



Standard Test Methods for Scavenger Content of Askarels¹

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1. Scope

1.1 These test methods cover the determination of the amount of an additive, either tin tetraphenyl or phenoxypropene oxide, (1,2-epoxy-3-phenoxypropane) or dicyclopentadiene carboxylate (3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexane-carboxylate), present in askarels as a scavenger for arc-formed gases. Separate procedures are described for each scavenger.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water²

3. Significance and Use

3.1 Measurement of scavenger content indicates the amount of protection available against arc-formed gases and permits estimation of the amount of makeup additive required.

3.2 Many askarels contain polychlorinated biphenyls (PCBs). Manufacture and sale of PCBs has been prohibited by Federal regulation in the United States since 1978. The conditions for continued use and the handling and disposal of PCBs are also regulated. Users of this and other methods of measuring the properties of askarels containing PCBs are responsible for following applicable Federal, state, and local regulations.

4. Identification of Additive

4.1 Tin tetraphenyl can be readily identified because it separates from askarel fluid on cooling; if a test specimen is cooled to – 20°C the tin tetraphenyl will appear on the surface as a crystalline layer.

4.2 No rapid identification test for phenoxypropene oxide or dicyclopentadiene carboxylate is as yet available; if a test specimen of askarel gives a negative result for tin tetraphenyl

upon cooling, it may be assumed to contain one of the epoxides and may be tested accordingly.

5. Purity of Reagents

5.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. 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 Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Use reagent grade water conforming to Specification D 1193, unless otherwise specified.

TIN TETRAPHENYL

6. Summary of Test Method

6.1 Reacted scavenger is measured by hydrolyzing the reaction product; the hydrochloric acid formed enters into the water phase and is titrated with standard potassium hydroxide solution. Total scavenger is measured by reacting the test specimen with dry hydrochloric acid gas, then measuring the reacted scavenger content. Unreacted scavenger content is equal to the total scavenger content minus the reacted scavenger content.

7. Apparatus

7.1 *Separatory Funnel*, Squibb, pearshaped, 125-mL capacity, with standard-taper joint.

7.2 *Standard-Taper Joint*, male, fitted with a side-arm tube above the joint for exit of gas, and a bubbling tube that reaches within 1 cm of the bottom of the funnel and is provided with a capillary tip.

7.3 *Drying Tower*, for air supply.

8. Reagents and Materials

8.1 *Hydrogen Chloride Gas, Dry*—Dry hydrogen chloride gas (HCl) from either a cylinder or generator. Dry the gas through a sulfuric acid drying tower.

¹ These test methods are under jurisdiction of ASTM Committee D-27 on Electrical Insulating Liquids and Gases and are the direct responsibility of Subcommittee D27.06 on Chemical Test.

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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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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8.2 *Phenolphthalein Indicator Solution* (10 g/L)—Dissolve 1 g of phenolphthalein in 50 mL of alcohol and add 50 mL of water.

8.3 *Potassium Hydroxide, Standard Solution* (0.1 N)—Prepare and standardize a 0.01 N solution of potassium hydroxide (KOH).

9. Procedure for Reacted Scavenger

9.1 Clean, dry, and weigh the separatory funnel. Pipet 10 mL of the test specimen into the funnel and determine the mass of the test specimen to the nearest 0.1 g.

NOTE 1—Askarel test specimens containing visible water may give misleading results and, therefore should be clarified by paper filtration before testing.

9.2 Add 50 mL of water to the funnel. Stopper the funnel and shake violently for 10 s for a minimum of five separate times over a period of not less than 5 min. Transfer the contents of the funnel to a 250-mL Erlenmeyer flask, rinse the funnel with 25 mL of water, and add this to the Erlenmeyer flask.

9.3 Add several drops of phenolphthalein indicator solution. Titrate with 0.01 N KOH solution until the first pink color is retained for 15 s. Swirl the flask gently with a minimum of surface agitation during the titration. The pink end point color will fade on standing, due to absorption of carbon dioxide from the air.

