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TT-T-656C
 5 OCTOBER 1992
 SUPERSEDING
 TT-T-656B
 15 MAY 1965

FEDERAL SPECIFICATION

TRICRESYL PHOSPHATE

This specification is approved by the Commissioner,
 Federal Supply Service, General Services Administration,
 for the use of all Federal agencies.

1. SCOPE

1.1 Scope. This specification covers tricresyl phosphate, a reaction product of cresylic acid and a phosphorus compound. Cresylic acid derived from petroleum or coal tar is acceptable.

2. APPLICABLE DOCUMENTS

* 2.1 Government documents.

* 2.1.1 Specifications and standards. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation (see 6.2).

SPECIFICATIONS

FEDERAL

TT-N-97	Naphtha; Aromatic
O-E-751	Ether, Petroleum; Technical-Grade

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Air Warfare Center Aircraft Division Lakehurst, Systems Requirements Department, Code SR3, Lakehurst, NJ 08733-5100, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

FSC 6810

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STANDARDS

FEDERAL

FED-STD-141	Paint, Varnish, Lacquer, and Related Materials; Methods of Inspection, Sampling, and Testing
FED-STD-791	Lubricants, Liquid Fuels, and Related Products; Methods of Testing

MILITARY

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-129	Marking for Shipment and Storage
MIL-STD-290	Packaging of Petroleum and Related Products

(Unless otherwise indicated, copies of federal and military specifications, standards, and handbooks are available from the Navy Publication and Printing Service Office, Standardization Documents Order Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.)

* 2.2 Non-government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS are the issues of the documents cited in the solicitation (see 6.2).

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D891	Standard Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals
ASTM D1208	Standard Test Method for Color of Halogenated Organic Solvents and their Admixtures (Platinum-Cobalt Scale)
ASTM D1296	Standard Test Method for Odor of Volatile Solvents and Diluents
ASTM D1721	Standard Test Method for Permanganate Time of Tricresyl Phosphate
ASTM D2369	Standard Test Method for Volatile Content of Coatings
ASTM E832	Standard Specification for Laboratory Filter Papers

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

(Non-Government standards and other publications are normally available from the organizations that prepare or distribute the documents. These documents also may be available in or through libraries or other informational services.)

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* 2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein (except for related associated detail specifications, specification sheets or MS standards), the text of this document shall take precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Quantitative requirements. The tricresyl phosphate shall be as specified in table I.

3.2 Qualitative requirements.

3.2.1 Appearance. The tricresyl phosphate shall be clear and free from sediment and suspended matter when examined by transmitted light.

3.2.2 Water. The tricresyl phosphate shall be miscible without turbidity with 19 volumes of 10 degree heptane at 20°C when examined as specified in 4.5.5.

4. QUALITY ASSURANCE PROVISIONS

* 4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements (examinations and tests) as specified herein. Except as otherwise specified in the contract or purchase order, 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 this specification where such inspections are deemed necessary to ensure supplies and services conform to prescribed requirements.

* 4.1.1 Responsibility for compliance. All items shall meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of ensuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling inspection, as part of manufacturing operations, is an acceptable practice to ascertain conformance to requirements, however, this does not authorize submission of known defective material, either indicated or actual, nor does it commit the government to accept defective material.

* 4.2 Classification of inspections. The inspection requirement specified herein is classified as follows:

- a. Quality conformance inspection (see 4.3).

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4.3 Quality conformance inspection. Quality conformance inspection shall consist of the examinations and tests specified herein.

4.3.1 Lot. A lot shall consist of all the tricresyl phosphate in a single storage tank or in containers into which tricresyl phosphate has been transferred from a single storage tank with no concurrent addition of new material taking place during transfer.

4.3.2 Sampling. Sampling for inspection and acceptance shall be in accordance with MIL-STD-105.

4.3.3 Inspection. The inspections specified herein shall be conducted in accordance with FED-STD-791, method 9601, "Inspection requirements".

4.3.4 Inspection level. The inspection level shall be S-2 and the Acceptable Quality Level (AQL) shall be 4.0, as specified by MIL-STD-105.

4.4 Quality conformance tests.

4.4.1 Quality conformance tests. The sample selected in accordance with 4.3.2 shall be subjected to the tests specified in 4.5.

4.5 Test procedures.

4.5.1 Tests shall be conducted as specified in table II and 4.5.2 through 4.5.5.

4.5.2 Acidity. Place 100 milliliters (ml) of the sample in a weighed stoppered 500 ml Erlenmeyer flask, stopper the flask, and reweigh. Add 100 ml of neutral methyl or ethyl alcohol and mix thoroughly by swirling. Titrate with 0.02 normal (N) potassium hydroxide (KOH) in 99 percent methyl or ethyl alcohol using phenolphthalein as the indicator. The end point shall be considered that point in the titration where the pink color persists after continuous swirling of the solution for 30 seconds. From the weight of the sample taken and the volume of the alcoholic KOH used, the volume (ml) of KOH required for the neutralization of 1 gram (g) of the sample shall be calculated.

