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METRIC
DOD-PRF-24574(SH)
29 September 1978

PERFORMANCE SPECIFICATION

LUBRICATING FLUID FOR LOW AND HIGH PRESSURE OXIDIZING GAS SYSTEMS

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

1. SCOPE

1.1 Scope. This specification covers lubricating fluid for use in oxidizing gas systems, including oxygen and compressed air. The lubricating fluid is intended for use with oxygen systems at all pressures, and for air and oil-free nitrogen systems at pressures above 10,300 kilopascals (kPa) (1500 pounds per square inch (lb/in²)).

1.2 Classification. Based on viscosity, volatility, and other physical properties, the lubricating fluid shall be of the following types, as specified (see 6.1 and 6.2.1):

Type I - for use at temperatures from -46°C (-50°F) to 0°C (32°F) (see 6.1.1).

Type II - for use at temperatures from -20°C (-4°F) to 40°C (104°F) (see 6.1.2).

Type III - for use at temperatures from 20°C (68°F) to 70°C (158°F) (see 6.1.3).

2. APPLICABLE DOCUMENTS

2.1 Issues of documents. The following documents, of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

SPECIFICATIONS

MILITARY

MIL-M-20693 - Molding Plastic, Polyamide (Nylon), Rigid.

MIL-G-23652 - Gasket and Packing Material Petroleum and Phosphate Ester Fluid Resistant.

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, drawings, and publications 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. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, Naval Ship Engineering Center, SEC 6124, 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.

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AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

- D 92 - Flash and Fire Points by Cleveland Open Cup
- D 97 - Pour Point of Petroleum Oils
- D 130 - Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test, Detection Of
- D 270 - Petroleum and Petroleum Products, Sampling For
- D 287 - API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method), Test for
- D 445 - Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity), Test for
- D 665 - Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water, Test for.
- D 892 - Foaming Characteristics of Lubricating Oils, Test for.
- D 974 - Neutralization Number by Color-Indicator Titration.
- D 1160 - Distillation of Petroleum Products at Reduced Pressures.
- D 1218 - Refractive Index and Refractive Dispersion of Hydrocarbon Liquids
- D 1500 - ASTM Color of Petroleum Products (ASTM Color Scale).
- D 2619 - Hydrolytic Stability of Hydraulic Fluids (Reverage Bottle Test Method)

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa 19103)

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

3. REQUIREMENTS

3.1 Qualification. Fluid lubricants for oxidizing gas systems 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 Composition. The composition of these fluids is not specified. However, in order to meet the ignition temperature test (see appendix to this specification) and other requirements, it shall be necessary to provide fully halogenated products in which all the hydrogen has been replaced with fluorine, or fluorine and chlorine. It shall also be necessary to have at least three times as many fluorine atoms as chlorine atoms in the polymer unit molecular structure. These fluids shall be of the generic type chlorotrifluoroethylene polymer, or be fully compatible and soluble with chlorotrifluoroethylene polymers.

3.3 Physical and chemical characteristics. Lubricating fluids shall conform to the requirements of table I for the type specified.

TABLE I. Physical and chemical characteristics.

