Designation: D 3030 – 95

An American National Standard

Standard Test Method for Volatile Matter (Including Water) of Vinyl Chloride Resins[1]

This standard is issued under the fixed designation D 3030; 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 quantitative determination of the volatile matter (including water) present in vinyl chloride resins.

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

1.3 *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.*

NOTE 1—This test method and ISO 1269–1980 are identical in all technical details.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[2]

D 883 Terminology Relating to Plastics[2]

D 1600 Terminology for Abbreviated Terms Relating to Plastics[2]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[3]

2.2 *ISO Standard:*

ISO 1269–1980 Homopolymer and Copolymer Resins of Vinyl Chloride—Determination of Volatile Matter Including Water[4]

3. Terminology

3.1 *Definitions:* Definitions are in accordance with Definitions D 883 and Terminology D 1600, unless otherwise indicated.

4. Summary of Test Method

4.1 This test method consists of heating at 110°C a known quantity of powdered resin to constant mass. The calculated mass loss defines quantitatively the volatile matter present in the sample.

5. Significance and Use

5.1 The quantity of volatile components in a vinyl chloride resin can be established by this test method. This test method does not identify the components.

6. Apparatus

6.1 *Oven*—A forced-ventilation oven conforming to the requirements for Type IIA in Specification E 145. The oven should be capable of maintaining a temperature of 110 ± 1°C when the damper is half open and the vent is wide open.

6.1.1 The oven shall be equipped with a calibrated ASTM thermometer and the proper stem correction shall be applied to the temperature measurement.

6.1.2 The oven temperature shall be controlled by an accurate, reliable thermoregulator, maintaining the set point within ±0.5°C or better.

6.1.3 The inside of the oven shall be free of contamination or surface deposits. Stainless steel oven liners should be used to reduce corrosion due to continued exposure to decomposition gases.

6.2 *Sample Container*—A weighing vessel of low shape design in glass, aluminum, or stainless steel of sufficient size to hold a 10-g resin sample. An aluminum weighing dish, 57 mm in diameter and 18 mm in depth, is suitable.

6.3 *Balance*—A weighing balance with a precision to within 0.001 g.

7. Preparation of Apparatus

7.1 Maintain the oven at the temperature of the test for at least 1 h prior to insertion of the specimens.

7.2 Prior to test operation, verify the uniformity of temperature within the oven according to Specification E 145. The airflow in the oven should be greater than 0.3 m^3 /min (10 ft^3 /min).

NOTE 2—The temperature uniformity can also be verified by placing 5 or more portions of the same resin sample in the oven, distributed over the test area, following Section 8. Calculate the percent of volatile loss (Section 9). If the differences in the volatile loss of the samples exceed the test reproducibility, the temperature in the oven is not uniform and must be corrected before proceeding with the test.

8. Procedure

8.1 Dry the weighing vessel in the oven at 110 ± 1°C for 30

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[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials (Section D20.15.08).

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This edition includes the addition of an ISO equivalency statement (Note 1).

[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

min and cool to room temperature in a desiccator. Tare the weighing vessel to the nearest 0.001 g.

8.2 Spread approximately 10 g of sample resin evenly over the bottom of the weighing vessel and weigh to the nearest 0.001 g. Record the sample mass.

8.3 Place the resin-filled weighing dish in the oven controlled at 110 ± 1°C and close the oven.

8.4 After 1 h, remove the weighing dish, cool to room temperature in a desiccator, and weigh to the nearest 0.001 g. Return the weighing dish to the oven and heat for an additional 15 min, cool to room temperature in the desiccator, and re-weigh. Constant mass is reached when the successive weighing differs by 0.001 g or less; if the mass variation is greater than 0.001, continue the 15 min heating-weighing cycle until constant mass is obtained.

NOTE 3—Prompt weighing after removal of the dish from desiccator will improve accuracy.

8.5 Test a minimum of two samples, and average the results.

9. Calculation

9.1 For each determination calculate the mass percent of volatile matter present as follows:

$$[(M - M')/M] \times 100$$

where:

M and M′ = respectively, the mass before and after heating of the resin determined at room temperature (a temperature in the range from 20 to 30°C (68 to 85°F), as defined in Methods D 618).

10. Report

10.1 Report the following information:

10.1.1 The arithmetic average of the determinations to the nearest 0.01 mass %.

11. Precision and Bias [5]

11.1 The following should be used for judging the acceptability of results (95 % confidence limits):

11.2 *Reproducibility*—The average of two determinations reported by one laboratory should be considered suspect if it differs from that of another laboratory by more than 22 % relative.

11.3 *Repeatability*—The average of two determinations obtained on two separate days at the same laboratory on the same sample should be considered suspect if it differs by more than 4.4 % relative.

11.4 *Bias*—No justifiable statement of bias can be made for this test method, since the true value of the property cannot be established by an accepted referee method.

NOTE 4—Other techniques of volatile determinations may be substituted at the option of the purchaser and seller, such as the use of a semi-automatic moisture tester or a procedure employing an infrared lamp as a heating source. With such methods, the temperature of the heated sample must be maintained at 110± 1°C.

12. Keywords

12.1 moisture content; test method; vinyl chloride resins; volatile matter

[5] Round-robin data for this test method may be obtained from ASTM Headquarters. Request RR: D-20-1025.