Designation: E649 – 00 (Reapproved 2011)

Standard Test Method for Bromine in Chlorine[1]

This standard is issued under the fixed designation E649; 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 bromine in liquid chlorine and in gaseous chlorine with a lower limit of detection of 4 ug/kg by weight.

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.* Specific hazard statements are given in Section 7.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]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 Chlorine gas is sampled by absorption in aqueous sodium hydroxide (NaOH). Liquid chlorine is first vaporized, and the vapor is absorbed in aqueous sodium hydroxide. An aliquot of the sample solution is reduced with an excess of sulfite ion, acidified, and excess sulfur dioxide (SO_2) boiled out. In a carefully buffered solution, bromide is oxidized to bromate by hypochlorite. Excess hypochlorite is reduced to chloride by formate. In the presence of molybdate catalyst, bromate is reduced to bromide by iodide, and the liberated iodine is titrated with standard sodium thiosulfate solution.

4. Significance and Use

4.1 Low levels of bromine contaminant in chlorine cause problems in some industrial uses. This test method may be used to determine bromine in liquid or gaseous chlorine at levels as low as 4 ug/kg.

5. Apparatus

5.1 The construction of the chlorine gas sampling apparatus and of the assembled sampling equipment is shown in Figs. 1 and 2. Modification of the equipment to deal with special sampling circumstances may be necessary. In Fig. 2, the control valve is shown with an adaptor for connection to a chlorine cylinder valve. Other adaptors will be required when sampling liquid chlorine in liquefaction plant streams or from large shipping or storage containers.

5.2 A 10-mL buret calibrated in 0.05-mL divisions is used when titrating with 0.01 *N* standard sodium thiosulfate solution.

6. Reagents

6.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] 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 *Chloride Solution*—Dissolve 200 g of sodium chloride (NaCl) in 940 mL of water.

[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 D16.16 on Industrial and Specialty Product 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] *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.

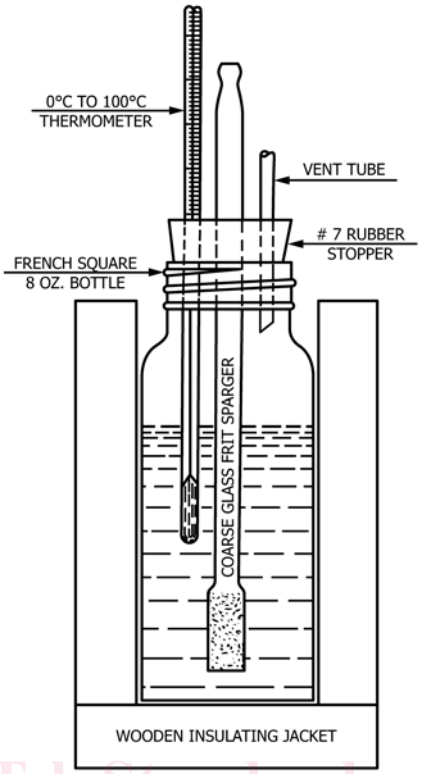
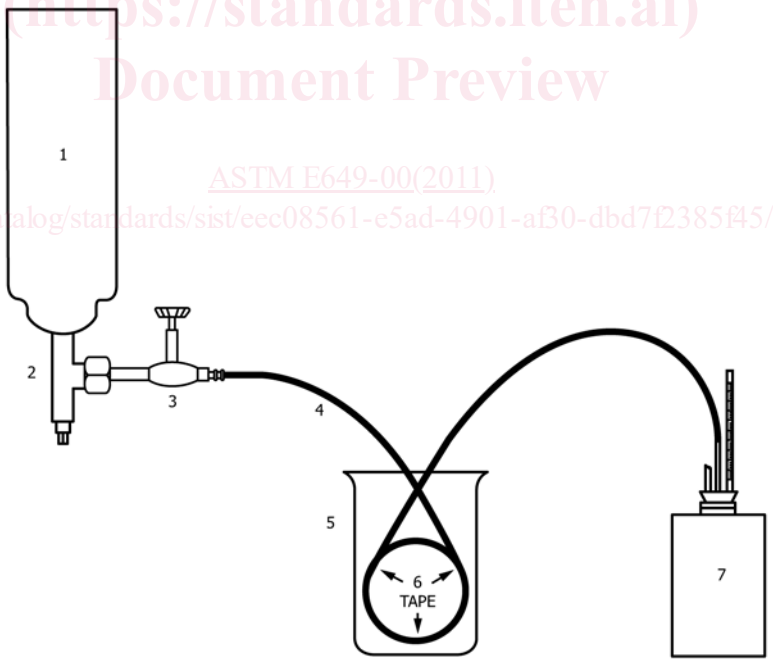


FIG. 1 Chlorine Gas Sampling Apparatus



1. Inverted liquid chlorine cylinder
2. Chlorine cylinder valve
3. Control valve (¼-in. 316 stainless steel needle valve)
4. ¼-in. PTFE instrument air tubing
5. Small pail or 2-L beaker filled with water
6. Evaporating coil (two loops held with tape at indicated points)
7. Gas sampling apparatus of Fig. 1

FIG. 2 Liquid Chlorine Sampling Equipment