Property	Requirement		Type III	Test method
	Type I	Type II		
Kinematic viscosity.				
Cs (mm ² /s) @ 100°C (212°F)	1.0-2.0	2.5-4.0	12.0-20.0	ASTM D 445
Cs (mm ² /s) @ 40°C (104°F)	4.0-5.0	13 - 35	100 - 450	
Cs (mm ² /s) @ -20°C (-4°F)	110 max	---	---	
Pour point, °C, (°F), max.	-62 (-80)	-20 (-4)	0 (32)	ASTM D 97
Distillation at reduced pressure			133(268)	ASTM D 1160
Vapor temperature, 1 mm Hg pressure, 1 percent, °C (°F), min	95(95)	80(176)	1	ASTM D 130
Corrosion test at 100°C (212°F), max rust protection,	1	1	pass	ASTM D 665, Proc A ² / ₁
Flash point, COC, °C (°F), min	pass	pass	pass	ASTM D 92 ³ / ₁
Fire point, COC, °C (°F), min	none	none	none	ASTM D 92
Autogenous ignition temperature	none	none	none	ASTM D 92
in oxygen at 10,300 kPa (1500 lb/in ²), °C (°F), min	350(662)	350(662)	350(662)	See appendix and 4.6.1
Wear test, scar diameter, mm, max	0.5	0.5	0.5	TM 6503 ¹ / ₁ ⁵ / ₄
Refractive index, Nd @ 20°C (68°F)	report ⁶ / ₁	report ⁶ / ₁	report ⁶ / ₁	ASTM D 1218 ⁴ / ₁
Specific gravity @ 15.6°C (60°F)	report ⁶ / ₁	report ⁶ / ₁	report ⁶ / ₁	ASTM D 287 ⁴ / ₁
Hydrolytic stability:				ASTM D 2619
Copper specimen wt loss, mg/cm ²	0.3	0.3	0.3	
Fluid acid No. increase, mg KOH/g	0.2	0.2	0.2	
Water acid No. increase, mg KOH/g	5.0	5.0	5.0	
Insoluble, percent max	0.5	0.5	0.5	
Color number, ASTM	report ⁶ / ₁	report ⁶ / ₁	report ⁶ / ₁	ASTM D 1500 ⁴ / ₁
Neutralization number, mg KOH/g, max	0.5	0.5	0.5	ASTM D 974
Compatibility	pass	pass	pass	see 4.6.2
Foaming characteristics:				ASTM D 892
Sequence 1	300	300	300	
Sequence 2	25	25	25	
Sequence 3	300	300	300	
Toxicity	pass	pass	pass	see 4.7

¹/ FED-STD-791 test method.²/ Use procedure for fluids heavier than water.³/ Use hood. Start testing at 150°C(302°F) or 30°C(86°F) below boiling, no flash or fire below 400°C (752°F).⁴/ No limiting requirement, test made for record.⁵/ Load - 15 Kg oil temp. 80°C (176°F); speed 600 r/min, duration 120 minutes.⁶/ See 4.8.

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3.4 Toxicity. The lubricating fluid shall have no adverse effect on the health of personnel when used for its intended purpose (see 4.7 and 6.1).

3.5 Lot acceptance requirements. Each sample selected in 4.5 when tested as specified in 4.3 and 4.4 shall meet the requirements of table I. In addition, the refractive index and specific gravity of lot to lot shall be within 0.005 units and 2.0 percent of the average.

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 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 inspections.

- (a) Qualification inspection (see 4.3).
- (b) Quality conformance inspection (see 4.4).

4.3 Qualification inspection. Qualification inspection shall be conducted at a laboratory satisfactory to the Naval Ship Engineering Center. Qualification inspection shall consist of the tests specified in table I.

4.3.1 Sampling for qualification tests. The qualification sample shall consist of 8 liters (L) (2 gallons) of lubricating fluid which shall be forwarded to a laboratory satisfactory to the Naval Ship Engineering Center for qualification test purposes.

4.4 Quality conformance inspection. Quality conformance inspection shall consist of the tests specified in table I, except that the corrosion, rust protection, wear, hydrolytic stability, compatibility, foaming characteristics, and autogenous ignition tests shall not be required.

4.5 Sampling and inspection.

4.5.1 At place of manufacture.

4.5.1.1 Lot and batch. A lot of lubricating fluid shall consist of a manufacturer's batch. When material cannot be identified by batch, a lot shall consist of not more than 350 kilograms (kg) (800 pounds (lbs)), and shall be from a single mixing container.

4.5.1.2 Sampling. A representative sample of .4L (1 gallon) of lubricating fluid shall be taken from each lot in accordance with ASTM D 270 for quality conformance testing.

4.5.2 At place of delivery.

4.5.2.1 Lot. A lot of lubricating fluid shall consist of all the fluid in a single shipment.

4.5.2.2 Sampling of filled containers. Where the lubricating fluid is contained in containers larger than 1 liter (1 quart), these containers shall be sampled by mixing thoroughly and withdrawing 1 liter (1 quart) from at least five containers, or all the containers, if fewer than five. However, the total sample drawn shall be at least 4L (1 gallon) for quality conformance testing.

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

4.5.3.1 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 acceptable quality level (AQL) = 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 of MIL-STD-105, this shall be cause for rejection of the lot represented by the sample.

4.5.3.2 Rejection of lots. If any sample of lubricating fluid fails any of the tests, this shall be cause for rejection of the lot represented by the sample.

