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Standard Test Methods for Rubber From Synthetic Sources—Volatile Matter[1]

This standard is issued under the fixed designation D5668; 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 These test methods cover a hot mill method, two oven methods, and a press method for the determination of moisture and other volatile matter content in synthetic rubber.

1.2 Either oven method shall be used especially when the rubber is too tacky to be handled satisfactorily on a hot mill.

1.3 The press method shall be used for rubbers that are too crumbly to be retained satisfactorily on a laboratory mill.

1.4 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Significance and Use

3.1 These test methods are mainly intended for referee purposes but can also be used for quality control of rubber production.

3.2 The amount of volatiles can affect processing and cure characteristics of compounded rubber.

4. Apparatus

4.1 *Mill,* as described in Practice D3182.

4.2 *Oven,* as described in Specification E145.

4.3 *Press,* as described in Practice D3182.

METHOD A—HOT-MILL TEST METHOD

5. Summary of Test Method

5.1 A weighed sample of rubber is sheeted out on a heated mill until all the volatile matter is driven off. The sample is weighed again and the percentage of volatile matter is calculated.

6. Procedure

6.1 Weigh a test sample of at least 250 g to the nearest 0.1 g.

6.2 Pass the weighed sample repeatedly for 4 min through a laboratory mill maintained at 100 ± 5°C (212 ± 9°F) with the distance between the rolls set at 0.50 ± 0.05 mm (0.020 ± 0.002 in.) as determined by a lead slug. Do not allow the sample to band, and take care to avoid any loss of sample. Weigh the sample to the nearest 0.1 g. Pass the sample through the mill for an additional 2 min and reweigh. If the weights at the end of the 4- and 6-min periods are within 0.1 g, calculate the volatile matter; if not, continue passing the sample through the mill for 2-min periods until the weight remains constant within 0.1 g.

7. Calculation

7.1 Calculate the percentage of volatile matter as follows:

$$\text{Volatile Matter, } \% = [(A - B)/A] \times 100 \quad (1)$$

where:

A = mass of original sample, g, and
B = mass of sample after milling, g.

METHOD B—OVEN TEST METHOD—LARGE TEST SPECIMEN

8. Summary of Test Method

8.1 A weighed sample of rubber is sheeted out on a laboratory mill, then placed in an oven and dried to constant weight. The difference in weight before and after drying is calculated as volatile matter.

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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[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.

9. Procedure

9.1 Sheet out a sample (at least 250 g) of rubber on a laboratory mill with the distance between the rolls set at 0.50 ± 0.05 mm (0.020 ± 0.002 in.) with the temperature of the roll being no greater than 50°C (122°F). Pass the rubber twice between the rolls.

9.2 Weigh the entire sheet to the nearest 0.1 g and place in a forced air circulation oven set at 100 ± 5°C (212 ± 9°F) so that both sides are exposed to the draft. Allow the sheet to remain in the oven until the mass is constant to within 0.1 g. One hour is usually sufficient for rubbers containing no more than 1.0 % moisture.

10. Calculation

10.1 Calculate the percentage volatile matter as follows:

$$\text{Volatile Matter, \%} = [(A - B)/A] \times 100 \quad (2)$$

where:

A = mass of sample before placing in the oven, g, and
B = mass of sample after drying in the oven, g.

METHOD C—OVEN TEST METHOD—SMALL TEST SPECIMEN

11. Summary of Test Method

11.1 A weighed sample of rubber is sheeted out on a laboratory mill, then placed in an oven and dried to constant weight. The difference in weight before and after drying is calculated as volatile matter.

12. Procedure

12.1 Sheet out about 250 g of a sample of rubber on a laboratory mill with the distance between the rolls set at 0.50 ± 0.05 mm (0.020 ± 0.002 in.) and the temperature of the roll being no greater than 50°C (122°F). Pass the rubber twice between the rolls.

12.2 Select a 10-g sample (cut in small pieces approximately 25 mm^3) weighed to the nearest 0.001 g and place in a forced air circulation oven set at 100 ± 5°C (212 ± 9°F) for 1 h so that the sample has the largest surface area exposed to the hot air. Allow to cool in a desiccator and weigh. Repeat the heating for further 30-min periods until the mass does not decrease by more than 0.01 % of the initial mass in successive weighings. One hour is usually sufficient for rubbers containing no more than 1.0 % moisture.

13. Calculation

13.1 Calculate the percentage volatile matter as follows:

$$\text{Volatile Matter, \%} = [(A - B)/A] \times 100 \quad (3)$$

where:

A = mass of sample before placing in the oven, g, and
B = mass of sample after drying in the oven, g.

METHOD D—PRESS TEST METHOD

14. Summary of Test Method

14.1 A sample of rubber is blended on a laboratory mill, weighed, then placed in a heated press under pressure until all the volatiles are driven off. The sample is weighed again and the loss of volatile matter is calculated from the loss in mass.

15. Procedure

15.1 Sheet out about 250 g of a sample of rubber on a laboratory mill with the distance between the rolls set at 1.4 ± 0.05 mm (0.055 ± 0.002 in.) and the temperature of the rolls being no greater than 50°C (122°F). Pass the sample twice between the rolls.

15.2 Weigh 5 to 7 g of the milled sample to the nearest 0.001 g.

15.3 Place between two polytetrafluoroethylene coated metal sheets in a press with the platens maintained at 150 ± 10°C (302 ± 18°F) and press at 5500 ± 275 kPa (800 ± 40 psi) for 3 min.

15.4 If the sample is cloudy or contains bubbles, press again for 3 min. Allow to cool in a desiccator and weigh.

16. Calculation

16.1 Calculate the percentage of volatiles as follows:

$$\text{Volatile matter, \%} = [(A - B)/A] \times 100 \quad (4)$$

where:

A = mass of the sample before heating in the press, g, and

B = mass of the sample after drying in the press, g.

17. Precision

17.1 Precision statements are being prepared in accordance with Practice D4483. They will be added as a revision to these test methods when they are completed.

18. Keywords

18.1 synthetic rubber; volatile