



Standard Test Method for Inorganic Chlorides in Askarels¹

This standard is issued under the fixed designation D 1821; 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 inorganic chloride ions in askarels, in the range 0.02 to 100 ppm.

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.* Specific precautionary statements are given in 8.2.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 Chloride ion is determined by potentiometric titration in an essentially nonaqueous medium.

4. Significance and Use

4.1 In the presence of water, chlorides can ionize. Their corrosive action can then be detrimental to the life of the apparatus in which the askarel is used.

4.2 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.

5. Interferences

5.1 Other halogens interfere but will not normally be encountered in making this test.

6. Apparatus

6.1 *Electrometric Titrimeter:*³

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

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² *Annual Book of ASTM Standards*, Vol 10.01.

³ Beckman Model GS, Beckman Model G, Leeds & Northrup Catalog No. 7664, and the Fisher Titrimeter have been found satisfactory for this purpose.

6.2 *Electrode System for Titrimeter*—The most satisfactory electrode system, with respect to length of life and ease of maintenance, is a silver-glass system.⁴ Other electrodes that have given satisfactory results are silver-silver chloride⁵ and silver (wire or billet)-mercurous sulfate, prepared as follows:

6.2.1 Dismantle the internal element from the salt bridge tube of a standard calomel reference electrode. Discard the saturated potassium chloride (KCl) solution from the tube, and clean out the mercurous chloride (Hg_2Cl_2) and mercury from the internal element. Clean the parts thoroughly. Add sufficient new clean mercury to the internal element to make contact with electrode wire, and repack the chamber of the internal element with mercurous sulfate (Hg_2SO_4) moistened with 0.5 M potassium sulfate (K_2SO_4) solution. Seal the chamber with nonabsorbent cotton. Fill the salt bridge tube with 0.5 M K_2SO_4 solution and reassemble the units.

6.2.2 The life of the silver-mercurous sulfate electrode is limited. It should be repacked when the mercurous sulfate turns from white to brown.

6.3 *Buret*—Microburet, 1.0-mL capacity.⁶

6.4 *Magnetic Stirrer*, with glass-covered or TFE-fluorocarbon-coated stirring bar.

7. Reagents and Materials

7.1 *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.⁷ 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.

7.2 *Purity of Water*—Use reagent grade water conforming to Specification D 1193, unless otherwise specified. In addition, the water shall be chloride-free, as detected by this test.

7.3 *Acetone*, containing no titratable chloride.

⁴ Beckman silver billet electrode No. 39261 and glass electrode No. 40498 have been found satisfactory for this purpose.

⁵ Obtainable from Fisher Scientific Co.

⁶ Fisher Catalog No. 20-110 and Scientific Glass Apparatus Co. No. JM-570 burets have been found satisfactory for this purpose.

⁷ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."