



Standard Test Method for Determination of Retained Blowing Agent in Extruded Polystyrene Foam¹

This standard is issued under the fixed designation D7132; 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.*

NOTE 1—There is no known ISO equivalent to this standard.

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 has the potential to contain a signal(s) from the polymer matrix that interferes with the

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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 is suitable for use. For example, if separating non-polar blowing agents, a 100 % dimethylpolysiloxane with 530- μ m thick film and 30-m length is a reasonable selection. If separating polar blowing agents, a 50 % diphenyl/50 % dimethyl polysiloxane column with 530- μ m thick film and 30-m length is suitable. Typical operating parameters for the GC are:

Head pressure (kPa)	50
Initial oven temperature ($^{\circ}$ C)	50
Initial hold time (min)	3.5
Temperature program rate ($^{\circ}$ C/min)	25
Final temperature ($^{\circ}$ C)	150
Final hold time (min)	3
Injector temperature ($^{\circ}$ C)	250
Detector temperature ($^{\circ}$ C)	300
Carrier gas	He
Carrier gas flow rate (cm ³ /min)	30
Injection sample size (μ L)	0.2

NOTE 2—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 ± 40 kPa, helium at 450 ± 40 kPa, and hydrogen at 380 ± 40 kPa.

4.4 100-cm³ volumetric flasks.

4.5 1-cm³ syringe with Chaney Adapter, and 10-cm³ gas tight syringe with Chaney Adapter.

4.6 1- μ L syringe.

4.7 Tall, wide mouth 125-cm³ sample bottles with caps and teflon/silicon septa.

4.8 30-cm³ serum vials with crimp on teflon/silicon septa.

4.9 Rubber sleeve stoppers for volumetric flask.

4.10 Septa for GC.

*A Summary of Changes section appears at the end of this standard

- 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%.
- 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 Record the manufacture date of the foam and the date of material sampling.
- 7.2 Tare 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 3—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.

NOTE 4—If testing foam board, the sample dimensions and location such as surface or core should be reported.

- 7.3 Typically, the sample weight is 1 gram.
- 7.4 Store the samples at about 4°C, and analyze within a week.
- NOTE 5—When samples need to be shipped, use an insulated container equipped with frozen ice packs. Shipping should be by overnight service.
- 7.5 Record the date of the blowing agent analysis.

8. Internal Standard Preparation

- 8.1 Affix a Chaney adapter to the 10-cm³ 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 cm³ of toluene in a 100-cm³ volumetric flask.
 - 8.1.2 Fill the 10-cm³ 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 gram.

8.2 Add about 90 cm³ of toluene to another 100-cm³ 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 6—The flask shall not cause the balance to respond before actually touching the balance pan.

8.4 Inject 0.2 to 0.4 cm³ 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-cm³ 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 (*W₁*).
- 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 (*W₂*). This solution is not to be retained for more than eight hours.

NOTE 7—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 cm³ of the ISTD solution and inject into

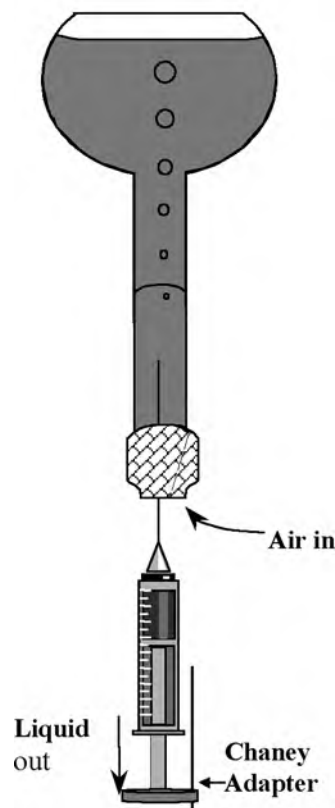


FIG. 1 Withdrawing Toluene Solution

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 cm³ 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 is not to be retained for more than 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 μ L of the calibration solution into the GC and calculate the response factor for the blowing agent using the following formula:

$$RF = \frac{W_i \times A_{st}}{W_{st} \times A_i} \quad (2)$$

where:

A_i = peak area of the internal standard

A_{st} = peak area of the blowing agent.

