Designation: D297 – 21

Standard Test Methods for Rubber Products—Chemical Analysis[1]

This standard is issued under the fixed designation D297; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover the qualitative and quantitative analysis of the composition of rubber products of the "R" family (see 3.1). Many of these test methods may be applied to the analysis of natural and synthetic crude rubbers.

1.1.1 *Part A* consists of general test methods for use in the determination of some or all of the major constituents of a rubber product.

1.1.2 *Part B* covers the determination of specific polymers present in a rubber product.

1.1.3 The test methods appear in the following order:

Part A. General Test Methods:	Sections
Rubber Polymer Content by the Indirect Method	11 – 13
Determinations and Report for the General Method	14 and 15
Density	16
Extract Analysis	17 – 26
Sulfur Analysis	27.1 – 33
Fillers Analysis	34 – 40
Ash Analysis	41 – 51
Part B. Determination of Rubber Polymers	52 – 58

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

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.* Specific precautionary or warning statements are given in 31.4.5, 31.6, 37.4.2, 38.4.2, 45.1.3, 53.2.3.5, 54.4.2, 54.6, 56.5.3, and 57.7.3; and X1.3.3 and X2.4.1.6.

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]

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D982 Test Method for Organic Nitrogen in Paper and Paperboard

D1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis (Withdrawn 1996)[3]

D1418 Practice for Rubber and Rubber Latices—Nomenclature

D1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)

D3040 Practice for Preparing Precision Statements for Standards Related to Rubber and Rubber Testing (Withdrawn 1987)[3]

D3156 Practice for Rubber—Chromatographic Analysis of Antidegradants (Antioxidants, Antiozonants and Stabilizers)

D3452 Practice for Rubber—Identification by Pyrolysis-Gas Chromatography

D3677 Test Methods for Rubber—Identification by Infrared Spectrophotometry

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

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

E442 Test Method for Chlorine, Bromine, or Iodine in Organic Compounds by Oxygen Flask Combustion (Withdrawn 1996)[3]

E443 Test Method for Sulfur in Organic Compounds by Oxygen Flask Combustion (Withdrawn 1996)[3]

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[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 D11.11 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.

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

3. Terminology

3.1 *Definitions*—The nomenclature and abbreviations used for natural and synthetic rubbers are in accordance with Practice D1418.

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 high 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 distilled water or water of equal purity.

5. Concentration of Reagents

5.1 *Concentrated Acids and Ammonium Hydroxide*—When acids and ammonium hydroxide are specified by name or chemical formula only, it is understood that concentrated reagents of the following densities or concentrations are intended:

	Density, Mg/m^3
Acetic acid, $HC_2H_3O_2$ (99.7 %)	1.05
Formic acid, HCOOH	1.22
Hydrochloric acid, HCl	1.19
Hydrofluoric acid, HF (49 %)	1.16
Nitric acid, HNO_3	1.42
Phosphoric acid, H_3PO_4 (85 %)	1.70
Sulfuric acid, H_2SO_4	1.84
Ammonium hydroxide, NH_4OH	0.90

The desired densities or concentrations of all other concentrated acids are stated whenever they are specified.

5.2 *Diluted Acids and Ammonium Hydroxide*—Concentrations of diluted acids and ammonium hydroxide, except when standardized, are specified as a ratio stating the number of volumes of the concentrated reagent to be added to a given number of volumes of water, as in the following example: HCl (1 + 9) means 1 volume of concentrated HCl (density 1.19) mixed with 9 volumes of water. Acids shall be added to water slowly, with stirring.

5.3 *Standard Solutions*—Concentrations of standard solutions are expressed as normalities or as volume of solution that reacts with or contains a given mass of material being used or determined, for example: 0.1 *N* $Na_2S_2O_3$ solution, or $CuSO_4$ solution (1 cm^3 = 0.001 g Cu).

5.4 *Nonstandardized Solutions*—Concentrations of nonstandardized solutions prepared by dissolving a given mass in a solvent are specified in grams of the reagent (as weighted out)/dm^3 of solution, and it is understood that water is the solvent unless otherwise specified, for example: NaOH (10 g/dm^3) means 10 g of NaOH dissolved in water and diluted with water to 1 dm^3 (Note 1). In the case of certain reagents the concentration may be specified as a percentage by mass, for example: ethanol (50 %) means a solution containing 50 g of ethanol per 100 g of solution. Other nonstandardized solutions may be specified by name only, and the concentration of such solutions will be governed by the instructions for their preparation.

NOTE 1—Whenever a hydrated salt is used in the preparation of a reagent (for example, $BaCl_2 \cdot 2H_2O$) the preparation of the reagent is described in detail. When an anhydrous salt is used in preparing a simple aqueous solution the reagent is listed by title only and details of the preparation are not given.

PART A. GENERAL TEST METHODS

6. Scope and Application

6.1 The general test methods described cover the analysis of many types of rubber products to determine the amount and type of nonrubber constituents and to calculate indirectly from these data the amount of rubber constituent.

6.2 The applications and limitations of the test methods to analysis of specific types of rubber products are given in the scopes of the various test methods. Application to types of rubber products not specified in the scope of a particular test method shall be verified by application to a control of known and similar composition.

6.3 Special test methods for analysis are given for rubber products containing glue, free carbon, antimony, lead, mineral oil, waxy hydrocarbons, and barium carbonate.

