



Designation: D5667 – 21

Standard Test Method for Rubber From Synthetic Sources—Total and Water Soluble Ash¹

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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 test temperature 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.

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

Current edition approved Nov. 1, 2021. Published December 2021. Originally approved in 1995. Last previous edition approved in 2019 as D5667 – 95 (2019). DOI: 10.1520/D5667-21.

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.

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*, having a minimum volume of 50 cm³.

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 $\pm$ 25 °C or 850 $\pm$ 25 °C, or both.

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.

7. Procedure

7.1 Accurately weigh 3 to 5 g of the dried sample in the tared crucible, which has been ignited to constant weight at 550 $\pm$ 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. Load the crucible with the dried sample at 300 °C. Ramp up the temperature to 550 °C. (Slow heating will reduce the possibility of fly loss during burning/charring of the sample). Allow it to remain there until the carbonaceous material has been completely oxidized.

NOTE 2—**Caution:** Do not open the furnace door for the first 30 min after reaching test temperature. It is recommended to open for a while and close the furnace door and continue heating the sample, to ease the combustion process.

NOTE 3—If fritted glass crucible is used, place the crucible in the muffle furnace at 300 $\pm$ 25 °C for 30 min. Then raise the temperature to 550 $\pm$ 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 material 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 a minimum of 60 min at 550 ± 25 °C, remove the crucible from the furnace, cool it to room temperature in the desiccator, and weigh it. If the mass of the ash differs by more than 1 % from the previous reading, repeat this step.

NOTE 4—It is advised to use same cooling time for crucible conditioning and crucible + ash cooling, for better repeatability.

7.4 For certain material like halogen containing polymers, ash test temperature 550 °C may not sufficient for complete combustion or may take long time to get constant weight, so suggested following method.

7.4.1 Accurately weigh 3 to 5 g of the dried sample in the tared crucible, which has been ignited to constant weight at 850 ± 25 °C.

7.4.2 Load the crucible with the dried sample at 300 °C. Ramp up the temperature to 850 °C. (Slow heating may reduce the possibility of fly loss during burning/charring of the sample.)

7.4.3 Allow it to remain there until the carbonaceous material has been completely oxidized.

NOTE 5—**Caution:** Do not open the furnace door for the first 30 min after reaching test temperature. It is recommended to open for a while and close the furnace door and continue heating the sample, this will ease the combustion process. It is advised to use same cooling time for crucible conditioning and crucible + ash cooling, for better repeatability. For safety, crucible may be removed at a lower temperature than the test temperature.

7.4.4 After the material 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.

7.4.5 Reheat the crucible and its contents for a minimum of 60 min at 850 ± 25 °C, remove the crucible from the furnace, cool it to room temperature in the desiccator, and weigh it. If the mass differs by more than 1 % from the previous reading, repeat this step.

7.5 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.

8.1.1 Report ash content with test temperature nearest two decimal points.

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 cm³ 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 cm³ 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 cm³ 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