



Designation: E 806 – 99

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 E 806; 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 (CCl₄) and chloroform (CHCl₃) 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 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 hazards statements are given in Section 7 and in Note 4.

2. Referenced Documents

2.1 ASTM Standards:

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

2.2 Federal Standard:³

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. 77 Sampling Liquid Chlorine⁴

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.

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 CCl₄ and CHCl₃ from Cl₂ and other impurities, using a suitable syringe. The amounts of CCl₄ and CHCl₃ 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 CCl₄ and CHCl₃ 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 CCl₄ and CHCl₃ in the liquid chlorine are 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,⁵ or nickel; or column must be installed for on-column injection.

5.1.2 *Septa*, from Viton.⁶ 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 Cl₂, CCl₄, and CHCl₃. Columns that have been found to be suitable are:

5.1.3.1 *Nickel Tubing*, 10 ft by 1/8 in. outside diameter, packed with 10 % sodium chloride solution on Porasil C (see Appendix X1 for packing preparation). This is the preferred packing.

5.1.3.2 *Polytetrafluoroethylene Tubing*, 10 ft by 2 mm inside diameter, packed with 20 % Kel-F[®] No. 10 oil on 60/80 mesh Chromosorb[®] W AW.

5.1.3.3 *Glass Tubing*, 10 ft by 2 mm inside diameter, packed

⁵ Available from the International Nickel Company, Park 80 West, Plaza 2, Saddlebrook, NJ 07662.

⁶ Viton[®] septa can be prepared from Pierce No. 13235 Viton[®] hypo vial seals, available from the Pierce Chemical Co., Rockford, IL 61105. The septum is prepared by using a sharp blade to cut off the tip of the seal and then punching out a septum from the remaining flat disc. A cork borer or leather punch can be used to punch out the septum. Viton[®] septa are also available from Canton Bio-Medical Products, P.O. Box 2017, Boulder, CO 80302, Catalog No. V-101.

with 20 % Halocarbon® 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.⁷

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.⁸ 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 ppm each of CCl₄ and CHCl₃. This may be prepared by condensing the gaseous phase above regular production chlorine.⁹

6.3 *Carbon Tetrachloride*, reagent grade.⁹

6.4 *Chloroform*, reagent grade.⁹

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

6.5.1 *Sample Cylinders*¹⁰; 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.¹¹

6.5.3 *Holder for a Septum*, that can be easily assembled.¹²

NOTE 1—Carbon or stainless steel cylinders and fittings are not suitable as CHCl₃ is unstable in the presence of FeCl₃ and Cl₂.

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.

6.7 *Syringe*, 10 to 100- μ L, capable of holding liquid chlorine under pressure, with 26-gage disposable needle.¹³

NOTE 3—Disposable needles are recommended because corrosion with permanent needles may cause problems.

7. Hazards

7.1 Chlorine is a corrosive and toxic material. A well-ventilated fume hood should be used to house all test equip-

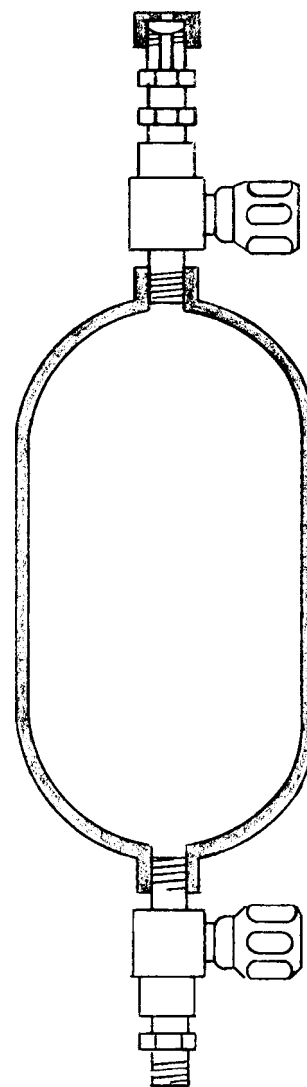


FIG. 1 Sample Cylinder Assembly

ment, except the gas chromatograph, when this product is analyzed in the laboratory.

7.2 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, will produce irritations and burns.

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

7.4 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.

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

⁷ A 400-mL nickel cylinder filled with liquid chlorine weighs about 4000 g.

⁸ *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.

⁹ This reagent is used for calibration purposes only.

¹⁰ If samples are to be shipped outside any plant, cylinders approved by the U.S. Department of Transportation must be used. DoT-approved (1979) nickel cylinders are available from Crown Controls, Inc., 388 Getty Ave., Clifton, NJ 07011, Hoke quotation SP78-12-26.

¹¹ Packing made from Teflon®, Viton®, or Kel-F® have been found suitable for this purpose.

¹² Swagelok® fittings have been found suitable for this purpose.

¹³ Two sources have been found to be suitable: Glenco Scientific Inc., 2802 Oak Drive, Houston, TX 77007, model 925-MV-2 micro valve and model KFLL Luer lock adapter; and Precision Sampling Co., P.O. Box 15119, Baton Rouge, LA 70815, Pressure-Lok, series A.