



## Standard Test Method for Residual Acrylonitrile Monomer Styrene-Acrylonitrile Copolymers and Nitrile Rubber by Headspace Gas Chromatography<sup>1</sup>

This standard is issued under the fixed designation D 4322; 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.

<sup>ε1</sup> NOTE—Several sections were changed editorially in March 2001.

### 1. Scope

1.1 This test method is suitable for determining the residual acrylonitrile (RAN) content of styrene-acrylonitrile (SAN) copolymer, rubber-modified acrylonitrile-butadiene-styrene (ABS) resins, and nitrile rubber (NBR).

1.2 *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 precautionary statements are given in Section 9.

NOTE 1—Although the packed column option of this test method and ISO 4581:1994 (E) differ in some details, data obtained using either test method should be technically equivalent. There is no equivalent ISO standard for the capillary column option of this test method.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 4526 Practice for Determination of Volatiles in Polymers by Headspace Gas Chromatography<sup>2</sup>
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>3</sup>
- IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI): The Modern Metric System<sup>4</sup>

### 3. Terminology

3.1 Units and Symbols used in this test method are those recommended in IEEE/ASTM SI-10.

#### 3.2 Abbreviations: Abbreviations:

3.2.1 AN—acrylonitrile.

3.2.2 RAN—residual acrylonitrile.

3.2.3 SAN—styrene-acrylonitrile copolymer.

3.2.4 ABS—acrylonitrile-butadiene-styrene copolymer.

3.2.5 NBR—butadiene-acrylonitrile rubber.

3.2.6 DMAC—*N,N*-dimethylacetamide.

3.2.7 PN—propionitrile (internal standard).

3.2.8 PC—propylene carbonate.

3.2.9 ppm—μg RAN/g polymer (parts per million).

### 4. Summary of Test Method

4.1 A dispersion of the polymer in a suitable solvent is prepared in a headspace vial and sealed. The vial is thermally equilibrated in a constant temperature bath.

4.2 After equilibrium, a given portion of the sample headspace is injected into a gas chromatographic column packed with porous polymer beads or a capillary column coated with an appropriate liquid phase. Sample injection is achieved using available commercial automatic equipment or a manual syringe injection technique. Passing through the column in a stream of carrier gas, acrylonitrile is separated from other components that may be present. The response of acrylonitrile is measured by a nitrogen-specific detector for packed column analysis or a flame ionization detector for capillary column analysis and this signal is recorded to indicate the retention time and relative concentration of acrylonitrile.

### 5. Significance and Use

5.1 For various reasons one may wish to measure the amount of unreacted or residual acrylonitrile monomer in styrene-acrylonitrile copolymers, nitrile rubbers, or ABS terpolymers.

5.2 Under optimum conditions, the lowest level of detection of AN in SAN or ABS copolymers and NBR rubbers is approximately 0.5 ppm for the packed column test method and 3 ppm for the capillary test method.

### 6. Interferences

6.1 The nitrogen-specific detector eliminates interference from all but compounds containing nitrogen or phosphorus. Any such material eluting at or near the AN or PN retention

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This revision includes the addition of an ISO equivalency statement and a suitable capillary column headspace GC test method.

<sup>2</sup> Annual Book of ASTM Standards, Vol 08.03.

<sup>3</sup> Annual Book of ASTM Standards, Vol 14.02.

<sup>4</sup> Annual Book of ASTM Standards, Vol 14.04.

