose of sampling, a lot of lubricating oil shall consist of all oil in a single shipment.

4.4.2 Sampling a storage tank. Unless otherwise specified (see 6.2), a representative sample of 5 gallons of lubricating oil shall be taken from each lot in accordance with ASTM D 270.

4.4.3 Sampling during loading. A sample shall be drawn at the discharge pipe where it enters the bulk carriers (tank cars, tank trucks and so forth). At least four samples shall be taken at regular intervals during the entire period of loading or filling, each sample being 1 pint.

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4.4.4 Sampling of filled containers.

4.4.4.1 Drums. Where the lubricating oil is contained in drums, samples shall be drawn in accordance with ASTM D 270, unless the lot consists of less than five drums, in which case one sample may be drawn from one drum. The contents of each drum to be sampled shall be shaken, rolled and stirred to mix the contents thoroughly. Equal portions shall be withdrawn from approximately the center of each drum by means of a thief or other sampling device.

4.4.4.2 Five-gallon cans. Where the lubricating oil is contained in 5-gallon cans, five samples shall be drawn in accordance with ASTM D 270. When the lot contains less than five containers, each container shall be sampled.

4.5 Quality conformance inspection. Each sample selected as specified in 4.4 shall be tested as specified in table I, except that the bearing compatibility, load carrying, wear oxidation, compatibility and valve sticking tests will not be required.

4.5.1 Rejection of lots. When the sample of lubricating oils fails any of the tests this shall be cause for rejection of the lot represented by the sample.

4.6 Inspection. Inspection shall be in accordance with method 9601 of FED-STD-791.

4.6.1 Examination of filled containers. A random sample of filled containers shall be selected from each lot in accordance with MIL-STD-105 at inspection level I and acceptance quality level (AQL) of 2.5 percent defective to verify compliance with this specification regarding fill, closure, marking, and other requirements not involving tests. Samples shall be examined for defects of the container and the closure, for evidence of leakage, and for unsatisfactory markings; each sample filled container shall also be weighed to determine the amount of the contents. Any container in the sample having one or more defects or under required fill shall be cause for rejection of the container, and if the number of defective containers in any sample exceeds the acceptance number for the appropriate sampling plan in accordance with MIL-STD-105, this shall be cause for rejection of the lot represented by the sample.

4.7 Test.

4.7.1 Homogeneity. After determining the pour point of the oil, return test jar and oil to cooling bath, cool to 6°C (10°F) below the pour point, and hold at that temperature for 3 hours. Remove jar of oil from cooling bath, and allow to warm to room temperature. Next place jar and oil in oil bath, bring temperature of bath to $121 \pm 2.8^\circ\text{C}$ ($250 \pm 5^\circ\text{F}$) and hold at test temperature for 1 hour. Remove jar of oil from oil bath, allow to cool in air to room temperature. Examine test oil visually for sediment or separation of insoluble material.

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4.8 Inspection of packaging. Sample packages and packs, and the inspection of the preservation-packaging, packing and marking for shipment and storage shall be in accordance with the requirements of section 5 and the documents specified therein.

5. PACKAGING

(The preparation for delivery requirements specified herein apply only for direct Government acquisition.)

5.1 Special requirements. Immediately before packaging, the lubricating oil shall be filtered. Before filling, all containers shall be thoroughly cleaned and inspected to insure absolute absence of dirt, corrosion products, water or other materials which would contaminate or interfere with satisfactory operation of the oil. Use of the zinc or cadmium containing materials in container areas which contact the lubricating oil is prohibited. This includes zinc plating and zinc phosphate coatings presently permitted as specified in PPP-D-729 and PPP-P-420 as well as all other container specifications as specified in MIL-STD-290. This lubricating oil is available in 1-gallon, 5-gallon and 55-gallon containers.

5.2 Packaging, packing and marking. The lubricating oil shall be packaged and packed for the level specified (see 6.2) and marked in accordance with MIL-STD-290. The type and size containers shall be as specified by the contracting activity (see 6.2).

6. NOTES

6.1 Intended use. This lubricant is intended for use in main and auxiliary turbines and gears, air compressors and certain hydraulic equipment, as well as general mechanical lubrication.

6.2 Ordering data. Acquisition documents should specify the following:

- (a) Title, number and date of this specification.
- (b) Type and size container required (see 5.2).
- (c) Packaging, packing and marking requirements other than those specified in 5.2 and 5.2.1.
- (d) Quantity. The material should be purchased by volume, the unit being a U.S. gallon at 15.6°C (60°F).

