



Standard Test Method for Nitrogen Trichloride 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 nitrogen trichloride levels in liquid chlorine at the 0.1 to 600 $\mu\text{g/g}$ (ppm) range. Solvent samples from chlorine production facilities containing very high concentrations of nitrogen trichloride may be analyzed by dilution with methanol.

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 Standard:³

49 CFR 173 Code of Federal Regulations 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; 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 are 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 nitrogen trichloride concentration.

3.2 In-process solvent samples from chlorine production facility may be analyzed by dilution in methanol followed by direct HPLC analysis of the diluted solution.

4. Significance and Use

4.1 This test method was developed for the determination of nitrogen trichloride in samples of carbon tetrachloride liquid taken from the compressor suction chiller bottoms of chlorine production plants and other places in the plants that may collect and concentrate nitrogen trichloride to levels that could explode. The test method was then modified to measure the lower levels of nitrogen trichloride observed in product liquid chlorine. This test method is sensitive enough to measure the levels of nitrogen trichloride observed in the normal production of liquid chlorine.

4.2 This test method for nitrogen trichloride will require the dilution (100:1) of highly concentrated in-process samples to bring them within the working range of the analysis.

5. Interferences

5.1 This test method is selective for nitrogen trichloride. At this time there is 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.

5.3 Nitrogen trichloride is decomposed by UV light, heat or strong acid. The presence of metal ions will increase the rate of

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 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.