Designation: E2649 – 12

Standard Test Method for Determining Argon Concentration in Sealed Insulating Glass Units Using Spark Emission Spectroscopy[1]

This standard is issued under the fixed designation E2649; 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 a spark emission spectroscope to determine the concentration of argon gas in the space between the lites of a sealed insulating glass unit.

1.2 This is a non-destructive test method.

1.3 This test method shall be used only in a controlled laboratory environment.

1.4 This test method is applicable for insulating glass units where argon has been added to the sealed insulating glass cavity and the balance of the gas is atmospheric air.

1.5 This test method is applicable for clear, double-glazed insulating glass units.

1.6 This test method is applicable for double-glazed insulating glass units with one lite having a metallic coating or tinted glass, or both, and with clear glass as the other lite.

1.7 This test method is applicable for triple-glazed insulating glass units only when the center lite of glass has a metallic coating (either low emissivity (low E) or reflective) and both of the other lites are clear glass.

1.8 This test method also includes a procedure for verifying the accuracy of the readings of the test apparatus.

1.9 The values stated in SI units are to be regarded as the standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

1.10 *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.* For specific warning statements, refer to 7.1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C162 Terminology of Glass and Glass Products
C717 Terminology of Building Seals and Sealants
E631 Terminology of Building Constructions
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E2190 Specification for Insulating Glass Unit Performance and Evaluation

3. Terminology

3.1 *Definitions:* For definitions of terms found in this test method, refer to Terminologies C162, C717, and E631.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *sealed insulating glass unit*—an assembled unit, comprising sealed lites of glass separated by dehydrated space(s), normally intended for clear vision areas of buildings.

4. Summary of Test Method

4.1 The spark emission spectroscope is placed against the glass surface of a sealed insulating glass unit in a prescribed manner. A high voltage, at low current, is applied to the glass surface. This voltage creates a spark which induces a plasma from the gas molecules inside the test specimen. This causes light emissions (photons) of characteristic wavelengths. The instrument then collects the photons and analyzes them by spark emission spectroscopy. The resulting spectrum is compared to calibration data internal to the instrument to determine the concentration of argon inside the unit.

5. Significance and Use

5.1 This test method is intended to provide a means for determining the concentration of argon in sealed insulating glass units under controlled conditions in compliance with the apparatus manufacturer's instructions.

[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.

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[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.

5.2 This is a non-destructive test method in that the edge seal of the test specimen is not breached in order to determine the argon gas concentration. However, damage to some glass coatings on the inner surfaces of the glass can occur.

5.3 This test method has been developed based on data collected in a controlled laboratory environment.

5.4 The device shall be used to determine the argon gas concentration in insulating glass units in a controlled laboratory environment. Refer to 12.3.

5.5 This test method may be used to determine the argon gas concentration before, during, or after the insulating glass unit is subjected to durability tests.

5.6 The accuracy of the test method is dependent upon the accuracy of the Spark Emission Spectroscope. When the concentration of the argon being measured is below certain levels, this test method is not applicable. See the spectroscope manufacturer's literature for recommended levels of accuracy of a given model.

6. Apparatus

6.1 *Spark Emission Spectroscope:*

6.1.1 The apparatus employs a high voltage, at low current, source and employs spark emission spectroscopy.

6.1.2 The head of the spark emission spectroscope contains an electrode which is used to apply the voltage to the glass surface of the test specimen. It also contains a light collector which transmits light emissions to a spectrometer for processing.

6.1.3 Different models[3] of the spark emission spectroscope shall be acceptable provided that new models demonstrate accuracy limits as defined in Section 10.

6.2 *Specimen Stand:*

6.2.1 The test specimen shall be supported in a vertical position or up to 30° off vertical position.

6.2.2 If necessary, a stand is used to support the test specimens. For example test stands, see Fig. 1 and Fig. 2.

6.3 *Background:*

6.3.1 A non-reflective black background shall be positioned behind the test specimen. Examples of background materials include photographic black fabric and black closed-cell foam.