6.3 With respect to products requiring qualification, awards will be made only for products which are, at the time set for opening of bids, qualified for inclusion in Qualified Products List QPL-17331 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 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. The activity responsible for the Qualified Products List is the Naval Sea Systems Command, SEA 5523, Department of the Navy, Washington, DC 20362 and information pertaining to qualification of products may be obtained from that activity. Application for qualification tests shall be made in accordance with "Provisions Governing Qualification SD-6" (see 6.3.1).

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6.3.1 Copies of "Provisions Governing Qualification SD-6" may be obtained upon application to Commanding Officer, Naval Publications and Forms Center, 5801 Tabor Avenue, Philadelphia, PA 19120.

6.3.2 Request for tests of oil will be considered only under the condition that the contractor submits the following information regarding composition of each symbol of oil proposed for qualification:

- (a) Crude source, complete manufacturing process for each stock, and types of base or blending stocks and percentages used in the finished oil. Changes in base or blending stocks must be reported to and approved by the qualifying activity.
- (b) Type of additive agents, typical chemical analysis and percentages used in the finished oil.
- (c) Typical analysis of the blending stocks, the base oil, and the finished oil, including percentage of metallic elements contributed by the additive.

6.3.3 Each symbol of oil, rebrand or experimental, submitted for qualification should be designated by the exhibitor by a different name, number or letter to denote any changes in base oil components or the amount or type of additive present.

6.4 The index of test methods in FED-STD-791 lists the corresponding equivalent test methods of the ASTM.

6.5 International interest. Certain provisions of this specification are the subject of international standardization agreement NATO-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 reconciliation action through international standardization channels including departmental standardization offices to change the agreement or make other appropriate accommodations.

6.6 Changes from previous issue. Asterisks are not used in this revision to identify changes with respect to the previous issue, due to the extensiveness of the changes.

Preparing activity:
Navy - SH
(Project 9150-N581)

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APPENDIX A

QUANTITY OF SLUDGE AND AMOUNTS OF COPPER AND IRON IN
THE SLUDGE, OIL AND WATER AFTER 1000 HOURS

10. SCOPE

10.1 This appendix intends to outline the procedures designed to provide information on the quantity of sludge and the amounts of copper and iron in the sludge, oil and water after 1000 hours in accordance with ASTM D 943.

20. APPLICABLE DOCUMENTS

20.1 Other publications. The following document forms 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.

ASTM

D 91 - Precipitation Number of Lubricating Oils. (DoD adopted)

(Application for copies should be addressed to the ASTM, 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.)

30. PROCEDURES FOR SLUDGE

30.1 Apparatus. Filtration apparatus (two pieces) shall be easily disassembled so that filter paper can be removed for weighing. The apparatus shall consist of the following:

- (a) Laboratory vacuum line (18-21 millimeters (mm) mercury).
- (b) 5-micrometer porosity filter paper.
- (c) Drying oven, $79 \pm 1^{\circ}\text{C}$ ($175 \pm 2^{\circ}\text{F}$).
- (d) Petri dish with cover or any suitable covered container for transporting filter membranes.
- (e) Antistatic device.
- (f) Eye dropper.
- (g) Wash bottles for solvents.

30.2 Solvents. The solvents shall consist of the following:

- (a) Distilled water (filter through 5-micrometer paper).
- (b) Naphtha equal to ASTM naphtha as specified in ASTM D 91 (filter through 5-micrometer paper).

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APPENDIX A

30.3 Determination of sludge.

30.3.1 Assemble chemically clean and dry filter apparatus using 5-micrometer paper which has been water-washed, naphtha-washed, dried at 79°C (174°F) and weighed to constant weight. (Deionize balance pan by exposing it to anti-static device before each weighing). Place paper in the filtration apparatus.

30.3.2 Remove catalyst coils from oxidation test cell while washing down onto oil sample with naphtha. (This dilutes oil portion for better filtration). Filter entire test cell contents, oil, naphtha and water. Wash sludge on filter paper with several portions of naphtha to remove the oil. (This is determined by observing color of solvent after it passes through the filter paper).

