



Designation: ~~D5194-11~~ Designation: D5194 – 11a

Standard Test Method for Trace Chloride in Liquid Aromatic Hydrocarbons¹

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1. Scope*

1.1 This test method covers the determination of total chloride (organic and inorganic) in liquid aromatic hydrocarbons and cyclohexane.

1.2 The test method is applicable to samples with chloride concentrations of 1 to 25 mg/kg.

1.3 Bromides and iodides, if present, will be calculated as chlorides.

1.4 Materials, such as styrene, that are polymerized by sodium biphenyl reagent cannot be analyzed by this test method.

1.5 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.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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.* For a specific hazard statement, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:²

D891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

D1193 Specification for Reagent Water

D1555M Test Method for Calculation of Volume and Weight of Industrial Aromatic Hydrocarbons and Cyclohexane [Metric]

D3437 Practice for Sampling and Handling Liquid Cyclic Products

D3505 Test Method for Density or Relative Density of Pure Liquid Chemicals

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

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

2.2 Other Documents:³

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200

3. Summary of Test Method

3.1 A known amount of hydrocarbon sample is transferred into a separatory funnel containing toluene. Sodium biphenyl reagent is added to convert organic halogens into inorganic halides. The excess reagent is decomposed with water and the phases are separated. The aqueous phase is acidified, washed, and concentrated. Acetone is added and the solution is titrated with silver nitrate solution.

4. Significance and Use

4.1 Organic and inorganic chlorine compounds can have a deleterious effect on equipment and reactions in processes involving aromatic hydrocarbons.

4.2 Maximum chloride levels are often specified for process streams and for aromatic hydrocarbon products.

¹ This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.04 on Instrumental Analysis.

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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 Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

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

5. Apparatus

5.1 *Titrator*, potentiometric, recording, + 2000 mV range, 1 mV resolution with dispenser having a volume readout of 0.00 to 9.99 mL or 0.00 to 99.99 mL and 0.01 % resolution.

5.2 *Electrode*, glass, reference.

5.3 *Electrode*, silver, billet type.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ 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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Types II or III of Specification D1193.

6.3 *Acetone*, 99.9 % purity.

6.4 *Congo Red Paper*.

6.5 *Detergent, residue free*.

6.6 *Isobutanol*, 99.9 % minimum purity.

6.7 *Isooctane*.

6.8 *Nitric Acid*, concentrated.

6.9 *Nitric Acid, 5-M*. Dilute 160 mL concentrated nitric acid to 500 mL with water.

6.10 *Potassium Chloride*, primary standard.

6.11 *Potassium Chloride Solution*, saturated.

6.12 *Scouring Powder*, cleanser.

6.13 *Silver Nitrate*, 99.99 % minimum purity.

6.14 *Silver Nitrate Solution*, 0.01 N, standardized to 0.1 %.

NOTE 1—This solution may be obtained as follows:

(1) Purchase from a laboratory supply company, (2) Weigh to four places, 1.680 to 1.720 g silver nitrate, transfer quantitatively into a 1000-mL volumetric flask, make to mark with water, and mix well.

$$\text{Normality of solution} = \frac{\text{Weight AgNO}_3}{169.9}$$

or (3) Dissolve 8.5 g silver nitrate in 500 mL water to give a 0.1 N solution. Weigh 0.09 to 0.10 g of dried (105°C) potassium chloride to the nearest 0.1 mg into a 250-mL electrolytic beaker, add 100 mL of water and a stirring bar. While stirring, titrate with the silver nitrate solution.

$$\text{Normality of AgNO}_3 \text{ solution} = \frac{\text{Weight KCl}}{0.0746 \times \text{mL AgNO}_3}$$

Pipet 50.00 mL of the solution into a 500-mL volumetric flask, dilute to mark with water, and mix well. Divide the calculated normality of the 0.1 N solution by 10 to give the normality of final AgNO₃ solution.

6.15 *Sodium Biphenyl Reagent*—The reagent is packed in 15-mL vials that contain 0.2 to 0.4 g of active sodium each.

6.16 *Toluene*, 99.9 % minimum purity.

7. Hazards

7.1 A material, such as styrene, which is polymerized by sodium biphenyl can cause a violent reaction and should never be used as the sample.

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

8. Sampling

8.1 Refer to Practice D3437 for proper sampling and handling of liquid hydrocarbons analyzed by this test method.

⁴ *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.

TABLE 1 Specimen Size

Estimated chloride, mg/kg	Specimen volume, mL
0 to 5	100
5 to 25	50