7. Hazards

7.1 **Warning—**The high voltage of the spark emission spectroscope used in this test method can be harmful. Appropriate protective measures shall be observed. Refer to the instrument manufacturer's instruction manual.

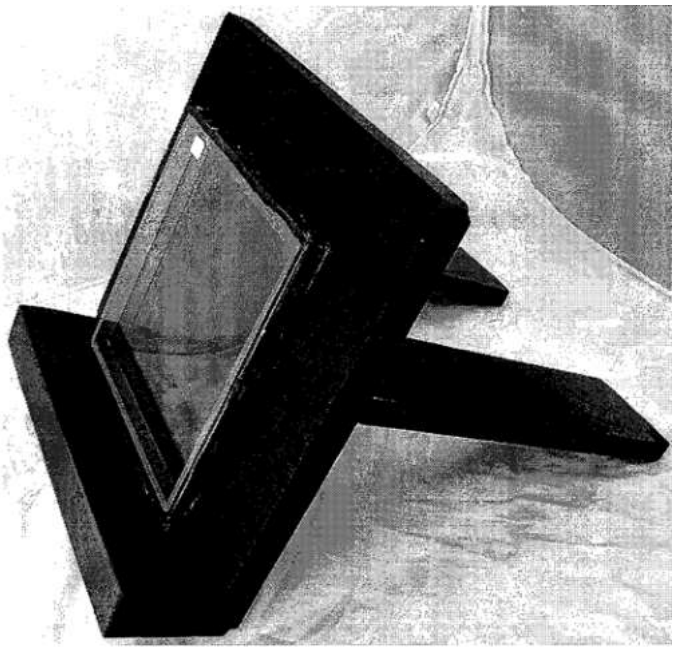

FIG. 1 Example of Test Stand

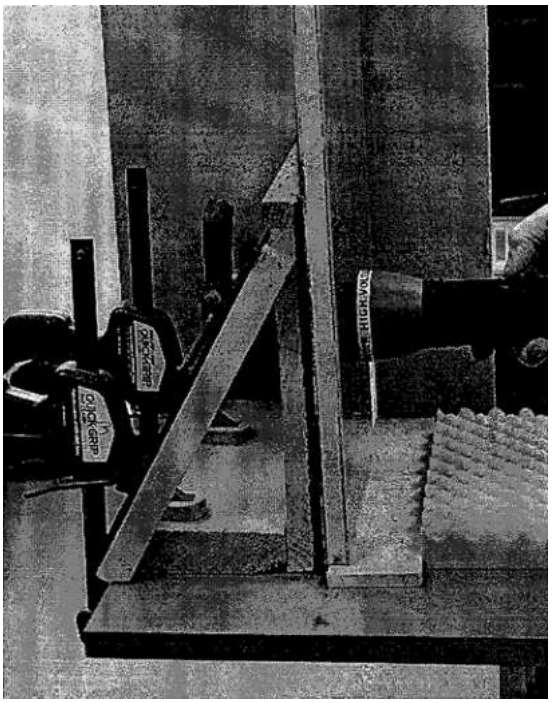

FIG. 2 Another Example of Test Stand

8. Test Specimens

8.1 Any sealed insulating glass unit that allows the spark emission spectroscope to excite the gas present in the airspace can be tested using this test method.

8.2 Typically, test specimens are 355 mm × 505 mm (14 in. × 20 in.) sealed insulating glass units constructed using one lite of 4 mm (5⁄32 in.) clear uncoated glass, a 12 mm (½ in.) air space, and one lite of 4 mm (5⁄32 in.) coated low E glass.

[3] This method was based on use of the Gasglass 1002 device (the wand model). As of this writing, there are other models of the device which include V1 and V2 (handheld models). The sole source of supply of these apparatuses known to the committee at this time is Sparklike, Ltd., Särkiniementie 5 C6, 00210 Helsinki, Finland, http://www.sparklike.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

Variations in the specimen construction may require a correction. See the instrument manufacturer's instruction manual for further information.

