

MIL-E-199A

25 MAY 1962

SUPERSEDING

JAN-E-199

1 APRIL 1945

MILITARY SPECIFICATION

ETHER, DIETHYL, TECHNICAL

This specification has been approved by the Department of Defense and is mandatory for use by the Departments of the Army, the Navy, and the Air Force.

1. SCOPE

1.1 This specification covers one grade of ether, diethyl as specified in the contract or order (see 6.2).

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

FEDERAL

- PPP-C-96 — Cans, Metal, 28 Gage and Lighter.
- PPP-D-729 — Drums, Metal, 55 Gallon for Shipment of Non-Corrosive Material.

MILITARY

- MIL-A-463 — Alcohol, Ethyl (For Ordnance Use).

STANDARDS

FEDERAL

- FED. TEST METHOD STD-141 — Paints, Varnish, Lacquer and Related Materials. Methods of Inspection, Sampling and Testing.

MILITARY

- MIL-STD-105 — Sampling Procedures and Tables for Inspection by Attributes.
- MIL-STD-109 — Inspection Terms and Definitions.
- MIL-STD-129 — Marking for Shipment and Storage.

PUBLICATIONS

ORDNANCE CORPS

- ORD-M608-11 — Procedures and Tables for Continuous Sampling by Attributes.

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(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 document forms a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date on invitation for bids shall apply.

CODE OF FEDERAL REGULATIONS

49-CFR 71-90 — Interstate Commerce Commission Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles.

(The Interstate Commerce Commission Regulations are now a part of the Code of Federal Regulations (1949 Edition-Revised 1956) available from the Superintendent of Documents, Government Printing Office, Washington 25, D.C. Orders for the above publication should cite "49 CFR 71-90 (Rev. 1956)".

3. REQUIREMENTS

3.1 Material. Diethyl ether shall be made from ethyl alcohol conforming to the requirements for grade 1 or grade 2 of Specification MIL-A-463, or diethyl ether shall be made as a co-product in the manufacture of ethyl alcohol from ethylene.

3.2 Appearance. Diethyl ether shall be clear and colorless when tested as specified in 4.3.1.

3.3 Chemical and physical properties. The diethyl ether shall conform to the chemical and physical requirements as specified in table I. When tested as specified in the applicable test method paragraphs.

TABLE I. Chemical and Physical Requirements

Properties	Test method
Specific gravity at 20/20 degrees centigrade (°C.)	0.712 to 0.723 4.3.1.2

Nonvolatile residue, percent by weight	0.002	4.3.1.3
Acidity as acetic acid, percent	0.006	4.3.1.4
Acetylene, percent	0.001	4.3.1.8
Peroxides	None	4.3.1.5
Chlorides	None	4.3.1.6
Aldehydes	None	4.3.1.7

4. QUALITY ASSURANCE PROVISIONS

4.1 General quality assurance provisions. The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examinations and tests shall be kept complete and available to the Government as specified in the contract or order. 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. Reference shall be made to Standard MIL-STD-109 in order to define the terms used herein. Inspection shall be performed in accordance with this specification and other specifications referenced in any of the contractual documents.

4.1.1 Contractor quality assurance system. If the contractor desires to utilize a quality assurance system, which is at variance with the quality assurance provisions of 4.2.2 and 4.3 and other documents referenced herein, he shall submit a written description of the system to the contracting officer for approval prior to initiation of production. It shall include a description covering controls for lot formation and identification, inspections to be performed, inspection stations, sampling procedures, methods of inspection, (measuring and testing equipment), and provisions for control and disposition of non-conforming material. The written description will be considered acceptable when, as a minimum, it provides the quality assurance provisions required by the provisions of 4.2.2 and 4.3

and the other documents referenced herein. The contractor shall not be restricted to the inspection station or the method of inspection listed in this specification provided that an equivalent control is included in the approved quality assurance procedure. In cases of dispute as to whether certain procedures of the contractor's system provide equal assurance, the comparable procedure of this specification shall apply. The contractor shall notify the Government of, and obtain approval for, any changes to the written procedure that effects the degree of assurance required by this specification or other documents referenced herein.