9.11 Repeat steps 9.4 – 9.10 with five different injection amounts of the solution from 9.3 (or Step 3 of Annex A1) to generate calibration solutions bracketing the expected amount of blowing agent in about 1 g of foam sample. Use these solutions to establish a five-point calibration curve.

9.12 Repeat steps 9.1 – 9.11 for each blowing agent.

10. Procedure

10.1 Using the calibrated syringe and the technique shown in Fig. 1, withdraw 10.0 cm³ of the ISTD solution, and inject into the bottle containing the foam sample. Shake the bottle to ensure that the entire sample is dissolved.

10.2 If the sample weighs more than 1.3 g, add toluene (not the ISTD solution) to reduce the concentration to about 10 % polystyrene.

10.3 Place the bottle in the ice water 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.

10.4 Allow the bottle to equilibrate in the water bath for a minimum of one hour, but not more than eight hours.

10.5 Inject 0.2 μ L of the solution into the GC. Measure the area of the peak(s) corresponding to each blowing agent and convert the areas into weight fraction(s) using the response factors determined in 9.10.

NOTE 8—Depending on the sensitivity of the GC detector and the accuracy desired, dissolve the foam sample using 10 to 25 cm³ of the ISTD solution in 10.1. It is imperative that if an amount of the ISTD solution other than 10 cm³ is used, then the same amount must be used in 9.4 and the calculation of W_i in 9.9 adjusted accordingly.

11. Daily Quality Assurance

11.1 Normally, no peaks in the ISTD or blowing agent peak region will be detected, if peaks greater than the detection limit are observed, another aliquot shall be injected. If contamination peaks are still observed, switch to a fresh bottle of toluene and monitor for peaks in the blowing agent region.

11.2 Prepare and analyze a calibration solution according to Section 9. The result(s) must agree within 10 % of the expected value or a new five-point calibration curve must be established.

12. Precision and Bias

12.1 Table 1 summarizes results of ten extruded polystyrene foam samples from the same population tested for their isobutane content. Each sample was injected twice. The results reported in Table 1 are typical of the method described here. Multiple injections of the same sample shall agree to ± 2.5 %. Multiple measurements from the same sample population shall agree to ± 5 %. The practical limit of quantitation of this method is 0.05 wt%.

13. Keywords

13.1 blowing agent retention; gas chromatography; polystyrene foam

TABLE 1 Test values and Repeatability Standard Deviation

Sample No.	Injection 1	Injection 2	Average (wt %)
1	1.703	1.700	1.702
2	1.711	1.756	1.734
3	1.696	1.704	1.700
4	1.686	1.645	1.666
5	1.687	1.669	1.678
6	1.655	1.686	1.671
7	1.673	1.711	1.692
8	1.717	1.647	1.682
9	1.643	1.663	1.653
10	1.664	1.674	1.669
	average		1.685
	standard deviation		0.023

ANNEX

(Mandatory Information)

