

MIL-P-8845(ASG)

20 APRIL 1959

Superseding

NARTS Specification

SL195-0066

13 September 1957

MILITARY SPECIFICATION

PROPELLANT, ETHYLENE OXIDE

This specification has been approved by the Department of the Air Force and by the Navy Bureau of Aeronautics.

1. SCOPE

1.1 This specification covers propellant ethylene oxide.

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:

SPECIFICATION

Military

MIL-S-4043

Steel; Corrosion-Resisting (Extra Low Carbon Type 304), Plate, Sheet and Strip

STANDARDS

Military

MIL-STD-105

Sampling Procedures and Tables for Inspection by Attributes

MIL-STD-129

Marking for Shipment and Storage

(Copies of documents 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 publication forms a part of this specification. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

Interstate Commerce Commission

49 CFR 71-78

Interstate Commerce Commission Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles

FSC 9135

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(The Interstate Commerce Commission regulations are now a part of the Code of Federal Regulations (1949 Edition-Revised 1950) available from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Orders for the above publications should cite "49 CFR 71-78 (Rev. 1950).")

American Society for Testing Materials

D268

Sampling and Testing Lacquer Solvents and
Diluents

(Copies of ASTM publications may be obtained from the Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pennsylvania.)

3. REQUIREMENTS

3.1 Form.- The ethylene oxide $\text{H}_2\text{C}-\text{O}-\text{CH}_2$ shall be clear and free of suspended matter; the color shall be not greater than 10 American Public Health Association color number.

3.2 Concentration.- The concentration shall be not less than 99.9 percent by weight of ethylene oxide. (Calculated by difference between 100 percent and total sum of foreign materials.)

3.3 Foreign materials.- Impurities shall not exceed limits specified below unless the using agency specifies otherwise.

PRIMARY SPECIFICATION FOR IMPURITIES

<u>Requirement</u>	<u>Maximum allowable limits</u>
Acidity - percent by weight as acetic acid	0.005 percent
Water - percent by weight	0.03 percent
Total iron	0.1 mg per liter at 0° C
Aldehydes - percent by weight as acetaldehyde	0.03 percent
Inorganic chlorides - percent by weight	0.02 percent
Acetylene	None (By method specified under 4.3.9)

3.4 Evaporation residue.- The nonvolatile residue, when tested as specified in 4.3.10 shall not exceed 100 mg per liter or 0.01 gram per 100 ml at 0° C unless otherwise specified by the using agency.

4. QUALITY ASSURANCE PROVISIONS

Unless otherwise specified herein, the supplier is responsible for the performance of all inspection requirements prior to submission for Government inspection and acceptance. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory 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.

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4.1 General.- All the tests required herein for the testing of ethylene oxide are classified as acceptance tests, for which necessary sampling techniques and methods of testing are specified in this section.

4.2 Sampling.-

4.2.1 Lot.- For purposes of sampling, a lot shall consist of all material of the same type manufactured in one production run, stored in one storage tank or in drums filled from one storage tank at one time, and offered for delivery at the same time. In the case of drum shipments, quality may be determined on a sample drawn from the storage tank from which the containers are filled provided that each container is inspected for cleanliness prior to filling and samples are drawn from a minimum of three drums for identification testing. Identification tests shall consist of the visual test specified in 4.3.2. In the event that the foregoing procedure is not practicable, a 1-quart sample shall be drawn from each of the containers selected in accordance with 4.2.2. These samples shall be visually inspected individually. If the samples pass the visual examination, a 1-quart composite sample shall be prepared for test purposes.

4.2.2 Sampling for inspection of filled containers.- A random sample of filled containers shall be selected from each lot by the Inspector in accordance with MIL-STD-105 at inspection level II and acceptable quality level (AQL) equal to 2.5 percent defective to verify compliance with this specification regarding container fill, closure, marking, and other requirements not involving tests.