30.3.3 Transfer filtrate to separatory funnel and separate oil and water layers. Dilute oil portion to known volume with naphtha and dilute water portion to known volume with water so that aliquots can be used to measure copper and iron in oil and water layer (see 40.2 and 40.3). Remove upper portion of filtration apparatus and gently wash edges of paper containing sludge. Place paper in loosely covered petri dish and dry at 79°C (174°F) until constant weight is obtained. Subtract weight of paper (see 30.3.1) and record result as the weight of insoluble sludge.

40. IRON AND COPPER CONTENT

40.1 Sludge. The entire sludge sample shall be ashed using the procedures in accordance with ASTM D 874 (weighing and calculating ash content is not necessary). The ash is dissolved in 5 milliliters (mL) of hydrochloric acid (HCl). Heating on a hot plate may be required. Transfer the acid solution to a 50 mL volumetric flask, dilute to volume with distilled water and retain for the atomic absorption determination of iron and copper in accordance with appendix B.

40.2 Oil portion. Take an aliquot of the oil-naphtha solution in accordance with 30.3.3. This aliquot can be retained and run in the atomic absorption in accordance with appendix D if standard solutions of iron and copper in organic solvents are available. Alternatively, solvent is evaporated from the aliquot, the residue ashed, and the ash dissolved in water as specified in 40.1 for sludge. The water is then analyzed in accordance with appendix B.

40.3 Water portion. Evaporate the entire water sample to dryness. It shall be dissolved in 5 mL of HCl (using heat and cover glass if necessary), diluted with distilled water to 50 mL in a volumetric flask and analyzed in accordance with appendix B.

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APPENDIX B

WATER SOLUBLE COPPER AND IRON IN THE ASHED SLUDGE, OIL AND
WATER OBTAINED FROM TURBINE OIL OXIDATION TESTING
(ATOMIC ABSORPTION METHOD)

10. SCOPE

10.1 Scope. This method is intended for the determination of copper and iron using an atomic absorption technique of the soluble copper and iron contained in the ashed sludge, oil and water after 1000 hours by ASTM D 943.

10.2 Summary of method. Aqueous solutions of the ashed sludge, ashed oil and the water in accordance with ASTM D 943 are aspirated into the flame of an atomic absorption spectrophotometer. The absorbances of the sample at the copper and iron wavelengths are compared with those of standard solutions whose copper and iron contents bracket those of the sample. Concentrations in the sample are then calculated by interpolation.

20. APPLICABLE DOCUMENTS

20.1 Other publications. The following document forms 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.

AMERICAN CHEMICAL SOCIETY (ACS)
Specifications - Reagent Chemicals

(Application for copies should be addressed to Applied Publications, American Chemical Society, 1155 16th Street, NW, Washington, DC 20036.)

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

30. APPARATUS

30.1 Atomic absorption spectrophotometer. The atomic absorption spectrophotometer shall operate with the following parameters:

- (a) Wavelength. 3247 angstroms (Å) for copper; 2483 Å for iron.
- (b) Source. Hollow cathode lamps for copper and iron.
- (c) Fuel. Acetylene (oxidizing flame).
- (d) Oxidizer. Air.

30.2 Hypodermic syringes. Hypodermic syringes shall have 5 and 10 mL capacity.

30.3 Volumetric flasks. Volumetric flasks shall have 25, 50 and 100 mL capacity.

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APPENDIX B

40. REAGENTS AND MATERIALS

40.1 Purity of reagents. Reagent grade chemicals shall be used in all tests. Unless otherwise specified, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

40.2 Purity of water. Unless otherwise specified, references to water shall be understood to mean distilled water or water of equal purity.

40.3 Stock copper solution. Dissolve copper nitrate in water in amounts to give a copper concentration of 1000 microgram/mL. The water should contain enough nitric acid (HNO_3) to make the solution slightly acid.

40.4 Copper standard. Dilute the stock copper solution 5:100 with water. This will give a copper concentration of 50 microgram/mL. This solution should be made daily as needed.

40.5 Copper working standard. Dilute the copper standard with water to obtain solutions containing 0, 10, 20, 30 and 40 microgram/mL copper.

40.6 Stock iron solution. Dissolve iron powder of known purity in dilute HCl and dilute with water to obtain an iron concentration of 1000 microgram/mL.

40.7 Iron standard. Dilute the stock iron solution 5:100 with water. This will give an iron concentration of 50 microgram/mL. This solution should be made daily as needed.