6.4 For the determination of the amount of a rubber polymer present in a rubber product, an indirect test method is given by which the nonrubber constituents are determined individually or in groups, and the rubber polymer content is determined by difference (Sections 11 – 13). If, in using this test method, fillers are determined by the ashing test method (Section 35 or 36), satisfactory results will be obtained, except where there are found to be present decomposable compounding ingredients such as carbonates that decompose at 550°C, clay, asbestine, talc, hydrated silica, antimony sulfide, halogen-containing components, and silicone polymers. No test method of filler determination herein described will give accurate results in the presence of clay, silica, talc, or any other hydrated

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[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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

filler unless a correction can be made for losses of water of hydration on ashing. This correction can be made only if the nature and quantity of these fillers are known. The indirect test method will not give accurate results in the presence of halogen-containing components or silicone rubber. In the presence of antimony sulfide or carbonates decomposing at 550°C, but in the absence of the above interfering constituents, approximate correction can be made by means of determination of total antimony (Section 50) or of the metal associated with the decomposable carbonate (usually calcium, Section 45) or (Section 49) and calculation of the original composition of the compounding ingredient from these data.

6.5 If factice or high percentages of mineral rubber are present, no accurate test method is known for determination of rubber content or for complete analysis of the rubber product.

6.6 For the determination of the rubber content of hard rubber products, no accurate test method is described herein if fillers decomposable at 550°C are present.

7. Blank Determinations

7.1 Blanks shall be run on all determinations to check the purity of the materials used and deductions shall be made accordingly.

8. Check Analyses

8.1 Duplicate determinations shall be made and shall check within the limits specified in the test method, when these are stated.

9. Preparation of Samples

9.1 Before preparing a sample for analysis, the analyst shall, by inspection, assure himself that it has not been contaminated. The sample to be analyzed shall be selected by taking pieces from various parts of the original sample and separating them from foreign matter. Because of the variety of rubber products to which this test method can be applied, no single procedure for reducing the sample to the required fineness is applicable to all samples. Therefore, several alternative procedures for this purpose are described in 9.2 to 9.7. The analyst is expected to select the one most suitable to the sample that he is analyzing and the equipment available.

9.2 For vulcanized soft rubber, unvulcanized rubber, crude rubber, and many samples of reclaimed rubber, it is preferable to mix the sample and grind it by passing it two or three times through a clean, cold, laboratory rubber mill. The rubber will come from the mill in the form of a coarse powder or a rough sheet. If the product is in the form of a sheet, the adjustment of the mill shall be such that the thickness of the final sheet is no greater than 0.5 mm. If the sample is sticky, it shall be rolled in a liner material that will not adhere to or contaminate the sample. If the milled sample is a powder, it shall be transferred to a No. 14 (1.40-mm) sieve[5] and rubbed through the sieve. Grinding shall be continued until the entire sample passes through the sieve.

9.3 In the absence of milling machinery, the sample may be prepared by cutting it with scissors so that it will pass a No. 14 (1.40-mm) sieve.[6,7] The sample may be cut into long strips that are fine enough to pass freely through the sieve and the strips fed through by hand, or the sample may be cut into small fragments and shaken through the sieve. The cutting shall be continued until the entire sample passes through the sieve. If necessary, to prevent sticking, different fragments of the sieved sample may be segregated by wrapping in a liner material that will not adhere to or contaminate the sample.

9.4 Certain very glutinous samples may be prepared for extraction analysis as follows: Place a weighed 2-g sample of the material between two pieces of ashless filter paper that has been extracted in accordance with Section 21. The papers should be approximately 500 by 100 mm (20 by 4 in.) and the sample should be placed near one end. Flatten the sample and spread it throughout the length of the filter paper by passing the "sandwich" lengthwise, through a cold, closely set, even-speed rubber calender. The gross thickness of the resulting sheet should not be greater than 1.0 mm. If a rubber calender is not available, a similar sheet may be obtained by placing the sample in a hydraulic press or a vise. In the latter case, the sample may be roughly spread by hand throughout the length of the filter paper and pressure applied to small areas at a time until the whole sample has been flattened.

9.5 Samples of rubberized cloth, whose overall thickness is no greater than 1.0 mm, may be prepared for analysis by cutting them into pieces 1.5 mm square and then mixing well. If the fabric is easily removed, it should be separated, unless an analysis of the whole cloth is desired.

9.6 Samples of rubber cements shall be evaporated to dryness in a vacuum oven at a temperature not higher than 30°C. The residue may then be analyzed as an unvulcanized sample. A separate sample of the cement shall be distilled under reduced pressure if examination of the solvent is desired.

9.7 Samples of hard rubber shall be reduced to powder form by filing, cleaned with a magnet, and sieved through a No. 30 (600-µm) sieve.[6] Residue retained on this sieve shall be reduced until the entire sample passes through the sieve.

9.8 Certain raw rubber samples may need to be re-shaped, especially when take from a solid bale or rubber crumbs, to approximately 2 mm sheet form for density measurement. In such cases, the raw rubber sample may be pressed between two heat resistant barrier films in a hydraulic press at 100°C for 5 min at a minimum pressure of 50 psi. Condition the molded rubber sheets at 23 ± 2°C for at least 1 h and remove the heat resistant film and subsequently conduct the density measurement.

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[5] Detailed requirements for these sieves are given in Specification E11.

[6] The sole source of supply of compressed volume densimeters known to the committee at this time is C. W. Brabender Instruments, Inc., 50 E. Wesley St., South Hackensack, NJ 07606.

[7] 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.