



Standard Test Method for Moisture in Aerosol Products¹

This standard is issued under the fixed designation D 3057; 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.

1. Scope

1.1 This test method covers the determination of moisture in aerosol products.

1.2 *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.* A specific warning statement is given in Note 1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 Moisture is determined in an aerosol product by back-titrating the specimen, to which Karl Fischer reagent has been added, with a methanol-water solution. A ratio between the methanol and Karl Fischer reagent is obtained, and the Karl Fischer reagent is standardized against sodium tartrate dihydrate.

4. Significance and Use

4.1 Moderate concentration of water in certain aerosol formulations can lead to metallic can corrosion.

NOTE 1—**Warning:** Contaminated primary alcohols with water levels of 1 to 2 % can produce hydrogen gas in aluminum containers causing them to rupture.

4.2 Ketones, such as acetone, can produce falsely high results.

5. Apparatus

5.1 *Closed Containers*, glass, three.

5.2 *Stirrer*, magnetic.

6. Reagents

6.1 *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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean water conforming to Specification D 1193.

6.3 *Karl Fischer Reagent*.

6.4 *Methanol*—Anhydrous methyl alcohol.⁴

6.5 *Methanol-Water Standard Solution*.

6.6 *Sodium Tartrate Standard* —(Na₂C₄H₄O₆·2H₂O).

7. Sampling

7.1 Obtain production made samples selected at random.

8. Standardization

8.1 *Ratio of Karl Fischer Reagent to Methanol-Water Solution:*

8.1.1 Place approximately 60 mL of methanol into a closed glass container, and add from 2 to 3 mL of Karl Fischer reagent. Agitate the solution with the magnetic stirrer for approximately 5 min.

8.1.2 Back-titrate the solution with the methanol-water solution until the end point is reached; then add 10 mL of Karl Fischer reagent. Back-titrate with the methanol-water solution until the end point is reached.

8.1.3 Calculate the ratio of Karl Fischer reagent to methanol-water solution, X , as follows:

$$X = A/B$$

where:

A = Karl Fischer reagent used, mL, and

B = methanol-water solution used, mL.

NOTE 2—A ratio close to 1 is recommended.

8.2 *Standardization of Karl Fischer Reagent:*

³ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

⁴ Baker's Analyzed Anhydrous Methanol (approximately 0.05 % water), available from Baker Chemical Co., has been found satisfactory for this method.

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² *Annual Book of ASTM Standards*, Vol 11.01.

8.2.1 Place approximately 60 mL of methanol into a closed glass container and add from 2 to 3 mL of Karl Fischer reagent. Agitate the solution for 5 min with the magnetic stirrer.

8.2.2 Back-titrate the solution with the methanol-water solution until the end point is reached. Add 0.32 g of sodium tartrate and approximately 6 mL of Karl Fischer reagent. Agitate until the sodium tartrate is completely dissolved, then back-titrate with the methanol-water solution until the end point is reached.

8.2.3 Calculate the millilitres of Karl Fischer reagent used to combine with the water in the sodium tartrate standard, C , as follows:

$$C = A - (BX)$$

8.2.4 Calculate the milligrams of water for 1 mL of Karl Fischer reagent, y , as follows:

$$y = W/C$$

where:

W = total water in the sodium tartrate, mg.

9. Procedure

9.1 Place approximately 60 mL of methanol in a closed glass container, and add from 2 to 3 mL of Karl Fischer reagent. Agitate the solution with the magnetic stirrer for 5 min, then back-titrate until the end point is reached.

9.2 Add a weighed amount of the specimen, and an excess of the Karl Fischer reagent. Agitate the solution for 5 min, then back-titrate with the methanol-water solution until the end point is reached.

10. Calculation

10.1 Calculate the water content (mass percent), M , as follows:

$$M = (A \times y \times 100)/S$$

where:

S = specimen, g.

11. Report

11.1 The report shall include the following:

11.1.1 Product being tested, and

11.1.2 Water content of product (mass percent).

12. Precision

12.1 *Precision*—This test method is being reviewed for development of a precision statement based upon intra-laboratory test data. Volunteers from various CSMA Aerosol Division, Test and Standard Methods subcommittee membership will provide the precision statement based upon their within-laboratory data.

12.2 The following criteria shall be used for judging the acceptability of results (95 % confidence) and apply only to aerosol products:

12.2.1 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than 5 %.

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