4.5.3 Ester content.

4.5.3.1 Reagents.

4.5.3.1.1 Potassium hydroxide. Reagent grade potassium hydroxide (KOH) pellets.

4.5.3.1.2 Ethyl ether. Reagent grade ethyl ether.

4.5.3.1.3 Nitric acid. Mixture of 1 ml of nitric acid HNO_3 (specific gravity (sp. gr.) 1.42) and 99 ml of distilled water.

4.5.3.1.4 Ammonium molybdate solution. Mix 100 g of pure molybdic anhydride or 188 g of 85 percent molybdic acid with 400 ml of distilled water and add 80 ml of ammonium hydroxide (sp. gr. 0.09). When solution is complete, filter and pour the solution slowly with constant stirring into a mixture of 400 ml of nitric acid (sp. gr. 1.42) and 600 ml of distilled water. Let settle for 24 hours and filter again.

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4.5.3.1.5 Potassium nitrate solution. Dissolve 10 g of potassium nitrate (KNO_3) in freshly distilled water and dilute to 1 liter.

4.5.3.1.6 Potassium hydroxide solution. Prepare a 0.5 N of KOH solution by dissolving 28 g of KOH in freshly distilled water and diluting to 1 liter. Add sufficient barium hydroxide (BaOH) to precipitate the carbonate. Allow the precipitate to settle out and decant the clear solution. Standardize the solution.

4.5.3.1.7 Sulfuric acid. Prepare 0.5 N solution of sulfuric acid (H_2SO_4) and standardize.

4.5.3.1.8 Phenolphthalein indicator. Dissolve 1 g of phenolphthalein in 100 ml of alcohol.

4.5.3.2 Procedure. Weigh 200.0 ± 0.1 mg of the tricresyl phosphate into a 100 ml silver or nickel crucible. Add 6 g of the KOH pellets and 5 drops of water. Heat the crucible over a low flame until the melt begins to bubble slowly. Maintain sufficient heat to ensure slow bubbling, swirling the contents of the crucible frequently until all the tricresyl phosphate has decomposed and the oily matter has disappeared, about 15 to 20 minutes. Allow to cool and add a small amount of water to dissolve the contents of the crucible. Transfer the contents of the crucible to a 300 ml beaker, wash the crucible with water to bring the solution to 50 to 75 ml. Neutralize the solution with HNO_3 (sp. gr. 1.42) and add 1 to 2 ml in excess. Transfer the solution to a 200 ml separatory funnel and extract with 50 ml portions of ether until the acid solution is clear and the ether layer is practically colorless. 1 to 3 extractions should be sufficient to accomplish this. Transfer the aqueous layer to a 500 ml Erlenmeyer flask and add water to bring its volume to 100 ml. Heat on a steam bath for 30 minutes to remove all traces of ether. Cool, neutralize with ammonium hydroxide (NH_4OH) (sp. gr. 0.90) and add HNO_3 (1 to 99) dropwise until the solution is slightly acid. Add 100 ml of the ammonium molybdate solution. Place the flask in a water bath maintained at 40° to 45°C and allow the precipitate to digest for 1 hour. Swirl the contents of the flask occasionally to agitate the precipitate during the digestion. Filter through a medium speed, ashless type filter paper, ASTM E832, Type II, Class F, (No. 40 Whatman or equal), by decanting the liquid from the precipitate. Wash the flask and precipitate 5 times with 10 to 20 ml portions of the 1 percent KNO_3 solution and filter the washings by decanting from the precipitate in the flask. Wash the filter paper containing most of the precipitate until the washings no longer show an acid reaction, with like portions of the KNO_3 solution, usually 5 to 10 times. When the filter paper has completely drained, transfer it with the precipitate to the 500 ml Erlenmeyer flask. Add 30 ml of the 0.5 N KOH to the contents of the flask. Allow the KOH solution to flow slowly down the inside of the flask to dissolve any precipitate that might be clinging to it. Use a glass rod to macerate the filter paper and break up any lumps of precipitate that might have formed, until all yellow particles have dissolved. Wash down the inside of the flask with approximately 100 ml of water, add 3 drops of phenolphthalein indicator and titrate the excess KOH with 0.5 N H_2SO_4 . Run a blank titration on 30 ml of the KOH solution.