9. Calibration

9.1 Adjustment of the instrument is recommended to be performed only by the manufacturer of the instrument or an authorized service representative. The user shall verify the accuracy of the instrument readings using Section 10.

10. Verification

10.1 Verification of the accuracy of the instrument readings shall be performed by the user.

10.2 *Verification Specimens:*

10.2.1 The verification specimens shall be comprised of two lites of 4 mm glass, and a 12.0 mm ± 0.8 mm cavity. One of the lites of glass shall have a metallic, low emissivity coating on its cavity facing surface. Specimen size is suggested to be 350 mm × 350 mm.

10.2.2 Follow the instrument manufacturer's instruction manual for gas filling of verification specimens. Fill the verification specimens with reference gas mixtures according to 10.3.

NOTE 1—Different models[3] of the spark emission spectroscope may have different requirements for gas filling of verification specimens. Consult the manufacturer's instruction manual specific to the model of use.

10.3 *Reference Gas Mixtures:*

10.3.1 At least two reference gas mixtures that contain known percentages of argon and atmospheric air are required for verification. For increased confidence in the measurements over the capability range of the instrument, additional reference gas mixtures are recommended.

10.3.2 The first reference gas mixture shall have an argon concentration of approximately 90 %.

10.3.3 The second reference gas mixture shall have an argon concentration of approximately 80 %.

10.3.4 If the user has defined a specific argon gas concentration, then a third reference gas mixture is recommended at the defined argon concentration.

NOTE 2—Suitable gas mixtures can be obtained with a certificate of analysis of the mixture from commercial gas suppliers. The accuracy of the results of this test method depends on the accuracy of the certified reference gas mixtures.

10.4 *Verification Procedure:*

10.4.1 Not less than five readings shall be taken on each verification specimen following the procedures outlined in the instruction manual of the spark emission spectroscope and following Sections 11-13 of this test method. The average of the readings is recorded as the verification specimen value.

10.4.2 The verification specimen value shall not differ from the reference gas mixture value by more than 2 %.

10.4.3 Frequent verification of the instrument shall be performed. Users of the instrument shall establish the frequency of verification.

