

Glass in building — Insulating glass units —

Part 3: Long term test method and requirements for gas leakage rate and for gas concentration tolerances

The European Standard EN 1279-3:2002 has the status of a
British Standard

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National foreword

This British Standard is the official English language version of EN 1279-3:2002.

The UK participation in its preparation was entrusted by Technical Committee B/520, Glass and glazing in building, to Subcommittee B/520/2, Insulating glass products, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

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Glass in building - Insulating glass units - Part 3: Long term test method and requirements for gas leakage rate and for gas concentration tolerances

Verre dans la construction - Vitrage isolant préfabriqué scellé - Partie 3: Méthode d'essai à long terme et prescriptions pour le débit de fuite de gaz et pour les tolérances de concentration du gaz

Glas im Bauwesen - Mehrscheiben-Isolierglas - Teil 3: Langzeitprüfverfahren und Anforderungen bezüglich Gasverlustrate und Grenzabweichungen für die Gaskonzentration

This European Standard was approved by CEN on 1 September 2002.

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Foreword

This document EN 1279-3:2002 has been prepared by Technical Committee CEN/TC 129, "Glass in building", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2003, and conflicting national standards shall be withdrawn at the latest by May 2003.

This document is part of the series EN 1279 "Glass in building - Insulating glass units" that contains the following Parts:

- *Part 1: Generalities, dimensional tolerances and rules for the system description.*
- *Part 2: Long term test method and requirements for moisture penetration.*
- *Part 3: Long term test method and requirements for gas leakage rate and for gas concentration tolerances.*
- *Part 4: Methods of test for the physical attributes of edge seals.*
- *Part 5: Evaluation of Conformity.*
- *Part 6: Factory production control and periodic tests.*

The annexe A is normative. The annexes B and C are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard is the product standard for insulating glass units, which defines insulating glass units, and ensures by means of an adequate evaluation of conformity to this standard that:

- energy savings are made because the U-value and solar factor do not change significantly;
- health is preserved because sound reduction and vision do not change significantly;
- safety is provided because mechanical resistance does not change significantly.

It covers additional characteristics that are of importance for trade. Marking conditions are included.

The main intended uses of the insulating glass units are installations in windows, doors, curtain walling, roofs and partitions where there exists protection against direct ultraviolet radiation at the edges.

NOTE In cases where there is no protection against direct ultraviolet radiation at the edges, such as structural sealant glazing systems, additional European technical specifications should be followed. See Bibliography [2] and [3].

This Part of this standard, which is inextricably bound up with the other Parts of the standard, covers:

- the gas leakage rate by testing;
- the gas concentration tolerances;

as one means of verifying whether a product made in accordance with its system description, conforms with the relevant aspects of the definition of insulating glass units.

2 Normative references

This European Standard incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated by amendment or revision. For undated references, the latest edition of the publication referred to applies (including amendments).

EN 572-1, *Glass in Building - Basic soda lime silicate glass products - Part 1: Definitions and general physical and mechanical properties.*

EN 572-2, *Glass in Building - Basic soda lime silicate glass products - Part 2: Float glass.*

EN 673, *Glass in Building - Determination of thermal transmittance (U value) - Calculation method.*

prEN 1279-1, *Glass in Building - Insulating glass units - Part 1: Generalities, dimensional tolerances and rules for the system description.*

EN 1279-2, *Glass in Building - Insulating glass units - Part 2: Long term test method and requirements for moisture penetration.*

EN 1279-4, *Glass in Building - Insulating glass units - Part 4: Methods of test for the physical attributes of edge seals.*

EN 1279-6, *Glass in Building - Insulating glass units - Part 6: Factory production control and periodic tests.*

prEN 12758, *Glass in Building - Glazing and airborne sound insulation - Definitions and determination of properties.*

3 Terms and definitions and symbols

For the purposes of this European Standard, the terms and definitions given in prEN 1279-1 together with the following apply.

3.1

gas-filled insulating glass units

insulating glass unit in which the cavity contains gas(es) in addition to air, usually for improving thermal and/or sound insulation

3.2

gas concentration c

percentage by volume of gas in the cavity

- c_i is the concentration of gas i ;
- $c_{i,0}$ is the nominal value for a system of insulating glass and gas i which is the basis for testing sound insulation and/or calculating or testing thermal insulation to fix the respective R_w and U -value;
- $c_{i,f}$ is the estimated final concentration in percent of gas i . See annex A

NOTE For use of $c_{i,0}$, see also EN 1279-6.

