DOD-T-82666A
6 November 1978
Superseding
(see 6.3)

MILITARY SPECIFICATION

TRIS-1-(2-METHYL AZIRIDINYL) PHOSPHINE OXIDE

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

1. SCOPE

1.1 Scope. This specification covers the requirements for 3 grades of tris-1-(2-methyl aziridinyl) phosphine oxide.

1.2 Classification. The tris-1-(2-methyl aziridinyl) phosphine oxide shall be of the following grades as specified in the contract (see 6.2):

Grade A - Distilled
Grade B - High Purity
Grade C - Crude

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.

STANDARDS

FEDERAL

FED-STD-313	Material Safety Data Sheets, Preparation and the submission of

MILITARY

MIL-STD-129	Marking for Shipping and Storage
MIL-STD-414	Sampling Procedures and Tables for Inspection by Variables for Percent Defective
MIL-STD-1218	ACS Chemicals

Beneficial comments (recommendation, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Ordnance Station, Standardization Division (611), Indian Head, Maryland 20640 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 6810

PUBLICATIONS

NAVAL SEA SYSTEMS COMMAND (Code Ident. 10001)

OD 18893 Water Content, Determination of

(Copies of specifications, standards, drawings, and publications required by suppliers 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 the date of the invitation for bids or the request for proposal shall apply.

AMERICAN NATIONAL STANDARDS INSTITUTE (ANSI)

ANSI Z 129.1-1976 Precautionary Labeling of Hazardous Industrial Chemicals

(Application for copies should be addressed to the American National Standards Institute, 1430 Broadway, New York, NY 10018.)

CODE OF FEDERAL REGULATIONS

49 CFR 100-199 Transportation

(The Code of Federal Regulations is available from the Superintendent of Documents, US Government Printing Office, Washington, DC 20402. Orders should specify "49 CFR 100-199 (latest revision)".)

(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 Chemical properties. The chemical properties shall conform to Table I.

* TABLE I. Chemical properties.

	Grade A		Grade B		Grade C	
PROPERTIES	MIN	MAX	MIN	MAX	MIN	MAX
Water content, Wt %	-	0.75	-	0.40	-	1.00
Assay, Wt %, by:						
Imine No.	96.0	-	98.0	-	92.0	-
Total Phosphorus	99.0	-	99.0	-	99.0	-
Total Nitrogen	98.0	-	98.0	-	98.0	-
Total Chlorides, wt %	-	0.75	-	0.50	-	2.00
Hydrolyzable Chlorides, wt %	-	0.25	-	0.10	-	1.50

3.2 Material safety data sheets. The contractor shall prepare and submit material safety data sheets in accordance with FED-STD-313 as specified in the contract (see 6.8).

3.3 Processing changes. The suppliers of crude and distilled material shall make no changes in processing techniques or other factors affecting the quality of the product without prior approval of the procuring activity.

3.4 Workmanship. The material shall be uniform, free from contamination, foreign material or any other defect that would prevent its use for the purpose intended.

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 that supplies and services conform to perscribed requirements.

4.2 Inspection conditions. Unless otherwise specified (see 6.2), all inspections shall be performed under the following conditions:

a. Temperature: Room ambient 18 to 35°C (65 to 95°F)
b. Altitude: Normal ground
c. Vibration: Not applicable
d. Humidity: Room ambient to 95 percent relative, maximum

4.3 Sampling. Sampling for quality conformance inspection shall be in accordance with MIL-STD-414, inspection level III, sections A and C. The unit of product shall be one container of material.

4.3.1 Lot. Unless otherwise specified in the contract (see 6.2), a lot shall consist of all material manufactured in one continuous production run or in one batch, under essentially identical conditions, from the same raw materials, and to be offered for acceptance at one time. Several batches, manufactured from the same raw materials, may be blended to form a uniform larger batch which shall then constitute a lot for inspection purposes.

4.4 Quality conformance inspection. Each sample obtained in accordance with 4.3 shall be subjected to the tests of 4.5. The acceptable quality levels (AQL's) for the lower specification limits (LSL) and the upper specification limits (USL) shall be as specified in TABLE II. When specified in the contract (see 6.2.2), three copies of the laboratory analysis shall be furnished to the procuring activity for each lot of material.

TABLE II. Quality conformance inspection.

