Designation: E2269 – 14

Standard Test Method for Determining Argon Concentration in Sealed Insulating Glass Units using Gas Chromatography[1]

This standard is issued under the fixed designation E2269; 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 procedures for using gas chromatographs to determine the concentration of argon gas in the space between the panes of sealed insulating glass.

1.2 This test method is not applicable to insulating glass units containing open capillary/breather tubes.

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 determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C162 Terminology of Glass and Glass Products
C717 Terminology of Building Seals and Sealants
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E355 Practice for Gas Chromatography Terms and Relationships
E631 Terminology of Building Constructions
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E773 Test Method for Accelerated Weathering of Sealed Insulating Glass Units (Withdrawn 2010)[3]
E2188 Test Method for Insulating Glass Unit Performance

3. Terminology

3.1 *Definitions*—For definitions of terms found in this standard, refer to Terminologies C162, C717, and E631.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *fill gas*—any gas or mixture of gases intended to replace atmospheric air in the space between the panes of a sealed insulating glass unit. A fill gas is typically inert; and the most commonly used fill gases include argon, krypton, and sulfur hexafluoride (SF_6).

3.2.2 *sealed insulating glass unit*—a pre-assembled unit, comprising sealed panes of glass separated by dehydrated space (s), intended for clear vision areas of buildings. the unit is normally used for windows, window walls, picture windows, sliding doors, patio doors, or other types of windows or doors.

4. Significance and Use

4.1 This test method is intended to provide a means for determining the concentration of argon, oxygen, and nitrogen gases in individual sealed insulating glass units, which were intended to be filled with a specific concentration of argon at the time of manufacture.

4.2 The argon, oxygen, and nitrogen are physically separated by gas chromatography and compared to corresponding components separated under similar conditions from a reference standard mixture or mixtures of known composition.

4.3 The composition of the sample is calculated from the chromatogram by comparing the area under the curve of each component with the area under the curve of the corresponding component on the reference standard chromatogram.

4.4 It is essential that the person or persons performing this test are very knowledgeable about the principles and techniques of gas chromatography, operation and calibration of gas chromatographs. More information can be found in Practice E355.

4.5 It takes time for the fill gas to equilibrate in any insulating glass unit. This is particularly important in insulating glass units using a tubular spacer and in units containing interior components such as tubular muntin bars. Performing this test before a unit has equilibrated could result in fill gas concentrations that are measurably different than the actual fill gas concentration.

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[1] This test method is under the jurisdiction of ASTM Committee E06 on Performance of Buildings and is the direct responsibility of Subcommittee E06.22 on Durability Performance of Building Constructions.

Current edition approved April 1, 2014. Published May 2014. Originally approved in 2003. Last previous edition approved in 2005 as E2269 – 05. DOI: 10.1520/E2269-14.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

4.6 This method may be used to determine the initial argon gas concentration achieved by the filling method, or the argon gas concentration in units which have been in service or which have been subjected to durability tests such as those described in Test Methods E773 and E2188.

4.7 This method is not applicable to units filled with mixtures of argon and gases other than air.

4.8 This is a destructive test method in that the edge seal of the insulating glass unit is breached in order to obtain a gas sample for analysis by gas chromatography.

4.9 The argon concentration in the gas fill is part of the information necessary to estimate the thermal performance of the sealed insulating glass unit.

NOTE 1—Other data necessary include gap width, glass thickness, coating type, film coefficients, and so forth, but are beyond the scope of this standard.

5. Apparatus

5.1 *Gas Chromatograph,* capable of separating argon from oxygen and nitrogen as indicated by the return of the recorded peak to the baseline between each successive peak and including the following components:

5.1.1 *Gas Sampling Valve,* with a capacity of 100 to 250 µL.

5.1.2 *Adsorption Column,* capable of separating argon from other gases.

NOTE 2—Examples of columns which may be used include: Haysep[4] and Restek, 5Å Plot.

5.2 *Detector,* such as a thermal conductivity detector (TCD).

5.3 *Integrator.*

5.4 Chromatograms must be reproducible so that successive runs of a reference standard agree on each component peak area within ±0.1 %.

