

MIL-P-8845A
30 June 1969
 Superseding
 MIL-P-8845(ASG)
 20 April 1959

MILITARY SPECIFICATION

PROPELLANT, ETHYLENE OXIDE

This specification is mandatory for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 This specification covers the requirements for propellant, ethylene oxide.

2. APPLICABLE DOCUMENTS

2.1 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-S-4043	Steel; Corrosion-Resisting (Extra Low Carbon Type 304), Plate, Sheet and Strip
MIL-P-27401	Propellant Pressurizing Agent, Nitrogen

STANDARDS

Military

MIL-STD-101	Color Code for Pipelines and for Compressed-Gas Cylinders
MIL-STD-129	Marking for Shipment and Storage
MIL-STD-172	Color Code for Containers of Liquid Propellants

FSC 9135

MIL-P-8845A

(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 specified, the issue in effect on date of invitation for bids shall apply.

Department of Transportation

49 CFR 170-190

Department of Transportation
Hazardous Materials Regulations

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402.)

Manufacturing Chemists' Association, Inc.

MCA Manual L-1

A Guide for the Preparation
of Warning Labels for Hazardous
Chemicals (1961)

(Application for copies should be addressed to the Manufacturing Chemists' Association, Inc., 1825 Connecticut Avenue, N.W., Washington, D.C. 20009.)

American Society for Testing and Materials Publications

ASTM Standards, Parts 20, 23, 30, and 31

(Copies of ASTM publications may be obtained upon application to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

2.2.1 Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among the technical groups and using Federal Agencies.

3. REQUIREMENTS

3.1 Chemical and physical properties. The chemical and physical properties of the propellant shall conform to those listed in table I when tested in accordance with the applicable test methods.

MIL-P-8845A

Table I. Chemical and Physical Properties

Properties	Limits	Test Paragraph
Ethylene Oxide %/wt	99.9 min.	4.5.2
Acidity, %/wt as acetic acid	0.005 max. <i>.005</i>	4.5.3
Water, %/wt	0.03 max. <i>.05</i>	4.5.4
Iron, ppm/wt	0.1 max.	4.5.5
Aldehyde, %/wt as acetaldehyde	0.03 max. <i>.001</i>	4.5.6
Chloride, %/wt	0.02 max.	4.5.7
Nonvolatile residue, %/wt	0.01 <i>.01</i>	4.5.8
Acetylene	None detectable	4.5.9

3.2 Limiting values. The following applies to all specified limits in this specification: For purposes of determining conformance with these requirements, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limitation value, in accordance with the rounding-off method of the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E29).

3.3 Filter. A filter with a 10-micron nominal and 40-micron absolute rating shall be installed between the manufacturer's plant system and the container to be filled for delivery.

3.4 Qualitative. The propellant shall be clear and free from suspended matter. The color shall not be greater than American Public Health Association color number 10.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for

MIL-P-8845A

the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier 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 tests. The inspection and testing of the propellant shall be classified as quality conformance tests.

4.3 Test conditions. The test conditions are described under the individual test to which they apply.

4.4 Quality conformance tests. Quality conformance tests shall consist of:

- (a) Individual tests 4.4.1
- (b) Sampling tests 4.4.2

4.4.1 Individual tests. The propellant shall be subjected to the following test as described under 4.5.

- Examination of product 4.5.1

4.4.2 Sampling tests. The propellant shall be sampled in accordance with 4.4.2.1 and subjected to the following tests as described under 4.5.

- (a) Ethylene oxide 4.5.2
- (b) Acidity 4.5.3
- (c) Water 4.5.4
- (d) Iron 4.5.5
- (e) Aldehyde as acetaldehyde 4.5.6
- (f) Chloride 4.5.7
- (g) Nonvolatile residue 4.5.8
- (h) Acetylene 4.5.9

4.4.2.1 Sampling plan.

MIL-P-8845A

4.4.2.1.1 Lot. A lot shall consist of one of the following:

(a) The propellant produced in not more than 24 consecutive hours from a continuous process which is used to fill shipping containers directly from the process output. A continuous process shall be the production of product by continuous input of raw materials and output of finished product by one manufacturer in one plant with no change in manufacturing conditions or materials.

(b) The propellant produced from individual runs of a batch process which is used to fill shipping containers directly from the process output. A batch process shall be the production of product from single additions of raw materials which are reacted and purified forming the product.

(c) The propellant from either or both the continuous and batch processes which is held in a single storage tank and subsequently withdrawn to fill shipping containers. The product shall be homogeneous at the time of withdrawal and shall not be added to while being withdrawn. After each addition to the storage tank, the contents shall constitute a separate lot.