Requirement	AQL's, % defective		Test Method
	LSL	USL	
Water	-	1.5	4.5.1
Assay			
Imine number	2.5	-	4.5.2
Total phosphorus	4.0	-	4.5.3
Total nitrogen	4.0	-	4.5.4
Total chlorides	-	1.5	4.5.5
Hydrolyzable chlorides	-	2.5	4.5.6

4.5 Tests. Unless otherwise specified herein, all chemicals shall be ACS grade in accordance with MIL-STD-1218.

* 4.5.1 Water content. The water content shall be determined in accordance with either the direct or the indirect titration method of OD 18893. The sample shall be approximately 10 grams (g) weighed to the nearest mg with 100 milliliters (mL) methanol as carrier.

4.5.2 Assay by imine number. The following procedure shall be used to determine the assay by imine number of tris-1-(2-methyl aziridinyl) phosphine oxide.

4.5.2.1 Special reagents.

a. Potassium thiocyanate solution - Dissolve 160 g of potassium thiocyanate in methanol and dilute to one liter (L) with additional methanol.

b. Hydrochloric acid in methanol, 0.4 Normal (N): 34 mL of concentrated hydrochloric acid in 1.0 L of reagent grade methanol.

c. Mixed indicator solution: 0.1% alcoholic bromothymol blue sodium salt and 0.1% alcoholic phenol red sodium salt in a 1:1 mixture.

d. Potassium hydroxide solution, 0.1 N.

4.5.2.2 Procedure.

a. Add 50 mL of potassium thiocyanate solution to a 250-mL Erlenmeyer flask fitted with a nitrogen purge to prevent carbon dioxide absorption. Pipet 10 mL of 0.4 N hydrochloric acid reagent into the flask and start the magnetic stirrer. It is very important that the two solutions are thoroughly mixed before addition of the sample. Weigh a 0.20 to 0.25 g sample to the nearest 0.1 mg, transfer to the flask, and stir for five to six minutes. Add six drops of mixed indicator and titrate visually with standard 0.1 N potassium hydroxide solution to a purple end point.

b. Run two blank standardizations by adding 50 mL of potassium thiocyanate solution to a 250-mL Erlenmeyer flask fitted with a nitrogen purge to prevent carbon dioxide absorption. Pipet 10 mL of 0.4 N hydrochloric acid reagent into the flask and start the magnetic stirrer. Titrate the same as for the sample. Record the volume of potassium hydroxide solution required for the blanks.

c. Calculate the assay by imine number as follows:

$$\text{Assay, weight percent (wt\%)} = \frac{(V_1 - V_2)\ (N)\ (7.168)}{W}$$

Where: V_1 = Average volume of potassium hydroxide used for blank, mL

V_2 = Volume of potassium hydroxide used for sample, mL

N = Normality of potassium hydroxide

W = Weight of sample, g.

4.5.3 Assay by total phosphorus content. The total phosphorus content shall be determined in accordance with the following:

4.5.3.1 Digestion of sample.

a. Weigh a 2.0 g sample, to the nearest 0.1 mg, into an iodine flask. Carefully add 10 mL of 1:1 nitric acid: distilled water, let flask stand until reaction subsides, and cool to room temperature.

b. Add 10 mL of 1:1 nitric acid: distilled water and 10 mL of 1:1 perchloric acid: distilled water to the flask and digest carefully on a hot plate. When sample has turned black remove from hot plate, cool, and add an additional 10 mL of 1:1 nitric acid: distilled water.

* c. Repeat digestion of sample and evaporate almost to dryness. Sample should be colorless (4.5.3.2.b.). Remove sample from hot plate, cool to room temperature, and transfer quantitatively to a 500-mL volumetric flask with distilled water. Dilute to volume.

4.5.3.2 Analysis of sample.

a. Pipet a 20 mL aliquot of the digested sample into a 400-mL beaker. Add three drops of methyl orange indicator solution and concentrated ammonium hydroxide, dropwise, until the color changes from pink to yellow. Add an excess of 9 mL of ammonium hydroxide.

b. Carefully add 10 mL of concentrated nitric acid while swirling the beaker. (This reaction is exothermic.) To the hot solution add 25 mL ammonium molybdate solution (4.5.3.2.c), stir vigorously, and let mixture stand 20 to 30 minutes. Filter precipitate through a gooch crucible containing plates and an asbestos mat. Wash precipitate with 1% ammonium nitrate solution until filtrate is neutral to litmus paper. If sample is still black at this point, add an additional 10 mL of 1:1 nitric acid:distilled water and repeat digestion.