6. Reagents and Materials

6.1 *Gas Cylinders:*

6.1.1 Helium carrier gas cylinder, analytical grade with purity 99.9 %.

6.1.2 Compressed air cylinder (for valve actuation).

6.1.3 Liquid CO_2 or N_2 cylinder with dip tube, or refrigeration system for cooling column oven if using a column that requires sub-ambient temperatures for operation.

6.2 10 mL gas-tight syringe(s) with closure valve and side port needle.

6.3 *Reference Standard Mixtures:*

6.3.1 At least two reference standard mixtures that contain known percentages of argon, oxygen, and nitrogen. These are required for calibration.

6.3.2 Reference standard mixture with argon concentration greater than 98 %.

6.3.3 Reference standard mixture with argon concentration equal to 50 ± 5 %.

6.3.4 If the argon concentration to be determined is less than 50 %, an additional reference standard mixture is necessary that would bracket the expected argon fill level.

NOTE 3—Suitable standard mixtures can be obtained with a certificate of analysis of the makeup of the mixture from commercial gas suppliers. The accuracy of the results of this method depends on the availability of accurate calibration standards.

7. Sampling

7.1 Condition the insulating glass unit so that at the time of sampling a positive pressure exists inside the unit. This is achieved by heating the unit above the sealing temperature or by placing the unit horizontally on a flat surface and applying a weight to the center of glass. If the gas sampling occurs with the unit under negative pressure, contamination of the gas sample can occur.

7.2 Wrap the shank of the sampling needle with PIB (poly isobutylene) sealant or other sealing mastic.

7.3 Drill or punch a 1.6 mm hole through the edge sealant and the spacer. The hole is drilled into one of the long sides of the unit approximately 76 mm from a corner. Drilling a hole is not necessary in spacers that allow the needle to pass through the spacer without damage or obstruction to the needle.

7.4 Remove the drill or punch and immediately plug the hole with a finger.

7.5 Slide the finger off the hole and immediately insert the PIB wrapped sampling needle, with the syringe evacuated (plunger forward).

7.6 Seal the needle into the hole with the PIB sealant.

8. Calibration and Standardization

8.1 *Apparatus Preparation:*

8.1.1 Prepare gas chromatograph as directed by the manufacturer.

NOTE 4—The following is an example of operating conditions that have been found to be satisfactory using the Haysep column for this test method:

Carrier Gas	Helium, 30mL/min
Column	Haysep DB, 100 to 120 mesh
Column Size	9.1 m by 3 mm stainless steel
Column (Oven) Temperature	–30°C
Sampling Loop Temperature	100°C
Sample Volume	250 µL

8.2 *Reference Standard Introduction and Separation:*

8.2.1 *Purging Syringe:*

8.2.2 Fill the 10 mL gas-tight syringe from the cylinder containing the reference standard. Filling and evacuating of the syringe must be done at a controlled rate to ensure proper sample collection.

8.2.3 Remove the syringe from the cylinder outlet and evacuate the syringe to purge any contaminants that it may have contained.

8.2.4 Repeat 8.2.2 and 8.2.3 at least two more times.

8.2.5 Refill the syringe with the reference standard gas.

8.2.6 Close the syringe valve and remove it from the cylinder.

8.2.7 Introduce the reference standard sample(s) into the gas chromatograph sampling port.

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[4] The term “Haysep” and the Haysep logo are trademarks of Valco Instruments Co. Inc., P.O. Box 1674, Bandera, TX 78003.

8.2.8 The reference standard introduction and separation shall be repeated before and after the Procedure in Section 9. For a group of samples, additional re-calibrations shall be conducted periodically (for example, after every 10 samples) to confirm uniformity.

8.2.9 As a minimum the reference standard mixtures shall be run twice within an 8-h period when no changes to the chromatograph conditions or configurations have occurred.

8.2.10 Record the chromatogram and the integrated percentages of oxygen, nitrogen and argon (O_2, N_2, and Ar).

9. Procedure

9.1 With the syringe evacuated (plunger forward), insert the syringe needle into the insulating glass unit as described in Section 7.

9.2 Fill the syringe with the airspace gas then evacuate its contents back into the airspace to purge any contaminants that it may have contained. Filling and evacuating of the syringe must be done at a controlled rate to ensure proper sample collection.