4.4.2.1.2 Sample. Two 300-milliliter (ml) samples shall be taken from each selected shipping container in accordance with 4.4.3. When required, the sample shall be forwarded to a laboratory designated by the procuring activity for subjection to the quality conformance tests specified herein.

4.4.2.1.3 Cylinders and drums. The number of cylinders and drums selected for sampling from each lot shall be in accordance with table II. The first and last containers to be filled within a given lot shall be sampled. Other samples may be selected at random. The propellant from each container sampled shall constitute a separate sample.

Table II. Sampling for Test

Number of containers in lot.	Number of containers to be sampled.
2-25	2
26-150	3
151-1,200	5
1,201-7,000	8

MIL-P-8845A

4.4.2.1.4 Portable tanks, cargo tanks, and tank cars. Each portable tank, cargo tank, or tank car shall constitute a lot. Unless otherwise specified, the sample shall be composited into one sample when one-third portions are withdrawn from the bottom, center, and top thirds of the tank.

4.4.2.1.5 Other containers. Unless otherwise specified, other containers of 100 gallons or less water capacity shall be sampled in accordance with 4.4.2.1.3. Containers greater than 100 gallons water capacity shall be sampled in accordance with 4.4.2.1.4.

4.4.3 Sampling procedure. Attach at V_1 a tared sampler, (designed in accordance with figure 1), to the container to be sampled. With V_1 and V_2 closed, cool the sampler to 32°F (0°C) with ice. Open the container valve and open V_1 . If 300 ml of propellant are not obtained, open V_2 slowly and fill the sampler to the 300-ml mark. Close the container valve, V_2 , and V_1 in that order and disconnect the sampler. Attach the "0" tube (figure 1) to the sampler.

4.4.4 Rejection. When any sample of the propellant tested in accordance with 4.5 fails to conform to the requirements specified herein, the entire lot represented by the sample shall be rejected.

4.5 Test methods.

4.5.1 Examination of product. Examine the propellant contained in the sampler for clarity and suspended matter. The test method described in ASTM D1209-62 shall be used for the color measurement. The color shall not exceed the requirement described in 3.4.

4.5.2 Ethylene oxide. Calculate the ethylene oxide concentration by the following formula:

$$\text{Ethylene oxide, \%wt} = 100.00 - \%wt (\text{acidity} + \text{water} + \text{aldehyde} + \text{chloride} + \text{nonvolatile residue})$$

4.5.3 Acidity. The acidity shall be determined in accordance with ASTM D1613-64T with the following exceptions:

4.5.3.1 Exceptions.

(1) The propellant sample size shall be approximately 50 ml. Determine the weight taken by weighing the sampler to the nearest 0.1 gram (g) before and after the liquid is removed.

(2) Calculate the results as follows:

$$\text{Acidity as acetic acid, \%wt} = \frac{\text{ml} \times N \times 0.06005}{W} \times 100$$