9.4 Perform a blank titration on 75 mL of water containing several drops of phenolphthalein indicator. Titrate to the same end point.

9.5 *Calculation*—Calculate the percentage of tin tetraphenyl as follows:

$$\text{Tin tetraphenyl, \%} = [(A - B)N \times 21.35]/W \quad (1)$$

where:

- A = millilitres of KOH solution used for titration of the test specimen,
- B = millilitres of KOH solution used for titration of the blank,
- N = normality of the KOH solution, and
- W = grams of test specimen used.

10. Procedure for Total Scavenger

10.1 Clean, dry, and weigh the separatory funnel. Pipet 10 mL of the test specimen (Note 2) into the funnel and determine the mass of test specimen taken to the nearest 0.1 g. Attach the male standard-taper joint and pass a rapid stream of dry air bubbles through the test specimen for 10 min. Rotate the funnel occasionally to wash down drops spattered on the sides.

NOTE 2—Askarel test specimens containing visible water may give misleading results and, therefore, should be clarified by paper filtration before testing.

10.2 Disconnect the air supply and connect the source of dry HCl gas. Bubble vigorously for 10 min, rotating the funnel occasionally to wash down spattered drops.

10.3 Disconnect the HCl gas source and reconnect the air supply. Bubble air for 10 min as before. Then complete the test method as in 9.2 to 9.5.

11. Unreacted Scavenger

11.1 Calculate the percentage of unreacted tin tetraphenyl as follows:

$$\text{Unreacted tin tetraphenyl, \%} = A - B \quad (2)$$

where:

- A = percentage of total tin tetraphenyl (Section 10), and
- B = percentage of reacted tin tetraphenyl (Section 9).

UNREACTED PHENOXYPROPENE OXIDE

12. Summary of Test Method

12.1 The phenoxypropene oxide is reacted with excess pyridinium chloride in the presence of pyridine, and the excess pyridinium chloride is titrated with standard sodium hydroxide solution to a cresol red-thymol blue end point.

13. Reagents and Materials

13.1 *Cresol Red-Thymol Blue Indicator Solution*—Mix 1 part of an aqueous solution of cresol red (1 g/L) and 3 parts of an aqueous solution of thymol blue (1 g/L).

13.2 *Pyridinium Chloride Reagent*—Slowly dissolve 16 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) in 1 L of pyridine.

13.3 *Sodium Hydroxide, Standard Solution* (0.1 N)—Dissolve 4 g of sodium hydroxide (NaOH) in 1 L of water. Standardize against potassium acid phthalate.

14. Procedure

14.1 Clean, dry, and weigh a 500-mL Erlenmeyer flask. Add approximately 100 g of the test specimen, weigh, and determine the mass of the test specimen taken by difference to the nearest 0.05 g.

14.2 With a pipet, add 20 mL of the pyridinium chloride reagent, attach a reflux condenser, and reflux 30 min. Measure the reflux time from the start of boiling. At the end of the reflux period, turn off the heat and pour into the top of the condenser 50 mL of water. Detach the flask from the condenser and, with a small stream of water from a wash bottle, wash the end of the condenser with a few millilitres of water. Let the washings drain into the flask. Cool the flask and contents to room temperature with ice or tap water.

14.3 Add 1 mL of cresol red-thymol blue indicator solution and titrate with 0.1 N NaOH solution to a distinct purple color. Swirl the flask rapidly so that there is thorough mixing of the contents (Note 3).

NOTE 3—If any doubt exists about the end point, allow the layers to separate completely after each addition of NaOH solution when close to the end point. The end point has been reached when the upper aqueous layer is blue.

14.4 Obtain a blank titration by repeating the procedures described in 14.1, 14.2, and 14.3, substituting 70 mL of an askarel known to contain no phenoxypropene oxide.⁴

14.5 *Calculation*—Calculate the percentage of phenoxypropene oxide as follows:

⁴ Type H askarel with no scavenger can be obtained from Standard Chlorine of Delaware, Governor Lea Road, Delaware City, DE 19706.