9.3 Repeat 9.2 at least two more times.

9.4 Fill the syringe with the gas sample.

9.5 Close the syringe valve.

9.6 Carefully grip the needle at its base and pull it out of the gas space.

9.7 Insert the needle into the gas sampling inlet and open the syringe valve.

9.8 Inject the contents of the syringe into the column via the septum connected at the inlet of the gas sampling valve.

9.9 Record the chromatogram with the integrated percentages of Ar, O_2, and N_2.

10. Calculation or Interpretation of Results

10.1 Refer to Practice E355.

11. Report

11.1 *Complete Description of Specimen Tested:*

11.1.1 Dimensions of the test specimen (width by height) and overall thickness.

11.1.2 Type and thickness of glass.

11.1.3 Glass coatings and surface locations if applicable.

11.1.4 Airspace thickness(es).

11.1.5 Describe the spacer composition(s) and configuration(s).

11.1.6 Describe the corner construction including the type and number of corner keys.

11.1.7 Desiccant type and quantity, if provided.

11.1.8 Presence and composition (if known) of muntin bars.

11.2 Report composition of reference standard gases used.

11.3 Report type of adsorption column used, column size, column temperature, sampling loop temperature, and sample volume.

11.4 Report argon concentration measured for each reference standard gas before and after measurement of the sample.

11.5 Report measured argon concentration for each sample.

12. Precision and Bias[5]

12.1 *Precision*—The precision of this test method is based on an interlaboratory study of E06 - ASTM E2269, Standard Test Method for Determining Argon Concentration in Sealed Insulating Glass Units using Gas Chromatography, conducted in 2013. A single laboratory participated in this study submitting a total of ten test results for Argon concentration. Because of the destructive nature of the testing, every "test result" reported represents an individual determination. Except for the use of only one laboratory, Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. RR: E06-1004.

12.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

12.1.1.1 Repeatability can be interpreted as the maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

12.1.1.2 The single-lab repeatability estimate is listed in Table 1.

12.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

12.1.2.1 Reproducibility can be interpreted as the maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

12.1.2.2 Reproducibility limits cannot be determined from single-lab data.

12.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

12.1.4 Any judgment in accordance with statements 12.1.1 and 12.1.2 will have an approximate 95 % probability of being correct. The precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable

TABLE 1 Argon Concentration (%)

Material	Average[A] $\bar{x}$	Repeatability Standard Deviation S_r	Repeatability Limit r
Sealed glass units	77.92	0.83	2.32

[A] The average of the laboratories' calculated averages.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: E06-1004. Contact ASTM Customer Service at service@astm.org.

to all circumstances and uses. The limited number of materials tested, and laboratories testing, may lead to times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply.

12.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

12.3 The precision statement was determined through statistical examination of 10 results, from one laboratory, on ten 14 in. by 20 in. sealed glass insulating units from the same batch and lot.

13. Keywords

13.1 fill gas; gas chromatography; gas concentration; sealed insulating glass unit

APPENDIX

(Nonmandatory Information)

