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~~An American National Standard~~

Standard Test Methods for Nonvolatile Matter in Halogenated Organic Solvents and Their Admixtures[1]

This standard is issued under the fixed designation D 2109; 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~~—A research report footnote was added in November 2001.~~
—Removed research report references in Sections 7, 10, and 13 editorially in March 2008.

1. Scope

1.1 These test methods cover the determination of nonvolatile matter in halogenated organic solvents and admixtures.

~~1.2Three~~1.2 Five test methods are covered, as follows:

1.2.1 *Test Method A*— For halogenated organic solvents or admixtures having less than 50 ppm nonvolatile matter; or where precision ~~greater~~better than ±10 ppm is required.

1.2.2 *Test Method B*— For halogenated organic solvents or admixtures having more than 50 ppm nonvolatile matter or where precision of ±0.001 % (10 ppm) is satisfactory.

1.2.3 *Test Method C*~~—For low-boiling halogenated organic solvents or their admixtures (for example, methylene chloride, trichlorotrifluoroethane) that may superheat and cause bumping while evaporating to dryness with steam. A precision of greater than ±10 ppm can be attained.~~ — For low-boiling halogenated organic solvents or their admixtures (for example, methylene chloride, trichlorotrifluoroethane) that may superheat and cause bumping while evaporating to dryness with steam. A precision of greater than± 10 ppm can be attained.

1.2.4 *Test Method D*— For rapid measurement of nonvolatile matter in halogenated organic solvents and their admixtures and where precision better than ±10 ppm is required.

1.2.5 *Test Method E*— For halogenated organic solvents or admixtures and where precision better than ±10 ppm is required.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 The term *nonvolatile matter* should not be construed as equivalent to *residue on ignition, ignition residue,* or *ash content*. Particulates, sediments, and suspended matter should not be considered part of nonvolatile ~~residue.~~matter. If these solids are present in the sample, they should be removed by filtration or decantation prior to beginning this test method. Nonvolatile matter is considered to be "in solution" with the solvent and that which will become residual upon drying the solvent at a specified temperature.

2.1.2 Nonvolatile matter and nonvolatile residue are interchangeable terms.

3. Significance and Use

3.1 Nonvolatile matter in solvents can adversely affect their cleaning properties. These test methods can be used to control soil contamination in the boiling solvent, which if allowed to become too high, can decrease the stability of the solvent.

3.2 These test methods can be used to establish manufacturing and purchasing specifications.

4. Apparatus

4.1 *Oven*, thermostatically controlled at 105 ± 5°C.

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

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4.2 *Evaporating Dish*, 125-mL capacity, platinum or high-silica glass. , 125-mL capacity, platinum or high-silica glass, Methods A, B, C.
4.3 *Evaporating Dish* (80 × 45 or 115 × 50) (Method D).
4.4 *Steam Bath* (or hot plate).
4.4*Analytical Balance*.
4.5 *Hot Plate*, (Method D).
4.6 *Heat Lamp*, 250 W, (Method E).
4.7 *Analytical Balance*, capable of measuring to 0.0001 g.
4.8 *Top Loading Balance*, capable of weighing to 0.01 g.
4.9 *Aluminum Weighing Dish*, 57 × 18 mm, (Method D).
4.10 *Aluminum Weighing Dish*, 200 ML capacity, (Method E).
4.11 *1000-mL Volumetric Flask* (Test Method A).
4.6
4.12 *100-mL Volumetric Pipet*(Test Method B).
4.7*1000-mL Graduated Cylinder* (Test Method B and E).
4.13 *1000-mL Graduated Cylinder* (Test Method C).
4.8
4.14 *1500-mL Erlenmeyer Flask* (Test Method C).

TEST METHOD A

5. Procedure

5.1 Dry a 125-mL capacity platinum (or high-silica glass) evaporating dish in an oven at 105 ± 5°C and cool in a desiccator. Repeat until the weight is constant or within 0.1 mg of the last weighing. Rinse a clean dry 1000-mL volumetric flask with the solvent and fill to the 1000-mL mark with the solvent to be tested. Invert the evaporating dish, place it over the mouth of the flask, hold it firmly in place, and invert the flask. In this position place both dish and flask on a steam bath. Adjust a ring support to hold the flask so the mouth of the flask is approximately 25 mm above the bottom of the evaporating dish. Thus held, the flask automatically feeds the solvent to the dish during the evaporation. (**Warning**—This test method must be run in a ventilated, dust-free area.)

5.2 Evaporate the 1000-mL sample to dryness. Remove the dish from the steam bath with metal tongs and blot the outside of the dish with lint-free paper tissue.

NOTE1—**Caution:**This test method must be run in a ventilated, dust-free area.

5.2Evaporate the 1000-mL sample to dryness. Remove the dish from the steam bath with metal tongs and blot the outside of the dish with lint-free paper tissue.

NOTE2—Hot plates develop high temperatures on the plate surface. If a hot plate is used to evaporate the solvent, the evaporating dish should be placed inside a water bath while on the hot plate to prevent the sample from reaching temperatures exceeding 105°C. 1—Hot plates develop high temperatures on the plate surface. If a hot plate is used to evaporate the solvent, the evaporating dish should be placed inside a water bath while on the hot plate to prevent the sample from reaching temperatures exceeding 105°C.

5.3 Place the dish and contents in an oven at 105 ± 5°C for approximately 1 h. Cool in a desiccator and weigh the dish and contents.

6. Calculation

6.1 Calculate the nonvolatile matter and report in weight percent or parts per million as follows:

$$\text{Nonvolatile matter, ppm by weight} = \frac{(A)\,(10^6)}{(B)\,(1000)} = \frac{(A)\,(1000)}{(B)} \quad (1)$$

where:
A = grams of residue, and
B = density of sample.

7. Precision and Bias

7.1 *Repeatability* (*Single Analyst*)—The standard deviation of results has been estimated to be 0.2 weight ppm. Two such values should be considered suspect (95 % confidence level) if they differ by more than 0.7 ppm.

7.2 *Reproducibility* (*Multilaboratory*)—The standard deviation of results has been estimated to be 1.0 weight ppm. Two such values should be considered suspect (95 % confidence level) if they differ by more than 4.6 weight ppm.

TEST METHOD B

8. Procedure

8.1 Dry a 125-mL capacity platinum (or high-silica glass) evaporating dish in an oven at 105 ± 5°C and cool in a desiccator.