



Designation: E806 – 21

Standard Test Method for Carbon Tetrachloride and Chloroform in Liquid Chlorine by Direct Injection (Gas Chromatographic Procedure)¹

This standard is issued under the fixed designation E806; 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 is designed for the determination of carbon tetrachloride (CCl_4) and chloroform (CHCl_3) in liquid chlorine. The lower limit of detection is dependent on the sample size and the instrument used; five ppm (w/w) is achievable.

1.2 In determining the conformance of the test results using this method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

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 hazards statements are given in Section 7 and in 9.2.3.

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

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

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.

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)³

2.2 Federal Standard:⁴

OSHA Regulations, 29 CFR paragraphs 19100.1000 and 1910.1200 Air contaminants – Table of exposure limits and Hazard Communication

49 CFR 173 Code of Federal Regulations Title 49 Transportation; Shippers' General Requirements for Shipments and Packagings, 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:

Chlorine Institute Pamphlet No. 1 Chlorine Basics⁵

3. Summary of Test Method

3.1 A sample of liquid chlorine is injected into a gas chromatograph (GC), equipped with a column capable of separating CCl_4 and CHCl_3 from Cl_2 and other impurities, using a suitable syringe. The amounts of CCl_4 and CHCl_3 in the sample are determined by comparison of the areas of the peaks, obtained with the samples, to areas of peaks obtained with suitable calibration standards, under the same conditions.

4. Significance and Use

4.1 CCl_4 and CHCl_3 may be present in trace amounts in liquid chlorine. The use of chlorine to purify water would then transfer these compounds to the water. Therefore, when the concentrations of the CCl_4 and CHCl_3 in the liquid chlorine are

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, <http://www.access.gpo.gov>.

⁵ Available from The Chlorine Institute Inc., 1300 Wilson Blvd., Suite 525, Arlington, VA 22209, <https://www.chlorineinstitute.org>.

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

known, the maximum amounts contributed to the water by the chlorine can be estimated.

5. Apparatus

5.1 *Gas Chromatograph*, equipped with:

5.1.1 *Injection Port*, must be lined with glass, Monel,⁶ or nickel; or column must be installed for on-column injection.

5.1.2 *Septa*, from Viton.⁷ Silicone septa may produce artifacts that may interfere with the analysis.

5.1.3 *Column, Column Materials, and Packing*, must be compatible with chlorine. Silanized supports and silanized glass wool must be avoided. Column must be able to separate Cl_2 , CCl_4 , and CHCl_3 . Columns that have been found to be suitable are:

5.1.3.1 *Nickel Tubing*, 3.05 m by 3.175 mm outside diameter, packed with 10 % sodium chloride solution on Porasil C⁸ (see [Appendix X1](#) for packing preparation). This is the preferred packing.

5.1.3.2 *Polytetrafluoroethylene Tubing*, 3.05 m by 2 mm inside diameter, packed with 20 % Kel-F⁹ No. 10 oil on 60/80 mesh Chromosorb¹⁰ W AW.

5.1.3.3 *Glass Tubing*, 3.05 m by 2 mm inside diameter, packed with 20 % Halocarbon¹¹ 1025 on 60/80 mesh Chromosorb W AW.

5.1.4 *Flame Ionization Detector*.

5.1.5 *Recorder*, compatible with the GC detector output.

5.1.6 *Electronic Integrator* (optional), compatible with the GC detector output.

5.2 *Balance*, capacity 5000 g, reading to ± 1 g.¹²

5.3 Autoinjectors are required for all gas chromatograph standards using an external standard to calculate results.

6. Reagents and Materials

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.¹³ 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 *Chlorine*, liquid, with less than 10 $\mu\text{g/g}$ each of CCl_4 and CHCl_3 . This may be prepared by condensing the gaseous phase above regular production chlorine.¹⁴

6.3 *Carbon Tetrachloride*, reagent grade.¹⁴

6.4 *Chloroform*, reagent grade.¹⁴

6.5 *Sample Cylinder Assembly* ([Fig. 1](#)), consisting of:

6.5.1 *Sample Cylinders*,¹⁵ nickel, Monel, or tantalum ([Note 1](#)), 400-mL capacity, double-ended, specially cleaned ([Note 2](#)).

6.5.2 *Valves*, having a packing resistant to liquid chlorine.¹⁶

6.5.3 *Holder for a Septum*, that can be easily assembled.¹⁷

NOTE 1—Carbon or stainless steel cylinders and fittings are not suitable as CHCl_3 is unstable in the presence of FeCl_3 and Cl_2 .

NOTE 2—A procedure for cleaning cylinders and valves, for use with liquid chlorine, is given in [Appendix X2](#).

6.6 *Fittings*, for transferring chlorine from one cylinder to another.

¹⁵ If samples are to be shipped outside any plant, cylinders approved by the U.S. Department of Transportation must be used.

¹⁶ Packing made from Teflon, registered trademark of The Chemours Company, Viton, Kel-F, or equivalent have been found suitable for this purpose.

¹⁷ Swagelok, registered trademark of Swagelok Company, or equivalent fittings have been found suitable for this purpose.

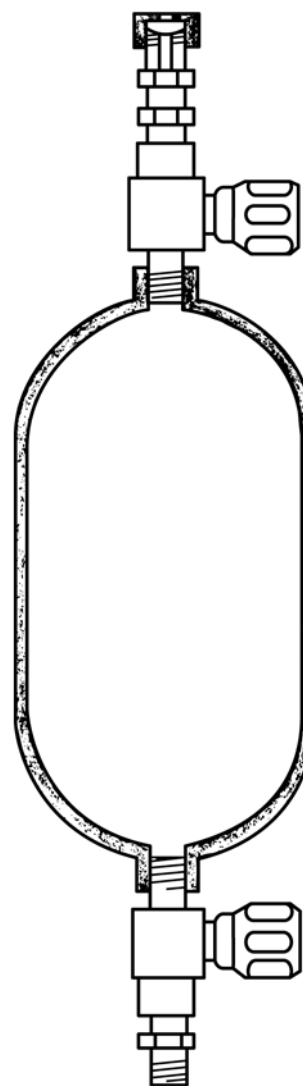


FIG. 1 Sample Cylinder Assembly

⁶ Monel is a registered trademark of Special Metals Corporation.

⁷ Viton is a registered trademark of The Chemours Company.

⁸ Porasil is a trademark of Waters Associates, Inc.

⁹ Kel-F is a registered trademark of 3M Company.

¹⁰ Chromosorb is a registered trademark of Imerys Minerals California, Inc.

¹¹ Halocarbon is a registered trademark of Halocarbon Products Corporation.

¹² A 400-mL nickel cylinder filled with liquid chlorine weighs about 4000 g.

¹³ *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.

¹⁴ This reagent is used for calibration purposes only.