4.1.2 Submission of product. At the time the completed lot of product is submitted to the Government for acceptance, the contractor shall supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product submitted:

- (a) A statement that the lot complies with all quality assurance provisions of the approved current written description of the system.
- (b) Quantity of product inspected.
- (c) Results obtained for all inspection performed.
- (d) Specification number and date, together with an identification and date of changes.
- (e) Certificates of analysis on all material procured directly by the contractor, when such material is controlled by Government specifications listed in any of the contractual documents.
- (f) Quantity of product in the lot.
- (g) Date submitted.
The certificate shall be signed by a responsible agent of the certifying organization. The initial certificate submitted shall be substantiated by evidence of the

agent's authority to bind his principal. Substantiation of the agent's authority will not be required with subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.

4.1.3 Government verification. Using the contractor's written quality assurance procedure (see 4.1.1), this detail specification, and other contractual documents as a guide, the Government inspector shall verify all quality assurance operations performed by the contractor. Verification shall be in accordance with a or b as applicable, the decision being the responsibility of the procuring activity. In either case, the inspector shall also ascertain, prior to acceptance, that all quality assurance provisions of other specifications referenced in any of the contractual documents have been complied with. Deviations from prescribed or agreed upon procedures discovered by the Government inspector shall be brought to the attention of the supplier. Disposition of the product and remedial action shall be as directed by the Government inspector and, depending on the nature of the deviation, may consist of lot rejection, screening, re-sampling, re-instruction of the supplier's employees, or other appropriate action:

- (a) Verification at the point of manufacture shall be accomplished at unscheduled intervals in accordance with 4.1.3.1 and 4.1.3.2.
- (b) Verification at the point of delivery shall be in accordance with 4.1.3.2.

4.1.3.1 Surveillance. Surveillance shall include, but is not limited to:

- (a) Observation of procedures concerning lot formation and identification.
- (b) Observation of sampling procedures and application of acceptance criteria.

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- (c) Determination that all required examinations and tests are performed in accordance with the prescribed procedures of this specification, or approved equivalents thereto.
- (d) Review of procedures for control and disposition of non-conforming material.

4.1.3.2 *Product inspection.* Product inspection shall consist of Government inspection of product which has been previously inspected by the contractor and found to meet the quality assurance provisions of this specification. The inspection by the Government shall be performed in order to determine that the product is of the quality required by this specification and that the contractor's records are reliable.

4.2 Inspection provisions

4.2.1 *Lot formation.* A lot shall consist of one or more batches of diethyl ether, pro-

Categories	Defects	Method of inspection
Critical: None defined.		
Major:	AQL 0.25 percent	
101.	Leakage test failure	Test
Minor:	AQL 0.40 percent	
201.	Exterior surface coating incorrect or inadequate	Visual

4.2.2.1.2 *Shipping container, prior to closing.*

Categories	Defects	Method of inspection
Critical: None defined.		
Major:	AQL 0.25 percent	
101.	Metal cans, not properly sealed	Visual-Manual
Minor:	AQL 1.00 percent	
201.	Number of metal cans incorrect	Visual
202.	Filler missing or insufficient	Visual-Manual

4.2.2.1.3 *Shipping container, after closing.*

Categories	Defects	Method of inspection
Critical: None defined.		
Major: None defined.		

duced by one manufacturer, in accordance with the same specification, or same specification revision, under one continuous set of operating conditions, and submitted for acceptance at one time. Each batch shall consist of that quantity of diethyl ether that has been subjected to the same unit chemical or physical mixing process intended to make the final product homogeneous.

4.2.2 *Examination.* Sampling plans and procedures for the following classification of defects shall be in accordance with Standard MIL-STD-105. Continuous sampling plans, in accordance with Handbook ORD-M608-11 may be used if approved by the procuring activity. Also, at the option of the procuring activity, AQL's and sampling plans may be applied to the individual characteristics listed using an AQL of 0.25 percent for each major defect and an AQL of 0.40 percent for each minor defect.

4.2.2.1 *Classification of defects.*

4.2.2.1.1 *Metal cans, prior to filling.*

Minor:	AQL 1.00 percent	
201.	Marking misleading or unidentifiable	Visual
202.	Shipping container not properly sealed	Visual-Manual

4.2.2.1.4 55 gallon metal containers, tank cars or tank vehicles, prior to filling.

Categories	Defects	Method of inspection
Critical: None defined.		
Major:	AQL 0.25 percent	
101.	Leakage test failure	Test
Minor:	AQL 0.40 percent	
201.	Interstate Commerce Commission markings missing, misleading or unidentifiable	Visual

4.2.2.1.5 Metal cans, 55 gallon metal containers, tank cars or tank vehicles, after filling, but prior to closing.

Categories	Defects	Method of inspection
Critical: None defined.		
Major:	AQL 0.25 percent	
101.	Outage under minimum	Gage
Minor: None defined.		