c. Dissolve 100 g of molybdic acid in a mixture of 400 mL of cold distilled water and 80 mL of ammonium hydroxide. Pour the solution slowly and with continuous stirring into a mixture of 400 mL of nitric acid and 600 mL of distilled water. Add 0.05 g of sodium ammonium phosphate dissolved in a few mL of distilled water and stir the mixture. After 24 hours, decant the clear solution through a filter paper into a reagent bottle.

d. Quantitatively transfer the precipitate, plates, and asbestos mat to the beaker which originally contained the precipitate. Carefully wash the crucible with a known volume of 0.5 N sodium hydroxide until all adhering precipitate is dissolved. Rinse crucible with distilled water. Collect all washings in the beaker. Dissolve the precipitate in the beaker with 0.5 N sodium hydroxide solution. Record total volume of sodium hydroxide solution used (25 to 30 mL is usually sufficient). Add 5 to 10 drops of phenophthalein indicator solution and titrate with 0.1 N hydrochloric acid to the colorless end point.

e. Calculate assay by total phosphorus content as follows:

$$\text{Assay, wt\%} = \frac{(A-B)\ (23.374)}{W}$$

Where: A = Mole equivalents (Meg) of sodium hydroxide used, mL x N

B = Meg of hydrochloric acid used, mL x N

W = Weight of sample, g.

4.5.4 Assay by total nitrogen content. The total nitrogen content shall be determined in accordance with the following:

a. Weigh a 0.2 g sample to the nearest 0.1 mg using a paper weighing disc (nitrogen-free). Place in a dry Kjeldahl flask and add approximately 18 g of potassium sulfate, one Hengar granule, and approximately 25 mL of concentrated sulfuric acid. Digest the sample for one hour after the mixture becomes clear.

b. Cool the sample, add 150 mL of distilled water, mix, and again cool to room temperature. Accurately deliver 35.0 mL of standard 0.1 N sulfuric acid into a 300-mL Berzelius beaker. Add two drops of methyl purple indicator and 40 mL of distilled water. Immerse the delivery tube of the distillation apparatus in the solution to about 1.3 centimeters from the bottom of the beaker.

c. Add 80 mL of saturated sodium hydroxide solution (55 g/100 mL water) to the Kjeldahl flask and immediately connect to the condenser of the distillation apparatus. Distill the solution for 20 to 25 minutes, or until the solution starts bumping. Disconnect the flask, rinse the condenser and delivery tube with a small amount of distilled water. Remove the beaker.

d. Titrate the excess acid in the beaker with standard 0.1 N sodium hydroxide solution to a faint green end point.

e. Calculate assay by total nitrogen content as follows:

$$\text{Assay, wt\%} = \frac{(A-B)\ (7.168)}{W}$$

Where: A = Meg of sulfuric acid used, mL x N

B = Meg of sodium hydroxide used, mL x N

W = Weight of sample, mg.

4.5.5 Total chlorides. The total chlorides shall be determined in accordance with the following:

a. Weigh a 0.25 g sample, to the nearest 0.1 mg, in a gelatin capsule (Parr No. 3601, size 00, or equivalent). Add to the fusion cup of the peroxide bomb, (Parr, or equivalent, electric ignition, 42-mL) 7.5 g of sodium peroxide and 0.3 g of potassium nitrate-benzoic acid mixture (2:1). Mix thoroughly. Place the capsule in the fusion cup and cover with 11.3 g of sodium peroxide and 0.3 g of potassium nitrate-benzoic acid (2:1) mixture. Sprinkle approximately 50 mg of benzoic acid on top of the fusion reagents.

b. Assemble the bomb and ignite electrically. Cool the bomb in a water bath for approximately 10 minutes. Dismantle the bomb and with a fine jet of distilled water wash the underside of the head collecting the washings in a 400-mL beaker. Wash again with 5 to 10 mL of 50% nitric acid and rinse with distilled water.

c. Remove the fusion cup from the bomb with a pair of tongs. Remove any materials adhering to the outer surface of the cup by washing with water and discard. Lay the cup on its side in the beaker containing washings from the underside of the head and cover beaker immediately with a watch glass. When the melt has dissolved, remove the cup and wash thoroughly with distilled water. Collect washings in the beaker.

d. Partly cover the beaker with a watch glass and slowly add 18 to 20 mL of concentrated nitric acid. Cool the solution and neutralize with either 50% nitric acid or 50% ammonium hydroxide to a methyl orange end point. Add a 3 mL excess of 50% nitric acid.

e. Total volume of solution should be about 200 mL. Dilute to volume with distilled water, if necessary. Titrate the solution potentiometrically with 0.02 N silver nitrate using a calomel/potassium nitrate bridge and silver/silver chloride electrode pair. Run a blank determination.

f. Calculate the total chlorides as follows:

$$\text{Total chlorides, wt \%} = \frac{(V_1 - V_2)\ (N)\ (3.546)}{W}$$

Where: V_1 = Volume of silver nitrate used for sample, mL

V_2 = Volume of silver nitrate used for blank, mL

N = Normality of silver nitrate

W = Weight of sample, g.