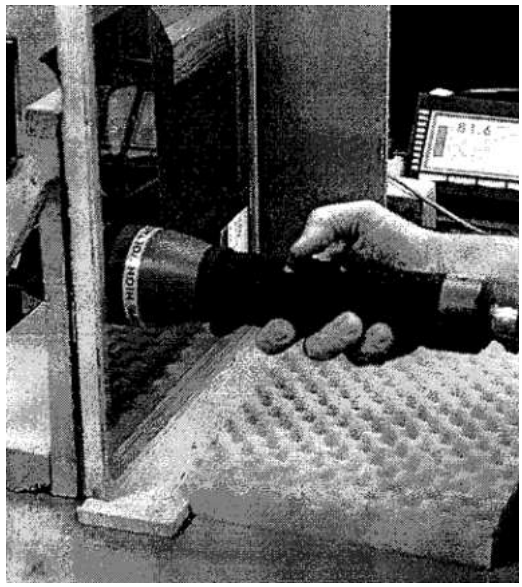

FIG. 3 Orientation of Sensor on Wand Model

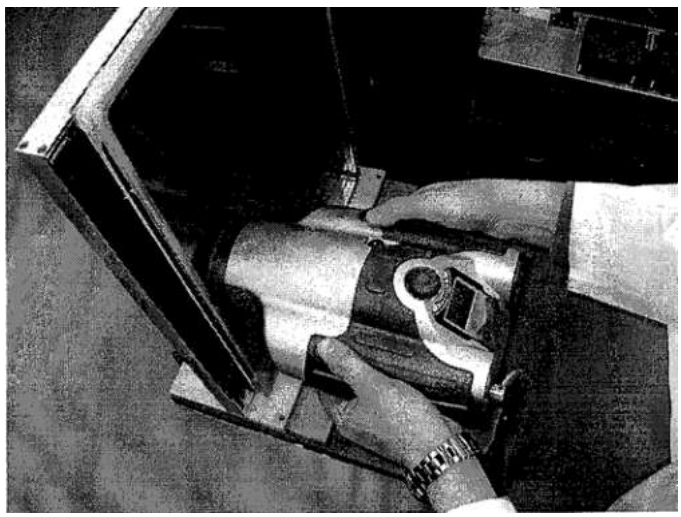

FIG. 4 Orientation of Sensor on Hand Model

11. Conditioning

11.1 The sealed insulating glass units shall be sealed for a minimum of four weeks from date of manufacture to allow for equilibration of the gas before testing.

NOTE 3—It takes time for the argon gas to equilibrate in any sealed insulating glass unit. This is particularly important in units using a tubular or porous spacer and in units containing interior components such as tubular or porous muntin bars. There can also be significant laminar stratification of the gasses inside the air space immediately following gas filling. Performing this test before a unit has equilibrated could produce results that are measurably different than the actual argon gas concentration. Some labs have found that mixing of the fill gas into the hollow tube spacer of an insulating glass unit can occur within 24 h. This will vary based on unit construction and gas filling methods.

12. Procedure

12.1 Turn on the instrument and allow it to warm up for at least 30 min.

FIG. 5 Positioning Sensor Head Near Edge of Glass

12.2 Orient the test specimen vertically against a dark background. Alternatively, place the test specimen on a stand. (See Fig. 1.)

12.3 Maintain controlled conditions in the room that include:

12.3.1 No direct sunlight,

12.3.2 No high intensity lamps in close proximity to the specimens, and

12.3.3 Air temperatures of 23 ± 3°C (73 ± 5°F).

12.4 For double-pane specimens that contain a metallic coating (either low E or reflective), this coated lite shall be placed against the dark background. A non-coated lite must be facing the instrument. For triple-pane specimens both outer lites are required to be clear glass and the middle lite must have a coating on one surface.

NOTE 4—Sealed insulating glass units with metallic coatings on both lites (both outer lites for triple-glazed units) cannot be tested with this test method.

12.5 Orient the sensor head so that the optical sensor is above the electrode in the face plate of the head. For the wand model, this puts the button/switch for the spark up at 12 o'clock. (See Fig. 3.) For the compact handheld model, the required orientation is shown in Fig. 4.

12.6 Locate the sensor head on the glass surface opposite the black background. The edge of the sensor head shall be at the inside edge of the insulating glass spacer of the test specimen. (See Fig. 5.)

12.7 Press the sensor head evenly against the glass so that the sensor head is perpendicular to the glass surface.

12.8 Press the button on the instrument to take a reading.

12.8.1 If the spark does not arc between glass Surfaces Two and Three on specimens without a metallic coating, place a grounding device against the glass surface opposite the sensor head and repeat 12.6 and 12.7. Examples of grounding devices include a finger, hand, or metal contact point. (See Fig. 6.) A black background shall be behind the grounding device to eliminate extraneous light from entering the spectrometer.

12.9 Observation of the following events is essential. If any of these events occurs during the sparking operation, reject the reading and take another reading:

12.9.1 Changes in ambient lighting,

12.9.2 Movement of the sensor head,

12.9.3 Inconsistent or excessive sound from the spark as compared to typical sounds from previous measurements,

12.9.4 Spark does not jump the gap in the test specimen for the duration of the "buzzing" sound from the instrument, and

12.9.5 For further information on these operational observations, see the instrument manufacturer's instruction manual.

12.10 Repeat 12.7-12.9 four more times for a total of five readings. After the third reading, relocate the sensor head by moving it along the insulating glass spacer approximately 75 to 100 mm (3 to 4 in.) from the original position.

12.11 Record all five readings.

13. Calculation of Results

13.1 Calculate and record the average and standard deviation for the data points. Report the calculated value rounded to the nearest whole percent.