4.2.2.1.6 55 gallon metal containers, tank cars or tank vehicles, after filling and closing.

Categories	Defects	Method of inspection
Critical: None defined.		
Major:	AQL 0.25 percent	
101.	Container not properly sealed	Visual-Manual
Minor:		
201.	Marking misleading or unidentifiable	Visual

4.2.3 Testing.

4.2.3.1 Appearance, chemical and physical properties

4.2.3.1.1 Containers. From each inspection lot, two containers shall be selected at random. From the two containers a 32 ounce composite sample shall be made and placed in an airtight glass bottle, having a glass stopper or a cap containing a cork or cardboard liner that is covered with metal foil. The bottle shall be labeled to show the name of the material, grade, manufacturer, plant, contract or purchase order number, and the lot number. If the sample fails to comply

with any one of the requirements, the lot shall be rejected.

4.2.3.1.2 Tank cars. By means of a clean, weighed, small-necked glass bottle, approximately one quart of the material shall be removed from the tank car. The unstoppered bottle shall be rinsed with the material being sampled, and lowered to the bottom of the tank by means of a cord, chain or rod. The bottle shall be immediately withdrawn to the surface. In order to obtain a representative cross-section of the material, the speed of lowering and raising the bottle shall be uniform and so regulated that the bottle is just filled as it reaches the surface of the liquid.

This sample shall be placed in an airtight glass bottle, having a glass stopper or a cap containing a cork or cardboard liner that is covered with metal foil. The bottle shall be labeled to show the name of the material, manufacturer, plant, contract or order number, lot number, and lot size. If the sample fails to comply with any one of the requirements, the lot shall be rejected.

4.3 Test method and procedure. The following tests shall be performed on each sample.

4.3.1 *Appearance.* Fill a 25 milliliter (ml.) colorless glass test tube with a specimen of the sample, and another with distilled water. Compare liquids in the two tubes. The specimen should not be darker in color than the water and should contain no visible suspended matter.

4.3.2 *Specific gravity.*

4.3.2.1 *Pycnometer method.* The specific gravity of diethyl ether shall be determined in accordance with Federal Test Method No. 141 Method 4183, except that the temperature shall be 20°C./20°C.

4.3.2.2 *Westphal balance method (alternative method).* The specific gravity of diethyl ether shall be determined in accordance with Federal Test Method No. 141 Method 4183, except that the temperature shall be 20°C./20°C.

4.3.3 *Nonvolatile residue.* Transfer 100 ml. of the sample to a clean, tarred evaporating dish. Evaporate to dryness on a steam bath, dry for one hour in an oven at 100°C., cool in a desiccator and weigh. Calculate the percentage of nonvolatile residue in the sample, as follows:

$$\text{Percentage of nonvolatile residue} = \frac{100A}{BC}$$

where:

A = weight of residue, grams (gm's.).

B = volume of sample.

C = specific gravity of sample.

4.3.4 *Acidity.* Introduce two drops of bromthymol blue indicator solution prepared by dissolving 100 milligrams (mg's.) of bromthymol blue in 20 ml. of acid-free alcohol and adding distilled water to make 100 ml. of solution. Add 10 ml. of carbon dioxide-free distilled water in a stoppered, colorless glass flask* or cylinder and add, dropwise from a 25 ml. burette, 0.001 N. sodium hydroxide solution until the first blue color is permanently developed. Add 50 ml. of the sample, shake well to mix the two layers and titrate with 0.001 N. sodium hydroxide solution to the first blue color that persists for several minutes. The final color of the solution should not be judged until the two layers have separated. Small amounts of alcohol in the solution dims the blue color; large amounts of alcohol give an indefinite end point. Calculate acidity as percentage of acetic acid in the sample as follows:

$$\text{Percentage of acetic acid} = \frac{6.0 VN}{AB}$$

where:

V = ml. of sodium hydroxide used in titration.

N = normality of sodium hydroxide solution.

A = volume of sample in ml's.

B = specific gravity of sample.

* It is necessary to use stoppers that fit well and to avoid introducing any carbon dioxide to the solution.

** This phenomenon occurs when ether is slightly alkaline.

4.3.5 *Peroxides.* Transfer 10 ml. of the sample to a 10 ml. colorless, glass-stoppered cylinder and add 1 ml. of a 10 percent potassium iodide (KI)-cadmium iodide (CdI₂) solution which has been prepared by dissolving 0.5 gms. KI and 0.5 gm. CdI₂ in 9 ml. of distilled water. Keep the cylinder and contents in a dark place for 1 hour, shaking

occasionally. If either layer darkens, peroxides are present.