3.3

gas leakage rate L_i

L_i : the proportion expressed as a percentage by volume of gas i leaking from a gas-filled unit per year calculated by the following formula:

$$L_i = 87,6 \cdot 10^6 \frac{m_i}{c_i \cdot V_{\text{int}} \cdot \rho_{o,i}} \cdot \frac{T}{T_o} \cdot \frac{P_o}{P} \quad \text{in } \% \cdot \text{a}^{-1} \quad (1)$$

where the meaning of the symbols is given in Table 1

Table 1 — Meaning and units of the symbols used in equation (1)

Symbol	Description	Units	
		SI system	for practical use in equation (1)
c_i	gas concentration determined according to 5.4.4	volume fraction in percentage	
m_j	mass of gas that has leaked from a gas-filled unit in a given time, and determined according to 5.4.3	kg/h	$\mu\text{g/h}$
$\rho_{O,i}$	density of gas i at temperature T_O and pressure P_O	kg/m^3	$\mu\text{g/mm}^3$
P	absolute atmospheric pressure at which the unit was sealed	Pa	same unit as P_O
P_O	atmospheric pressure at which ρ_O was determined	Pa	e.g. hPa
T	temperature at which unit was sealed	K	K
T_O	temperature at which ρ_O was determined	K	K
V_{int}	internal volume of the test specimen according to 5.4.1	m^3	mm^3
a	one year		

NOTE $\rho_{O,i}$ is often given at $T_O = 273 \text{ K}$ ($0 \text{ }^\circ\text{C}$) and $P_O = 1014 \text{ hPa}$

3.4

U_p

U -value (see EN 673) for publication: the thermal transmittance value to be published, normally determined with the gas concentration $c_{i,O}$. See also annex A

3.5

$R_{w,p}(C/C_{tr})$

$R_w(C/C_{tr})$ -value (see prEN 12758) for publication: the weighted sound reduction index to be published, normally determined with the gas concentration $c_{i,O}$. See also annex A

4 Requirements

4.1 Gas leakage rate

The gas leakage rate, L_j , for gases with concentrations higher than 15 %, and also for air, measured as described in clause 5 shall be

$$L_j < 1,00 \quad \text{in \% a}^{-1} \quad (2)$$

NOTE For most insulating glass units, measured L_j values are much higher than actual L_j values will be after 10 years natural ageing. Therefore the limiting value should not be used for calculating the gas concentration during the lifetime of the unit. See annex B.

In the case of sealants based on polysulfide, polyurethane, silicone or polyisobutylene, determining the gas leakage rate of argon (Ar) may replace the measurement of the gas leakage rate for sulfurhexafluoride (SF₆) and air.

4.2 Tolerances on gas concentration

For tolerances on gas concentration, refer to EN 1279-6.

4.3 Dew point and moisture penetration index

For testing and requirements on dew point and moisture penetration, refer to EN 1279-2.

4.4 Edge seal strength

For the requirements on edge seal strength, refer to EN 1279-4.

4.5 Additional requirements for other gases than argon, sulfurhexafluoride and air

For those requirements, refer to annex A.

5 Testing

5.1 Principle of testing

In the test, the gas leakage rate at 20 °C is measured after subjecting the test specimen to a climate as specified in EN 1279-2 with the following modifications:

- the number of cycles is reduced to 28; and
- the time at a constant temperature of 58 °C is reduced to 4 weeks.

For measuring the gas leakage rate, the unit is placed in a gastight container and, after a given time, the amount of gas which has leaked from the unit is measured. After this measurement, the gas concentration in the unit is analysed and the gas leakage rate calculated.

5.2 Apparatus

5.2.1 Climate exposure equipment

Test apparatus for the climate exposure as specified in EN 1279-2.

5.2.2 Container for gas leakage rate measurement

A controlled temperature container shall be used for measuring the gas leakage rate, which shall be hermetically sealable, and capable of receiving the unit to be tested while inducing as little stress as possible, so that the residual volume in the container is as small as possible while the sealed edge zones of the unit are exposed to the circulation of purging gas.

The quantity of ambient air penetrating into the container from outside, or the quantity of each constituent leaking from the container, shall be measured in a blank test using a solid glass body of approximately the same dimensions as the test specimens.

The container shall be deemed to have an adequate degree of tightness if the measured quantity of gas during the measurement does not exceed 10 % of the mass of gas leaking from the test specimen.

The container shall have fittings for introducing specific gases and for taking gas specimens.