40.8 Iron working standards. Dilute the iron standard with water to obtain solutions containing 0, 10, 20, 30 and 40 micrograms/mL iron.

50. PROCEDURE

50.1 Using the appropriate light source, aspirate the 0.0 microgram/mL copper standard and adjust the instrument to give zero absorbance. Aspirate the sample and record absorbance. Aspirate two copper working standards selected to give absorbances higher and lower than that of the sample. Repeat the aspiration of standards and sample several times to average out absorbance variations. Record and average the absorbances obtained for the sample and standards.

50.2 Repeat in accordance with 50.1 using the light source and working standards appropriate for the determination of iron.

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APPENDIX B

60. CALCULATION

60.1 Calculate the concentration of copper in the sample as follows:

$$\text{Copper, microgram/mL} = L + \frac{(A_s - A_1)}{(A_u - A_1)} (U - L)$$

Where:

L = concentration of lower working standard, microgram/mL

U = concentration of upper working standard, microgram/mL

A₁ = absorbance of lower working standard

A_u = absorbance of upper working standard

A_s = absorbance of sample

60.2 Calculate the concentration of iron in the sample as specified in 60.1, substituting the absorbance values obtained with the iron source and working standards.

60.3 Convert concentrations of copper and iron obtained as specified in 60.1 and 60.2 to total copper and iron content of the sample by multiplying the concentrations by the total volume of the sample.

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APPENDIX C

VALVE-STICKING CHARACTERISTICS

10. SCOPE

10.1 Scope. This appendix outlines a procedure to determine the valve-sticking tendency of candidate oils when used with shipboard hydraulic control valves.

10.2 Summary. The procedure consists of measuring the actuation force required to shift the spool of shipboard hydraulic control valves after 24 hours of static contact with candidate fluid when tested as specified herein.

20. APPLICABLE DOCUMENTS

20.1 Government documents.

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

SPECIFICATION

MILITARY

MIL-C-81302 - Cleaning Compound, Solvent Trichlorotrifluoroethane.

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

30. MATERIALS AND APPARATUS

30.1 Apparatus. The test apparatus is shown schematically on figure 1. It consists of the following:

- (a) Sump with 5 gallon capacity.
- (b) Air operated piston pump capable of 1 liter/minute (L/min) capacity at 20.7 megapascals (MPa) (3000 pounds per square inch (lb/in²)).
- (c) Accumulator bladder type, 6.5 L capacity.
- (d) Flow control valves, eight, each 20.7 MPa (3000 lb/in²) rating.
- (e) Test valves, one each Bendix models 3191350-1 and 3188561-4.
- (f) Pressure control switch capable of maintaining 20.7 ± 1.7 MPa (3000 ± 250 lb/in²).
- (g) Pressure gage, 0-34.5 MPa (0-5000 lb/in²).
- (h) Enclosure capable of maintaining $38 \pm 1^\circ\text{C}$ ($100 \pm 1.8^\circ\text{F}$).

30.2 Materials. The materials shall consist of the following:

- (a) Test oil, 10 gallons meeting cleanliness requirement as specified in table I.
- (b) Solvent in accordance with MIL-C-81302.

