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**Designation: D 2109 – 96 (Reapproved 2000)<sup>e1</sup>**



# Standard Test Methods for Nonvolatile Matter in Halogenated Organic Solvents and Their Admixtures<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

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~~<sup>ε1</sup> Note—A research report footnote was added in November 2001.~~

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<sup>1</sup> 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.

Current edition approved ~~June Dec.~~ 10, 1996; 2001. Published ~~August 1996~~ February 2002. Originally published as D 2109 – 62 T. Last previous edition D 2109 – 92ε.

## 1. Scope

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

~~1.2 Three~~

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  $\pm 10$  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  $\pm 0.001$  % (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  $\pm 10$  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  $\pm 10$  ppm is required.

1.2.5 *Test Method E*— For halogenated organic solvents or admixtures and where precision better than  $\pm 10$  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  $105 \pm 5^\circ\text{C}$ .

4.2 *Evaporating Dish*, 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).
- 4.612 *100-mL Volumetric Pipet* (Test Method B and E).
- 4.713 *1000-mL Graduated Cylinder* (Test Method C).
- 4.814 *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  $105 \pm 5^\circ\text{C}$  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~~.

NOTE 1—**Caution:** This 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.

NOTE 2<sup>1</sup>—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  $105^\circ\text{C}$ .

5.3 Place the dish and contents in an oven at  $105 \pm 5^\circ\text{C}$  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 <sup>2</sup>

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  $105 \pm 5^\circ\text{C}$  and cool in a desiccator. Repeat until the weight is constant or within 0.1 mg of the last weighing. Pipet 100 mL of sample into the dish and place on a steam bath.

8.2 Evaporate the sample to dryness as in Section 5 (see Note 1 and Note-2); 1). Remove the dish from the steam bath with metal tongs and blot the outside of the dish with lint-free paper tissue.

8.3 Place the dish and contents in an oven at  $105 \pm 5^\circ\text{C}$  for approximately 1 h. Cool in a desiccator and weigh the dish and contents.

<sup>2</sup> A research report is available from ASTM headquarters. Request RR: D26-1017.

## 9. Calculation

9.1 Calculate the nonvolatile matter and report in weight percent as follows:

$$\text{Nonvolatile matter, ppm by weight} = \frac{(A)(100)}{(B)(100)} = \frac{(A)}{(B)} \quad (2)$$

where:

$A$  = grams of residue, and

$B$  = density of sample.

## 10. Precision and Bias <sup>2</sup>

10.1 *Precision*—As the only significant difference between Test Method B and Test Methods A and C is the sample size (100 mL versus 1000 mL), it is reasonable to assume that the precision is roughly ten times that of Test Methods A and C or 2 ppm for repeatability and 10 ppm for reproducibility.

10.2 The bias of this test method ~~is being~~ has not been determined.

## **TEST METHOD C**

### 11. Procedure

11.1 Dry a 125-mL capacity platinum (or high-silica glass) evaporating dish in an oven at 105°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 graduated cylinder and a 1500-mL Erlenmeyer flask with the solvent to be tested.

11.2 Measure 1000 mL of the sample into the cleaned graduate and transfer 970 mL of it into the clean Erlenmeyer flask. Evaporate to about 40 mL on a steam bath or warm hot plate in a fume hood. Do not evaporate to dryness.

11.3 Transfer the residue to the evaporating dish. Rinse the flask twice with a 15-mL portion of the sample retained in the graduate (see Note-3). 2. Add the rinsings to the evaporating dish.

NOTE 3—All of the retained sample is used to rinse the beaker and must be included in the sample volume.

11.4 Allow the sample to evaporate by gently heating on a warm hotplate. Protect the evaporating dish from external contamination and do not exceed 60°C (140°F). When evaporation is complete, dry the evaporating dish in the oven at 1405°C for 30 min, cool in a desiccator for 30 min, and reweigh.

### 12. Calculation

12.1 Calculate the nonvolatile matter and report in parts per million as follows:

$$\text{Nonvolatile matter, ppm by weight} = \frac{(A)(10^6)}{(B)(1000)} = \frac{(A)(1000)}{(B)} \quad (3)$$

where:

$A$  = grams of residue, and

$B$  = density of sample.