For multiple glass units with at least one outer pane made of organic material, it shall be ensured that the gas diffusion through this (these) pane(s) is included in the measurement.

5.2.3 Gas analysis equipment

A gas analysis equipment shall be used which is capable of:

- a) analysis of the gaseous constituents essential to the insulation function of the glass unit, for concentrations of $50 \cdot 10^{-6}$;
- b) determination of percentages by volume of gas of up to 100 % within ± 3 % (relative).

These tasks shall not necessarily be performed using the same equipment.

5.2.4 Gas sampling device

A device shall be used for taking gas specimens from the glass unit, ensuring that the result is not distorted by ingress of air, segregation phenomena, or similar.

5.3 Test specimens

5.3.1 Preparation of test specimens

The test specimens shall consist of two panes of 4 mm clear float glass in accordance with EN 572-1 and EN 572-2. The length shall be (502 ± 2) mm and the width (352 ± 2) mm. The gap shall be nominal 12 mm, or if not manufactured, a gap as near to 12 mm as possible. The test specimens shall be representative of the system description as defined in prEN 1279-1.

If glass/plastic units are produced in such a way that gas leakage out of the unit through the plastic is possible, deviating from the paragraph above, glass shall be substituted by plastic.

Unless otherwise agreed, the design of the insulating glass unit, including the type and quantity of desiccant and of gas, shall conform to that manufactured in normal production (except for the measurement of the air loss rate, where 100 % air is allowed). The panes of the test specimen shall be flat when the unit is sealed. During sealing, the temperature, T in K, and the absolute pressure, P in hPa, shall be measured to the nearest 1 K and 3 hPa respectively.

The test specimens have to be manufactured in such a way that the gas concentration meets $c_j = c_{j,0}$ (+ 10 % to - 5 % absolute), for each gas when gas mixtures are used.

For the production of the test specimens, EN 1279-6 is mandatory.

5.3.2 Number of test specimens

At least six test specimens shall be prepared of which at least two shall be tested as described in 5.4 after climate exposure.

NOTE It is recommended to take more test specimens to test the gas filling before climate exposure. The gas leakage can be measured on further units before the climate exposure, at the earliest four weeks after they have been filled with gas and sealed. This will achieve the objective of keeping test costs and time scale to an acceptable maximum.

5.3.3 Construction and appearance

The test specimens shall be examined visually for the following criteria and/or defects:

- a) construction of insulating glass unit;
- b) damaged edges;
- c) edge cracks;
- d) fractures;
- e) specking in the cavity;
- f) congruence of panes;
- g) other visible defects.

5.4 Procedure

5.4.1 Determination of internal volume of a test specimen

Measure the clear distance between opposite spacers, s_1 and s_2 , to the nearest 1 mm, e.g. by means of a gauge graduated in millimetres. Determine the clear distance between the inner pane surfaces, s_3 , by measuring the distance between the inner pane surfaces at mid-length on the four edges of the test specimen, to the nearest 0,1 mm, and calculate the mean. The internal volume, V_{int} in mm^3 , is obtained as the product of s_1 , s_2 and s_3 .

5.4.2 Climate exposure

The climate exposure as specified in 5.1 shall be carried out on four test specimens, not sooner than one week after preparation of the test specimens. On completion of the climate exposure the test specimens are stored to achieve stabilisation, with free circulation of air around the edges at $(23 \pm 2)^\circ\text{C}$ and $(50 \pm 5)\%$ relative humidity, for at least four weeks, and no longer than seven weeks, up to the time of measuring the gas leakage rate as described in 5.4.3.

NOTE If the units are stored vertically, the free circulation of air around the edges may be realized by using two blocks at least 20 mm high, each covering not more than 30 mm of the edge.

5.4.3 Measuring the gas leakage

Measure the gas leakage of at least two test specimens at $(20 \pm 1)^\circ\text{C}$ after the climate exposure, leaving the test specimen in the container for as long as it takes for the mass of gas that has leaked from the unit to be determined quantitatively, in $\mu\text{g}/\text{h}$, using the gas analysis equipment described in 5.2.3.

The measurement of the gas leakage has to be repeated until sufficient constancy of the values has been achieved. Sufficient constancy is reached when the standard deviation over the last four measurements, which in the case of natural gases such as the components of air are made with at least one day between them, is less than $0,25 \mu\text{g}/\text{h}$, and with at least one measurement being higher than the one immediately preceding it (see annex C).