4.5.6 Hydrolyzable chlorides. The hydrolyzable chlorides shall be determined as follows:

a. Weigh a 1 g sample, to the nearest 0.1 mg, into an iodine flask. Add 50 mL of approximately 0.5 N alcoholic potassium hydroxide, insert a condenser, and reflux for 45 minutes.

b. Cool the solution, rinse the condenser with distilled water, and disconnect the flask. Transfer the solution with methanol to a 400-mL beaker, neutralize with 50% nitric acid, and add 3 mL in excess.

c. Total volume of solution should be about 200 mL. Dilute to volume with distilled water, if necessary. Titrate the solution potentiometrically with 0.02 N silver nitrate using a calomel/potassium nitrate bridge and silver/silver chloride electrode pair. Run a blank determination.

d. Calculate hydrolyzable chlorides as follows:

$$\text{Hydrolyzable chlorides, wt \%} = \frac{(V_1 - V_2)\ (N)\ (3.546)}{W}$$

Where: V_1 = Volume of silver nitrate used for sample, mL

V_2 = Volume of silver nitrate used for blank, mL

N = Normality of silver nitrate

W = Weight of sample, g.

4.6 Packaging inspection. The packaging, packing and marking shall be inspected to verify conformance with the requirements of Section 5.

5. PACKAGING

5.1 Packaging and packing. Unless otherwise specified in the contract (see 6.2), packaging and packing shall be level C.

5.1.1 Level C. Tris-1-(2-methyl aziridinyl) phosphine oxide shall be packed in containers not over 5 gallons capacity each for the anticipated mode of transporation in accordance with 49 CFR 171-178.

5.2 Marking. In addition to any special marking required by the contract (see 6.2), each container shall be marked in accordance with MIL-STD-129 and 49 CFR 171-178. Precautionary labels shall be in accordance with ANSI Z 129.1-1976. Marking shall include but not be limited to, the following information:

a. Manufacturer's name and location.

b. Material trade name.

c. Net weight and volume.

d. Lot number, batch number, and date of manufacture.

e. Storage conditions (see 6.5 and 6.6).

f. Toxicity precautions (see 6.4).

g. Number and date of this specification.

h. Contract number.

i. Grade of tris-1-(2-methyl aziridinyl) phosphine oxide (A, B, or C).

* 6. NOTES

6.1 Intended use. The material is intended for use as an ingredient in the manufacture of solid propellant for rocket motors. Grade A material is intended for use in catapults Mk 12 and 18, and the Sidewinder missile. Grade B material is intended for use in the rocket motor for the CKU-5/A rocket assembly, in the M270 impulse cartridge, and in the dual thrust rocket motor Mk 56 Mods 0, 1 and 2. Grade C material is intended to be further purified prior to use in propellant compositions.

6.2 Ordering data. Procurement documents should specify the following:

6.2.1 Procurement requirements.

a. Title, number and date of this specification.

b. Grade of material required (A, B or C) (see 1.2).

c. Quantity required.

d. Place of delivery.

e. Inspection conditions when other than as specified (see 4.2).

f. Lot size if other than as specified (see 4.3.1).

g. Packaging requirements if other than as specified (see 5.1).

h. Size of container required (see 5.1).

i. Any special markings required (see 5.2).

* j. Safety precautions (see 6.4 and 6.8).

* 6.2.2 Data requirements. When this specification is used in a procurement which incorporates a DD Form 1423 and invokes the provisions of 7-104.0(n) of the Armed Services Procurement Regulations, the data requirements identified below will be developed as specified by an approved Data Item Description (DD Form 1664) and delivered in accordance with the approved Contract Data Requirements List (DD Form 1423) incorporated into the contract. When the provisions of ASPR-7-104.9(n) are not invoked, the data specified below will be delivered by the contractor in accordance with the contract requirements. Deliverable data required by this specification is cited in the following paragraph:

PARAGRAPH	DATA REQUIREMENT	APPLICABLE DID
4.4	Quality conformance inspection data	DI-T-3721

(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.)

