



Standard Test Method for Determination of Residual Acrylonitrile Monomer in Styrene- Acrylonitrile Copolymer Resins and Nitrile-Butadiene Rubber by Headspace-Capillary Gas Chromatography (HS- CGC)¹

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1. Scope

1.1 This test method covers the determination of the residual acrylonitrile (RAN) content in nitrile-butadiene rubbers (NBR), styrene-acrylonitrile (SAN) copolymers, and rubber-modified acrylonitrile-butadiene-styrene (ABS) resins.

1.2 Any components that can generate acrylonitrile in the headspace procedure will constitute an interference. The presence of 3-hydroxypropionitrile in latices limits this procedure to dry rubbers and resins.

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given Note 4 and Note 5.

NOTE 1—There is no current ISO equivalent test method for residual acrylonitrile (RAN) determinations.

2. Referenced Documents

2.1 ASTM Standards:

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method²

3. Summary of Test Method

3.1 Two dispersions (in o-dichlorobenzene) are prepared and sealed in headspace vials for each polymer; one vial contains the polymer in solvent while the second vial contains the polymer, solvent, plus a known standard addition of acrylonitrile (AN). Both vials are agitated for a specified time under ambient conditions. After agitation, the vials are thermally equilibrated in a constant-temperature bath.

3.2 After completion of the timed equilibration, an aliquot of the heated headspace gas from each vial is injected into a capillary gas-chromatographic column. An automated injection system is used to effect the transfer. The capillary column will provide the chromatographic resolution necessary to isolate the

AN from other volatiles that may be present. The AN response is measured using a nitrogen-specific detector (NPD). The raw data signal is converted to a relative RAN concentration through a standard addition calculation.

4. Significance and Use

4.1 A measurement of the residual acrylonitrile in nitrile rubbers (NBR), styrene-acrylonitrile copolymers or ABS terpolymers will determine the polymer's suitability for various applications.

4.2 Under optimum conditions, the minimum level of detection of RAN in NBR, SAN, or ABS terpolymers is approximately 50 ppb.

5. Apparatus

5.1 *Gas Chromatograph*, equipped with a nitrogen-phosphorus specific detector, backflush valve (see Fig. 1), split injector, and capable of accepting megabore (0.53 mm inside diameter) fused silica capillary columns. Detector make-up gas is required.

NOTE 2—The use of a backflush configuration will provide for operating advantages, but its use is optional. Chlorinated solvents quench the alkali bead in a nitrogen-phosphorous detector, producing a loss of signal. While the bead (signal) will recover as the solvent evacuates the detector, repeated quenching during a multi-run sequence may produce instabilities in the signal (and precision) over the sequence period.

5.2 *Automated Headspace Sampler*, should have a thermostatted sample tray capable of 90°C heating with constant heating times. Automated sampling of the headspace gas in the sample vials via a heated, constant-volume sample loop or pressure balancing sampling mechanism is required. Sampling to the gas chromatograph should be via a heated transfer line of minimum dead volume.

5.3 *Fused Silica Porous-Layer-Open-Tubular (PLOT) Capillary Column*³, GS-Q, 30 m × 0.53 mm inside diameter.

NOTE 3—The column should be cut so as to have a 3 m section for the pre-column (Column 1) and a 27-m section for the analytical column (Column 2).

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

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² *Annual Book of ASTM Standards*, Vol 14.02.

³ J & W Scientific Catalog Number 115-3432, available from J & W Scientific, 91 Blue Ravine Road, Folsom, CA 95630-4714, or equivalent, has been found suitable for this purpose.