Designation: D2022 – 89 (Reapproved 2024)

Standard Test Methods of Sampling and Chemical Analysis of Chlorine-Containing Bleaches[1]

This standard is issued under the fixed designation D2022; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover the sampling and chemical analysis of chlorine-containing bleaches. The methods appear in the following order:

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, health, and environmental 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.

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

3. Terminology

3.1 *Definitions:*

3.1.1 *available chlorine*—the measure of the oxidizing power of the chlorine present as hypochlorite. It is expressed in terms of chlorine with a gram-equivalent weight of 35.46.

4. Reagents

4.1 *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.

4.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193.

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

Current edition approved Nov. 1, 2024. Published November 2024. Originally approved in 1962. Last previous edition approved in 2016 as D2022 – 89 (2016). DOI: 10.1520/D2022-89R24.

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

[3] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

SODIUM HYPOCHLORITE (SODA BLEACH) SOLUTIONS

5. Sampling

5.1 The stability of soda bleach is influenced to a considerable degree by the purity of the alkali used in its preparation, by the excess of alkali remaining, and by the kind and amount of metal contamination from equipment. Owing to the relatively unstable nature of bleach solutions, special attention shall be given to the collection and preservation of the sample. Exposure to heat and sunlight promotes decomposition, and shall be avoided. Samples shall be kept cool in a dark place (or in dark-colored bottles) until analyzed, which shall be done without unnecessary delay.

5.2 Strong solutions of bleach shall be accurately diluted and aliquots taken for determination of available chlorine, chlorate, and total chlorine. The size of aliquots shall be such that approximately 40 mL of the 0.1 *N* reagent is required. The alkali determinations shall be made directly on the sample received and sample sizes to require about 10 mL of 0.1 *N* reagent are recommended.

5.3 Precision results will require sampling at a standard temperature such as 20 °C. Results expressed in terms of weight percent will require determination of the density or specific gravity. This may be determined with a hydrometer or by weighing the sample, after pipetting the amount to be diluted for analysis into a tared weighing bottle. The weighed sample may be transferred to a volumetric flask and used for subsequent analysis.

AVAILABLE CHLORINE

6. Summary of Test Method

6.1 The sample is added to an acidified solution of potassium iodide and the released iodine is titrated with standard sodium thiosulfate solution to the usual starch end point.

7. Reagents

7.1 *Acetic Acid,* glacial.

7.2 *Potassium Iodide* (KI), crystals, iodate-free.

7.3 *Sodium Thiosulfate Solution Standard,* (0.1 *N*)—Dissolve 25 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) crystals in freshly boiled and cooled water and dilute to 1 L. The solution is more stable if the glassware is cleaned with sulfuric-chromic acid and thoroughly rinsed with water. Standardize against potassium iodate as follows: Weigh out accurately 3.567 g of dry potassium iodate (KIO_3) and transfer to a 1 L volumetric flask. Dissolve with water, make up to the mark, and mix thoroughly. This solution will be exactly 0.1000 *N*. To standardize the $Na_2S_2O_3$ solution, carefully pipet a 50 mL aliquot of the KIO_3 solution into a 250 mL Erlenmeyer flask and dilute to 100 mL with water. Add 1 g of KI crystals. When it is dissolved, add 15 mL of 1.0 *N* hydrochloric acid and titrate immediately with the $Na_2S_2O_3$solution. When the solution becomes light yellow, add 1 mL of starch indicator solution and complete the titration to the disappearance of the blue color. Standardize at least monthly. Calculate the normality of the $Na_2S_2O_3$solution as follows:

$$\text{Normality} = (50 \times 0.1)/A \quad (1)$$

where:

A = $Na_2S_2O_3$ solution required for titration of the KIO_3solution, mL.

7.4 *Starch Indicator Solution (0.5 %)*—Mix 0.5 g of soluble starch with 5 mL of cold water and add to 95 mL of boiling water. Mix, cool, and store in a sterilized bottle. Replace frequently or add 0.1 % salicylic acid to minimize deterioration.

8. Procedure

8.1 Dissolve 2 g to 3 g of KI crystals in 50 mL of water in a 250 mL Erlenmeyer flask. Add 10 mL of acetic acid, then pipet the aliquot of sample into the solution, keeping the tip of the pipet beneath the surface of the solution until drained. Titrate at once with 0.1 *N* $Na_2S_2O_3$solution until the iodine color is nearly gone, then add 1 mL of starch indicator solution and complete the titration to the disappearance of the blue color. Record the titration as *A* (see Section 14).

9. Calculation

9.1 Calculate the available chlorine as follows:

$$\text{Available chlorine as Cl, g/L} = (AN \times 35.46)/V \quad (2)$$

$$\text{Available chlorine as Cl, weight \%} = [(AN \times 0.03546)/VS] \times 100$$

9.2 Calculate the sodium hypochlorite content as follows:

$$\text{Sodium hypochlorite (NaOCl), g/L} = (AN \times 37.22)/V \quad (3)$$

$$\text{Sodium hypochlorite (NaOCl), weight \%} = [(AN \times 0.03722)/VS] \times 100$$

where:

A = $Na_2S_2O_3$ solution required for titration of the sample, mL

N = normality of the $Na_2S_2O_3$solution,

V = original sample in aliquot used, mL, and

S = specific gravity of the sample.

SODIUM CHLORATE

10. Summary of Test Method

10.1 Sodium chlorate is reduced with sodium bromide in 8 *N* hydrochloric acid.[4,5] After dilution and addition of potassium iodide, the released iodine (equivalent to the hypochlorite plus chlorate) is titrated with standard sodium thiosulfate solution and starch indicator.

[4] Ditz, Hugo, "Determination of Chlorates in Electrolytic Bleaching Lyes and in Lyes Obtained from Absorption Vessels During the Production of Potassium Chlorate," *Chemiker Zeitung*, Vol 25, 1901 p. 727.

[5] White, J. F., "Determination of Available Chlorine in Solutions Containing Textone ($NaClO_2$)," *American Dye-stuff Reporter*, Vol 31, 1942 pp. 484–7.