### 13. Precision and Bias <sup>2</sup>

13.1 *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.

13.2 *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 D**

### 14. Procedure

14.1 Weigh 100 g of sample on a top loading balance into an evaporating dish (80 × 45 or 115 × 50) and place it on the hot plate. Record the sample weight. Adjust the heat so that the sample boils away without spattering or splashing out of the dish. Evaporation of the solvent must be carried out in a fume hood.

14.2 While the sample is evaporating, weigh an aluminum weighing dish to 0.0001 g and record the weight  $W_1$ .

14.3 Evaporate the sample to within 8 to 12 mL of dryness and transfer it to the weighed aluminum weighing dish using metal tongs.

14.4 Place the aluminum weighing dish on the hot plate and evaporate until only a small amount of liquid (about 1 mL) remains.

14.5 Transfer the aluminum weighing dish to an oven set at 105°C and leave it for 30 min.

14.6 Remove the dish from the oven, allow it to cool for 1 min and weigh the dish. Record the weight as  $W_2$ . It is not necessary to cool the dish in a desiccator.

14.7 Calculate the nonvolatile matter (NVM) by the formula:

$$PPM\ NVM = \frac{(W_2 - W_1) \times 1\ 000\ 000}{\text{sample weight}} \quad (4)$$

14.8 In many cases the analysis can be done faster in the following way:

14.8.1 Leave the sample in the 105°C oven until the dish is free of liquid but not more than 30 min.

14.8.2 When the dish is free of liquid, remove it from the oven and cool it for 1 min. Weigh the dish. If the calculation shows the result to be less than one ppm, the test may be stopped and the results recorded.

14.8.3 If there is measurable NVM (>1 ppm) in the dish, return it to the oven for 30 min and proceed beginning with 14.6.

### 15. Precision and Bias <sup>2</sup>

15.1 The standard deviation of results of a single analyst over different days was determined to be 1.8 ppm at a level of 10 ppm nonvolatile matter.

15.2 The standard deviation of results from multiple laboratories over multiple days was determined to be 2.2 ppm at a level of 10 ppm nonvolatile matter.

15.3 Overall recovery for this method was 104.5 % from approximately 1 ppm to approximately 1000 ppm nonvolatile matter.

15.4 The low quantification limit is approximately 1 ppm.

## TEST METHOD E

### 16. Procedure

16.1 Dry a 200 mL capacity aluminum dish overnight (16 to 24 h), handling with metal tongs.

16.2 Place in a dessicator for at least 5 min. If the conditioned dish is not used within 24 h of cooling, repeat the conditioning process.

16.3 Weigh a conditioned aluminum pan to the nearest 0.0001 g,  $W_1$ .

16.4 Place the aluminum pan under the heat lamp in a fume hood. The heat lamp should be positioned approximately 5 in. (12.7 cm) above the bottom of the aluminum dish. Slowly add 100 mL of the solvent to the pan using a volumetric pipet.

16.5 Turn on the heat lamp and evaporate until only a small amount (about 1 mL) of liquid remains.

16.6 Place the pan and contents in an oven at 105°C for 10 to 15 min.

16.7 Remove the pan with tongs and place in a dessicator for at least 5 min to cool. Weigh the pan and contents  $W_2$ .

16.8 Calculate the nonvolatile matter (NVM) by the formula:

$$PPM\ NVM = \frac{(W_2 - W_1) \times 1\ 000\ 000}{(\text{Density} \times 100)} \quad (5)$$

where density is the density of the solvent being analyzed.

### 17. Precision and Bias <sup>2</sup>

17.1 The standard deviation of results of a single analyst over different days was determined to be 1.9 ppm at a level of 10 ppm nonvolatile matter

17.2 The standard deviation of results from multiple laboratories over multiple days was determined to be 3.6 ppm at a level of 10 ppm nonvolatile matter.

17.3 Overall recovery for this method was 123.0 % from approximately 1 ppm to approximately 100 ppm nonvolatile matter.

17.4 The lower quantification limit is approximately 1 ppm.

### 18. Keywords

148.1 halogenated organic solvents; nonvolatile matter; nonvolatile residue; residue on evaporation

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