



Designation: D 3634 – 99 (Reapproved 2005)

## Standard Test Method for Trace Chloride Ion in Engine Coolants<sup>1</sup>

This standard is issued under the fixed designation D 3634; 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 approval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

### 1. Scope

1.1 This test method covers the determination of chloride ion in engine coolants in the range from 5 to 200 ppm in the presence of up to 0.6 weight % mercaptobenzothiazole.

1.2 Other materials that react with silver ion will interfere.

1.3 Chloride in engine coolants containing an aryltriazole instead of mercaptobenzothiazole can be determined directly by this test method without pretreatment with hydrogen peroxide.

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

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 7.

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

**D 1176** Practice for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes

**D 1193** Specification for Reagent Water

**E 200** Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

2.2 *Manufacturing Chemists Association Document:*<sup>3</sup>

MCA Chemical Safety Data Sheet SD-53

### 3. Summary of Test Method

3.1 The sample is first treated at a pH of 12 to 13 with aqueous hydrogen peroxide to oxidize the mercaptobenzothiazole to soluble, noninterfering sulfonate. The treated sample is dissolved in glacial acetic acid and titrated potentiometrically with dilute standard silver nitrate solution. Since the solubility of silver chloride in this system is sufficient to prevent obtaining a suitable inflection at the very low limit of the test method, some chloride is deliberately added to the glacial acetic acid solvent and then corrected for by a blank titration.

### 4. Significance and Use

4.1 This test method permits the determination of very low levels of chloride ion in engine coolants containing the common corrosion inhibitor, mercaptobenzothiazole, or related mercaptans, which would normally interfere with the titration by also forming insoluble silver salts with silver nitrate.

### 5. Apparatus

5.1 *Manual Titrations:*

5.1.1 *pH Meter*—An expanded scale pH meter which can be read to 1 or 2 mV is desirable but not required. A silver billet indicator electrode<sup>4</sup> and glass reference electrode<sup>5</sup> are used for the chloride titration. The silver electrode should be polished occasionally with fine steel wool or scouring powder and thoroughly rinsed.

5.1.2 *Buret*, 10-mL, micro, Class A, calibrated in 0.02-mL divisions.

5.1.3 *Beakers*, electrolytic, 250-mL tall form.

5.2 *Automatic Titrators* are satisfactory for this test method, provided they exhibit resolution and accuracy equivalent to that specified for manual titrations.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D15 on Engine Coolants and is the direct responsibility of Subcommittee D15.04 on Chemical Properties.

Current edition approved Oct. 1, 2005. Published November 2005. Originally approved in 1977. Last previous edition approved in 1999 as D 3634 – 99<sup>ε1</sup>.

<sup>2</sup> 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.

<sup>3</sup> Available from the Manufacturing Chemists Association, 1825 Connecticut Ave., Washington, DC 20009.

<sup>4</sup> The sole source of supply of the Silver Billet Electrode known to the committee at this time is Fisher Scientific (www.fishersci.com, Catalog No. 13-620-122). If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>4</sup> which you may attend.

<sup>5</sup> The sole source of supply of the reference electrode known to the committee at this time is Fisher Scientific (www.fishersci.com, Catalog No. 13-620-216). If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>5</sup> which you may attend.