Designation: E2037 – 15

Standard Test Method for Bromine Chloride in Liquid Chlorine by High Performance Liquid Chromatography (HPLC)[1]

This standard is issued under the fixed designation E2037; 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 uses high performance liquid chromatography (HPLC) to determine bromine chloride levels in liquid chlorine at the 10 to 1400 µg/g (ppm) range.

1.2 Review the current safety data sheet (SDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 8.

1.5 *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]

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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

E806 Test Method for Carbon Tetrachloride and Chloroform in Liquid Chlorine by Direct Injection (Gas Chromatographic Procedure)

2.2 *Federal Standards:*[4]

CFR 173 Title 49 Transportation; Shippers' General Requirements for Shipments and Packaging, including Sections:

173.304 Charging of Cylinders with Liquefied Compressed Gas

173.314 Requirements for Compressed Gases in Tank Cars

173.315 Compressed Gases in Cargo Tanks and Portable Tank Containers

2.3 *Other Document:*[5]

Chlorine Institute Pamphlet No. 1 Chlorine Basics

3. Summary of Test Method

3.1 Weighed samples of chlorine delivered into a cooled graduated centrifuge tube. One mL of cooled HPLC eluent is added before the chlorine is allowed to evaporate. After the chlorine has evaporated the remaining eluent is analyzed directly on the HPLC for bromine chloride concentration.

4. Significance and Use

4.1 This test method was developed for the determination of bromine chloride in liquid chlorine. Bromide is a common contaminant in all salt sources that are used in the production of chlorine. This bromide content of the salt is converted into bromine chloride in the liquid chlorine product. This test method is sensitive enough to measure the levels of bromine chloride observed in normal production chlorine.

5. Interferences

5.1 This test method is selective for bromine chloride. At this time there are no known interference in the materials used in this test method.

5.2 Contact with any metal surfaces should be avoided due to the corrosive nature of the sample.

6. Apparatus

6.1 A high performance liquid chromatograph (HPLC) composed of the following:

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.13 on Chlorine.

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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] Available from U.S. Government Publishing Office, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.gpo.gov.

[5] Available from The Chlorine Institute, 1300 Wilson Blvd., Ste. 525, Arlington, VA 22209, http://www.chlorineinstitute.org.

6.1.1 *HPLC Pump,* capable of 1 mL/min flow,

6.1.2 *HPLC UV Detector,* capable of operating at 221 nm with a 1-cm cell,

6.1.3 *HPLC Injection Valve,* 20 µl loop, all nonmetal, and

6.1.4 *HPLC Column,* C18 reverse phase, 25 cm by 4.6 mm.

6.2 *Plastic Syringes,* 1, 2.5, 5, 10, 20, and 60 mL.

6.3 *Nonmetallic Syringe Needles.*

6.4 *Top Loader Balance,* capable of 0.01 g resolution with a 1-kg capacity.

6.5 *TFE-Fluorocarbon Tubing,* 1.59 mm outside diameter.

6.6 *Stainless Steel Sample Cylinder,* with a needle valve on one end.

6.7 *Graduated Centrifuge Tube,* 15 mL.

7. Reagents

7.1 *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.[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 *Water,* HPLC grade.

7.3 *Methanol,* HPLC grade.

7.4 *Sodium Acetate,* reagent grade.

7.5 *Glacial Acetic Acid,* reagent grade.

7.6 *Dry Ice.*

7.7 *Potassium Bromide,* reagent grade.

7.8 *Chlorine,* reagent grade.

8. Hazards

8.1 Chlorine is a corrosive and toxic material. A well-ventilated fume hood should be used to house all sample handling and to vent the test equipment when this product is analyzed in the laboratory.

8.2 The analysis should be attempted only by persons who are thoroughly familiar with the handling of chlorine, and even an experienced person should not work alone. The operator must be provided with adequate eye protection and a respirator. Splashes of liquid chlorine destroy clothing and if such clothing is next to the skin, it will produce irritations and burns.

8.3 If liquid samples are to be taken in cylinders, do not allow the sample cylinder to become liquid full. Test Method E806, 49 CFR 173.314, 173.315, and 173.304 advise that the weight of the chlorine in the cylinder should not be more than 125 % of the weight of the water that the cylinder could contain. See the Chlorine Institute Pamphlet No. 1 for additional technical information regarding liquid chlorine.

8.4 When sampling and working with chlorine out-of-doors, people downwind from such an operation should be warned of the possible release of chlorine vapors.

8.5 In the event chlorine is inhaled, first aid should be summoned immediately and oxygen administered without delay.

8.6 Store pressurized samples where involuntary release would not cause excessive risk to people or property.

8.7 It is recommended that means be available for disposal of excess chlorine in an environmentally safe and acceptable manner. If chlorine cannot be disposed of in a chlorine consuming process, a chlorine absorption system should be provided. When the analysis and sampling regimen requires an initial purging of chlorine from a container, the purged chlorine should be similarly handled. Purging to the atmosphere should be avoided.

8.8 *Safety and Health Precautions*—Exposure to all solvents used in this test method should be avoided.

9. Typical Instrument Parameters

9.1 Adjust the chromatograph in accordance with the following parameters and allow the instrument to equilibrate until a steady baseline is obtained:

9.1.1 *Column*—C18 reverse phase ODS (C18) 25 cm by 4.6 mm, 10 µm,

9.1.2 *Eluent*—60 % by volume methanol, 40 % by volume 0.1 mol/L (M) acetate buffer, pH 4.5, helium sparged,

9.1.3 *Flow Rate*—1 mL/min,

9.1.4 *Injection Volume*—20-µl sample loop, and

9.1.5 *Detector Wavelength*—UV at 221 nm.

10. Preparation of Buffer Solution

10.1 *Sodium Acetate Buffer Stock Solution (1 mol/L (M))*—Dissolve 136 g of sodium acetate ($NaOOCCH_3 \cdot 3H_2O$) and 60 g of glacial acetic acid ($HOOCCH_3$) in water and dilute to 1 L.

10.2 *Sodium Acetate Buffer (0.1 mol/L (M))*—Transfer 100 mL of the stock buffer solution into a 1-L volumetric flask and dilute to volume with water.

11. Preparation of Eluent

11.1 Add 600 mL of methanol to 400 mL of 0.1 mol/L (M) sodium acetate buffer solution and mix well. Before use, purge the solution with helium for 20 min to remove dissolved oxygen.

12. Preparation of the Sample Cylinder

12.1 Clamp the sample cylinder in a vertical position with the needle valve in the downward position. Insert the 6.35 mm end of the reducing tube fitting into the needle valve and set the 6.35 mm nut and ferrule of the fitting. Insert the 1.59 mm fluoropolymer tubing in the reducing tube fitting and tighten the 1.59 mm nut and ferrule. See Fig. 1. It may be helpful to cut a 6.35 mm circle of fluoropolymer frit material and place it into the reducing fitting prior to assembly to prevent plugging of the

[6] *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.