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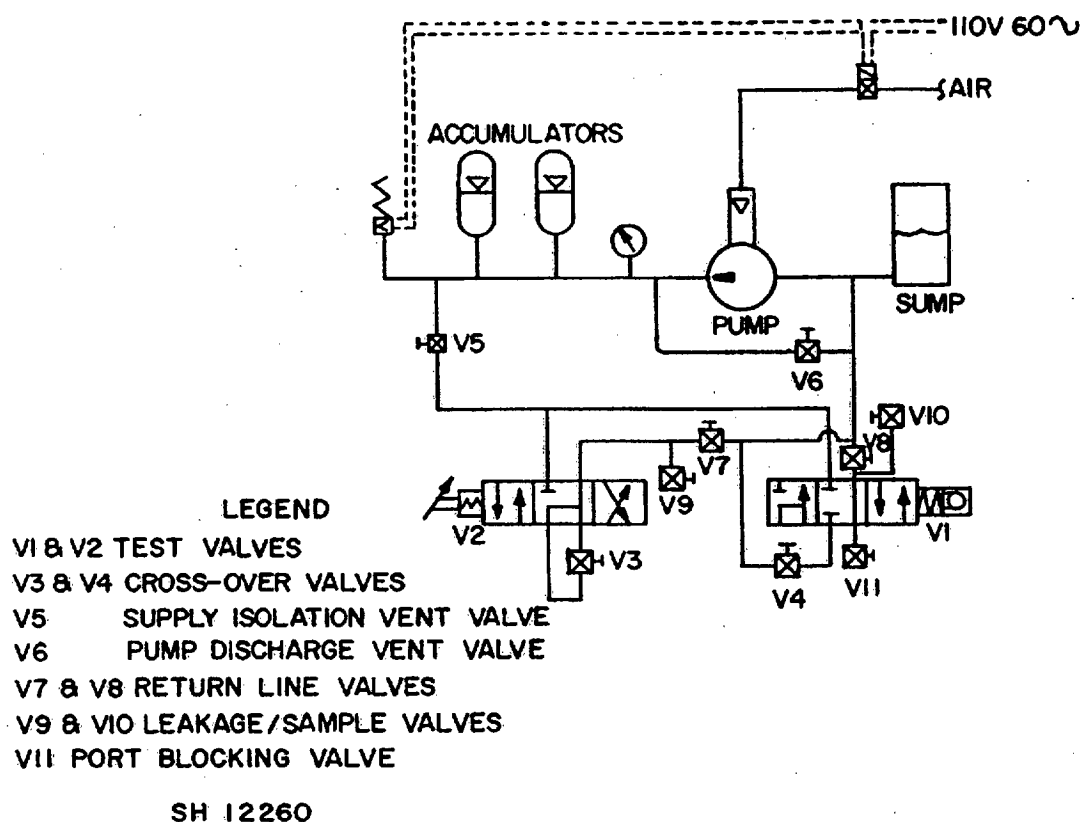


FIGURE 1. Hydraulic valve test loop.

40. TEST PROCEDURE

40.1 Test valve cleaning.

40.1.1 The pipe loop is disassembled at all take-down joints and the fluid allowed to drain. The test valves are disassembled, the spool and sleeve assemblies examined, cleaned by washing with precipitation solvent in accordance with MIL-C-81302, wiping with a chloroform saturated lint-free tissue to remove any naphtha insolubles, and rinsing again with naphtha. Adequate ventilation and fire precautions are necessary. Following cleaning, the valves are assembled according to the manufacturer's specifications and the spool stroke is adjusted to proper stroke in each direction.

40.1.2 Sump cleaning. The sump is drained of oil and rinsed four times with solvent in accordance with MIL-C-81302 and air dried.

40.2 System flushing.

40.2.1 Following assembly of all system joints and installation of the test valves, 10 L of the test oil is placed in the sump. Close the supply valve, V5; open pump discharge vent valve, V6, to the sump. Start pump and purge air from accumulators and system lines. Secure pump.

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40.2.2 Close valve V6 and open valves V3, V4, V5, V7 and V8. Test valve V1 has through flow (opened) only when it is shifted to one of its extreme positions. This is the same direction the valve is pulled while making the valve actuating force determinations. With test valve V1 in the open position and test valve V2 centered, start the pump. Adjust system pressure to 3.4 MPa (500 lb/in²) pressure using valve V4. Cycle test valve V1 ten times. Test valve V2 has through flow (opened) when in either extreme position. With test valve V2 in one of its extreme positions and test valve V1 centered, the system pressure is adjusted to 3.4 MPa (500 lb/in²), using valve V3. Cycle test valve V2 ten times. A valve cycle is described by starting at the center position, moving the spool to one extreme position, and reversing to the other extreme position, and reversing again to recenter the spool.

40.2.3 Center test valves V1 and V2, close valves V3 and V4, and charge accumulators to 10.3 MPa (1500 lb/in²) with pump. Secure pump. Cycle test valve V1, dropping system pressure 1.4 MPa (200 lb/in²) per cycle until 0 MPa (0 lb/in²) is reached. Valve V4 is used to control the rate of pressure drop. Recharge accumulators and repeat for test valve V2, using valve V3 to control the rate of pressure drop. The same number of cycles are used for each test valve.

40.2.4 Repeat procedure as specified in 40.2.3 above except beginning with a system pressure of 20.7 MPa (3000 lb/in²).

40.2.5 Close valve V5, start pump and charge system to 20.7 MPa (3000 lb/in²), secure pump, and discharge fluid in accumulators through valve V6. Disassemble all system takedown joints and drain sump and system.