A1. VOLATILE BLOWING AGENT STANDARD PREPARATION

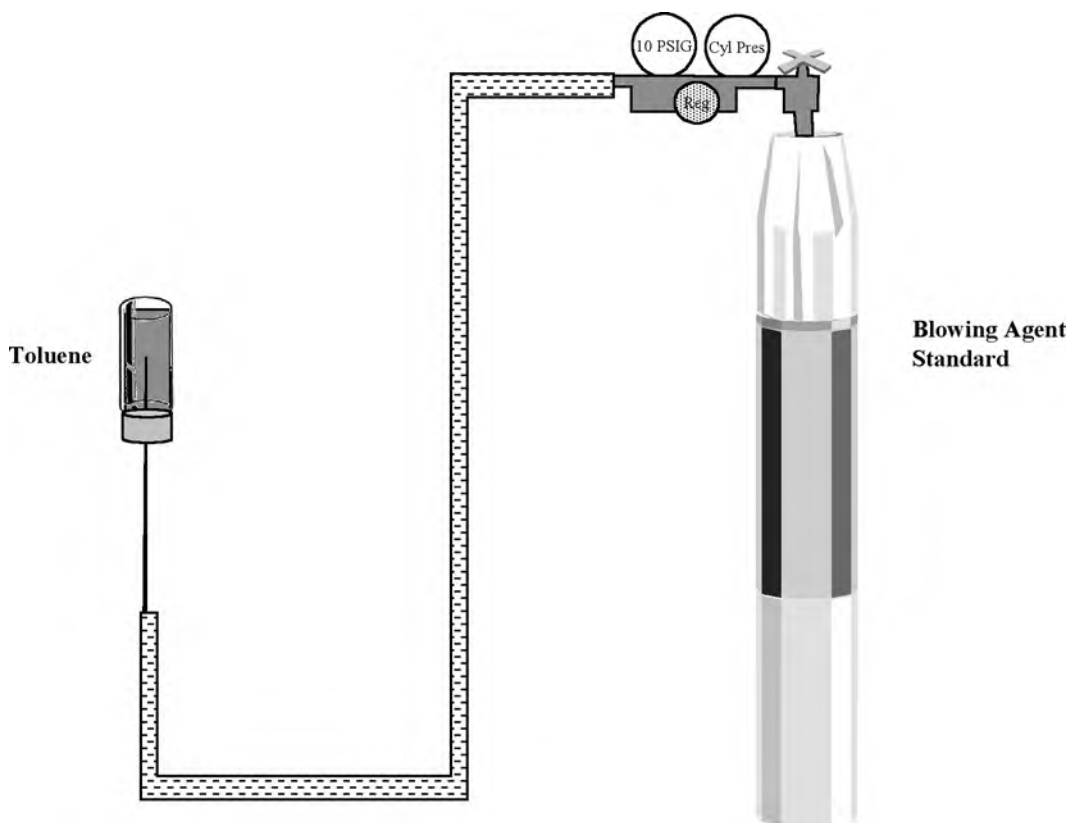


FIG. A1.1 Volatile Blowing Agent Standard Preparation

A1.1 Tare an empty 30-cm³ serum vial (with septa and seal).

A1.2 Fill the serum vial with toluene to about 90 % capacity. Affix septa and seal. Record the weight of the toluene to 0.1 mg (*W_T*). Tare the balance with the vial containing the toluene.

A1.3 Open the blowing agent standard cylinder. Set the delivery pressure to 70 kPa. To ensure gas purity, purge the line for at least 30 seconds.

A1.4 While the gas is flowing, insert the needle into the vial containing the toluene, as shown in Fig. A1.1, and inject 1 to 2 g of blowing agent. Weigh the vial and record weight to the nearest 0.1 mg (W_2).

A1.5 Proceed to 9.4.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D7132 – 13) that may impact the use of this standard. (March 1, 2014)

(1) Added 7.1, 7.5, and Note 4.

(2) Revised 11.1.

Committee D20 has identified the location of selected changes to this standard since the last issue (D7132 – 05) that may impact the use of this standard. (November 1, 2013)

(1) Edited standard for permissive language, removed in mandatory situations and moved non-mandatory options to notes.

(2) Removed references to trade names in 4.1, 4.7, and 7.2

(3) Updated ISO equivalency statement and moved to Note 1.

(4) Revised 11.1 to: “Normally, no peaks in the ISTD or blowing agent peak region are detected. If peaks are observed...”

(5) Editorially added units of measurement statement in 1.3.

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