



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 (Hg₂Cl₂) 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 (Hg₂SO₄) moistened with 0.5 M potassium sulfate (K₂SO₄) solution. Seal the chamber with nonabsorbent cotton. Fill the salt bridge tube with 0.5 M K₂SO₄ 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."

7.4 *Nitric Acid* (1 + 69)—Mix 1 volume of concentrated nitric acid (HNO_3 , sp gr 1.42) in 69 volumes of water.

7.5 *Silver Nitrate, Standard Solution* (0.005 N)—Prepare by dissolving 0.8495 g of silver nitrate (AgNO_3) in 1000 mL of water containing 3 mL of concentrated nitric acid (HNO_3 , sp gr 1.42), or by diluting 5 mL of 0.1 N AgNO_3 to 100 mL.

NOTE 1—Dry the AgNO_3 overnight in a desiccator before preparing the solution. Both the solid material and the solution must be protected from light by storage in brown glassware in the dark.

8. Preparation of Apparatus

8.1 In the determination of low levels of chloride content, use extreme care in the cleaning and handling of all glassware. Glassware should receive a final rinse in water and should not be touched with the hands afterwards.

NOTE 2—**Caution:** Test specimens should not be exposed nor the test conducted in an atmosphere containing appreciable quantity of chlorides, as contamination will occur and erroneous results will be obtained.

9. Procedure

9.1 Weigh 100 g of askarel into a clean 250-mL beaker. Weigh to ± 0.5 g.

9.2 Add 100 mL of acetone measured in a 100-mL graduate. Add 2 mL of HNO_3 (1 + 69).

9.3 Titrate with 0.005 N AgNO_3 solution, using a magnetic stirrer with glass-covered or TFE-fluorocarbon-coated stirring bar and an electrode system as described in 6.2. Normal test specimens of askarel require extremely small amounts of AgNO_3 solution, and for this reason titrate using 0.01-mL additions and allowing sufficient time for equilibrium to be established before recording the emf change. If a change of less than 1 mV/0.01-mL addition is observed for three or four 0.01-mL increments, larger additions of AgNO_3 solution may be used, for instance 0.05 mL, until such a change is observed. Then reduce the additions to 0.01 mL again to complete the titration. The end point normally is defined by two 50-mV changes. A normal titration would yield data such as are shown in Table 1.

9.4 To calculate the change per 0.01 mL observed, divide

the change in millivolts by the volume of AgNO_3 solution. By plotting $\Delta \text{mV}/\Delta \text{mL}$ versus millilitres, the end point may be found to the nearest 0.001 mL. This gives a sensitivity of ± 0.0017 ppm. To define the end point to ± 0.01 mL no plotting is necessary, and a sensitivity of ± 0.02 ppm is assumed.

9.5 Repeat 9.2 and 9.3, using an acetone blank. The value obtained on the blank, in addition to its use as a correction to the value obtained on the test specimens, can also serve as an indicator of cleanliness of the glassware, purity of the reagents, and absence of chlorides from the atmosphere. Normally 1 drop of 0.005 N AgNO_3 solution should be sufficient to titrate the blank.

10. Calculation

10.1 Calculate the chloride content as follows:

$$\text{Chloride content, ppm} = 1.77(A - B)$$

where:

A = millilitres of 0.005 N AgNO_3 solution used to titrate the test specimen, and

B = millilitres of 0.005 N AgNO_3 solution used to titrate the blank.

11. Precision and Bias

11.1 *Repeatability*—Interlaboratory testing indicates that the difference between two measurements in one laboratory should not exceed (95 % of the time):

Approximate chloride content, ppm	Repeatability, ppm
0.1	0.002
1.0	0.003
4.0	0.084

11.2 *Reproducibility*—The difference between two laboratories, each reporting the average of two determinations, should not exceed (95 % of the time):

Approximate chloride content, ppm	Reproducibility, ppm
0.1	0.050
1.0	0.082
4.0	0.189

11.3 *Bias*—No justifiable statement can be made on the bias of this test method for testing the property involved. The true value of the property cannot be established by acceptable referee methods.

11.4 The statement of precision and bias was developed at the time that this test method was originally published and, although a supporting research report is no longer available, is considered to be valid. A supplementary inter-laboratory test cannot be conducted due to the unavailability of unused PCB-containing askarels to serve as standards.

12. Keywords

12.1 askarels; inorganic chlorides; potentiometric titration

TABLE 1 Typical Data for Titration

mV	ΔmV	mL	ΔmL	$\frac{\Delta \text{mV}}{\Delta \text{mL}} \times 10^{-2}$
400	0	0.06	0	0
392	8	0.07	0.01	8
352	8	0.08	0.01	8
341	11	0.09	0.01	11
321	20	0.10	0.01	20
271	50	0.11	0.01	50
221	50	0.12	0.01	50
201	20	0.13	0.01	20
285	16	0.14	0.01	16

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