40.3 Test operation.

40.3.1 Assemble system joints and charge sump with 12 L of test oil.

40.3.2 Purge system of air as specified in 40.2.1 above.

40.3.3 Close valves V6, V7, V8, and open valves V3, V4, V6, V9 and V10. Start pump and slowly cycle each test valve (V1 and V2) three times to purge air from the system. Secure pump, close valves V9 and V10 and open valves V7 and V8.

40.3.4 Repeat procedure as specified in 40.2.2, except cycle test valves V1 and V2 five times, maintaining system pressure at 3.4 MPa (500 lb/in²). Secure pump and reduce pressure to 0 MPa (0 lb/in²) through valve V6.

40.3.5 Measure each test valve actuation force at 0 MPa (0 lb/in²), returning spool to the center position each time. Repeat for each test valve until three consecutive measurements are within a 0.02 kilogram (kg) (0.5 pounds) range which does not exceed 4.5 kg (10 pounds).

40.3.6 Center test valves V1 and V2, close valves V3, V4, V7 and V8, and open valves V9 and V10. Start pump and charge accumulators to 20.7 MPa (3000 lb/in²).

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40.3.7 Repeat procedure as specified in 40.3.5 at 20.7 MPa (3000 lb/in²).

40.3.8 Center test valve spools, bring the system to test temperature of $38 \pm 1^\circ\text{C}$ ($100 \pm 1.8^\circ\text{F}$), and allow valves to remain at these conditions undisturbed for 24 hours. Measure valve fluid leakage rates during the first 2 hours and at end of the 24 hour period.

40.3.9 After the 24-hour period, measure actuation force for each test valve (V1 and V2) by pulling on valve spool. Complete valve cycle to return the spool to center position.

40.3.10 Close valves V9 and V10, open valves V7 and V8, and secure pump. Using valve V4 to control rate of pressure drop through test valve V1 and valve V3 to control rate of pressure drop through V2, reduce system pressure to 0 MPa (0 lb/in²) by alternately cycling each test valve (V1 and V2) three times, and allowing a 2.8 MPa (400 lb/in²) pressure drop for each cycle. Open valve V6.

40.3.11 Measure actuation force for each test valve at 0 MPa (0 lb/in²).

40.3.12 Repeat steps as specified in 40.3.4 through 40.3.11 two times, except in 40.3.4 each valve will be cycled ten times at 3.4 MPa (500 lb/in²) in lieu of five times.

50. RATING

50.1 A pass rating is given to a candidate oil when the initial actuation force after the 24-hour aging for each test valve (see 40.3.9) averages 9 kg (20 pounds) or below with no individual initial actuation force exceeding 11 kg (25 pounds).

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APPENDIX D

ALTERNATE METHOD FOR
COPPER AND IRON IN TURBINE OIL BY ATOMIC ABSORPTION

10. SCOPE

10.1 Scope. This method is intended for the determination of copper and iron using an atomic absorption technique, of copper and iron in filtered turbine oil following its testing in accordance with ASTM D 943. Sensitivity of the method (for 1 percent absorption) is about 0.15 microgram/mL for each metal.

10.2 Summary of method. An aliquot of the filtered turbine oil is diluted with xylene and aspirated into the flame of an atomic absorption spectrophotometer, sequentially using appropriate hollow cathode lamp sources for copper and iron. Concentrations of copper and iron are determined by the method of standard additions of a series of known amounts of the metals to the sample and the plotting of response versus amounts added. Extrapolation of the plot back to zero response then gives a measure of the materials originally present before the additions.

20. APPLICABLE DOCUMENTS

20.1 Other publications. The following document forms 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.

AMERICAN CHEMICAL SOCIETY (ACS)
Specifications - Reagent Chemicals

(Application for copies should be addressed to Applied Publications, American Chemical Society, 1155 16th Street, NW, Washington, DC 20036.)

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

30. APPARATUS

30.1 Atomic absorption spectrophotometer. The atomic absorption spectrophotometer shall operate with the following parameters:

- (a) Wavelength. 3247 Angstroms (Å) for copper; 2483 Å for iron.
- (b) Source. Hollow cathode lamps for copper and iron.
- (c) Fuel. Acetylene (oxidizing flame).
- (d) Oxidizer. Air.

