



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

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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 $\mu\text{g/g}$ (ppm) range.

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

1.3 *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 8.

2. Referenced Documents

2.1 ASTM Standards:

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals²

E 806 Test Method for Carbon Tetrachloride and Chloroform in Liquid Chlorine by Direct Injection (Gas Chromatography Procedure)²

2.2 Federal Standards³

49 CFR 173 Code of Federal Regulations Title 49 Transportation: Shipper's 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; and, 173.315 Compressed Gases in Cargo Tanks and Portable Tank Containers.

2.3 Other Document

Chlorine Institute Pamphlet No. 77 Sampling Liquid Chlorine⁴

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:

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*, $\frac{1}{16}$ in. 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

¹ This test method is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and is the direct responsibility of Subcommittee E15.02 on Product Standards.

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² *Annual Book of ASTM Standards*, Vol 15.05.

³ Available from the U.S. Government Printing Office, Superintendent of Documents, Washington, DC 20402.

⁴ Available from The Chlorine Institute Inc., 70 W. 40th St., New York, NY 10018.