



Standard Test Method for Hydrolyzable Chlorine Compounds in Chlorinated Aromatic Hydrocarbons (Askarels) by Refluxing¹

This standard is issued under the fixed designation D 2441; 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 hydrolyzable chlorine compounds in chlorinated aromatic hydrocarbons (askarels).

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 a specific hazard statement, see Section 8.

1.3 The values stated in SI units are to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water^{2,3}

D 1820 Test Method for Hydrolyzable Chlorine Compounds in Chlorinated Aromatic Hydrocarbons (Askarels)⁴

3. Summary of Test Method

3.1 This test method uses a quantitative potentiometric titration using silver nitrate in an essentially nonaqueous medium to determine chloride ions. These are obtained after methanolic sodium hydroxide hydrolysis of unstable chlorine compounds in chlorinated aromatic hydrocarbons of the askarel dielectric class.

4. Significance and Use

4.1 The measured chloride ion, reported as parts per million in the askarel test specimen, is indicative of the relative stability of the askarel under prescribed conditions of test.

4.2 Refluxing with methanolic sodium hydroxide offers the detection of certain chlorine addition compounds that could be present as impurities and do not respond fully to hydrolysis at the lower temperature used in Test Method D 1820.

4.3 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 for measuring the properties of askarels containing PCBs are responsible for following applicable Federal, state, and local regulations.

5. Interferences

5.1 The presence of compounds containing hydrolyzable halogen other than chlorine, that is, bromine and iodine, will give positive results, as will also any other foreign substance capable of forming an insoluble compound with silver nitrate. Such material would not normally be present in an askarel.

5.2 The amount of chloride ions measured when the test is applied to normally stable askarels is very small, ranging near 1 ppm. Therefore, the test must be made in an area where the atmosphere is free of contaminating influences and it is essential to make a blank determination. The value obtained on the blank will indicate the degree to which the test environment, reagents, and glassware are satisfactory for this type of testing.

6. Apparatus

6.1 *Flask*, borosilicate glass, 500-mL, with 24/40 standard-taper joint.

6.2 *Reflux Condenser*, with mated 24/40 standard-taper joint.

6.3 *Combination Magnetic Stirrer*⁵ and *Hot Plate*.

6.4 *Magnetic Stirring Bar*.⁶

6.5 *Microburet*, 1.0-mL capacity, graduated in 0.01-mL divisions.

6.6 *Electrodes*, silver and glass.⁷

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 11.01.

³ *Annual Book of ASTM Standards*, Vol 10.01.

⁴ *Annual Book of ASTM Standards*, Vol 10.03.

⁵ A Tempco, Aloe Scientific Co., St. Louis 3, MO, Catalog No. V-75655 or equivalent is satisfactory.

⁶ A TFE-fluorocarbon-covered, 1-in. long magnetic stirring bar from Fisher Scientific Co., Catalog No. 9-311-9 or equivalent is satisfactory.

⁷ Beckman Catalog No. 39261 silver billet electrode and Catalog No. 40498 glass electrode have been found satisfactory for this purpose. Although the silver billet-glass electrode system is preferred, a silver electrode with a mercurous sulfate reference electrode may be used. However, a silver-silver chloride electrode system should not be used.