



Standard Test Method for Hydrolyzable Chloride in Peroxy Esters and Peroxy Dicarbonates¹

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1. Scope *

1.1 This test method covers determination of the hydrolyzable chloride content of peroxy esters and peroxy dicarbonates.

NOTE 1—There are no ISO standards covering the primary subject of this test method.

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

1.3 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of this standard.

1.4 *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 hazards statements are given in Section 8.

2. Referenced Documents

2.1 ASTM Standards:

D 883 Terminology Relating to Plastics²

D 1193 Specification for Reagent Water³

D 1600 Terminology for Abbreviated Terms Relating to Plastics²

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis⁴

3. Terminology

3.1 *General*—Unless otherwise indicated, definitions are in accordance with Terminology D 883 and abbreviations are in accordance with Terminology D 1600.

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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² *Annual Book of ASTM Standards*, Vols 08.01 and 08.04.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

4. Summary of Test Method

4.1 The hydrolyzable chloride content of the peroxy ester or peroxy dicarbonate is determined volumetrically using silver nitrate titrant after the alkaline hydrolysis of the material. The end point of the titration is detected potentiometrically.

5. Significance and Use

5.1 Peroxy esters and peroxy dicarbonates are used widely as initiators and chemical intermediates in the production and modification of polymers, including PVC, polyethylene, and polypropylene. The level of hydrolyzable chloride in the peroxide species may affect its performance in some applications. This test method provides a procedure for determining the hydrolyzable chloride content of peroxy esters and peroxy dicarbonates.

5.2 As measured by this test method, hydrolyzable chloride is defined as the sum of the inorganic chloride content plus the chloride content generated upon alkaline hydrolysis of the peroxy ester or peroxy dicarbonate.

6. Apparatus

6.1 *Automatic Potentiometric Titrimeter*, consisting of a potentiometer with a ± 2 -mV sensitivity, a buret graduated in 0.001-mL divisions, and a mechanical stirrer.

6.1.1 A system consisting of a Metrohm AG Model 682 Titroprocessor with a Model 664 Control Unit and a Model 665 Dosimat⁵ has been used successfully.

6.2 *Combined Silver Electrode*—Brinkman Instruments Catalog No. 020-92-460-8 (Metrohm AG 6.0404.100)⁵ or equivalent.

7. Reagents

7.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. 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

⁵ Available from Brinkman Instruments, Inc., One Cantiague Road, Westbury, NY 11590-0207.

*A Summary of Changes section appears at the end of this standard.

available.⁶ 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*—Unless otherwise indicated, references to water shall be understood to mean Type II or III reagent water conforming to Specification D 1193.

7.3 *Isopropyl Alcohol*, pesticide residue grade.

7.4 *Nitric Acid* (46 g/L)—Dilute 3.0 mL of concentrated nitric acid to 100 mL with water.

7.5 *Silver Nitrate* (0.1 mol/L)—Prepare and standardize in accordance with the appropriate sections of Practice E 200.

7.6 *Silver Nitrate* (0.005 mol/L)—Dilute 50.00 mL of silver nitrate (0.1 mol/L) to 1 L in a volumetric flask.

7.7 *Sodium Hydroxide* (1:50)—Dissolve 2.0 g of sodium hydroxide pellets in 100 mL of water.

8. Hazards

8.1 Organic peroxides are strong oxidizing agents and present potential fire and explosion hazards. Avoid contact with reducing agents and sources of heat, sparks, or open flames. Reactivity varies widely, and some compounds may explode when shocked. Organic peroxides are irritating to the skin, eyes, and mucous membranes. Avoid bodily contact and handle only in a well-ventilated area.

9. Procedure

9.1 Conduct the following procedure for the sample and a reagent blank.

9.2 Transfer approximately 5 g of accurately weighed sample into a 250-mL beaker.

9.3 Add 50 mL of isopropyl alcohol.

9.4 Add 15 mL of sodium hydroxide (1:50) solution. Stir the solution for 30 min.

9.5 Add 25 mL of nitric acid (46 g/L) and an additional 50 mL of isopropyl alcohol.

9.6 Titrate with the 0.005 N silver nitrate solution to the potentiometric end point.

10. Calculation

10.1 Calculate the hydrolyzable chloride content:

$$Cl = \frac{(A - B) \times C \times 35.45 \times 10^3}{W} \quad (1)$$

where:

- Cl* = hydrolyzable chloride content, ppm,
- A* = sample titration volume, mL,
- B* = blank titration volume, mL,
- C* = concentration of silver nitrate titrant, mol/L,
- W* = sample weight, g, and
- 35.45 = equivalent weight of chloride ion.

11. Precision and Bias

11.1 A task force is continuing to attempt to develop a precision and bias statement for this test method. For this reason, data on precision and bias cannot be given. Because this test method does not contain a numerical precision and bias statement, it shall not be used as a referee test method in case of dispute. Anyone wishing to participate in the development of precision and bias data should contact the chairman, Subcommittee D20.15, ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

12. Keywords

12.1 hydrolyzable chloride; peroxy dicarbonates; peroxy esters; PVC

⁶ "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 "Analar Standards for Laboratory U.K., Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D-20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 5318 – 97:

(I) Added 1.3.

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