Designation: D5667 – 95 (Reapproved 2015)

Standard Test Method for Rubber From Synthetic Sources—Total and Water Soluble Ash[1]

This standard is issued under the fixed designation D5667; 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 a test method for the determination of ash content in synthetic rubber.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D5668 Test Methods for Rubber From Synthetic Sources—Volatile Matter

PART A—TOTAL ASH

3. Summary of Test Method

3.1 A dried sample is accurately weighed into a tared crucible and ignited in a muffle furnace at about 550 °C until all the carbonaceous material is oxidized. The crucible is then allowed to cool in a desiccator and weighed, and the percentage of ash is calculated.

4. Significance and Use

4.1 These tests are mainly intended for referee purposes but can also be used for quality control of rubber production.

4.2 The total and water soluble ash content of a rubber can affect the properties of compounded, vulcanized rubber such as water absorption, swell, electrical properties, etc.

5. Apparatus

5.1 *Crucible,* tared, having a minimum volume of 25 cm^3 of sample.

NOTE 1—If water soluble ash is to be determined, use a fine-porosity fritted glass or sintered porcelain filtering crucible, placing a small amount of ashless filter paper in the bottom of the crucible in order to prevent loss of liquid pyrolysis products.

5.2 *Ashless Filter Paper,* 150 mm in diameter.

5.3 *Muffle Furnace,* capable of being maintained at 550 ± 25 °C.

5.4 *Glass Stirring Rod.*

5.5 *Rubber Policeman.*

5.6 *Drying Oven.*

6. Sample Preparation

6.1 Prepare the sample by drying in accordance with Test Methods D5668—Method A or Method B.

7. Procedure

7.1 Accurately weigh 3 to 5 g of the dried and sheeted sample in the tared crucible, which has been ignited to constant weight at 550 ± 25 °C. In the case of alum-coagulated rubbers, which tend to boil over during ashing, wrap the sample tightly in a 150-mm ashless filter paper before ashing. Place the crucible containing the sample directly into the muffle furnace at 550 ± 25 °C and allow it to remain there until the carbonaceous material has been completely oxidized.

NOTE 2—**Caution:** Do not open the door of the furnace for at least 1 h after the crucible has been placed in the furnace.

NOTE 3—If fritted glass crucible is used, place the crucible in the muffle furnace at 300 ± 25 °C for 30 min. Then raise the temperature to 550 ± 25 °C and heat for at least 1 h or until all the carbonaceous material has been oxidized. Reduce temperature to 300 °C before removing.

7.2 After the carbon has been completely oxidized, remove the crucible from the furnace, cool it to room temperature in the desiccator, and weigh it. If the ash line is within 3 mm of the rim of the crucible, discard the ash and rerun the analysis.

7.3 Reheat the crucible and its contents for about 30 min at 550 ± 25 °C, remove the crucible from the furnace, cool it to

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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.

room temperature in the desiccator, and weigh it. If the mass differs by more than 1 % from the previous reading, repeat this step.

7.4 If water soluble ash is to be determined, save the ashed sample and proceed to Method B.

8. Calculation

8.1 Calculate the percentage of total ash as follows:

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

where:

A = mass of crucible plus original dried sample, g, and
B = mass of crucible, g, and
C = mass of crucible plus ash, g.

PART B—WATER SOLUBLE ASH

9. Summary of Test Methods

9.1 The ash obtained in Part A is digested and washed with three 30 cm^3 portions of water, the crucible and contents are dried in an oven, and the crucible is then placed in a muffle furnace. The crucible is then cooled, weighed, and the percentage of soluble ash is calculated.

10. Procedure

10.1 Add 30 cm^3 of hot deionized water, at a temperature of 80 to 90°C, to the crucible. Allow to stand for 5 min, stirring with the glass rod tipped with the rubber policeman. Filter with suction and repeat the digestion and filtration with two additional 30 cm^3 portions of hot water. Dry the crucible for 1 h in an air oven at 105 ± 5 °C.

10.2 If the sintered porcelain crucible was used, place in a muffle furnace at 550 ± 25 °C for 1 h. Cool the crucible in a desiccator and weigh it.

10.3 If the fritted glass crucible was used, follow the heating and cooling procedure described in Note 3. Finally, weigh the crucible and contents at room temperature.

11. Calculation

11.1 Calculate the percentage water soluble ash as follows:

$$\text{Water Soluble Ash, \%} = [(C - D)/A - B] \times 100 \quad (2)$$

where:

A = mass of crucible plus original dried sample, g,
B = mass of crucible, g,
C = mass of crucible plus total ash, g, and
D = mass of crucible plus insoluble ash, g.

12. Keywords

12.1 ash; synthetic rubber; total ash; water soluble ash