Designation: D 2180 – 89 (Reapproved 2008)

Standard Test Method for Active Oxygen in Bleaching Compounds[1]

This standard is issued under the fixed designation D 2180; 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" active oxygen" in bleaching compounds such as perborates, percarbonates, and peroxides but not in persulfates or monopersulfates.

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.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 459 Terminology Relating to Soaps and Other Detergents
D 1193 Specification for Reagent Water

3. Terminology

3.1 *Definition:*

3.1.1 *active oxygen*—the measure of the oxidizing power of compounds such as inorganic perborates, percarbonates, or peroxides which, in effect, release hydrogen peroxide in acid solutions. It is expressed in terms of oxygen (O) with gram-equivalent weight of 8.00.

4. Summary of Test Method

4.1 Active oxygen is determined by titration of an acidified aqueous solution of the compound with a standard solution of potassium permanganate.

NOTE 1—Use of a molybdate-catalyzed iodometric method for this purpose has been suggested, but the possible reaction of the liberated iodine on organics present must be considered. It is believed there is less likelihood of reaction with permanganate, provided the titration is performed in such a manner as to avoid any considerable excess of permanganate.

5. Interferences

5.1 The possibility of interference from organic constituents, which may react with permanganate, must be considered with each compound encountered. A sluggish reaction or vague end point, will suggest interference. Inorganic builders or detergents, such as silicates, phosphates, or carbonates, do not interfere. Organic detergents or wetting agents may interfere. EDTA-type sequestrants do interfere (see Terminology D 459).

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used 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.[3] 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

6.3 *Potassium Permanganate, Standard Solution* (0.1 *N*)—Dissolve 3.2 g of potassium permanganate ($KMnO_4$) in 100 mL of water and dilute the solution with water to 1 L. Allow the solution to stand in the dark for two weeks and then filter through a fine-porosity sintered-glass crucible. *Do not wash the filter.* Store the solution in glass-stoppered, amber-colored glass bottles.

NOTE 2—Do not permit the filtered solution to come into contact with paper, rubber, or other organic material.

6.3.1 Weigh accurately to the nearest 0.1 mg about 0.3 g standard sodium oxalate ($Na_2C_2O_4$, previously dried at 105°C). Transfer to a 400-mL beaker. Add 250 mL of H_2SO_4 (5+95), freshly boiled for at least 10 min and cooled to 27 ± 3°C. Stir until the oxalate has dissolved. Add 0.1 *N* $KMnO_4$ solution rapidly from a buret (25 to 30 mL/min) while stirring slowly but constantly until about 40 mL have been added. Let stand

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[1] This test method is under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.12 on Analysis and Specifications of Soaps, Synthetics, Detergents and their Components.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] *Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

until the pink color disappears. Heat the solution from 55 to 60°C and complete the titration promptly, adding the last 0.5 to 1 mL dropwise, with particular care to let the color disappear before adding the next drop. Continue until a faint pink color persists for 30 s. Determine the amount of $KMnO_4$ solution required to produce the same color in a blank of an equal amount of special dilute acid at the same temperature. Calculate the normality of the $KMnO_4$ solution, N as follows:

$$N = W/[(V - B) \times 0.0670] \tag{1}$$

where:

W = grams of $Na_2C_2O_4$ used,

V = millilitres of $KMnO_4$ solution required for the titration of the standard, and

B = millilitres of $KMnO_4$ solution required for titration of the blank.

6.4 *Sodium Oxalate,*[4,5] primary standard grade.

6.5 *Sulfuric Acid (1+9)*—To 900 mL of cool water, carefully add 100 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84). Mix well and cool.

6.6 *Sulfuric Acid (5+95)*—To 950 mL of cool water, carefully add 50 mL of concentrated H_2SO_4. Mix well and cool.

7. Sampling

7.1 The material shall be well mixed just prior to sampling to ensure a representative sample for analysis, especially with commercial products compounded of several ingredients. If sufficient product is available, a relatively large sample should be weighed and put into solution. An aliquot should then be used for the titration. See Section 8 for the recommended amount.

8. Procedure

8.1 From a well mixed sample, weigh to the nearest 0.1 mg, a sample of sufficient size so that a 1⁄20 aliquot will require a titration of at least 10 mL of 0.1 N $KMnO_4$ solution. For example, use 3.5 to 4.0 g of a sample containing 5 % active oxygen. Transfer this sample quantitatively to a 500-mL volumetric flask, using 250 mL of water. Swirl the flask gently to dissolve the sample and avoid producing a foam should the sample contain foam-producing ingredients. Dilute to volume with H_2SO_4 (1+9) and mix thoroughly (Note 3). Immediately pipet a 25-mL aliquot and titrate (Note 4) with standard 0.1 N $KMnO_4$ solution to a faint pink color. Prepare a blank and determine the amount of $KMnO_4$ solution necessary to produce the same pink color. Record the volume of titrant required for sample and blank. Replicate samples are advisable.

NOTE 3—**Caution:** When the dilute, H_2SO_4, is added, there is a possibility of vigorous CO_2 evolution from carbonates or bicarbonates in the sample.

NOTE 4—Titrate slowly so as to avoid any considerable temporary excess of permanganate.

9. Calculation

9.1 Calculate the weight percentage of active oxygen as follows:

$$\text{Active oxygen as O, weight } \% = [(V - B)NA \times 0.008/W] \times 100 \tag{2}$$

where:

V = millilitres of $KMnO_4$ solution required for titration of the sample,

B = millilitres of $KMnO_4$ solution required for titration of the blank,

N = normality of the $KMnO_4$ solution,

A = aliquot factor (500/25 = 20), and

W = grams of sample used.

10. Keywords

10.1 active oxygen; bleaches; perborate; percarbonate; peroxide

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[4] This reagent is used for standardization purposes only.

[5] National Institute of Standards and Technology Standard Sample 40°C has been found satisfactory for this purpose.