4.6 Test procedures. Test procedures shall be in accordance with table I, with the following additions:

4.6.1 Autogenous ignition temperature (see appendix to this specification).

4.6.2 Compatibility. Each manufacturer's product shall be mixed with three (or all, if less than three) other manufacturers' products in clean glass containers (50:50), and stored at room temperature at $60^{\circ}\text{C} + 3^{\circ}\text{C}$ ($140^{\circ}\text{F} + 5^{\circ}\text{F}$) for seven days. Any precipitate or cloud shall be evidence of incompatibility. Also, specimens of fluorocarbon rubber in accordance with MIL-G-23652, type I or II, and specimens of polyamide in accordance with MIL-M-20693, composition A, shall be immersed in portions of the sample and held at 60°C (140°F) for seven days. Softening or swelling of the specimens shall constitute evidence of incompatibility.

4.7 Toxicity. A manufacturer of material shall disclose the formulation of his product to the Navy Bureau of Medicine and Surgery, Navy Department, Washington, DC 20372. The disclosure of proprietary information, which will be held in confidence by the Bureau of Medicine and Surgery, shall include the name, formula, and approximate percentage by weight and volume of each ingredient in the product, the results of any toxicological testing of the product; identification of its pyrolysis products, and any such other information as may be needed to permit an accurate appraisal of any toxicity problem associated with the handling, storage, application, use, or disposal of the material.

4.8 Certificate of compliance. The contractor shall prepare a certificate of compliance in accordance with the data ordering document included in the contract (see 6.2.2).

4.9 Inspection of preparation for delivery. Packaging, packing, and marking shall be examined to determine conformance with the requirements of section 5 of this specification.

5. PREPARATION FOR DELIVERY

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

5.1 General requirements. Before filling, all containers shall be thoroughly cleaned and inspected to insure the absence of residue from the container manufacturing process, dirt, corrosion products, water, or other materials which would contaminate or interfere with the satisfactory operation of the oil.

5.2 Packaging, packing, and marking. The lubricating fluid in the quantity and container specified shall be packaged level A, B, or "commercial", packed level A, B, or "commercial", and marked in accordance with MIL-STD-290 (see 6.2.1)

6. NOTES

6.1 Intended use.

6.1.1 Type I lubricating fluid is intended for use where a moderate viscosity is required at operating temperatures from -46°C (-50°F) to 0°C (32°F), for example, as a crankcase lubricant in compressors for low temperature liquid air distillation plants.

6.1.2 Type II lubricating fluid is intended for use where this viscosity is required at operating temperatures from -20°C (-4°F) to 40°C (104°F), for example, as a compressor lubricant within the prescribed temperature range, such as with liquid oxygen transfer pumps.

6.1.3 Type III lubricating fluid is intended for use where this viscosity is required at operating temperatures from 20°C (68°F) to 70°C (158°F), for example, as a crankcase lubricant in nominal temperatures within the prescribed temperature range.

6.2 Ordering data.

6.2.1 Procurement requirements Procurement documents should specify the following

- (a) Title, number, and date of this specification.
- (b) Type of fluid required (see 6.1 and 1.2).
- (c) Level of packaging and packing required (see 5.2).
- (d) Quantity. The material should be purchased by volume, the unit being 3.785L (1 gallon) at 15.6°C (60°F), or a specific number of standard size containers (see MIL-STD-290).

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6.2.2 Data requirements. When this specification is used in a procurement which invokes the provision of the "Requirements for Data" of the Armed Services Procurement Regulations (ASPR), the data identified below, which are required to be developed by the contractor, as specified on an approved Data Item Description (DD Form 1664), and which are required to be delivered to the Government, should be selected and specified on the approved Contract Data Requirement List (DD Form 1423) and incorporated in the contract. When the provisions of the "Requirements for Data" of the ASPR are not invoked in a procurement, the data required to be developed by the contractor and required to be delivered to the Government should be selected from the list below and specified in the contract.

<u>Paragraph</u>	<u>Data requirements</u>	<u>Applicable DID</u>	<u>Option</u>
4.8	Certificate of compliance	DI-E-2121	

(Copies of data item descriptions required by the contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.)

6.2.2.1 The data requirements of 6.2.2 and any task in section 3, 4, or 5 of the specification required to be performed to meet a data requirement may be waived by the procuring/purchasing activity upon certification by the offeror that identical data were submitted by the offeror and accepted by the Government under a previous contract for identical item procured to this specification. This does not apply to specific data which may be required for each procurement regardless of whether an identical item has been supplied previously (for example, test reports).