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4.5.3.3 Calculation. Calculate the percentage of tricresyl phosphate as follows:

$$\text{Percent tricresyl phosphate} = \frac{(A - B) \times C \times 0.0165}{W} \times 100$$

where:

- A = Volume difference (in ml) of 0.5 N H₂SO₄ required for titration of 30 ml blank of 0.5 N KOH and volume (ml) of H₂SO₄ required to titrate the excess 0.5 N KOH in sample.
- B = Volume (ml) of 0.5 N KOH required for titration of the free acid in a sample equal in weight to that used for the ester determination.
- C = Normality (N) of the KOH solution.
- W = Weight (g) of sample used.

Note. The cresols generally used for the manufacture of tricresyl phosphate are a mixture of cresols and xylenols. Therefore, the average molecular weight is 112 not 108. Since 23 mols of KOH are required for the titration of 1 mol ammonium phosphomolybdate, 1 ml of 1.0 N KOH is equivalent to 0.165 g tricresyl phosphate.

4.5.4 Phosphite content.

4.5.4.1 Reagents.

- a. 10 percent solution of hydrochloric acid.
- b. Saturated solution of sodium chloride.
- c. 0.05 N solution of iodine.
- d. 0.05 N solution of sodium thiosulfate.

4.5.4.1.1 Procedure. Weigh 10 g of the sample, add 25 g of the hydrochloric acid (HCl) solution, and reflux the mixture for 2 hours. Cool the mixture and transfer to a separatory funnel. Add 25 ml of petroleum ether conforming to O-E-751 or aromatic naphtha conforming to TT-N-97 and 25 ml of the sodium chloride solution. Mix the contents of the separatory funnel by shaking and allow to stand until the nonaqueous layer completely separates from the water layer. Draw off and retain the water layer. Wash the nonaqueous layer four successive times with 25 ml portions of the saturated sodium chloride solution, adding each portion of the separated salt solution to the original water layer. Discard the nonaqueous solution. Neutralize the combined aqueous salt solution with solid sodium bicarbonate and dilute to a volume of 2300 ml with distilled water in a glass-stoppered flask or bottle. Add 10 ml of 0.05 N iodine solution from a burette, thoroughly mix, and allow to stand in the dark for 30 minutes. Cautiously acidify the mixture with 25 percent acetic acid and titrate with 0.05 N sodium thiosulfate solution. Run a blank determination as specified above, using the same reagents.

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4.5.4.1.2 Calculation. Calculate the percentage of tricresyl phosphite as follows:

$$W = \frac{17.6 \times A \times (B - C - D)}{E}$$

where:

- W = Percent by weight of tricresyl phosphite.
- A = Normality (N) of iodine solution.
- B = Volume (ml) of iodine solution originally added.
- C = Volume (ml) of iodine solution titrated with sodium thiosulfate solution.
- D = Volume (ml) of iodine solution used for blank.
- E = Weight (g) of sample.

4.5.5 Water. Transfer 5 ml of the sample to a 100 ml glass-stoppered cylinder and add 10 degree heptane having an aromatic content of not more than 5 percent in 5 ml portions until the total volume specified is obtained. Both the sample and the heptane shall be kept at 20°C during the test and the mixture shall be shaken well after each addition. Turbidity of the mixture indicates the presence of water in the sample.

5. PACKAGING

* 5.1 Packaging and packing. Tricresyl phosphate shall be packaged and packed in accordance with MIL-STD-290, in unit quantities as specified by the procuring activity (see 6.2).

* 5.2 Marking and labeling. In addition to the marking specified in MIL-STD-129 and any special marking required by the procuring activity, each unit and shipping container shall have marking and/or labelling bearing the following:

"WARNING: TOXIC, CONTAINS TRICRESYL PHOSPHATE. AVOID INHALING, SWALLOWING, OR CONTACT WITH SKIN. IN CASE OF CONTACT REMOVE SOILED CLOTHING AND THOROUGHLY WASH EXPOSED SKIN."

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. The uses of tricresyl phosphate are as follows:

- a. Antiwear agent for lubricants.
- b. Manufacture of organic coatings.
- c. Lubrication of vacuum pumps in conjunction with oxygen systems.
- d. Air filter adhesive.

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6.2 Acquisition requirements. Acquisition documents must specify the following:

- a. Title, number and date of this specification.
- b. Issue of DODISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.1.1 and 2.2).
- c. Type and size of container required (see 5.1).
- d. Level of packaging and packing required (see 5.1).
- e. Any special marking and/or labelling required (see 5.2).