5.4.4 Analysis of gas

Determine the volume fraction in percent of the gaseous constituents essential to the insulation function of the unit, using the gas analysis equipment described in 5.2.3.

Take a gas specimen for this analysis from the cavity of the unit after the last measurement of the gas leakage rate.

5.5 Evaluation

Calculate the gas leakage rate, L_j , according to 3.3.

6 Precision of test method

In an inter-laboratory test involving four laboratories, four to eight units of a uniform production were exposed to a climate in accordance with 5.1. Afterwards the gas leakage rate was determined according to 5.4.3 and annex C. The standard deviation of all individual values was 20 %.

7 Test report

The test report shall evaluate the test in detail and shall include the following summary:

Name, address and logo of the test laboratory.

Summary of report n° Date

Insulating glass units - Evaluation of the gas leakage rate and gas concentration measured according to prEN 1279-3

For details, see the test report

Company: Name:
 Address:

Plant: Name:
 Address:

System description, file number:

Product name:

Reference to test report for moisture

penetration index according to EN 1279-2:

Applied gas(es) i :

Unit number:

Measured concentration c_i , (in %):

Nominal concentration $c_{i,o}$, (in %):

Gas leakage rate L_i , (in %·a⁻¹):

1	2	1	2	1	2	1	2

NOTE 1 If for certain gases the gas leakage rate L_i is not relevant, fill in the cell for L_i "NR" (= not relevant)

System conforms:

YES NO (Delete whichever is not applicable)

.....
 Name and signature

NOTE 2 For comparisons of gas leakage rates of different insulating glass unit systems, the standard deviation indicated in clause 6 of EN 1279-3:2002, should be taken into consideration.

Annex A (normative)

Requirements for other gases

A.1 Durability of the gas and interaction with insulating glass components

If it is not evident that the durability of the gas is adequate for the proposed applications, it shall be assessed by investigations. The same shall be done concerning the interactions with the insulating glass components.

A.2 Effect on thermal- and sound insulation

For most insulating glass unit types, the thermal transmittance U -value and/or the weighted sound reduction index $R_w(C/C_{tr})$, depending on the gas concentration, shall be determined.

According to the scope of this standard, thermal transmittance and sound reduction shall not change significantly over the working life of the insulating glass unit. That means that the increase of the U -value shall be maximum 0,1 W/(m²·K) and the deterioration of $R_w(C/C_{tr})$ shall be maximum 1 dB.

Those requirements are fulfilled under one of the following two conditions:

1) When gas filling improves the U -value by a maximum of 0,4 W/(m²·K) and when gas filling improves the $R_w(C/C_{tr})$ index by a maximum of 5 dB.

The U -value and $R_w(C/C_{tr})$ -value for publication are:

$$U_p = U(c_{i,o}) \text{ and } R_{w,p}(C/C_{tr}) = R_w(C/C_{tr})(c_{i,o}).$$

2) When gas filling improves the U -value by more than 0,4 W/(m²·K) or when gas filling improves the $R_w(C/C_{tr})$ index by more than 5 dB, then it shall be checked that:

$$a) \quad U(c_{i,f}) - U(c_{i,o}) \leq 0,1 \text{ W/(m}^2\cdot\text{K)}, \text{ and/or} \tag{A.1}$$

$$R_w(C/C_{tr})(c_{i,o}) - R_w(C/C_{tr})(c_{i,f}) \leq 1 \text{ dB} \tag{A.2}$$

The U -value and $R_w(C/C_{tr})$ -value for publication are:

$$U_p = U(c_{i,o}) \text{ and } R_{w,p}(C/C_{tr}) = R_w(C/C_{tr})(c_{i,o}).$$

And when a) is not fulfilled, the following calculation shall be carried out:

$$b) \quad U_p = U(c_{i,f}) - 0,1 \text{ W/(m}^2\cdot\text{K)}, \text{ and/or} \tag{A.3}$$

$$R_{w,p}(C/C_{tr}) = R_w(C/C_{tr})(c_{i,f}) + 1 \text{ dB} \tag{A.4}$$

$$\text{with } c_{i,f} = (c_{i,o} - 5) (1 - 0,22 \cdot L_{i,m}) \tag{A.5}$$

where

$L_{i,m}$ is the maximum gas leakage rate in percent as measured according to this standard, plus 5 % relative.

In case of gas mixtures, only the gas(es) with the maximum influence on the U -value and/or $R_{w}(C/C_{tr})$ -value shall be considered.

