



# Standard Test Methods for Determining the 24-Hour Gas (AIR) Space Acetaldehyde Content of Freshly Blown PET Bottles<sup>1</sup>

This standard is issued under the fixed designation D 4509; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These test methods cover the 24-h gas-space acetaldehyde (AA) content of freshly blown polyethylene terephthalate (PET) bottles.

1.2 These test methods, containing internal or external standard calibration, are applicable to all PET bottles.

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

NOTE 1—There is no similar or equivalent ISO standard.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 883 Terminology Relating to Plastics<sup>2</sup>

D 1193 Specification for Reagent Water<sup>3</sup>

D 1600 Terminology for Abbreviated Terms Relating to Plastics<sup>2</sup>

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods<sup>4</sup>

E 355 Practice for Gas Chromatography Terms and Relationships<sup>4</sup>

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)<sup>4</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>4</sup>

## 3. Terminology

3.1 The gas chromatographic terms employed in these test methods are those recommended by Practice E 355.

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D-20 on Plastics and are the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.03).

This standard has been reviewed and the following items added: an ISO equivalency statement; a material specification reference statement; and a Keywords Section.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 08.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

3.2 Units, symbols, and abbreviations used in these test methods are those recommended by Practice E 380.

3.3 For further information on abbreviation, PET, refer to Terminology D 1600.

3.4 Additional terms relative to plastics are explained in Terminology D 883.

## 4. Summary of Test Methods

4.1 A molded preform (any size) is blown into a bottle that is purged with nitrogen, capped, and aged. After 24 h, a headspace gas sample is taken from the bottle, and the gas sample is injected into a gas chromatograph for comparison with known external standards (Sections 9-11) or internal standards (Sections 12-14).

## 5. Significance and Use

5.1 Before proceeding with these test methods, reference should be made to the specification of the material being tested. Any test specimens preparation, conditioning, dimensions, and testing parameters covered in the materials specification, shall take precedence over those mentioned in these test methods. If there is no material specification, then the default conditions apply.

5.2 Acetaldehyde is a decomposition product of the polycondensation reaction and is a by-product of melt processing of polyethylene terephthalate (PET). It adds undesirable flavor to some beverages.

5.3 The level of acetaldehyde in PET blown containers is monitored by these test methods.

## 6. Apparatus

6.1 *Gas Chromatograph*, with flame ionization detector, equipped with a six-port gas-sampling valve and a 5-mL gas-sampling loop for sampling the headspace of the beverage bottle, as shown in Fig. 1.

6.2 Any suitable system of peak integration can be used for measurement of the acetaldehyde.

6.3 *Gas Sampling Apparatus* as shown in Fig. 1, including:

6.3.1 *Trap*, 25.4 mm (1 in.) outside diameter by 0.305 m (1 ft) long containing 1 part silica-gel absorbant and 1 part 5A molecular-sieve packing.

6.3.2 *Pressure-Vacuum Gage*, 0–30 psig and 30 in. Hg.

6.4 *Gastight GC Syringe*, 10  $\mu$ L.