* 6.3 Consideration of data requirements. The following data requirements should be considered when this specification is applied on a contract. The applicable Data Item Descriptions (DID's) should be reviewed in conjunction with the specific acquisition to ensure that only essential data are requested/provided and that the DID's are tailored to reflect the requirements of the specific acquisition. To ensure correct contractual application of the data requirements, a Contract Data Requirements List (DD Form 1423) must be prepared to obtain the data, except where DOD FAR Supplement 27475-1 exempts the requirements for a DD Form 1423.

<u>Reference Paragraph</u>	<u>DID Number</u>	<u>DID Title</u>	<u>Suggested Tailoring</u>
3.1	DI-E-3130	Process Specification	Use 10.2.7.1 for certification.

6.4 Purchase volume. Tricresyl phosphate should be purchased by volume, the unit being one U.S. gallon of 231 cubic inches at 20°C. The volume of deliveries may be determined by dividing the net weight, in pounds, by the weight per gallon. To obtain the weight per gallon, multiply the specific gravity at 20°/20°C by 8.322. One gallon of tricresyl phosphate at 20°C weighs between 9.57 and 9.82 pounds.

* 6.5 Subject term (key word) listing.

Adhesive
Antiwear agent
Lubrication
Petroleum additive

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* 6.6 Changes from previous issue. The margins of this specification are marked with asterisks 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 this document based on the entire content irrespective of the marginal notations and relationship to the last previous issue.

Custodians:

Navy - AS
Air Force - 68

Preparing Activity:

Navy - AS
(Project No. 6810-1147)

Review activities:

Navy - SH, OS
DLA - GS

Civil Agency Coordinating Activity:

GSA - FSS

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TABLE I. Quantitative requirements.

Characteristics	Requirements
Color (Pt-Co scale)	100, maximum
Odor	No objectionable odor
Oxidizable substances (Permanganate time)	No color change after 30 minutes ^{1/}
Specific gravity, 20°/20°C	1.15 - 1.18
Nonvolatile matter (percent by weight)	99.8
Acidity, mg. of KOH per gram of sample	0.20, maximum
Refractive index	1.55 - 1.56
Ester content, percent by weight (as tricresyl phosphate)	99.0, minimum
Ortho-tricresyl phosphate, percent by weight	1.0, maximum.
Phosphite content, percent by weight	0.05, maximum

^{1/} Color of permanganate solution remains pink after a thirty minute dwell time.

TABLE II. Test requirements.

Test	Reference paragraph	Test paragraph method	FED-STD-141 method	ASTM method
Appearance	3.2.1	---		
Odor	Table I	---		D1296
Acidity	Table I	4.5.2		
Ester content	Table I	4.5.3		
Water	3.2.2	4.5.5		D891
Specific gravity	Table I			D2369
Nonvolatile matter	Table I		4371	
Refractive index	Table I		4261	
Phosphite content	Table I			D1208
Color	Table I			D1721
Oxidizable substance (Permanganate time)	Table I			
Ortho-tricresyl phosphate	Table I	^{1/}		

^{1/} Unless otherwise specified by the procuring activity, a certificate of compliance shall be submitted by the manufacturer or supplier confirming compliance to this requirement.

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.
2. The submitter of this form must complete blocks 4, 5, 6, and 7.
3. The preparing activity must provide a reply within 30 days from receipt of the form.

NOTE: This form may not be used to request copies of documents, nor to request waivers, or clarification of requirements on current contracts. 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.

I RECOMMEND A CHANGE:

 1. DOCUMENT NUMBER
 TT-T-656C

 2. DOCUMENT DATE (YYMMDD)
 921005

3. DOCUMENT TITLE

TRICRESYL PHOSPHATE

4. NATURE OF CHANGE (Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.)

5. REASON FOR RECOMMENDATION

6. SUBMITTER

a. NAME (Last, First, Middle Initial)

b. ORGANIZATION

c. ADDRESS (Include Zip Code)

d. TELEPHONE (Include Area Code)

 7. DATE SUBMITTED
 (YYMMDD)

(1) Commercial

(2) AUTOVON

(If applicable)

8. PREPARING ACTIVITY

 a. RECOMMENDING OFFICER, NAVAL AIR
 FARE CENTER AIRCRAFT DIVISION LAKEHURST
 SYSTEMS REQUIREMENTS DEPARTMENT

b. TELEPHONE (Include Area Code)

(1) Commercial

(908) 323-7488

(2) AUTOVON

624-7488

c. ADDRESS (Include Zip Code)

 CODE SR3
 LAKEHURST, NJ 08733-5100

 IF YOU DO NOT RECEIVE A REPLY WITHIN 45 DAYS, CONTACT:
 Defense Quality and Standardization Office
 5203 Leesburg Pike, Suite 1403, Falls Church, VA 22041-3466
 Telephone (703) 756-2340 AUTOVON 289-2340