



Designation: D1278 – 91a (Reapproved 2020)

Standard Test Methods for Rubber from Natural Sources—Chemical Analysis¹

This standard is issued under the fixed designation D1278; 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 the sampling and chemical analysis of solid natural rubber in the forms supplied to the rubber industry.

1.2 The analytical procedures appear in the following order:

	Sections
Sampling	5
Volatile Matter	6 – 8
Dirt	9 – 13
Ash	14 – 17
Copper (Referee Colorimetric Method)	18 – 22
Copper (Alternative Colorimetric Method)	23 – 27
Copper (Alternative Flame Atomic Absorption Method)	28
Manganese (Colorimetric Method)	29 – 33
Manganese (Alternative Flame Atomic Absorption Method)	34
Iron (Colorimetric Method)	35 – 39
Acetone Extract	40 – 41
Rubber Hydrocarbon	42 – 43
Nitrogen	44 – 45

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

1.4 *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.*

1.5 *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

D297 Test Methods for Rubber Products—Chemical Analysis

¹ These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and are the direct responsibility of Subcommittee D11.22 on Natural Rubber.

Current edition approved June 1, 2020. Published June 2020. Originally approved in 1953. Last previous edition approved in 2015 as D1278 – 91a (2015). DOI: 10.1520/D1278-91AR20.

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.

D1193 Specification for Reagent Water

D1485 Practice for Rubber from Natural Sources—Sampling and Sample Preparation

D3533 Test Method for Rubber—Nitrogen Content (Withdrawn 2006)³

D4004 Test Methods for Rubber—Determination of Metal Content by Flame Atomic Absorption (AAS) Analysis

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

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E131 Terminology Relating to Molecular Spectroscopy

3. Significance and Use

3.1 These test methods are intended for quality control acceptance of natural rubber and may be used for referee purposes.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently higher purity to permit its use without lessening the accuracy of the determination.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification **D1193**.

5. Sampling

5.1 A lot of natural rubber shall be sampled, the sample prepared for test, and the acceptability of the lot determined from tests on the sample in accordance with Test Methods **D1485**.

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

VOLATILE MATTER

6. Procedure

6.1 Weigh a 10 to 12-g specimen of homogenized rubber to the nearest 1 mg and then dry it in a circulating-air oven at $100 \pm 5^\circ\text{C}$ to constant mass. If it is suspected that volatile hydrocarbon oils are present, they should be determined by heating the rubber in a circulating-air oven at $160 \pm 5^\circ\text{C}$ to constant weight. The drying is facilitated by cutting the rubber into 25-mm strips having a maximum width and thickness of 2.5 by 1.25 mm or by passing it through a laboratory mill with a clearance between rolls set at 0.5 ± 0.1 mm (0.020 ± 0.004 in.). Keep the dried specimen in a covered weighing vessel or in a desiccator after its removal from the oven until it is ready to be weighed.

7. Calculation

7.1 Calculate the percentage of volatile matter as follows:

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

where:

V = percentage of volatile matter,
 A = mass of piece taken from bale,
 B = mass of piece after homogenizing,
 C = mass of specimen before oven drying, and
 D = mass of specimen after oven drying.

8. Precision and Bias

8.1 Task groups for the purpose of obtaining precision and bias data in accordance with Practice D4483 are being organized and precision and bias statements will be added to this test method when available.

DIRT

9. Apparatus

9.1 *Sieve*—A45- μm (No. 325) sieve conforming to Specification E11.

10. Reagents

10.1 *Petroleum, Light*, boiling between 60 and 80°C .

10.2 *Rubber Peptizing Agent*.

NOTE 1—The peptizing agent selection is not critical but, where one is used that contains inert mineral filler, the peptizer should be added to the rubber solvent and filtered⁵ prior to addition of the rubber.

10.3 *Rubber Solvent, Xylene, or a Hydrocarbon Solvent*, with a distillation range within 135 to 220°C .

11. Procedure

11.1 Weigh a 10 to 12-g specimen of homogenized rubber to the nearest 0.1 g and cut into pieces having a maximum dimension of less than 3 mm. Place the pieces in a 250-cm³

⁵ The sole source of supply of the apparatus (Number 40 Watman filter paper) known to the committee at this time is Fischer Scientific, 1600 W. Glenlake Ave., Itasca, IL 60143. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

conical flask and cover with 150 cm³ of rubber solvent containing about 0.5 g of peptizing agent. Heat the mixture and maintain it at a temperature of 125 to 130°C (Note 2) until dissolution is complete (about 3 h).

NOTE 2—Overheating or boiling may cause gelling or charring. Infrared heating lamps and magnetic stirring are aids that will induce rubber solution.

11.2 Pour the hot solution through a 45- μm (No. 325) sieve previously weighed to the nearest 0.1 mg. Rinse the flask three times with about 25 cm³ of hot rubber solvent and pour the rinsings through the sieve. Transfer any dirt remaining in the flask to the sieve by means of a jet of light petroleum and wash the dirt on the sieve until free of rubber solution. Dry the sieve and contents at $100 \pm 5^\circ\text{C}$ and weigh to the nearest 0.1 mg.

12. Calculation

12.1 Calculate the dirt content as follows:

$$D = [(C - B)/A] \times 100 \quad (2)$$

where:

D = percentage of dirt,
 A = mass of the specimen,
 B = mass of the clean, dry sieve, and
 C = mass of the sieve plus dirt.

13. Precision and Bias

13.1 Task groups for the purpose of obtaining precision and bias data in accordance with Practice D4483 are being organized and precision and bias statements will be added to this test method when available.

ASH

14. Apparatus

14.1 *Crucible*—An unetched porcelain crucible having a capacity of 50 cm³. If copper is subsequently to be determined, a smooth unetched silica crucible is preferred, but a Vycor crucible or an ignited, acid-washed unetched No. 2 Coors porcelain crucible may be used.

NOTE 3—In cases of dispute where the greatest accuracy is required, use a new, smooth silica crucible each time the test is run.

14.2 *Muffle Furnace*, with temperature indicator and control.

14.3 *Filter Paper, Ashless*, about 150 mm in diameter.

15. Procedure

15.1 Weigh a 5 to 6-g specimen of homogenized rubber to the nearest 1 mg and place it in a crucible previously ignited and weighed to the nearest 0.1 mg. Place the crucible and its contents in a furnace controlled at a temperature of $550 \pm 25^\circ\text{C}$ until free from carbon (Note 4). When ashing is complete, cool the crucible in a desiccator and then weigh it to the nearest 0.1 mg.

NOTE 4—The rubber may be charred over a small flame or on a hot plate before it is placed in the furnace. When the rubber is not previously charred before placing it in the furnace, the crucibles shall be placed on a suitable tray to permit placing them in the furnace simultaneously, and the door of the furnace shall then be kept closed for at least 1 h while