30.2 Hypodermic syringes. Hypodermic syringes shall have 5 and 10 mL capacity.

30.3 Volumetric flasks. Volumetric flasks shall have 25, 50 and 100 mL capacity.

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40. REAGENTS AND MATERIALS

40.1 Purity of reagents. Reagent grade chemicals shall be used in all tests. Unless otherwise specified it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

40.2 Purity of water. Unless otherwise specified, references to water shall be understood to mean distilled water or water of equal purity.

40.3 Stock copper solution. Transfer approximately 0.5 grams of NBS Standard 1056, copper cyclohexanecarboxylate, to a small beaker and dry over fresh phosphorus pentoxide (P_2O_5) in a desiccator for 2 hours. Quickly and accurately transfer 0.313 grams (equivalent to 50 mg of copper) of the dried salt to a weighed 200 mL Erlenmeyer flask. Add 2 mL of xylene and 4 mL of 2 ethyl hexylamine; and, while swirling and without charring, heat the flask on a hot plate until a clear solution is obtained. Add 2 mL of 2 ethylhexanoic acid. Add 80 to 90 mL of nonadditive lubricating oil to the hot solution and gently shake the flask to mix the contents. Allow the flask to cool to room temperature and add enough oil to bring the total weight of the flask contents to 100 ± 0.5 grams. Stopper the flask and gently shake to insure a homogeneous solution. The concentration of copper in this solution is 500 microgram/gram. Alternatively, prepared metal-organo standards may be purchased and diluted with white oil to obtain the desired 500 microgram/mL concentrations as specified in 40.3 and 40.5.

40.4 Copper standard. Dilute the stock copper solution 1:10 with xylene. This will give a nominal copper concentration of 50 microgram/mL.

40.5 Stock iron solution. Transfer approximately 0.6 gram of NBS Standard 1079B, tris (1, phenyl-1, 3-butanedione) iron, to a small beaker and dry over fresh P_2O_5 in a desiccator for 2 hours. Quickly and accurately transfer 0.478 gram of this salt to a weighed 200 mL Erlenmeyer flask. Add 3 mL of xylene and 4 mL of 2 ethylhexanoic acid and heat the flask on a hot plate, while swirling and without charring, until the compound dissolves. Add to the hot solution 80 to 90 mL of iron and copper free lubricating oil and gently shake the flask to mix the contents. Allow the flask to cool to room temperature and add enough oil to bring the total weight of the contents of the flask to 100 ± 0.5 grams. Stopper the flask and shake gently to insure a homogeneous solution. The concentration of iron in this solution is 500 microgram/grams.

40.6 Iron standard. Dilute the stock iron solution 1:10 with xylene. This will give a nominal iron concentration of 50 microgram/mL.

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50. PROCEDURE

50.1 Determine the total volume of filtered turbine oil, either by actual measurement or by dilution with naphtha to known volume.

50.2 Using a 5 mL syringe without needle, put 5 mL aliquots of the turbine oil into five 25 mL volumetric flasks. To the series of flasks, add 0.00, 0.25, 0.50, 1.0 and 1.5 mL of the nominally 50 microgram/mL copper standard solution. To the same series of flasks and in the same order, add 0.00, 0.25, 0.50, 1.0 and 1.5 mL of the nominally 50 microgram/mL iron standard solution. Dilute each to 25 mL with xylene. This procedure results in a five times dilution of the turbine oil with 0.0, 0.5, 1.0, 2.0 and 3.0 microgram/mL of added copper and iron.

50.3 Burn xylene for 2 minutes in the spectrophotometer. Aspirate each sample for 1.5 minutes at each wavelength and record signal while aspirating. Burn xylene for 1.5 minutes between each sample.

50.4 For each metal, plot sample signal as the ordinate versus the corresponding standard metal added, in microgram/mL, as the abscissa. Draw the best fit straight line through the points and extend it to pass through the abscissa. The distance of this intercept from the ordinate is equivalent to the concentration of the metal whose signal has been plotted. Plot the data for each metal in this fashion.

50.5 Multiply the concentrations of copper and iron as specified in 50.4 by 5 to obtain their concentrations in the filtered oil.

60. CALCULATION

60.1 Calculate the total copper and iron content of the turbine oil, in mg, as follows:

- (a) Total copper, mg = $AC/1000$
- (b) Total iron, mg = $BC/1000$

Where:

- A = concentration of copper in the filtered oil, microgram/mL,
- B = concentration of iron in the filtered oil, microgram/mL and
- C = total volume of filtered turbine oil.