A.3 Assessment example with krypton gas filling

Assumptions:

- insulating glass unit with a cavity width of 8 mm, two panes of 4 mm, one coated with an emissivity of:
 $\varepsilon = 0,1$
- krypton concentration: $c_{Kr,o} = 90 \%$
- measured krypton leakage rate: $L_{Kr,m} = 0,5 \% \cdot a^{-1} (= 1,05 \cdot L_{Kr})$
- demonstrated argon leakage rate: $L_{Ar} < 1,0 \% \cdot a^{-1}$

Answer to 4.1: The gas leakage rates satisfy the requirements.

Answers to 4.2, 4.3 and 4.4: In this example, the requirements are assumed to be satisfied.

Answer to A.1: Krypton reacts chemically similar to argon. No special investigations are necessary.

Answer to A.2: The krypton gas filling improves the U -value by more than 0,4 W/(m²·K), however the $R_{w}(C/C_{tr})$ -value improves by less than 5 dB.

In accordance with condition 1) of A.2, the weighted sound reduction index for publication $R_{w,p}(C/C_{tr})$ is equal to $R_{w}(C/C_{tr})(c_{Kr,o})$.

For the U -value, check against to condition 2 a) of A.2:

- gas concentration: $c_{Kr,f} = (90-5) (1 - 0,22 \times 0,5) = 75,65$
- increase in U -value (calculations according to EN 673):
 $U(c_{Kr,f}) - U(c_{Kr,o}) = 1,54 - 1,38 = 0,16 \text{ W/(m}^2\cdot\text{K)}$

That increase is greater than 0,1 W/(m²·K), so that condition 2)b) of A.2 is performed.

- the U -value for publication shall be:

$$U_p = 1,54 - 0,1 = 1,44 \text{ W/(m}^2\cdot\text{K)} \text{ and rounded to one decimal } U_p = 1,4 \text{ W/(m}^2\cdot\text{K)}$$

Annex B (informative)

Relationship between artificial and natural ageing with regard to thermal and sound insulation

The gas leakage rate of insulating glass units which had been installed in windows of buildings for 10 years was determined. The measured values were smaller by a factor of 10 compared to values measured on units with the same construction according to DIN 52293 (see Bibliography [1]) after artificial ageing. A comparison of this artificial ageing with the one described in 5.4 showed no significant difference with respect to the gas leakage rate.

With this experience, it may be assumed that a type of insulating glass with a gas leakage rate $L_j < 1,0 \text{ \%} \cdot \text{a}^{-1}$ after artificial ageing according to this standard, loses less than 5 % relative of gas over 25 years installed in a building. For this estimate, to be on the safe side, it was assumed that in buildings the gas leakage rate is doubled every 10 years. In a unit with a typical nominal value for the argon concentration of $c_{Ar,O} = 90 \text{ \%}$, and with a possible real concentration of 85 %, the gas concentration after 25 years is higher than 80 %. On the assumption that the improvement of the U -value with 100 % argon filling is $0,4 \text{ W}/(\text{m}^2 \cdot \text{K})$, this results in a deterioration of

$$\Delta U < 0,04 \text{ W}/(\text{m}^2 \cdot \text{K})$$

from the value calculated with $c_{Ar,O} = 90 \text{ \%}$. For the final assessment, the U -value is rounded to $0,1 \text{ W}/(\text{m}^2 \cdot \text{K})$.

Similar considerations can be made for the sound insulation. It can be expected that insulating glass units fulfilling the requirements of this standard will show no significant change.

Annex C (informative)

Determination of the gas leakage rate by gas chromatography

C.1 Principle of method

The test method described below is one method for measuring the gas tightness according to clause 5. Other methods can be adopted.

If one follows this method, it should be followed strictly in order to achieve the correct result.

The test method is only applicable to gas-filled insulating glass units made with panes of inorganic materials. The gas leakage, m_j , in mass of gas per hour, will be determined in order to calculate the gas leakage rate L_j , expressed as a percentage by volume per year ($\% \cdot a^{-1}$), according to clause 5.

The test specimen or its edges is enclosed by a container, of which the inner volume is only slightly greater than the outer volume of the unit. The gas which has leaked from the unit in a given time is transported by a helium stream to a gas chromatograph with a thermal conductivity or electron capture detector. The mass of the leaked gas is then determined.

