Designation: D4755 – 95 (Reapproved 2015)ε1

Standard Test Method for Free Halogens in Halogenated Organic Solvents and Their Admixtures[1]

This standard is issued under the fixed designation D4755; 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.

ε1 NOTE—Units statement was inserted in Section 1.2 editorially in June 2015.

1. Scope

1.1 This test method covers the evaluation of free halogens in halogenated organic solvents and their admixtures.

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

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D1193 Specification for Reagent Water

3. Summary of Test Method

3.1 Any free halogens present in the halogenated solvent are extracted with water and reacted with potassium iodide test solution containing starch indicator. A blue color indicates that presence of free halogens.

4. Significance and Use

4.1 Free halogens will react with any residual moisture in a solvent to form acid. These acids can cause corrosion to process and storage equipment used for halogenated solvents.

4.2 The presence of free halogens in halogenated solvents is often an indication that the stabilizers in the solvent system are breaking down.

4.3 This test method provides a means of detecting the presence of free halogens in halogenated solvents and their admixtures.

5. Interferences

5.1 The following materials interfere with this test giving a false positive: ferric ion; phosgene; iodine; oxidizing agents; and strong acid (catalyze atmospheric oxidation of the KI).

6. Apparatus

6.1 *Glass-Stoppered Graduate,* 25-mL.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. 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.[3] 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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type III of Specification D1193.

7.3 *Potassium Iodide (KI) Test Solution*—Dissolve 16.5 g of potassium iodide in water to make 100 mL. Store in light-resistant containers.

7.4 *Starch Test Solution*—Mix 1 g of soluble starch[4] with 10 mg of red mercuric iodide and sufficient cold water to make a thin paste. Add 200 mL of boiling water, and boil for 1 min with continuous stirring. Cool, and use only the clear solution.

[1] This test method is under the jurisdiction of ASTM Committee D26 on Halogenated Organic Solvents and Fire Extinguishing Agents and is the direct responsibility of Subcommittee D26.04 on Test Methods.

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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] *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.

[4] Commercially available, stabilized starch indicator solutions may be used.

8. Procedure

8.1 Transfer 10 mL of sample to a glass-stoppered graduate and add 10 mL of potassium iodide test solution and 1 mL of starch indicator solution. Shake vigorously for 2 min and observe color of the aqueous layer. The presence of free halogens is indicated by a blue coloration.

NOTE 1—The potassium iodide solution can generate free iodine via air oxidation. For this reason, the test procedure should first be performed on a reagent blank (10 mL of potassium iodide and 1 mL of starch). If a faint blue color is observed in the blank, a fresh potassium iodide solution should be prepared and a reagent blank retested before proceeding with the sample solution.

9. Report

9.1 Report the following information:

9.1.1 Report as either positive or negative.

10. Precision and Bias

10.1 This is a pass/fail test. It is not the intent of this procedure to provide a test method for determining the concentration of free halogens.

10.2 The lower limit of detection of this test method has not been documented by formal precision tests. Detection limit is below 1 ppm, by weight as Cl_2 based on repeatability of a variety of standards.

11. Keywords

11.1 free halogens; halogenated organic solvent; halogens; solvent