4.2.3 Sampling for tests of ethylene oxide.- From each sampling lot of ethylene oxide offered for inspection, a water-pumped nitrogen line shall be attached through a dryer and regulating valve to the liquid vent of the container and both the liquid and vapor vent shall be opened. The contents of the container shall be mixed by vigorously bubbling dry nitrogen gas through the ethylene oxide for 1 minute. After completion of the 1-minute period of mixing, the vents shall be closed. A 1-quart specimen shall be transferred into a suitable sampling pressure cylinder or flask that has been properly flushed and rinsed twice with small portions of ethylene oxide liquid. Flasks should be kept lightly stoppered and immersed in an ice water bath while being held for subsequent analyses. All tests on samples taken for inspection and analyses shall be performed within the 48-hour period from the time the samples are taken. Each specimen shall be kept in separate containers, and labeled for identification of the container and the lot represented. Each of the samples shall be subjected to all the tests specified in 4.3 (4.3.1 through 4.3.11) in duplicate, unless otherwise specified by the using agency.

4.2.4 Place of test. Unless otherwise specified in the contract or order, lot acceptance inspection tests shall be conducted by the contractor under supervision of the Government Inspector. The contractor shall provide the necessary laboratory facilities satisfactory to the Inspector, or shall engage the services of a commercial testing laboratory acceptable to the Inspector.

4.2.5 Reports on inspection and tests.- Two copies of each lot acceptance inspection and test report shall be forwarded to the procuring activity and other agencies or contractors as may be designated by the procuring activity.

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4.3 Tests.-4.3.1 Inspection.-

4.3.1.1 Filled containers.- Each sample filled container selected in accordance with 4.2.2 shall be examined by the Inspector for defects of the container and the enclosures, and for unsatisfactory markings. Each sample filled container shall also be weighed to determine the amount of the contents. The tare weights furnished by the manufacturer shall be accepted.

4.3.1.1.1 Rejection and retest.- Any container in the sample having one or more defects, or under required fill, shall be rejected, and if the number of defective containers in any sample exceeds the acceptance number for the appropriate sampling plan of MIL-STD-105, the lot represented by the sample shall be rejected. Rejected lots may be resubmitted for acceptance tests provided the contractor has removed, repaired, or corrected nonconforming containers.

4.3.2 Visual.- One hundred milliliters (ml) of ethylene oxide from each of the samples shall be measured into a prechilled (below 0° C) 250-ml graduate and examined for suspended particles, sediment, and color. Samples should be clear, water white, and free of suspended matter as required in 3.1.

4.3.3 Color.- Reference should be made to American Society for Testing Materials Method D268 using American Public Health Association color standards.

4.3.4 Acidity.- The acidity shall be determined as follows.

4.3.4.1 Preparation of reagents.-

Phenolphthalein indicator solution - dissolve 0.5 gram of phenolphthalein indicator in 100 ml of alcohol.

0.01 N Sodium hydroxide solution - add 0.8 gram of a 50-percent NaOH solution (CO₂ free) to 1 liter of boiled distilled water. Standardize against dry potassium acid phthalate using phenolphthalein indicator solution.

4.3.4.2 Titration procedure.-

Place 68 ml of the ethylene oxide sample in a 100-ml graduate and allow to evaporate to about 50 ml. Transfer to a 125-ml Erlenmeyer flask, add 1 ml of phenolphthalein indicator solution, and titrate with 0.01 N sodium hydroxide to a faint pink which persists for at least 15 seconds.

Calculation:

$$\frac{\text{ml of (NaOH) x (N) of (NaOH) x 6.005}}{68 \times \text{specific gravity of ethylene oxide}} = \frac{\text{percent acidity as wt percent acetic acid}}{\text{acetic acid}} \quad (1)$$

4.3.5 Water.- The water shall be determined as follows.

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4.3.5.1 Procedure for standardization of reagents.- 1/

Iodine solution: Dissolve 150 grams of reagent-grade iodine crystals in 3 liters of anhydrous methanol.

Pyridine solution: To 3,400 ml of freshly distilled pyridine add 400 grams of refrigerant-grade sulfur dioxide dried through concentrated sulfuric acid and allow the solution to cool.

4.3.5.2 Analytical procedure.- Transfer 50 ml of anhydrous methanol to a 500-ml electrometric titration assembly. Transfer 25 ml of pyridine reagent to the titrating flask. Fresh portions of methanol and pyridine should be used for each series of determinations.

Add the iodine solution dropwise from the buret until the ammeter reads a maximum value, about 100 microamperes. Introduce 0.08 to 0.10 gram of distilled water weighed to the nearest 0.1 mg by means of a suitable weighing pipet. Titrate with the iodine solution, approaching the end-point dropwise until the maximum value, about 100 microamperes, is obtained on the ammeter.

