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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Rubber—Nitrogen Content¹

This standard is issued under the fixed designation D 3533; 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.

^{ε1} NOTE—Keywords were added and editorial changes were made throughout in June 1995.

1. Scope

1.1 This method outlines two procedures for the determination of total nitrogen in natural and synthetic rubbers and latexes, using variants of the Kjeldahl process.

1.2 It is applicable to raw rubbers, cured or uncured compounds, and finished articles.

1.3 Procedure A, the referee method, is a macro procedure. Procedure B, the alternative method, is a semimicro procedure using the same reagent as in Procedure A.

1.4 In the absence of other nitrogen-containing materials, the method can be used for the estimation of the NBR content of NBR rubbers and rubber products.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *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. Specific cautions are given in 8.2.1 and 9.2.1.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1076 Specification for Rubber—Concentrated, Ammonia Preserved, Creamed, and Centrifuged Natural Latex²
- D 3040 Practice for Preparing Precision Statements for Standards Related to Rubber and Rubber Testing³
- D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²
- E 147 Specification for Apparatus for Microdetermination of Nitrogen by Kjeldahl Method⁴

NOTE 1—The specific dated edition of Practice D 3040 that prevails in this method is referenced in the Precision section.

3. Summary of Method

3.1 The procedures used are modifications of the Kjeldahl

method using sulfuric acid, potassium sulfate, and catalytic amounts of copper sulfate and selenium in the digestion mixture. The distillate from a strongly alkaline solution of the digested sample is caught in boric acid solution and titrated with standard acid or in excess standard acid and back titrated with standard base.

4. Significance and Use

4.1 The determination of nitrogen in natural rubber is usually carried out in order to arrive at an estimate of the protein content. Minor amounts of non-proteinous nitrogen-containing constituents are also present, however, and in the dry solids prepared from natural rubber latex, these materials can make a substantial contribution to the total nitrogen content.

4.2 In the absence of other nitrogen-containing materials, the method can be used for the estimation of the NBR content of NBR rubbers and rubber products.

4.3 In the absence of other nitrogen-containing materials and if the acrylonitrile content of the NBR rubber is known, the method can be used to estimate the amount of NBR rubber in mixtures.

4.4 This method may be used for quality control, for purchase and raw material uses, for processing studies, and for research and development.

5. Apparatus

5.1 Kjeldahl Digestion and Distillation Apparatus:

5.2 For Procedure A—Macro-Kjeldahl apparatus, preferably having ground-glass joints and including an 800-cm³ Kjeldahl flask, with electrical heating equipment for the digestion apparatus.

5.3 For Procedure B—Micro-Kjeldahl digestion and distillation apparatus in accordance with Specification E 147, or semimicro Kjeldahl digestion and distillation apparatus⁵ in which the digestion and distillation are carried out in the same flask (30 to 100-cm³ Kjeldahl flask that may be attached to the distillation apparatus by means of a standard-taper joint).

6. Reagents

6.1 *Boric Acid Solution* (40 g/dm³)—Dissolve 40 g of boric

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

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² Annual Book of ASTM Standards, Vol 09.01.

³ Discontinued—see 1986 Annual Book of ASTM Standards, Vols 09.01 and 09.02.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Suitable semimicro apparatus may be obtained from the American Instrument Co., 8030 Georgia Ave., Silver Spring, Md., 20910, and from the Scientific Glass Apparatus Co., 737 Broad St., Bloomfield, NJ 07003.