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.

Designation: D1123 – 22a

Standard Test Methods for Water in Engine Coolant Concentrate by the Karl Fischer Reagent Method[1]

This standard is issued under the fixed designation D1123; 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 U.S. Department of Defense.

1. Scope*

1.1 These test methods cover the determination of the water present in new or unused glycol-based coolant concentrates using a volumetric (Test Method A) or an automatic coulometric titrator procedure (Test Method B).

1.2 Many carbonyl compounds react slowly with the Fischer reagent, causing a fading end point and leading to high results. A modified Fischer reagent procedure is included that minimizes these undesirable and interfering reactions.

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 *This standard does not purport to address all of the safety problems, 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.* For specific hazards statements see Sections 8 and 16.

1.5 *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]

D156 Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)

D1176 Practice for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes

D1193 Specification for Reagent Water

E203 Test Method for Water Using Volumetric Karl Fischer Titration

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *color end point*—that point during the titration when the color change from yellow to orange-red is sharp and easily repeated. The orange-red color must persist for at least 30 s in order to indicate an end point.

3.1.1.1 *Discussion*—View the color by transmitted daylight or by transmitted light from an artificial daylight lamp, such as one that complies with the specification given in Test Method D156.

3.1.2 *instrument end point*—for the purpose of these tests, that point in the titration when two small platinum electrodes, upon which a potential of 20 mV to 50 mV has been impressed, are depolarized by the addition of 0.05 mL of Fischer reagent (6 mg of water/mL), causing a change of current flow of 10 µA to 20 µA that persists for at least 30 s.

3.1.2.1 *Discussion*—This end point is sometimes incorrectly called the "dead stop," which is the reverse of the above.

4. Summary of Test Method

4.1 These test methods are based essentially on the reduction of iodine by sulfur dioxide in the presence of water. This reaction can be used quantitatively only when pyridine and an alcohol are present to react as follows:

$$I_2 + SO_2 + H_2O \rightarrow SO_3 + 2HI \quad (1)$$

4.2 In order to determine water, Karl Fischer reagent is added to a solution of the sample in anhydrous high-purity methanol until all water present has been consumed. This is evidenced by the persistence of the orange-red end point color, or alternatively by an indication on a galvanometer or similar current-indicating device that records the depolarization of a pair of noble metal electrodes. The reagent is standardized by the titration of water.

[1] These test methods are under the jurisdiction of ASTM Committee D15 on Engine Coolants and Related Fluids and are the direct responsibility of Subcommittee D15.04 on Chemical Properties.

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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.

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

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NOTE 1—It is believed that these methods give all the information required for determining the water in coolant formulations. Should additional information on water determinations be needed, reference should be made to Test Method E203.

5. Significance and Use

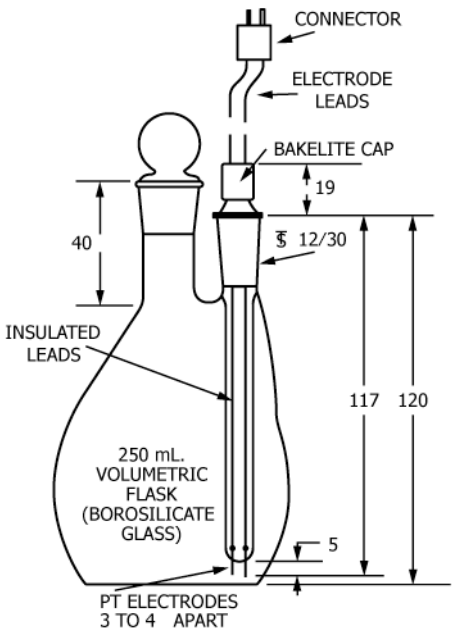
5.1 The total apparent water in engine coolant concentrate as determined by Karl Fischer titrations consists of the following: (*1*) water present in the original glycol base; (*2*) water added (for example, inhibitor solutions); (*3*) water of hydration of inhibitors (for example, $Na_2B_4O_7{\cdot}5H_2O$); (*4*) water formed in the chemical reaction between borate and ethylene glycol, producing boratediol condensate and water; and (*5*) quantitative interference by the reaction of the reagent with inhibitors such as tetraborate or sodium hydroxide.

TEST METHOD A—VOLUMETRIC TITRATION

6. Apparatus

6.1 *Titration Vessel*—For color end point titrations, use a 100 mL or 250 mL volumetric flask, which need not be calibrated. For instrument end point, a 250 mL flask fitted with interchangeable electrodes (Fig. 1) may be used. This is particularly good for titrations of coolant concentrate that is deeply colored from dye or any other cause. For permanently mounted assemblies, the vessel should have a capacity about equal to that of a 300 mL tall-form beaker and should be provided with a tight-fitting closure to protect the sample and reagent from atmospheric moisture, a stirrer, and a means of adding sample and reagents and removing spent reaction mixture. It is desirable to have a means for cooling the titration vessel to ice temperature.

6.2 *Instrument Electrodes,* platinum with a surface equivalent to two No. 26 wires, 4.76 mm long. The wires should be 3 mm to 8 mm apart and so inserted in the vessel that the liquid will cover them.



NOTE 1—All dimensions in millimetres.

FIG. 1 Titration Flask Assembly

6.3 *Instrument Depolarization Indicator,* having an internal resistance of less than 5000 Ω and consisting of a means of impressing and showing a voltage of 20 mV to 50 mV across the electrodes and capable of indicating a current flow of 10 μA to 20 μA by means of a galvanometer or radio tuning eye circuit.

6.4 *Buret Assembly* for Fischer reagent, consisting of a 25 mL or 50 mL buret connected by means of glass (not rubber) connectors to a source of reagent; several types of automatic dispensing burets may be used. Since the reagent loses strength when exposed to moist air, all vents must be protected against atmospheric moisture by adequate drying tubes containing anhydrous calcium sulfate. All stopcocks and joints should be lubricated with a lubricant not particularly reactive with the reagent.

6.5 *Weighing Bottle,* of the Lunge or Grethen type, or equivalent.

6.6 Some laboratory equipment suppliers offer a Karl Fischer apparatus.

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 Unless otherwise indicated, references to water shall be understood to mean reagent water, Type IV, conforming to Specification D1193.

7.3 *Karl Fischer Reagent,* pyridine-free, equivalent to 5 mg of water/mL.

7.4 *Methanol* (**Warning—**See 8.1)—Anhydrous, high purity.

8. Hazards

8.1 *Methanol*—Poison; flammable; may be fatal or cause blindness if swallowed; cannot be made non-poisonous; harmful if inhaled.

9. Sampling

9.1 A representative sample of the contents of the original container shall be obtained as directed in Practice D1176; even if two phases are present, the water-insoluble phase should not be separated.

[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.