Calculation:

$$\frac{\text{g water} \times 100}{\text{ml titration}} = \text{Factor} = \text{g water consumed by 100 ml of reagent.} \quad (2)$$

4.3.5.3 Procedure.- For the sample, proceed as directed above to the addition of the water. Quickly introduce the prescribed amount of sample by means of a graduate. Titrate to the electrometric end-point.

Precaution: After titrating the sample, immediately empty the contents of the titrating flask into the sink and flush down the drain with copious quantities of cold water.

Calculation:

$$\frac{\text{ml titration} \times \text{factor}}{\text{g sample}} = \text{Water, percent by weight} \quad (3)$$

Sample size:

50 ml (use a chilled graduate which has been rinsed with a portion of the sample immediately before use.)

4.3.6 Total iron (tentative method).- The iron shall be determined as follows.

4.3.6.1 Reagents.-

Special solutions -

Hydroquinone - 1 percent solution in water.

Sodium citrate - 250 g per liter H₂O.

O - Phenanthroline - 0.5 percent aqueous.

(Keep in dark - discard if any color develops.)

Hydrochloric acid - 1 N.

1/ Commercial preparations of Karl Fisher reagent may be used.

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4.3.6.2 Procedure.-

- (a) Measure 100 ml of the ethylene oxide in graduated cylinder cooled to 0° C and evaporate in a small porcelain dish in a hood.
- (b) To the evaporation residue, add 5 ml of concentrated hydrochloric acid. Swirl the dish to assure complete digestion of the residue by the HCl. Evaporate to dryness and cool.
- (c) Transfer the residue with three 2-ml washes of 1 N HCl to a 25-ml volumetric flask.
- (d) Add in the order listed:
 - (1) Hydroquinone - 1 ml
 - (2) O - phenanthroline - 1 ml
 - (3) Sodium citrate - sufficient to bring the pH of the solution between 3.0 and 4.0.
- (e) Allow the solution to stand at room temperature (20° C) for 1 hour to insure complete reduction of iron to the ferrous state.
- (f) Dilute to the 25-ml mark.
- (g) Determine the absorbance of the unknown solutions against that of a blank in a colorimeter with a filter showing ~~maximum~~ transmission at 480-520 mμ, or in spectrophotometer at 508 mμ.
- (h) Prepare a working curve of absorbance versus concentration of iron by treating a series of standards containing 0 to 2 mg iron per filter as indicated above.
- (i) Determine concentration of iron in ethylene oxide from the working curve.

4.3.7 Aldehydes.- The aldehyde content shall be determined as follows:

4.3.7.1 Reagents.-

Iodine (approximately 0.1 N standard solution.) 1/
 Sodium bisulfite (approximately) 0.2 N 2/
 Starch indicator, 0.5 g per liter of water.

4.3.7.2 Procedure.- Using a buret, measure 20 ml of sodium bisulfite solution into each of four Erlenmeyer flasks and cool by immersing flasks in a water-ice bath between 0° C and 4° C. Cool a 10-ml graduated cylinder by rinsing with a portion of the oxide sample and measure 10-ml samples, (3/, 4/) into two of the flasks. The remaining two flasks are reserved as blanks. Immediately stopper securely all the flasks and swirl to mix the contents. Remove the flasks from the cold bath, and let stand for 30 minutes at room temperature. Titrate each flask with standard iodine solution adding 5 ml of starch indicator towards the end of the titration. The end point is the first appearance of blue color.

Calculation:

$$\text{percent aldehyde as acetaldehyde} = \frac{(\text{ml } I_2 \text{ for blank} - \text{ml } I_2 \text{ for sample}) \times N \text{ of } I_2 \times 2.2}{10 \times \text{sp gr of sample}} \quad (4)$$

See footnotes 1, 2, 3, and 4 on page 7.

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4.3.8 Inorganic chlorides.- Inorganic chlorides shall be determined as follows:

Principle

The chloride present in a sample of ethylene oxide is reacted with excess solid silver chromate, liberating chromate ion:



The chromate ion concentration is measured by comparison of the transmission of the sample solution with that of a blank prepared under the same conditions.