X1. GAS FILL CORRECTION FOR HOLLOW TUBE SPACERS PRIOR TO EQUILIBRIUM

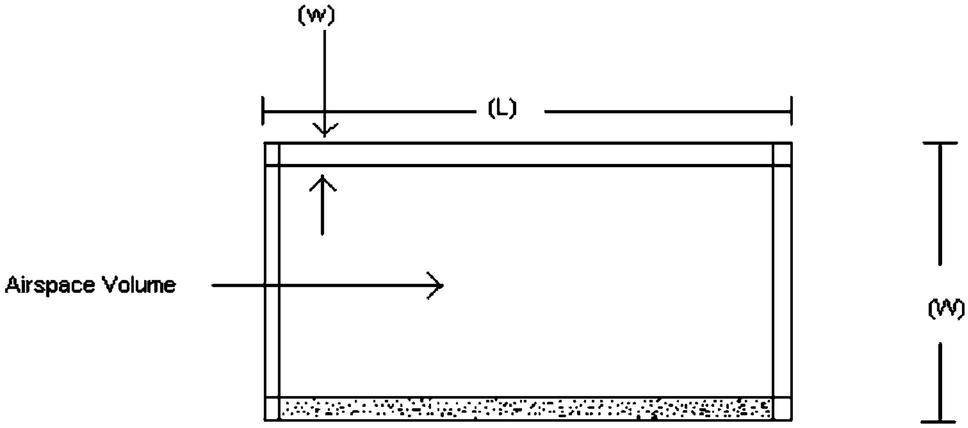

FIG. X1.1 IG Unit Dimensions

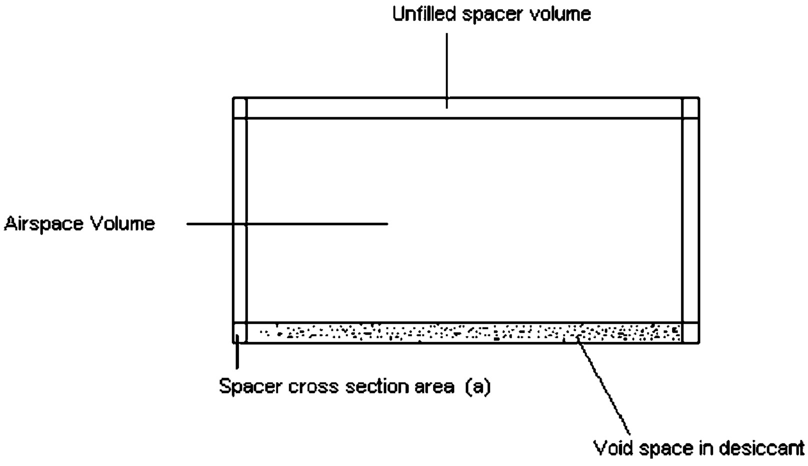

FIG. X1.2 Unfilled Spacer Volume

X1.1 Initial Gas Fill Correction Only for Hollow Tube Spacers

X1.1.1 This correction is intended for use prior to gas equilibrium.

X1.1.2 This correction is intended for use in insulating glass units made with hollow tube spacers.

X1.1.3 The correction given below may be suitable for use when gas-filling insulating glass units are made with hollow tube spacers. Air can remain in the hollow tubes after gas completely fills the airspace. When this air and the fill gas subsequently mix together, the actual percentage of fill gas in the insulating glass unit can be of lesser concentration than expected.

$$\text{Corrected Initial Gas Fill \%} = \frac{\text{Volume Fill Gas Initial}}{\text{Total Gas Space Volume}} \quad (X1.1)$$

$$\text{Volume Fill Gas Initial} = (\text{Airspace Volume})(\text{\% Gas Fill Initial}) \quad (X1.2)$$

$$\text{Airspace Volume} = (L - 2w)(W - 2w)(t) \quad (X1.3)$$

$$\text{Total Gas Space Volume} \quad (X1.4)$$

$$= \text{Airspace Volume} + \text{Unfilled Spacer Volume} + \text{Void Spaces in Desiccant}$$

$$\text{Unfilled Spacer Volume} \quad (X1.5)$$

$$= a(1 - \text{\% Desiccant Fill})[2(L - w) + 2(W - w)]$$

$$\text{Void Space in Desiccant} \quad (X1.6)$$

$$= a(\text{\% Desiccant Fill})(\text{\% Desiccant Voids})[2(L - w) + 2(W - w)]$$

where:

L = spacer length in the long leg
W = spacer length on the short leg
w = width of hollow tube spacer
t = airspace thickness
a = spacer corner cross section

Corrected Initial Gas Fill %

$$= \frac{(L - 2w)(W - 2w)(t)(\text{\% Gas Fill})}{(L - 2w)(W - 2w)(t) + a(1 - \text{Desiccant Fill})[2(L - w) + 2(W - w)] + a(\text{Desiccant Fill})(\text{Desiccant Voids})[2(L - w) + 2(W - w)]} \quad (X1.7)$$

NOTE X1.1—The voids present in the desiccant fill are represented in the formula in order to acknowledge their presence. The % desiccant voids are not actually calculated when using the formula to determine the final % gas fill.

Ignoring the void space in the desiccant, the equation reads:

Corrected Initial Gas Fill %

$$= \frac{(L - 2w)(W - 2w)(t)(\text{\% Gas Fill})}{(L - 2w)(W - 2w)(t) + a(1 - \text{Desiccant Fill})[2(L - w) + 2(W - w)]} \quad (X1.8)$$

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