

Designation: D 5318 - 97

# Standard Test Method for Hydrolyzable Chloride in Peroxy Esters and Peroxy Dicarbonates<sup>1</sup>

This standard is issued under the fixed designation D 5318; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 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 Plastics<sup>2</sup>
- D 1193 Specification for Reagent Water<sup>3</sup>
- D 1600 Terminology for Abbreviated Terms Relating to Plastics<sup>2</sup>
- E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis<sup>4</sup>

#### 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.

# 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  $\pm 2$ -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 Dosimat<sup>5</sup> has been used successfully.
- 6.2 Combined Silver Electrode—Brinkman Instruments Catalog No. 020-92-460-8 (Metrohm AG 6.0404.100)<sup>5</sup> 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

 $<sup>^{\</sup>rm 1}$  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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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vols 08.01 and 08.04.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 15.05.

<sup>&</sup>lt;sup>5</sup> Available from Brinkman Instruments, Inc., One Cantiague Road, Westbury, NY 11590-0207.