Designation: D 3634 – 99$^{\epsilon 1}$

Standard Test Method for Trace Chloride Ion in Engine Coolants[1]

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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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

ε[1] NOTE—Editorial changes were made in November 2001.

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 the 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:*

D 1176 Test Method for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes[2]

D 1193 Specification for Reagent Water[3]

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis[2]

2.2 *Manufacturing Chemists Association Document:*[4]

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[5] and glass reference electrode[6] 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.

5.3 *Pipets*, 10, 20, and 100-mL, Class A.

5.4 *Flask, Erlenmeyer*, 250 mL, with a 24/40 standard taper, female ground glass joint.

[1] This test method is under the jurisidiction of ASTM Committee D15 on Engine Coolants and is the direct responsibility of Subcommittee D15.04 on Chemical Properties.

Current edition approved May 10, 1999. Published August 1999. Originally published as D 3634 – 77. Last previous edition D 3634 – 93.

[2] *Annual Book of ASTM Standards,* Vol 15.05.

[3] *Annual Book of ASTM Standards,* Vol 11.01.

[4] Available from the Manufacturing Chemists Association, 1825 Connecticut Ave., Washington, DC 20009.

[5] The 1993–1994 Fisher Scientific Company Catalog number for the Silver Billet Electrode is: 13-620-122 and it is available from 10700 Rockley Road, P.O. Box 1307, Houston, TX 77251.

[6] The 1993-1994 Fisher Scientific Company Catalog number for the reference electrode is 13-620-216 and it is available from 10700 Rockley Road, P.O. Box 1307, Houston, TX 77251.