



## Standard Test Method for Determination of Retained Blowing Agent in Extruded Polystyrene Foam<sup>1</sup>

This standard is issued under the fixed designation D 7132; 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.

### 1. Scope

1.1 This test method covers the determination of retained blowing agent in extruded polystyrene foam.

1.2 This test method applies to organic blowing agents which lend themselves to a convenient analysis by gas chromatography. The method is not applicable to blowing agents such as nitrogen, carbon dioxide, or water.

1.3 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 to determine the applicability of regulatory limitations prior to use.*

### 2. Summary of Test Method

2.1 Polystyrene foams are made with a variety of blowing agents such as hydrocarbons, hydrofluorocarbons, ethers, ketones, and other volatile organic chemicals. A fraction of the blowing agent used in the manufacture of foam is retained in the product, some residing in the cell walls and some entrapped inside the cells. This test method is based on releasing the retained blowing agent by dissolving the polystyrene foam in a solvent, such as toluene, and then analyzing the solution for its components by gas chromatography. An internal standard is used as a reference analyte and calibration of the gas chromatograph (GC) with standard solutions allows conversion of the GC response to the amount of blowing agent retained in the foam matrix.

### 3. Interferences

3.1 The gas chromatogram may contain a signal(s) from the polymer matrix that interferes with the signal(s) from the blowing agent components. Test calibration solutions with and without the polymer matrix to rule out any interference. These calibration solutions shall contain about the same amount of dissolved resin as in the foam sample being analyzed.

### 4. Apparatus

4.1 Any GC equipped with a column suitable for resolving blowing agent components can be used. For non-polar blowing agents, a column such as HP-1 with 530-μm thick film and 30-m length can be used. For polar blowing agents, a column such as HP-17 with 530-μm thick film and 30-m length can be used. Typical operating parameters for the GC are:

Head pressure (kPa)	50
Initial oven temperature (°C)	50
Initial hold time (min)	3.5
Temperature program rate (°C/min)	25
Final temperature (°C)	150
Final hold time (min)	3
Injector temperature (°C)	250
Detector temperature (°C)	300
Carrier gas	He
Carrier gas flow rate (cm <sup>3</sup> /min)	30
Injection sample size (μL)	0.2

NOTE 1—An autosampler is not recommended as the transfer of a solution containing volatile components can lead to loss of the volatile analyte.

4.2 Analytical balance with at least 150 g full scale and 0.1 mg resolution.

4.3 Instrument grade air supplied at  $515 \pm 40$  kPa, helium at  $450 \pm 40$  kPa, and hydrogen at  $380 \pm 40$  kPa.

4.4 100-cm<sup>3</sup> volumetric flasks.

4.5 1-cm<sup>3</sup> syringe with Chaney Adapter, and 10-cm<sup>3</sup> gas tight syringe with Chaney Adapter.

4.6 1-μL syringe.

4.7 I-Chem Tall 200 series wide mouth 125-cm<sup>3</sup> sample bottles with caps and teflon/silicon septa.

4.8 30-cm<sup>3</sup> serum vials with crimp on teflon/silicon septa.

4.9 Rubber sleeve stoppers for volumetric flask.

4.10 Septa for GC.

4.11 Ice water bath.

4.12 Weights to keep sample bottle submerged.

4.13 Refrigerator for sample storage.

4.14 Insulated mailing containers with ice packs for shipping samples.

### 5. Reagents and Materials

5.1 Certified grade toluene with minimum purity of 99.8 mol%.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods. Current edition approved July 1, 2005. Published August 2005.

5.2 Certified grade normal heptane with minimum purity of 99.8 mol%.

5.3 Instrument grade standards for blowing agents to be analyzed.

## 6. Hazards

6.1 Some blowing agents are compressed liquids or gases at room temperature. Appropriate care shall be taken when preparing calibration solutions from such blowing agents.

## 7. Sampling, Test Specimens, and Test Units

7.1 Tare an I-Chem sample bottle with cap and septum. Cut a piece of foam using a sharp knife, transfer it immediately to the sample bottle, and seal with cap and septum. Record the sample weight.

NOTE 2—The cutting action produces open cells and the blowing agent trapped in these cells is lost immediately. The sample size and shape shall be such as to minimize the ratio of area of cut surfaces to total surface area. The foam sample shall also be of dimensions such that it can easily be placed in the sample bottle without causing any break or tear in the sample. To establish the optimum sample size, make some preliminary measurements as a function of sample size/shape until a constant value of the retained blowing agent is obtained.