NOTE 5—The terms *average* and *standard deviation* are common mathematical terms and are found as functions in most spread sheet programs.

14. Precision and Bias

14.1 The precision of this test method is based on an interlaboratory study of ASTM E2649 – 09 conducted in 2011. Seven laboratories tested a total of thirteen different materials

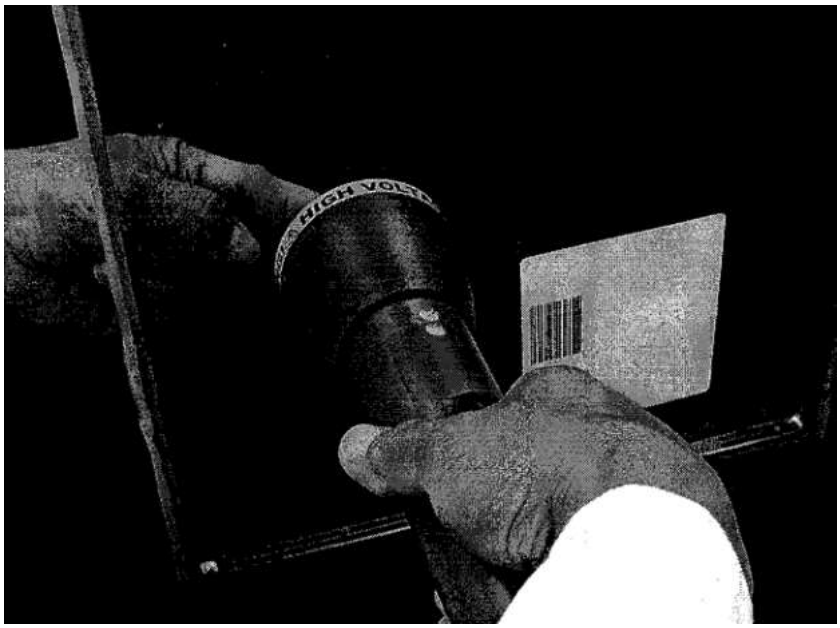

FIG. 6 Grounding with Finger or Hand

in triplicate. Every "test result" represents an individual determination. Practice E691 was followed for the analysis of the data; the details are given in ASTM Research Report No. E06-1003.[4]

14.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

14.1.1.1 Repeatability limits are listed in Table 1.

14.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

14.1.2.1 Reproducibility limits are listed in Table 1.

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

14.1.4 Any judgment in accordance with statements 9.1.1 would have an approximate 95 % probability of being correct.

14.2 *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.

14.3 The precision statement was determined through statistical examination of 249 results, produced by seven laboratories using V1 and V2 instruments, reporting the Argon concentrations of thirteen samples, in triplicate.

15. Keywords

15.1 argon gas; fill gas; gas concentration; sealed insulating glass unit; spark emission spectroscopy (SES)

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E06-1003.

TABLE 1 Argon Concentration (%, Absolute)

Material[A]	Average,[B] $\bar{X}$	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, r	Reproducibility Limit, R
A	91.852	0.442	0.606	1.239	1.698
B	90.648	0.242	0.652	0.678	1.826
C	86.686	0.393	0.685	1.102	1.919
D	68.392	1.090	2.270	3.051	6.357
E	51.333	1.482	2.768	4.149	7.750
F	89.948	0.373	0.496	1.044	1.390
G	91.239	0.404	0.459	1.132	1.284
H	83.562	0.358	1.019	1.002	2.853
I	77.228	1.121	1.463	3.137	4.095
J	80.976	0.588	1.535	1.645	4.298
K	87.511	0.581	1.078	1.626	3.018
Q	88.914	0.337	0.745	0.945	2.085
R	80.014	0.593	1.551	1.661	4.342

[A] Specimens Q and R were verification specimens, filled with known, premixed gases of 90 % and 80 % argon, respectively. All other specimens were exposed to accelerated weather cycle testing, in compliance with Specification E2190.
[B] The average of the laboratories' calculated averages.