Designation: E2036 – 24

Standard Test Method for Nitrogen Trichloride in Liquid Chlorine by High Performance Liquid Chromatography (HPLC)[1]

This standard is issued under the fixed designation E2036; 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 uses high performance liquid chromatography (HPLC) to determine nitrogen trichloride levels in liquid chlorine at the 0.1 µg/g (ppm) to 600 µg/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 safety data sheet (SDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.

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 The following applies for the purposes of determining the conformance of the test results using this test method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

1.5 *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 hazard statements are given in Section 8.

1.6 *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

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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E806 Test Method for Carbon Tetrachloride and Chloroform in Liquid Chlorine by Direct Injection (Gas Chromatographic Procedure)

2.2 *Federal Standards:*[4]

CFR 173 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

173.315 Compressed Gases in Cargo Tanks and Portable Tank Containers

2.3 *Other Document:*[5]

Chlorine Institute Pamphlet No. 152 Safe Handling of Chlorine Containing Nitrogen Trichloride

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

[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.

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

[4] Available from U.S. Government Publishing Office, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.gpo.gov.

[5] Available from The Chlorine Institute (CI), 1300 Wilson Blvd., Ste. 525, Arlington, VA 22209, http://www.chlorineinstitute.org.

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

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 decomposition. Special care should be taken to avoid exposure of the samples to direct sunlight. Samples and standards should be analyzed immediately upon preparation. Samples not immediately analyzed must be stored in dry ice until just before analysis.

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 light path,

6.1.3 *HPLC Injection Valve,* 20 µL sample loop, all nonmetal, and

6.1.4 *HPLC Column,* C18 reverse phase, 25 cm by 4.6 mm.

6.2 *Plastic Syringes.* 1 mL, 2 mL, 5 mL, 10 mL, 20 mL, and 60 mL.

6.3 *Nonmetallic Syringe Needles.*

6.4 *Top Loader Balance,* capable of 0.01 g resolution and 1 kg capacity.

6.5 *TFE-fluorocarbon Tubing,* 1.59 mm outside diameter.

6.6 *Stainless Steel Sample Cylinder,* with a needle valve on one end.

6.7 *Graduated Centrifuge Tube,* 15 mL.

6.8 *Reducing Tube Fitting,* 6.35 mm to 1.59 mm tubing.

7. Reagents

7.1 *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.[6] 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.

7.2 *Water,* HPLC grade.

7.3 *Methanol,* HPLC grade. CAS# 67-56-1.

7.4 *Sodium Acetate,* reagent grade. CAS# 127-09-3.

7.5 *Glacial Acetic Acid,* reagent grade. CAS# 64-19-7.

7.6 *Dry Ice.* CAS# 124-38-9.

7.7 *Ammonium Sulfate,* reagent grade. CAS# 7783-20-2.

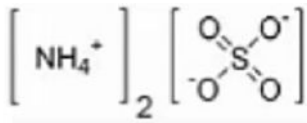

FIG. 1 Ammonium Sulfate, reagent grade: CAS# 7783-20-2

7.8 *Chlorine,* reagent grade. CAS# 7782-50-5.

7.9 *Helium.*

8. Hazards

8.1 Consult current OSHA regulations, suppliers' Safety Data Sheets, and local regulations for all materials used in this test method.

8.2 *Safety and Health Precautions*—Exposure to all solvents used in this test method should be avoided.

8.3 Concentrations of nitrogen chloride over 2 % pose the risk of explosion and should be considered dangerous. Never allow any solutions containing nitrogen chloride to evaporate, concentrating the nitrogen chloride in the remaining residue. The concentrations of nitrogen trichloride synthesized in this test method are small and should not pose any significant risk of explosion. All of the solutions prepared in this test method can be disposed of by flushing them down any laboratory sink using a reasonable amount of water.

8.4 Chlorine is a corrosive and toxic material. A well-ventilated fume hood should be used to house all sample handling and to vent the test equipment when this product is analyzed in the laboratory.

8.5 This analysis should only be performed 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.

[6] *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.