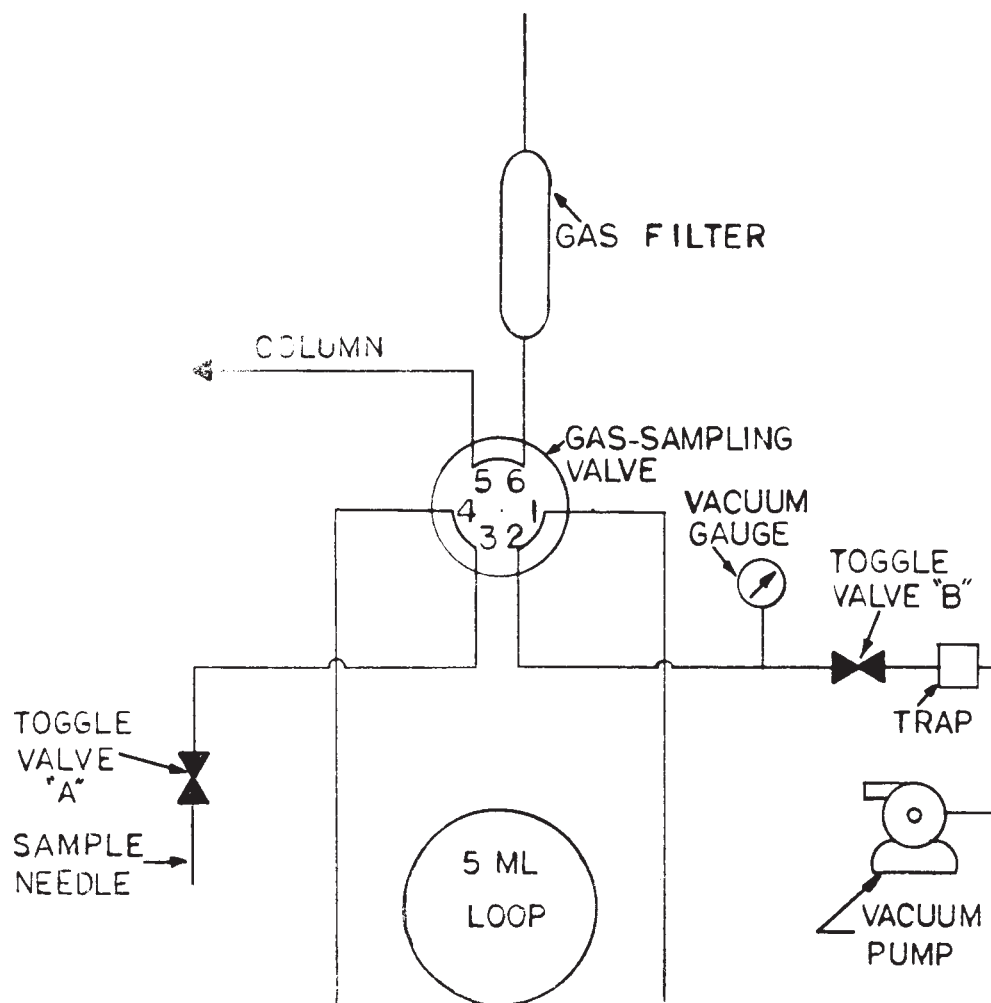


FIG. 1 Schematic Drawing of Gas-Sampling Apparatus

6.5 *Gastight GC Syringe*, 0 to 5.0 cc (internal standard method only).

## 7. Reagents and Materials

7.1 A 2.0 m by 6 mm outside diameter, 4-mm inside diameter glass column, packed with Porapak Q or QS (100 to 120 mesh) porous polymer or Tenax GC porous polymer (60 to 80 mesh), packed into a 3.2 or 3.18 mm ( $\frac{1}{8}$  in.) outside diameter by 3.66 m (12 ft) long stainless-steel tube.<sup>5</sup>

7.2 *Acetaldehyde Standard Solution*, prepared and analyzed by the procedure described in Annex A1.

7.3 *Acetaldehyde/Propionaldehyde Standard Solution*, prepared by Annex A2 (internal standard method only).

7.4 *Phenolic Polymer Bottle Cap*, 28 mm outside diameter, containing a 6.35-mm ( $\frac{1}{4}$ -in.) hole drilled in the top and snugly fitted with a seal cut from 1.59-mm ( $\frac{1}{16}$ -in.) butyl rubber and lined with a liner cut from 0.08-mm (0.003-in.) fluoropolymer film to prevent absorption of acetaldehyde into the butyl rubber seal.

NOTE 2—The phenolic caps and the butyl rubber seals may be reused after the test, but the fluoropolymer liner must be discarded after it is punctured.

7.5 *Nitrogen* (oxygen-free) or helium (GC).

7.6 *Hydrogen*, prepurified or zero-gas.

7.7 *Air*, breathing, water-pumped.

7.8 *Acetaldehyde*, reagent-grade (internal standard method only).

7.9 *Propionaldehyde*, reagent-grade (internal standard method only).

7.9.1 *High-purity 1-propanol*, distilled in glass (internal standard method only).

7.10 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>6</sup>

<sup>5</sup> Poropak is a registered trademark of Waters Associates, Inc., Framingham, MA. Tenax is a registered trademark of Enka Glanzstoff B.V. of Arnhem, Netherlands. Both polymers are available from laboratory supply houses.

<sup>6</sup> "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."