4.3.6 Chlorides. Transfer 10 ml. of the sample to a test tube and add 4 to 5 ml. of a 10 percent solution of silver nitrate. Should opalescence** appear add 1 drop of nitric acid by means of a stirring rod. Persistence of opalescence indicates the presence of chloride.

4.3.7 Aldehydes. Transfer 10 ml. of the solution to a colorless, glass stoppered cylinder and add 1 ml. of a solution prepared by mixing 1 ml. of alkaline mercuric potassium iodide with 17 ml. of a saturated aqueous solution of sodium chloride. Stopper the cylinder and shake vigorously for 10 seconds, then set it aside for 1 minute. Persistence of turbidity in the aqueous layer indicates the presence of aldehydes.

4.3.8 Acetylene. Add 10 drops of freshly prepared 0.1 percent methyl red indicator solution to each of two 250 ml. Erlenmeyer flasks containing 50 ml. of 50 percent by volume alcohol. Reserve one of the flasks as a blank and add 10 ml. of the sample to the other. Neutralize the acidity of the liquids in each of the flasks and match their colors exactly by adding 0.02 N. alcoholic silver nitrate to each flask and titrate the sample and blank with standard 0.02 N. sodium hydroxide to the same yellow end point. Calculate the percentage of acetylene as follows:

$$\text{Percent acetylene} = \frac{1.30 (V-v)N}{SD}$$

where:

V = ml. standard sodium hydroxide required for the sample.

v = ml. standard sodium hydroxide required for the blank.

N = normality of the sodium hydroxide.

S = ml. of sample.

D = specific gravity of sample.

5. PREPARATION FOR DELIVERY

5.1 Packaging. (see 6.2)

5.1.1 Level A. As specified ethyl ether shall be packaged in 1 pound capacity metal cans or 55 gallon capacity metal drums. Cans shall be round, conical, or dome shaped and shall comply with type VIII, Specification PPP-C-96. The cans shall be provided with inner seals and screw cap closures. Exterior surfaces of the cans shall be coated in accordance with finish plan B, Specification PPP-C-96, with side seams striped. Steel drums shall comply with type III, Specification PPP-C-729.

5.1.2 Level C. Ethyl ether shall be packaged in accordance with the manufacturer's commercial practice.

5.2 Packing. (see 6.2)

5.2.1 Level A. Ethyl ether packaged in cans as specified in 5.1.1, shall be packed in accordance with the overseas requirements of the Appendix of Specification PPP-C-96. Metal drums specified in 5.1.1 require no overpacking.

5.2.2 Level B. Ethyl ether shall be packed in cans as specified in 5.1.1, and shall be overpacked in accordance with the domestic requirements of the Appendix of Specification PPP-C-96. Metal drums specified in 5.1.1 require no overpacking.

5.2.3 Level C. Ethyl ether shall be packed in a manner such as to insure carrier acceptance and safe delivery at destination. Containers shall be in accordance with Interstate Commerce Commission Regulations and other regulations applicable to the mode of transportation.

5.3 Marking. In addition to any special marking required by the contract or order, unit packages and shipping containers shall be marked in accordance with Interstate Commerce Commission Regulations and Military Standard. MIL-STD-129.

5.3.1 *Precautionary marking.* All individual containers shall be marked with the following precautionary marking:

Danger! Extremely Flammable.

Highly Volatile.

Tends to form explosive peroxides especially when anhydrous.

Keep away from heat, sparks, and open flame.

Keep containers tightly closed.

Do not allow to evaporate to near dryness. Addition of water or appropriate reducing agents will lessen peroxide formation.

6 NOTES

6.1 *Intended use.* Diethyl ether is intended for use in the manufacture of smokeless powder, in the cleaning of optical instruments, and where a high purity ether is required.

6.2 *Ordering data.* Procurement documents should specify the following:

* U.S. GOVERNMENT PRINTING OFFICE: 1974-603-108/1361

(a) Title, number and date of this specification.

(b) Levels of protection (see 5.2). Level B packing is intended to provide economical but limited protection and should be specified only when it is determined that the ethyl ether will be held in covered storage approximately one year from date of initial packing.

Notice. When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any other way supplied said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian:

Army—Ord
Navy—Wep.

Preparing activity:

Army—Ord