



Standard Test Method for Evaporating Residue of Naphthalene¹

This standard is issued under the fixed designation D2232; 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.

1. Scope*

1.1 This test method covers the determination of the evaporation residue of naphthalene.

1.2 This test method has been found applicable to determining residue in the range of 0.3 and 1.5 wt %.

1.3 In determining the conformance of the test results using this method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.* For specific hazard statements, see Section 7 and 8.1.

2. Referenced Documents

2.1 ASTM Standards:²

D3438 Practice for Sampling and Handling Naphthalene, Maleic Anhydride, and Phthalic Anhydride

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

¹ This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.07 on Styrene, Ethylbenzene and C9 and C10 Aromatic Hydrocarbons.

Current edition approved July 1, 2014. Published August 2014. Originally approved in 1967. Last previous edition approved in 1990 as D2232 – 81 (1990) which was withdrawn April 1995 and reinstated in July 2014. DOI: 10.1520/D2232-14.

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.

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 Other Document:

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200³

3. Terminology

3.1 See Terminology D4790 for the definition of terms used in this test method.

4. Summary of Test Method

4.1 A weighed quantity of naphthalene is heated in a tared dish for 3 h at 105°C in a forced-draft oven and the residue is weighed.

5. Significance and Use

5.1 Evaporation residue is an empirical measure of nonvolatile impurities in naphthalene. This test method is suitable for setting specifications and for use as an internal quality control tool.

6. Apparatus

6.1 *Evaporating Dishes*, porcelain, shallow form or aluminum weighing dishes, low form, fluted, 60 mm diameter, 15 mm high, 42 mL capacity.

6.2 *Drying Oven*, forced-ventilation, conforming to Specification E145 Type II, Grade A or B.

7. Hazards

7.1 Consult current OSHA regulations, supplier's Safety Data Sheets and local regulations for all materials used in this test method.

8. Sampling and Handling

8.1 Refer to Practice D3438 for proper sampling and handling of this product analyzed by this test method. (**Caution**—Sampling should follow safe rules in order to adhere to all safety precautions as outlined in the latest OSHA regulations.)

³ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

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

9. Procedure

9.1 Place the evaporating dish in the drying oven and heat at $105 \pm 5^\circ\text{C}$ for at least 30 min. Remove from the oven and cool in a desiccator. Weigh the cooled dish to the nearest 0.001 g.

9.2 Transfer to the tared dish 9.5 to 10.5 g weighed to the nearest 0.001 g of the thoroughly mixed naphthalene specimen. (**Caution**—Specimens for this determination should be handled in the solid state.)

9.3 Place the dish and contents in the forced draft oven. Maintain at $105 \pm 5^\circ\text{C}$ for 180 ± 5 min. (**Caution**—Specimens should be placed as near the center of the oven as possible (both horizontally and vertically). The oven should not contain other volatile materials during this determination, nor should it contain objects large enough to change the flow pattern of the circulating air. The draft control on the oven should be fully open.)

9.4 Remove the dish from the oven, cool in a desiccator, and weigh to the nearest 0.001 g.

10. Calculation

10.1 Calculate the evaporation residue content of the specimen, E , as follows:

$$E = [(B - A) / C] \times 100 \quad (1)$$

where:

- E = evaporation residue, wt %,
- B = weight of evaporating dish and residue, g,
- A = tare weight of evaporating dish, g, and
- C = weight of specimen used, g.

11. Precision and Bias⁴

11.1 An ILS was conducted which included six laboratories analyzing four samples three times. Practice E691 was followed for the design and analysis of the data. The details are given in ASTM Research Report No. RR:D16-1051.

11.2 Repeatability:

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1051. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Evaporation Residue (wt. %)

Material	Average ^A \bar{X}	Repeatability Limit r	Reproducibility Limit R
A	0.07	0.09	0.09
B	0.26	0.04	0.11
C	0.35	0.14	0.34
D	1.48	0.27	1.36

^A The average of the laboratories' calculated averages.

11.2.1 Results should not be suspect unless they differ by more than shown in Table 1. Results differing by less than “ r ” have a 95 % probability of being correct.

11.3 Reproducibility:

11.3.1 Results submitted by two laboratories should not be considered suspect unless they differ by more than shown in Table 1. Results differing by less than “ R ” have a 95 % probability of being correct.

11.4 Bias:

11.4.1 Since there is no accepted reference material suitable for determining the bias in this test method, bias has not been determined.

12. Quality Guidelines

12.1 Laboratories shall have a quality control system in place.

12.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

12.1.2 A quality control sample is a stable material isolated from the production process and representative of the sample being analyzed.

12.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

12.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical control practices.

13. Keywords

13.1 coal tar; naphthalene; petroleum; residue

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D2232–81(1990)) that may impact the use of this standard. (Approved July 1, 2014.)

(1) Method was withdrawn in 1995 and was still in use in 1995 and is being updated to meet D16 editorial guidelines.

(2) Changes included updating footnotes, adding Quality Guidelines, keywords and mandatory statements at the end of the method.

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