MIL-P-8845A

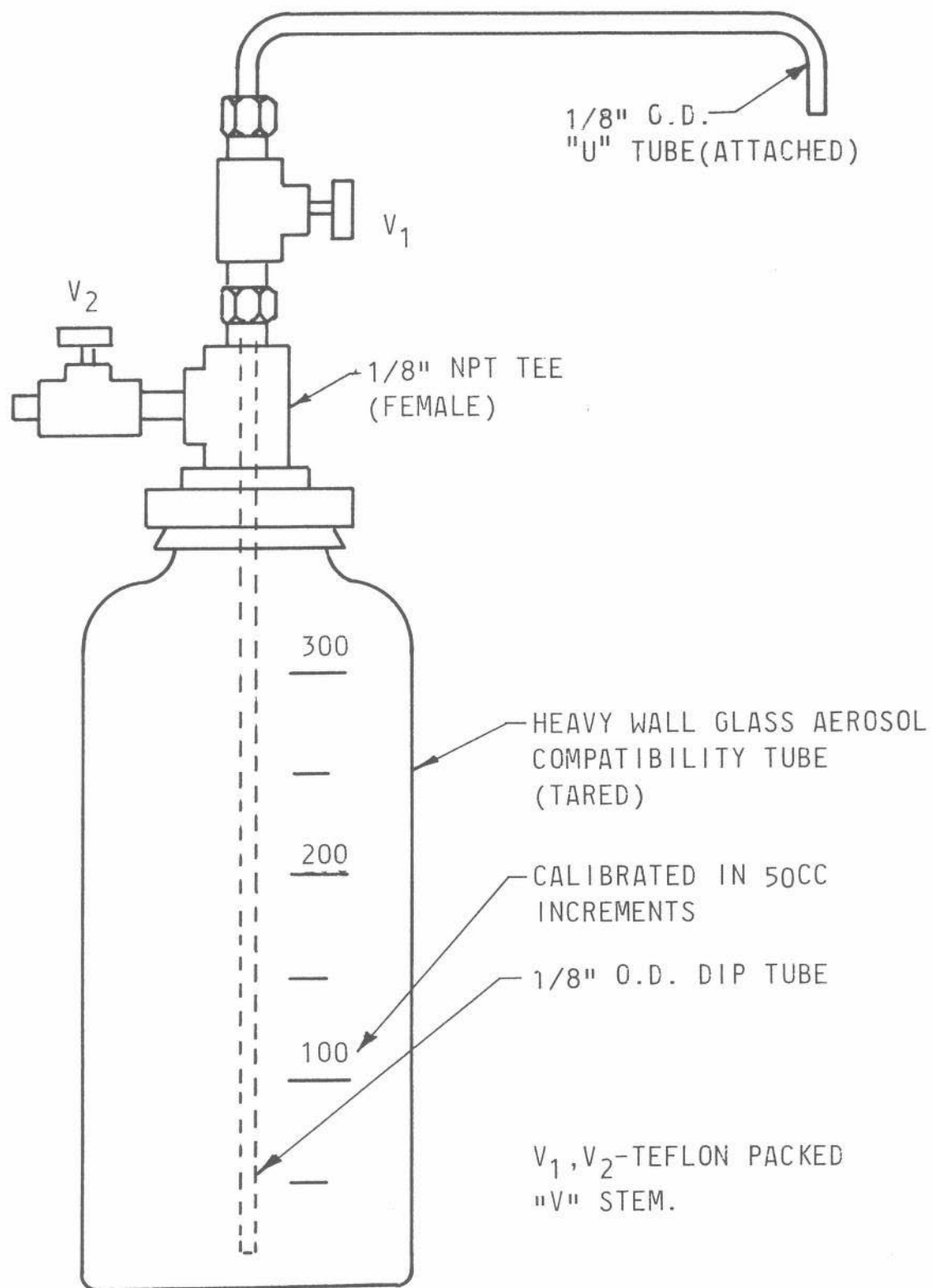


Figure 1. Sampler

MIL-P-8845A

Where:

ml = milliliters of NaOH used
 N = normality of NaOH
 W = propellant sample weight titrated

4.5.4 Water. The water content shall be determined in accordance with ASTM E203-64, with the following exception:

4.5.4.1 Exception. The propellant sample size shall be approximately 50 ml. Determine the weight taken by weighing the sampler to the nearest 0.1 g before and after the liquid is removed.

4.5.5 Iron. The iron content shall be determined in accordance with D1068-62T, Method A, with the following exceptions:

4.5.5.1 Exceptions:

(1) The propellant sample size shall be approximately 100 ml. Determine the weight taken by weighing the sampler to the nearest 0.1 g before and after the liquid is removed.

(2) Place the propellant sample in an evaporating dish and evaporate to dryness. Add 5 ml of concentrated HCl to the residue and evaporate the mixture to dryness. Swirl the mixture during evaporation to assure quantitative digestion of the residue. Quantitatively transfer the residue to a 100-ml volumetric flask with three 2-ml portions of 0.5N HCl. Proceed with the Method A analysis.

(3) An absorption cell with a 10-cm optical path shall be used.

(4) Calculate the results as follows:

$$\text{Iron, ppm/wt} = \frac{W}{W_s} \times 1000$$

Where:

W = milligrams of iron found
 W_s = propellant sample wt in grams

4.5.6 Aldehyde. The aldehyde content of the propellant shall be determined in accordance with ASTM D2191-65 with the following exceptions:

4.5.6.1 Exceptions.

(1) The propellant sample size shall be approximately 50 ml. Determine the weight taken by weighing the sampler to the nearest 0.1 g before and after the liquid is removed.

MIL-P-8845A

(2) Substitute a 12-oz heavy-wall aerosol compatibility tube with closure for the 500-ml glass-stoppered erlenmeyer flask used for the titration.

(3) Substitute "W", the propellant sample weight in grams taken, for "W x M" in the formula for aldehyde content.

4.5.7 Chloride. The chloride content of the propellant shall be determined in accordance with ASTM D512-62T, Method A, with the following exception:

4.5.7.1 Exceptions.

(1) The propellant sample size shall be approximately 50 ml. Determine the weight taken by weighing the sampler to the nearest 0.1 g before and after the liquid is removed.