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 the applicable Qualified Products List QPL 24574 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 orders for the products covered by this specification. The activity responsible for the Qualified Products List is Naval Ship Engineering Center, 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).

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, Pennsylvania 19120.

Preparing activity:
Navy - SH
(Project 9150-N496)

APPENDIX

DETERMINATION OF AUTOGENOUS IGNITION TEMPERATURE

AT HIGH PRESSURE

10. SCOPE

10.1 This appendix specifies the procedure for determining the high pressure autogenous ignition temperature (AIT) of lubricating fluids for low and high pressure oxidizing gas systems.

20. APPLICABLE DOCUMENTS

20.1 Issues of documents. The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

SPECIFICATION

FEDERAL

BB-O-925 - Oxygen, Technical, Gas, and Liquid.

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

30. EQUIPMENT

30.1 Suitable components shall be assembled so that the specified reaction vessel (bomb), including sample holding assembly, can be charged with oxygen and heated, and a record kept of the time and temperature at which ignition occurs. One possible assembly is illustrated on figure 1.

30.2 The reaction vessel shall be the American Instrument Company (AMINCO) Model No. 41-12555. The thermocouple assembly shall be AMINCO No. 45-17620.

30.3 The sample holding assembly shall be as shown on figure 2. The test tube stopper is fabricated from borosilicate glass tubing of such a size that it just fits inside the larger test tube, as shown on figure 3. The supporting spring is wound from B and S 22-24 gauge nickel-chromium resistance wire by forming several turns on a mandrel of such a size that the resulting spring fits snugly over the larger test tube. The support loop is formed so as to hold the test tube at the proper height in the bomb, for example, so that the sample and thermocouple tip are roughly equidistant from the center of the zone heated by the heating jacket. The thermocouple tip should be 15 ± 2 millimeters (mm) inside the top of the small, inner test tube. The entire assembly is hung from the bomb cover by a bushing in the bomb cover.

40. PROCEDURE

40.1 Clean all components of the assembly. Glass parts should be soaked in chromic acid cleaning solution, rinsed in distilled water, and dried. Metal components should be clean and bright. They should be rinsed in trichlorotrifluoroethane and/or acetone, polished with steel wool, and re-rinsed with solvents. However, all traces of steel wool must be eliminated, since it is highly flammable.

40.2 Weigh out a 0.20 ± 0.03 gram(g) sample into the inner culture tube.

40.3 Assemble the equipment as shown on figures 1 and 2, and as directed by the bomb manufacturer.

40.4 Flush the bomb twice with oxygen meeting the requirements of BB-O-925, type I, by pressurizing to 5000 kPa (725 lb/in^2), and releasing the pressure.

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40.5 Fill the bomb with the above specified oxygen to a pressure of 11,500 kPa (1650 lb/in²) and allow to stand at room temperature for 15 minutes. The pressure will drop about 300 kPa (45 lb/in²) during the first 5 minutes while the gas cools, but should be nearly constant thereafter. Any additional steady pressure drop indicates a leak in the system which should be corrected before proceeding. After assuring the absence of leaks, the pressure should be adjusted to 10,300 kPa (1500 lb/in²).

40.6 Heat the bomb at a rate of 5°C ± 1°C (9°F ± 2°F) per minute. It will take a while for the heating rate to become steady, but the specified rate should be maintained from 60°C to 260°C (140°F to 500°F). Above 260°C (500°F), it may be difficult to maintain this heating rate, but it should not drop below 3°C (5°F) per minute.

40.7 Ignition of the sample is indicated by a very rapid temperature rise of at least 20°C (36°F). As soon as ignition is complete, but not less than 3 minutes after it starts, turn off the heater and recorder, and release the bomb pressure under a laboratory-type hood, some decomposition products are corrosive, irritating, or even poisonous.

40.8 If no ignition occurs below the maximum safe operating temperature of the bomb (425°C) (800°F), stop heating and report as above 425°C (800°F).

40.9 Report the sample size, initial and final gas pressure, and the ignition temperature.

50. LIMITATIONS

50.1 Occasionally, the decomposition and oxidation of fully fluorinated materials releases so little energy that there is no clear-cut indication of ignition. Also, if the sample distills into other parts of the bomb before the ignition temperature is reached, there will be no clear indication of ignition. Neither of these situations will arise with the chlorotrifluoroethylene polymers desired under this specification.