* 6.3 Supersession information. DOD-T-82666A includes the requirements of and supersedes the following documents:

a. Grade A of DOD-T-82666A supersedes:

1. Grade A of DOD-T-82666(NAVY), dated 31 January 1977, with Amendment 2, dated 1 March 1978, and Interim Amendment 3 (OS), dated 15 June 1978.

2. MIL-M-23945, dated 19 August 1964, with Amendment 1, dated 1 September 1966.

3. Naval Sea Systems Command (Code Ident 10001) purchase descriptions WS16104, Grade A, dated 25 July 1975; XWS3591A, dated 13 March 1968; WS8504A, dated 13 September 1971; and WS9254B, dated 24 December 1970.

4. Naval Air Systems Command (Code Ident 30003) purchase description WS7173, dated 16 November 1967, with Amendment 1, dated 26 May 1970.

b. Grade B of DOD-T-82666A supersedes Grade B of DOD-T-82666(NAVY) and Naval Sea Systems Command purchase description WS 16104, Grade B, dated 25 July 1975.

c. Grade C of DOD-T-82666A supersedes Naval Air Systems Command (Code Ident 30003) drawing 707AS111.

6.4 Safety considerations. The material is a highly-toxic liquid when absorbed through the skin. Care should be exercised in handling this compound. Wear synthetic rubber gloves and avoid any contact with skin, eyes, and clothing. Dispense under nitrogen atmosphere.

6.5 Storage conditions. The material should be stored in a temperature regulated area at -37 to +24°C (-35 to +75°F). Material in opened containers should be stored in a humidity regulated area with a 30% minimum relative humidity.

6.6 Shelf life. Material should be retested to the requirements of TABLE I within the 3 month period preceding use to assure the shelf life has not been exceeded.

6.7 Suggested source of supply. A Grade C product that has met the requirements of this specification in past procurement actions is tris-1-(2-methyl-aziridinyl) phosphine oxide (MAPO) manufactured by ARSYNCO, INC., P.O. Box 8, Foot of 13th Street, Carlstadt, NJ 07072. The Grade C material is then distilled to meet the requirements of Grade A or B at the Naval Ordnance Station, Indian Head, MD 20640. This information is for the convenience of the procuring activity and is not to be construed as a waiver of any requirement of this specification nor as any limitation of additional potential sources of supply.

* 6.8 Material safety data. Material safety data sheet requirements are applicable and should be specified in the contract as required by the Armed Services Procurement Regulations (ASPR) 1-323.2.

* 6.9 Changes from previous issue. The margins of this specification are marked with an asterisk to indicate where changes (additions, modifications, corrections, deletions) from the previous issue were made. This was done as a convenience only and the Government assumes no liability whatsoever for any inaccuracies in these notations. Bidders and contractors are cautioned to evaluate the requirements of the document based on the entire content irrespective of the marginal notation and relationship to the last previous issue.

Custodian:
NAVY - OS

AIR FORCE - 68

Preparing Activity:
NAVY - OS

Review Activity:
NAVY -AS

DLA - GS

Project Number:
6810-B133

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

OMB Approval
No. 22-R255

INSTRUCTIONS: The purpose of this form is to solicit beneficial comments which will help achieve procurement of suitable products at reasonable cost and minimum delay, or will otherwise enhance use of the document. DoD contractors, government activities, or manufacturers/vendors who are prospective suppliers of the product are invited to submit comments to the government. Fold on lines on reverse side, staple in corner, and send to preparing activity. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements. Attach any pertinent data which may be of use in improving this document. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity.

DOCUMENT IDENTIFIER AND TITLE
DOD-T-82666A, TRIS-1-(2-METHYL AZIRIDINYL) PHOSPHINE OXIDE

NAME OF ORGANIZATION AND ADDRESS

CONTRACT NUMBER

MATERIAL PROCURED UNDER A

☐ DIRECT GOVERNMENT CONTRACT ☐ SUBCONTRACT

1. HAS ANY PART OF THE DOCUMENT CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?
   A. GIVE PARAGRAPH NUMBER AND WORDING.

   B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES

2. COMMENTS ON ANY DOCUMENT REQUIREMENT CONSIDERED TOO RIGID

3. IS THE DOCUMENT RESTRICTIVE?

   ☐ YES ☐ NO *(If "Yes", in what way?)*

4. REMARKS

SUBMITTED BY *(Printed or typed name and address - Optional)*

TELEPHONE NO.

DATE

DD FORM 1 JAN 72 1426 REPLACES EDITION OF 1 JAN 66 WHICH MAY BE USED

S/N 0102-LF-014-1802