4.3.8.1 Reagents.- Silver chromate - prepared as follows: heat 100 ml of 10 percent silver nitrate solution to boiling and slowly add 200 ml of 5.5 percent potassium chromate solution, maintaining the solution at the boiling point to prevent formation of silver dichromate. When the precipitate settles, the supernatant liquid should be yellow in color, indicating the presence of excess potassium chromate. Filter the hot solution, wash the precipitate thoroughly with distilled water, and air dry.

4.3.8.2 Procedure.- Introduce a small amount of solid silver chromate (a volume the size of a pea) into a clean, dry test tube. Add 5.0 ml of distilled water from a 10-ml graduated cylinder and cool to 0° C in an ice bath. By means of a cold 10-ml graduated cylinder, add 10 ml of ethylene oxide sample to the water and silver chromate, mix well, and allow the sample to warm to room temperature. After nearly all the ethylene oxide has evaporated, add 5.0 more ml of distilled water and heat in a boiling bath for 5 minutes. Cool to room temperature (25° C) and filter all of the sample by suction through a fine fritted disk funnel, directly into a clean, dry 18 mm x 150 mm test tube in the filter flask. It is important that the filter disk be drawn nearly dry prior to this filtration. Do not rinse the funnel into the test tube before or after filtering the sample.

By means of forceps, remove the test tube from the flask, add 1 drop of concentrated ammonium hydroxide, mix, and compare the transmission of the sample at 410 mμ with that of a blank prepared in the same way but without the sample. A standard curve is prepared by using solutions of known chloride content and treating the same way as above. The total amount of water added in all cases should be 10.0 ml.

Calculation:

$$\frac{\text{g chloride found} \times 10^6}{8.9} = \text{ppm chloride in sample} \quad (5)$$

- 1/ Dissolve 12.692 g of resublimed iodine in a saturated solution of 24 g of potassium iodide and dilute to 1 liter. This solution may be used as a standard only if the weighing is carefully done. The solution may be standardized against a standard sodium thiosulfate solution using starch indicator.
- 2/ Sodium bisulfite solution is prepared preferably from sodium metabisulfite.
- 3/ Larger or smaller samples may be used if necessary.
- 4/ Obtain the specific gravity of the sample as near as possible to the temperature at which the samples are measured for analysis. If the sample is a substantially pure compound, its specific gravity may be taken from suitable tables.

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4.3.9 Acetylene.-- The acetylene content shall be determined as follows.

4.3.9.1 Reagents.--

Mixed indicator

Dissolve 0.10 gram of methyl red and 0.050 gram of methylene blue in 100 ml of 190-proof ethanol. Mix thoroughly and store in a dark colored bottle.

Silver nitrate

Dissolve 25 grams of chemically pure silver nitrate in 150 ml of distilled water contained in a 1,000-ml volumetric flask. Mix thoroughly and dilute to the mark with 190-proof ethanol.

4.3.9.2 Procedure.-- Transfer 150 ml of the silver nitrate reagent to each of two 250-ml glass stoppered Erlenmeyer flasks. Place the flasks in a suitable ice bath until the temperature of the contents is 0° to 4° C. Add 7 to 9 drops of the mixed indicator to each flask and, if necessary, neutralize to a gray-green color with 0.02 N sodium hydroxide, if acid, or with 0.02 nitric acid, if alkaline. Reserve one of the flasks for the blank. Into the second flask introduce 50 ml of the sample, using a suitable chilled graduate. Compare the color of the sample with that of the blank. A purple color in the sample solution indicates the presence of acetylene.

NOTE: If a positive test is obtained, destroy the silver acetylide by adding an excess of acid ferrous sulfate solution to the sample flask before discarding the solution.

4.3.10 Nonvolatile matter.-- Evaporation residue shall be determined as follows.

4.3.10.1 Procedure.-- Weigh accurately a pyrex evaporating dish which has previously been dried at 105° to 110° C for 1 hour. Transfer 100-ml ethylene oxide sample which has been cooled to 0° to 5° C to the evaporating dish. Allow the dish to dry at room temperature in a dust-free hood until the liquid has evaporated, then set the dish in a drying oven at 105° to 110° C for 1 hour. Cool the dish in a desiccator and weigh to the nearest 0.1 mg.