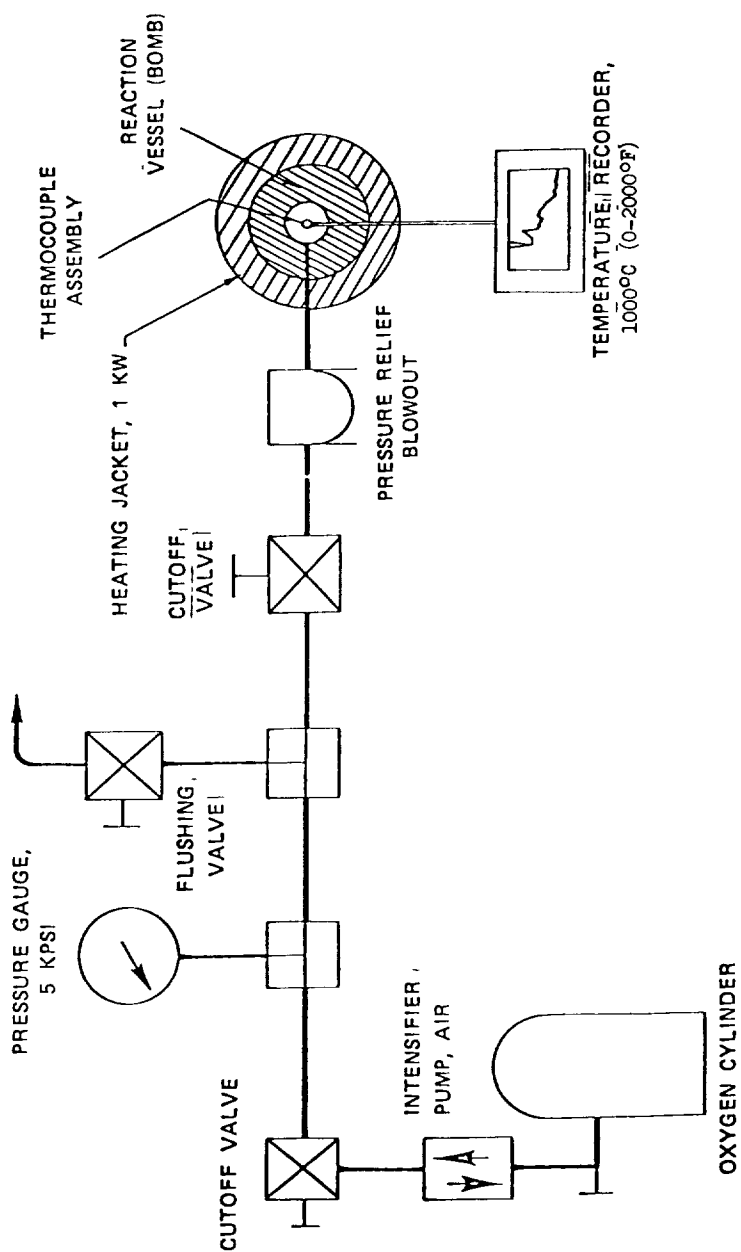


FIGURE 1. AIT equipment assembly.

