Designation: E256 – 09

Standard Test Method for Chlorine in Organic Compounds by Sodium Peroxide Bomb Ignition[1]

This standard is issued under the fixed designation E256; 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 This test method covers the determination of chlorine in organic compounds by sodium peroxide bomb ignition. It is intended for application to samples of organic materials containing more than 0.5 % chlorine. The procedure assumes that compounds containing halogens other than chlorine will not be present.

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 specific safety precautions, see 6.5.2, Section 7, 8.3, and 8.9.

1.3 Review the current Material Safety Data Sheets (MSDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.

NOTE 1—Other test methods based on oxygen bomb combustion for analysis for chlorine are described in Test Method D808, Test Method D2361, and Test Method D4208.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D808 Test Method for Chlorine in New and Used Petroleum Products (High Pressure Decomposition Device Method)

D1193 Specification for Reagent Water

D2361 Test Method for Chlorine in Coal (Withdrawn 2008)[3]

D4208 Test Method for Total Chlorine in Coal by the Oxygen Bomb Combustion/Ion Selective Electrode Method

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

3. Summary of Test Method

3.1 The sample is heated in a closed bomb with sodium peroxide in the presence of an accelerator (potassium nitrate) and an oxygen-bearing compound (sucrose). The chlorine is converted to chloride. The contents of the bomb are dissolved in water. The solution is acidified and the chloride is determined by titrating the excess of silver nitrate solution used to precipitate the chloride.

4. Significance and Use

4.1 This test method may be used to determine the total chlorine content of unknown organic samples or to assay known chlorine containing organic compounds.

4.2 This test method may be used on organic materials in which the complete conversion to chloride can be accomplished by sodium peroxide bomb ignition, and which contain no other halogens.

5. Apparatus

5.1 *Sodium Peroxide Bomb,* [4] flame ignition type, 22-mL capacity. This consists of a 99 % nickel fusion cup, 99 % nickel cap cover, lead gaskets, a bomb body, a screw cap, a wrench, an ignition housing, a bomb socket, glass mixing rod, and a sodium peroxide measuring dipper. A less expensive nickel-plated brass cap cover may be substituted.

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.15 on Industrial and Specialty General Standards.

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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] The last approved version of this historical standard is referenced on www.astm.org.

[4] The sole source of supply of the ignition bomb and assembly known to the committee at this time is Parr Instrument Co., Moline, IL. The 22-mL fusion cup is part No. N-200. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

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

5.2 *Safety Shield*—Any heavy-duty commercially available shield should suffice to confine the results of an explosion in the event of bomb body rupture during flame ignition.

5.3 *Burner,* blast-type, using gas and air. For some bomb work, the cheaper bunsen burner has been found to be satisfactory.

5.4 *Capsules,* gelatin, size No. 00, for liquid samples.

6. Reagents

6.1 *Purity of Reagents*—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.[5] 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 Type II or Type III reagent water conforming to Specification D1193.

6.3 *Ammonium Thiocyanate, Standard Solution* (0.1 meq/mL*)*—Prepare in accordance with Practice E200.

6.4 *Ferric Ammonium Sulfate Indicator Solution* (80 g/L)—Prepare in accordance with Practice E200.

6.5 *Mixture, Bomb Accelerator:*

6.5.1 Dry 900 g of potassium nitrate (KNO_3) crystals in a 110 to 120°C oven for 2 days. Grind the dried crystals to a powder.

6.5.2 Dry 300 g of granulated sucrose in a 50 to 60°C oven for 2 days. Grind the dried sugar to a powder (**Warning—**Do not grind potassium nitrate with sugar. To avoid the possibility of an explosion, grind these materials separately.).

6.5.3 Mix the powdered KNO_3 and powdered sugar in a ball mill for 20 to 30 min. Balls should be omitted from the mill during this blending operation. Store in a closed jar kept in a desiccator.

6.6 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

6.7 *Nitrobenzene*—The use and disposal of nitrobenzene must be done in accordance with its hazardous properties (see 7.2). Benzyl alcohol may be a safer, but less effective, coagulant than nitrobenzene.

6.8 *Silver Nitrate, Standard Solution* (0.1 meq/mL*)*—Prepare in accordance with Practice E200.

6.9 *Sodium Peroxide,* 30- to 40-mesh, calorific grade. See the safety precautions on the label concerning this strongly caustic oxidant.

7. Safety Precautions

7.1 This test method should not be applied to samples containing appreciable amounts of water (0.1 % or greater). Water reacts spontaneously with sodium peroxide and may cause premature reaction of the sample and peroxide. Also, oxygen is released which may develop dangerously high internal bomb pressure.

7.2 Nitrobenzene is very hazardous when absorbed through the skin or when its vapor is inhaled. Do not get nitrobenzene in the eyes, on the skin, or on clothing. Avoid breathing its vapor. Use only with adequate ventilation.

7.3 Sodium peroxide is a potentially dangerous chemical. Avoid scattering the reagent or leaving the container exposed to the air or moisture. Spilled sodium peroxide should be washed down with large amounts of water, and not wiped up with paper or cloth.

7.4 Fusion cups may develop holes or cracks at any time (even when relatively new, but especially after long and continued use) and should be examined before use; cups not suitable for use should be discarded promptly. Fusion cup cover gaskets must be replaced when damaged. Bomb bodies and screw caps must be discarded when the threads become worn. This must be checked frequently.

7.5 Samples of unknown or unfamiliar composition or samples suspected of containing water or other material reactive with sodium peroxide should be tested before mixing with sodium peroxide. Place sodium peroxide in an empty fusion cup to a depth of 5 mm. Cautiously (wear gloves and safety glasses) add small quantities (approximately 25 mg) of the sample and mix. If the sample ignites spontaneously upon contact with the sodium peroxide, use a gelatin capsule for weighing the sample and introducing it into the bomb mixture. If the sample does not react with sodium peroxide, then proceed with the weighing of the sample, omitting the use of a gelatin capsule.

8. Procedure

8.1 Weigh (to the nearest 0.0001 g) a sample containing the equivalent of 0.1 g of chlorine. In any case, do not exceed a sample weight of 0.5 g. Solid samples should be pulverized before weighing. Liquid samples and samples that react on contact with sodium peroxide (see 7.4) should be weighed in a gelatin capsule.

8.2 Weigh approximately 1.0 g of KNO_3-sugar mixture (see 6.5) into a clean, dry fusion cup. Add one dipper (approximately 15 g) of sodium peroxide to the fusion cup and mix the contents with a clean, dry, glass stirring rod.

8.3 Transfer approximately one half of the mixture momentarily from the fusion cup above into a second fusion cup (**Warning—**During the following charging procedure, the analyst should be prepared for a premature ignition. The use of gloves, safety spectacles, and a protective shield is urged.). Place the weighed sample (see 8.1) into the first fusion cup. In the case of a sample that is not in a capsule, stir the contents of the first cup with a clean, dry, glass rod while slowly returning the balance of the peroxide-accelerator mixture from the

[5] *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.