Calculation:

$$\frac{\text{g residue} \times 100}{\text{ml of sample}} = \text{g per 100 ml} \quad (6)$$

4.4 Action in case of failure.-- If any one of the samples tested in duplicate is found to be not in conformance with this specification, the lot which it represents shall be rejected. A rejected lot may be resubmitted for inspection only after the manufacturer, having been informed of the reasons for rejection, has reworked the entire lot as to remove or correct all nonconforming material.

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5. PREPARATION FOR DELIVERY

5.1 Ethylene oxide shall be furnished in accordance with Interstate Commerce Commission Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles, Section 73.124 and with the following procedures, and in the type and size container indicated, unless otherwise specified by the using agency.

5.1.1 Drums.- The material shall be furnished in ICC 5P-400-type drums of 55-gallon capacity lined with stainless steel type 304 conforming to MIL-S-4043. The drums shall be equipped with proper fixtures for safe and convenient transfers of ethylene oxide and shall be constructed in accordance with ICC regulations. (Drums shall not be filled to more than 90 percent of capacity at 15.6° C). Closure shall be wire sealed to prevent removal in transit. Unless otherwise specified by the bureau or agency concerned, materials of construction allowed to be in contact with ethylene oxide are stainless steel type 304 and Teflon.

5.1.2 Cleaning - Procedures for cleaning drums prior to re-use.- All containers shall be cleaned before re-use and rinsed with water until the rinse water is neutral as indicated by litmus or other suitable pH paper. The interior surface of the drum shall be visually inspected for the presence of rust, polymer, and other foreign matter and if evidence of such contamination is found, the drum shall be re-cleaned in whatever manner deemed suitable to remove the contamination. A final rinse with ethylene oxide conforming to the provisions of this specification shall be made to remove residual moisture and to passivate the cleaned surfaces.

5.2 Filling.-

5.2.1 Fittings.- Only fittings and piping free of rust, scale, or other sediment shall be used in the filling operation. As necessary, cleaning and passivation will be performed as specified in 5.1.2.

5.2.2 Transfer.- Transfer and drum pressurization shall be performed using only water-pumped nitrogen, methane, or natural gas. These gases must be free of such impurities as air, oxygen, acetylene, sulfur, hydrogen sulfide, water, ammonia, and carbon dioxide. The transfer operations shall be conducted at a minimum total pressure of 35 psig at 21° C.

5.2.3 Pressurization.- A minimum total pressure of inert gas and ethylene oxide vapor phase of 35 psig at 21° C shall be maintained in the drum at all times when it contains ethylene oxide.

5.3 Marking of shipments.- In addition to any special marking required in the contract or order, marking for shipment shall be in accordance with ICC regulations and MIL-STD-129.

5.3.1 The following marking data required by MIL-STD-129 not required by ICC regulations shall be stenciled or printed in accordance with MIL-STD-129:

- (a) Stock number.
- (b) Specification number
- (c) Ethylene oxide concentration (wt percent).
- (d) Name and address of contractor.
- (e) Contract or order number.
- (f) Bill of lading or shipping order number.
- (g) Domestic or overseas address (as applicable).

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6. NOTES

6.1 Intended use.- The ethylene oxide covered by this specification is intended for use in auxiliary power units.

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

- (a) Title, number, and date of this specification.
- (b) Special requirements (see sections 4 and 5).

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 way supplied the 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.

Custodians:

Navy - Bureau of Aeronautics
Air Force

Preparing activity:

Navy - Bureau of Aeronautics

SPECIFICATION ANALYSIS SHEET		Form Approved Budget Bureau No. 119-R004
INSTRUCTIONS		
<p>This sheet is to be filled out by personnel either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity (as indicated on reverse hereof).</p>		
SPECIFICATION		
ORGANIZATION (Of submitter)	CITY AND STATE	
CONTRACT NO.	QUANTITY OF ITEMS PROCURED	DOLLAR AMOUNT \$
MATERIAL PROCURED UNDER A		
<input type="checkbox"/> DIRECT GOVERNMENT CONTRACT <input type="checkbox"/> SUBCONTRACT		
1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE? A. GIVE PARAGRAPH NUMBER AND WORDING.		
B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES.		
2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID		
3. IS THE SPECIFICATION RESTRICTIVE? <input type="checkbox"/> YES <input type="checkbox"/> NO IF "YES", IN WHAT WAY?		
4. REMARKS (Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)		
SUBMITTED BY (Printed or typed name and activity)		DATE

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