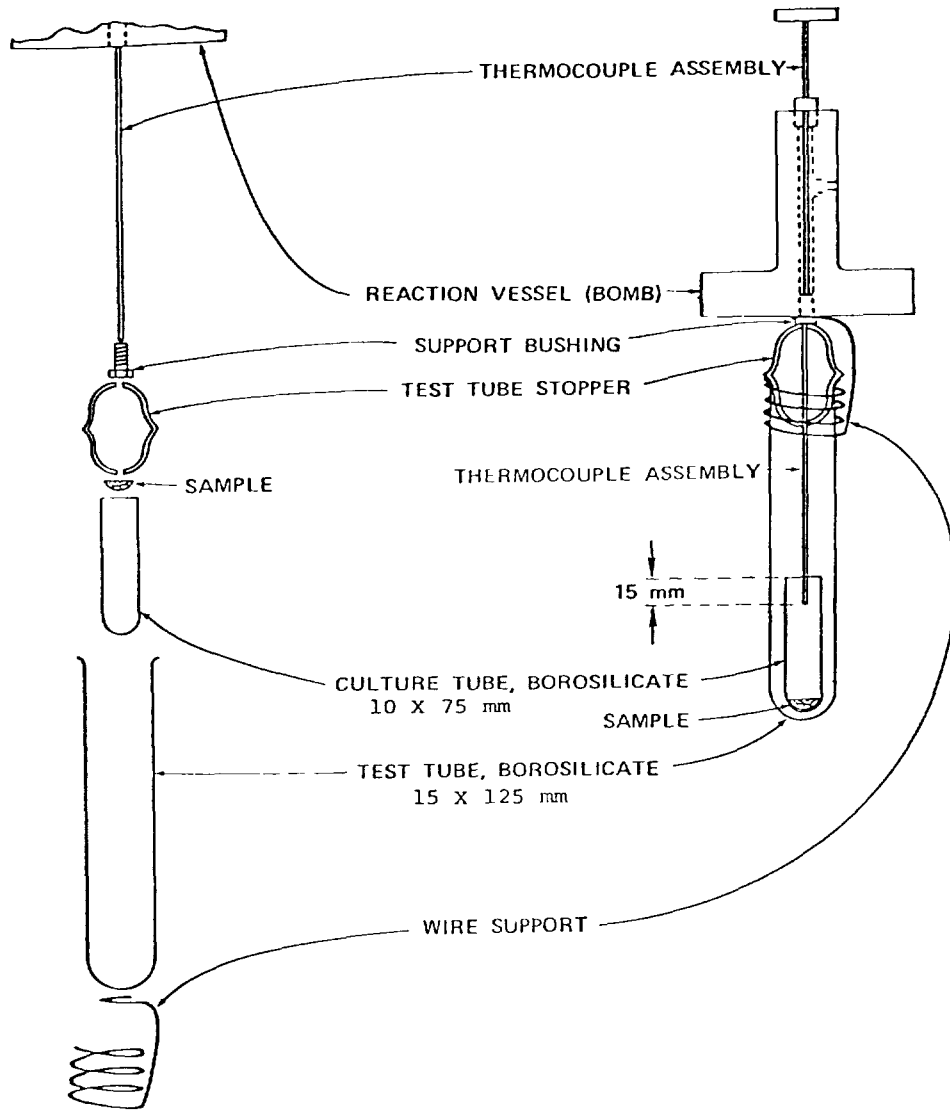


FIGURE 2. Sample holding assembly.

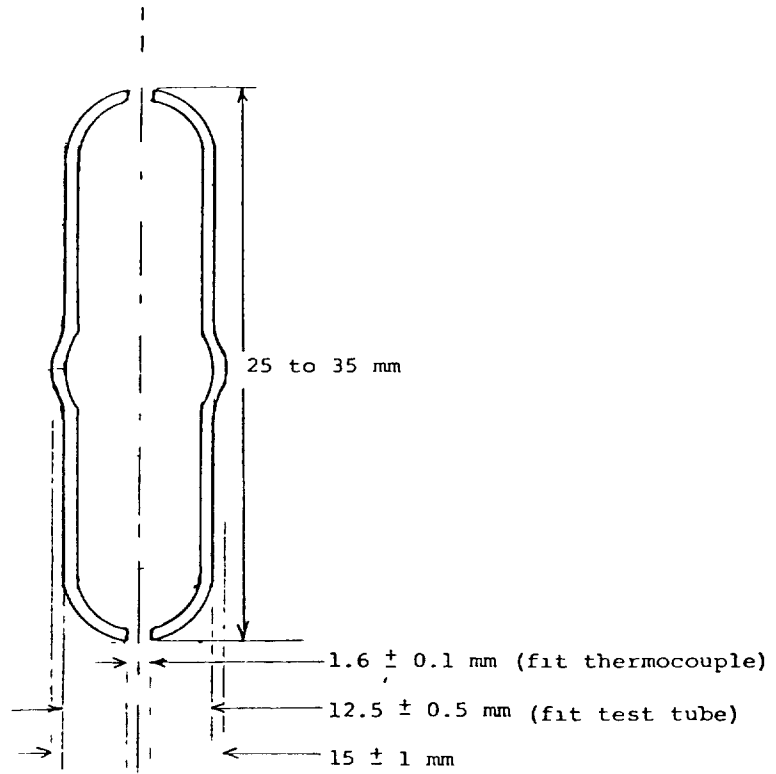


FIGURE 3. Test tube stopper.

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