Designation: D 1399 – 95 (Reapproved 2000)

Standard Test Method for Unsaponifiable Contents of Tricresyl Phosphate[1]

This standard is issued under the fixed designation D 1399; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the amount of unsaponifiable impurities in tricresyl phosphate.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 7.

1.3 For hazard information and guidance, see the supplier's Material Safety Data Sheet.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

3. Summary of Test Method

3.1 The specimen is refluxed in the presence of an aqueous solution of sodium hydroxide which converts the phosphate ester to water-soluble salts. The saponified material is extracted with petroleum ether which dissolves any unsaponifiable or water-insoluble material. The combined extracts are evaporated in a tared evaporating dish, and the residual nonvolatile material is determined.

4. Significance and Use

4.1 This test method determines the amount of unsaponifiable impurities in tricresyl phosphate remaining after manufacture or that may be introduced during handling and storage.

4.2 Water-insoluble materials, which do not evaporate on a steam bath or in a drying oven operated at 75 ± 5°C for 1 h, will be included as unsaponifiable material.

5. Apparatus

5.1 *Reflux Apparatus*, consisting of a 250-mL Erlenmeyer flask equipped with a 24/40 standard-taper joint attached to an Allihn-type reflux condenser similarly equipped with a 24/40 standard-taper joint.

5.2 *Separatory Funnel*, having a capacity of 250 mL.

5.3 *Evaporating Dish*, porcelain or glass, having a capacity of 120 mL.

5.4 *Steam Bath*.

5.5 *Drying Oven*, thermostatically controlled, at a temperature of 75 ± 5°C.

6. Reagents

6.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] Otherwise, the best available grade shall be used.

6.2 *Purity of Water*—References to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

6.3 *Petroleum Ether*, having a boiling range from 30 to 65°C.

6.4 *Sodium Hydroxide (NaOH) Pellets*.

7. Hazards

7.1 Tricresyl phosphate is hazardous through inhalation or skin absorption. Care should be taken in handling the material.

7.2 Ortho-isomer of tricresyl phosphate is considered toxic. Trace amounts may be present in tricresyl phosphate specimens.

8. Procedure

8.1 Place approximately 30 mL of water in the 250-mL Erlenmeyer flask and to this carefully add approximately 10 g of NaOH pellets. Carefully swirl the flask to dissolve the NaOH, and cool the solution to room temperature.

8.2 By means of a weighing pipet, transfer a 10-g specimen, weighed to 1 mg, to the flask. Add several boiling-stones, connect the flask to the reflux condenser, and reflux for 1 h. (**Warning—**See 7.1 and 7.2.)

8.3 At the end of the refluxing period remove the flask from the heat and wash the condenser down with 50 mL of water.

8.4 Cool the flask to room temperature and transfer its content to the 250-mL separatory funnel. Extract the saponified

---

[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

Current edition approved Aug. 15, 1995. Published October 1995. Originally published as D 1399 – 56 T. Last previous edition D 1399 – 90.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

material three times with 25-mL portions of petroleum ether. Exercise particular care in separating the two phases so that none of the aqueous phase is occluded in the ether phase.

8.5 Combine the ether extract and place in the 120-mL evaporating dish, weighed to 1 mg. Place on the steam bath and carefully evaporate to dryness. Dry the bottom of the evaporating dish with a clean lint-free cloth and place in the drying oven set at 75 ± 5°C for 1 h. Cool in a desiccator and weigh the evaporating dish and its contents to 1 mg.

9. Calculation

9.1 Calculate in weight percent the unsaponifiable content, *U*, of the specimen as follows:

$$U = (R/S) \times 100 \quad (1)$$

where:

R = residue weight from evaporation, g, and

S = specimen weight, g.

10. Precision and Bias

10.1 *Precision*—Results should not differ from the mean by more than the following amounts:

	Repeatability (One operator and apparatus)	Reproducibility (Different operators and apparatus)
Unsaponifiable content, weight % absolute	0.05	0.1

10.2 *Bias*—Bias cannot be determined because there is no available material having an accepted reference value.

11. Keywords

11.1 tricresyl phosphate; unsaponifiable content