7.2 Typically, the sample weight is 1 g.

7.3 Store the samples at about 4°C, and analyze within a week.

NOTE 3—When samples need to be shipped, use an insulated container equipped with frozen ice packs. Shipping should be by overnight service.

## 8. Internal Standard Preparation

8.1 Affix a Chaney adapter to the 10-cm<sup>3</sup> syringe and calibrate the syringe in accordance with steps 8.1.1-8.1.3.

8.1.1 Measure and record the weight of 100 cm<sup>3</sup> of toluene in a 100-cm<sup>3</sup> volumetric flask.

8.1.2 Fill the 10-cm<sup>3</sup> syringe from the flask using the Chaney adapter for volume control.

8.1.3 Inject the toluene into a pre-weighed sample bottle. Weigh the bottle. The weight shall be 1/10 of the weight obtained in step 8.1.1 within 0.01 g.

8.2 Add about 90 cm<sup>3</sup> of toluene to another 100-cm<sup>3</sup> volumetric flask. Cap the flask with an unused rubber sleeve stopper. Remove any static and dry the outside of flask with an ionized air blower.

8.3 Place the flask on the balance pan, allow it to stabilize, and tare the balance.

NOTE 4—The flask shall not cause the balance to respond before actually touching the balance pan.

8.4 Inject 0.2 to 0.4 cm<sup>3</sup> heptane into the flask and weigh to the nearest 0.1 mg ( $W_{istd}$ ).

8.5 Dilute to the mark with toluene. It is acceptable to retain this internal standard (ISTD) solution for 24 hours.

## 9. Calibration

9.1 Tare an empty 30-cm<sup>3</sup> serum vial (with a septa and seal).

9.2 Fill the serum vial with toluene to about 90 % capacity. Affix the septa and seal. Obtain the weight of the toluene to 0.1 mg ( $W_l$ ).

9.3 Tare the balance with the vial containing the toluene. Inject 1 to 2 g of blowing agent into the vial. Weigh the vial and record the weight to the nearest 0.1 mg ( $W_2$ ). This solution may be retained for eight hours.

NOTE 5—See Annex A1 for injecting volatile or gaseous blowing agents.

9.4 Using the calibrated syringe and the technique shown in Fig. 1, withdraw 10.0 cm<sup>3</sup> of the ISTD solution and inject into a tightly capped empty sample bottle.

9.5 Place the sample bottle containing the ISTD on the balance and tare the bottle.

9.6 Inject 0.2 to 1.0 cm<sup>3</sup> of the solution prepared in step 9.3 into the tared sample bottle and record weight of the solution added ( $W_3$ ).

9.7 Place the bottle in the ice bath. Place a weight on the bottle so it will remain upright. Be sure that the water level is even with the shoulder of the bottle.

9.8 The sample shall remain in the water bath for a minimum of one hour to equilibrate. This calibration solution may be retained for eight hours.

9.9 The amount of internal standard ( $W_i$ ) in the calibration solution is  $W_{istd}/10$  and the amount of blowing agent ( $W_{st}$ ) in the calibration solution is:

$$W_{st} = \frac{W_2 \times W_3}{W_1 + W_2} \quad (1)$$

9.10 Inject 0.2 µL of the calibration solution into the GC and calculate the response factor for the blowing agent using the following formula:

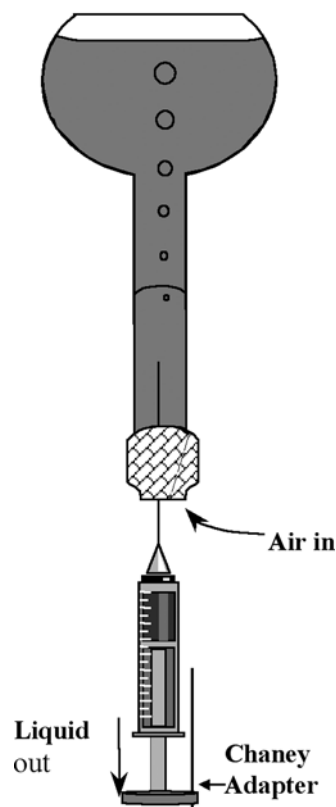


FIG. 1 Withdrawing Toluene Solution