(2) Place the sample in an evaporating dish and add 5 ml of 0.25N NaOH. Evaporate the mixture to dryness and with distilled water quantitatively transfer the residue to a 250-ml erlenmeyer flask. Proceed with the Method A analysis.

4.5.8 Nonvolatile residue. The nonvolatile residue of the propellant shall be determined in accordance with ASTM D1353-64 with the following exception:

4.5.8.1 Exception. The propellant sample shall be approximately 100 ml. Determine the weight taken by weighing the sampler to the nearest 0.1 g before and after the liquid is removed.

4.5.9 Acetylene. The acetylene content of the propellant shall be determined by the following procedure.

4.5.9.1 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 32° to 39.2°F (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 0.02N sodium hydroxide if acid; or with 0.02N nitric acid if alkaline. Reserve one of the flasks for the blank and into the second flask pour 50 ml of propellant. 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 acetylde by adding an excess of acid ferrous sulfate solution to the sample flask before discarding the solution.)

MIL-P-8845A

4.5.9.2 Reagents and equipment.

(a) Reagents

(1) Mixed indicator. Dissolve 0.10 g of methyl red and 0.050 g of methylene blue in 100 ml of 95% ethanol. Mix and store in a brown bottle.

(2) Silver nitrate solution. Dissolve 25 g of ACS reagent grade AgNO_3 in 150 ml distilled water contained in a 1000-ml volumetric flask. Mix thoroughly and dilute to the mark with 95% ethanol.

(3) Sodium hydroxide solution, 0.02N. Dissolve 0.8 g of ACS reagent grade NaOH in distilled water in a 1000-ml volumetric flask. Dilute to the mark with distilled water.

(4) Nitric acid solution, 0.02N. Dilute 1.3 ml of ACS reagent grade concentrated HNO_3 to the mark with distilled water in a 100-ml volumetric flask.

(5) Acid ferrous sulfate solution, 0.5N. Dilute 8 g of ACS reagent grade ferrous sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) to the mark in a 1000-ml volumetric flask with distilled water. Add these drops of concentrated H_2SO_4 .

(b) Equipment

- (1) Volumetric flasks, 1000 ml and 100 ml.
- (2) Graduated cylinder, 250 ml.
- (3) Erlenmeyer flasks, 250 ml, glass-stoppered
- (4) Ice bath.

4.6 Preparation for delivery inspection. The preservation, packaging, packing, and marking for shipment and storage of the propellant shall be inspected to determine compliance with the requirements of section 5 of this specification.

5. PREPARATION FOR DELIVERY

5.1 Packaging. The propellant shall be packaged in cylinders, drums, portable tanks, cargo tanks, tank cars, or other containers as specified by the procuring activity. All containers shall conform to requirements specified herein, and with the requirements of the Department of Transportation (DOT) as contained in the Code of Federal Regulations 49 CFR 170-190, or special permit obtained from DOT by the shipper in conjunction with Commander, Headquarters Military Traffic

MIL-P-8845A

Management and Terminal Service, Washington, D.C. 20315.

5.1.1 Cylinders. Cylinders shall conform to any DOT specification cylinders of seamless construction and not exceeding 30 gallons (250 lb.) water capacity nominal. The cylinders shall have approved fusible plug-type safety devices.

5.1.2 Drums. Drums shall conform to DOT specification 5P and be not over 61 gallons capacity. The drums shall be lagged. The inner body shall be of stainless steel conforming to MIL-S-4043 and the outer body may be of any weldable carbon steel. The drums shall have approved safety relief devices.

5.1.3 Portable tanks. Portable tanks shall not be used unless authorized by DOT special permit.

5.1.4 Cargo tanks. Cargo tanks shall not be used unless authorized by DOT special permit.

5.1.5 Tank cars. Tank cars shall conform to DOT specifications 105A100-W or 111A100-W-4; or 105A200-W, 105A300-W, 105A400-W, 105A500-W or 105A600-W if equipped with safety relief devices as required for 105A100-W tank cars and are restenciled 105A100-W.

5.1.6 Other containers. Other containers shall conform to the requirements of DOT special permit.

5.2 Preparation of containers. Prior to filling, the contractor shall establish the condition of all containers to insure that they are free from contamination and suitable for shipment and storage. Contractor owned containers shall be cleaned and repaired by the contractor at his expense. Leased or Government owned containers shall be cleaned and repaired in accordance with the schedule established in the contract or purchase order.

5.2.1 Cleaning and repair. All cylinders and drums shall be visually inspected internally and externally for the presence of rust, polymer, other foreign matter, and physical damage. Any physical damage which would endanger safe transportation of the propellant shall be repaired prior to reuse. If evidence of internal contamination is found, the containers shall be cleaned by a suitable method to remove the contamination. A final rinse with ethylene oxide conforming to this specification shall be made to remove residual moisture and to passivate the cleaned surfaces. Other types of containers shall be cleaned as specified in the contract or purchase order.

MIL-P-8845A

5.2.2 Testing. Each cylinder or drum shall be leak tested at a pressure of at least 15 psi with nitrogen gas conforming to MIL-P-27401 before each refilling.

5.2.3 Gaskets. Gaskets used to seal container openings shall be polytetrafluoroethylene or polymonochlorotrifluoroethylene materials, or other materials compatible with the propellant and approved for use by the procuring activity. The contractor shall assure that all gaskets are serviceable and furnish new gaskets when necessary so that a tight seal is assured.

5.3 Filling.

5.3.1 Transfer system. Only materials free of rust, scale, or other sediment and compatible with the propellant shall be used in the transfer system. Cleaning and passivation as necessary shall be performed as specified in 5.2.1.

5.3.2 Transfer. The propellant shall be transferred to the container by pressurization which shall be performed using nitrogen gas conforming to MIL-P-27401, or commercially available methane or natural gas. However, 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 70°F (21°C). Filling shall be such that the container will not be liquid full below 185°F (85°C), or shall not be filled to more than 90 percent of capacity at 68°F (15.6°C) whichever is least. The closure shall be wire sealed to prevent removal in transit.

5.3.3 Pressurization atmosphere. A minimum total pressure of inert gas and propellant (vapor phase) of 35 psig at 70°F (21°C) shall be maintained in the container at all times when it contains propellant.

5.4 Labeling and marking. Each container shall be labeled and placarded in accordance with MIL-STD-129 and established DOT requirements or DOT special permit. In addition, an identification tag and container color code as follows shall be used.

5.4.1 Identification tag. An identification tag impervious to climatic conditions shall be wired to the outlet port of each

MIL-P-8845A

container and shall contain the following information: Propellant name, specification number with revision letter, FSN number, quantity, name of manufacturer, name of contractor (if different from manufacturer), and date of manufacture.

5.4.2 Container color code. Each cylinder and drum shall be color coded in accordance with MIL-STD-101 or MIL-STD-172 as applicable. The exact name identification to be marked on the outside of the container shall be "Ethylene Oxide". Any other name identifications shall be obliterated by removing or over-painting.

6. NOTES

6.1 Intended use. The propellant 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. Method of shipment, type, and capacity of containers
- c. Quantity by weight in pounds (avoirdupois).
- d. That two copies of the test report, signed by the contractor's representative, listing values obtained on all tests (qualitative values where method provides), should accompany each shipment delivered to the consignee. In addition, one copy should be furnished the AFRPL (RPORS), Edwards, California, 93523.

6.3 Highway safety. To promote safety in the transportation of propellants in interstate commerce by motor vehicle, the shipper should assure that each driver possesses an MCA Chem-Card-Transportation Emergency Guide No. CC36. A complete manual of cards or the individual cards are available from the Manufacturing Chemists' Association, 1825 Connecticut Avenue, N.W., Washington, D.C., 20009.

6.4 Pollution control. U. S. Public Laws dictate increased effort to improve air, land, and water pollution control of toxic propellant vapors, leaks, spills, and disposal during all phases of manufacture, transfer, storage, and transportation operations. The manufacturer/supplier is enjoined to approach the appropriate pollution control

MIL-P-8845A

district to mutually resolve all problem areas, and to develop adequate control and disposal methods for situations which are likely to develop in any of the phases.

Custodians:

Army - MI
Navy - AS
Air Force - 12

Preparing Activity:

Air Force - 12

Civilian Agency Interest:

Review Activities:

Air Force - 19, 68

NAS

Project No. 9135-0018

Reviewer/user information is current as of the date of this document. For further coordination of changes to this document, draft circulation should be based on the information in the current DOD Index of Specifications and Standards.

☆ U. S. GOVERNMENT PRINTING OFFICE: 1969-393-066/S-2478

SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 119-R004INSTRUCTIONS

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).

SPECIFICATION

MIL-P-8845A, Propellant, Ethylene Oxide

ORGANIZATION (Of submitter)

CITY AND STATE

CONTRACT NO.

QUANTITY OF ITEMS PROCURED

DOLLAR AMOUNT

\$

MATERIAL PROCURED UNDER A

 DIRECT GOVERNMENT CONTRACT 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?

 YES 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

DD FORM 1426
1 APR 63

REPLACES NAVSHIPS FORM 4863, WHICH IS OBSOLETE

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