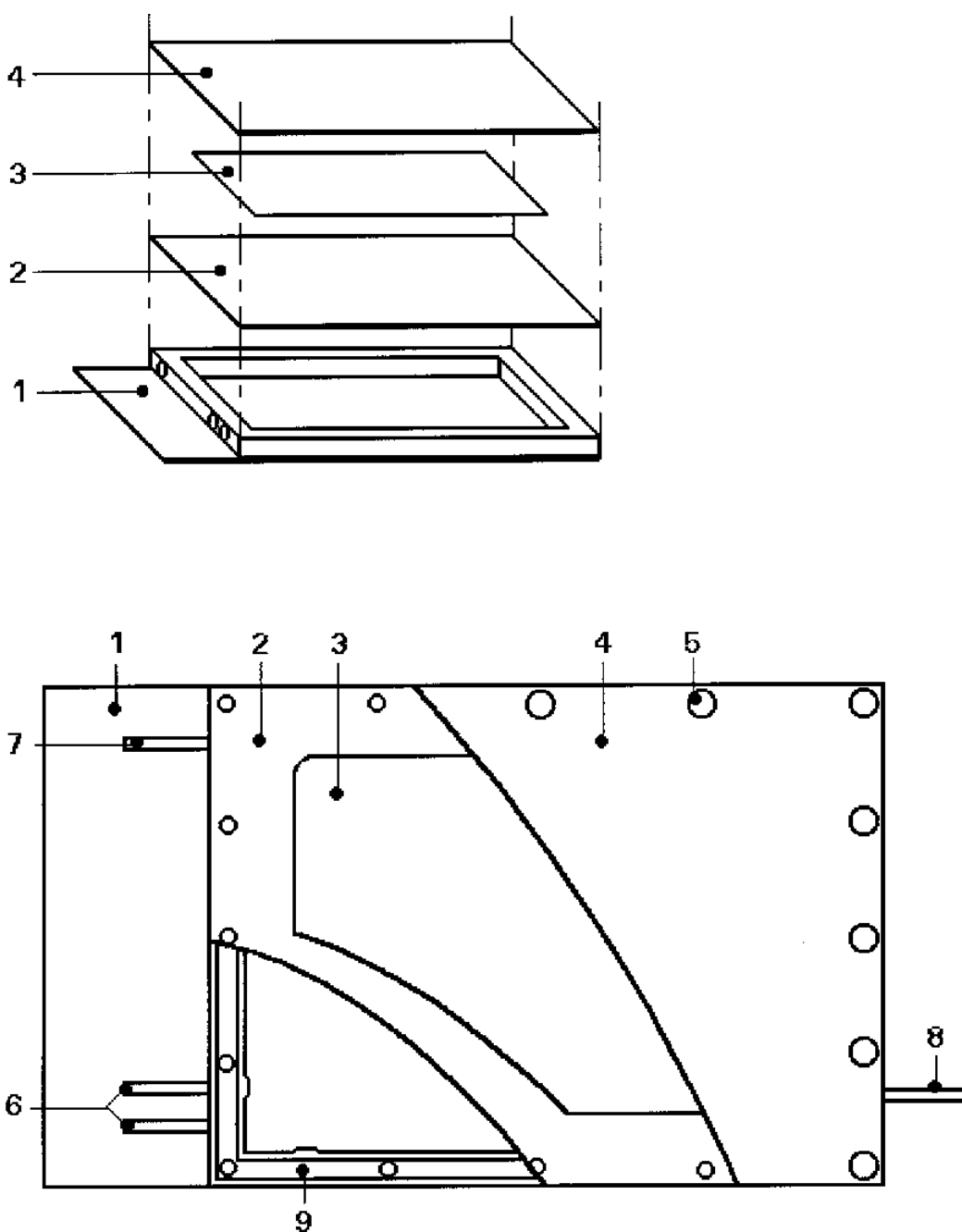
C.2 Equipment

C.2.1 Full container

The container in Figure C.1 consists of:

- 1) a lower part made of metal;
- 2) a foil made of copper;
- 3) a mat made of foam plastic, 3 mm thick, with dimensions according to the test specimen;
- 4) a cover made of metal;
- 5) elements for tightening.

The lower part has a flat inner bottom with the dimensions approximately 360 mm width and approximately 510 mm length. The dimensions of the full container and the test specimen have to be reconciled, so that after installation of the test specimen the residual volume is as small as possible. The inner height of the lower part is 22 mm. The walls may have ledges for fixing the test specimen, but they may not interrupt the gas stream at the edges of the test specimen. The walls contain two bore holes for the supply pipe and the extraction of the purging gas. The lower part can have an annular groove, through which a protective gas stream can flow.



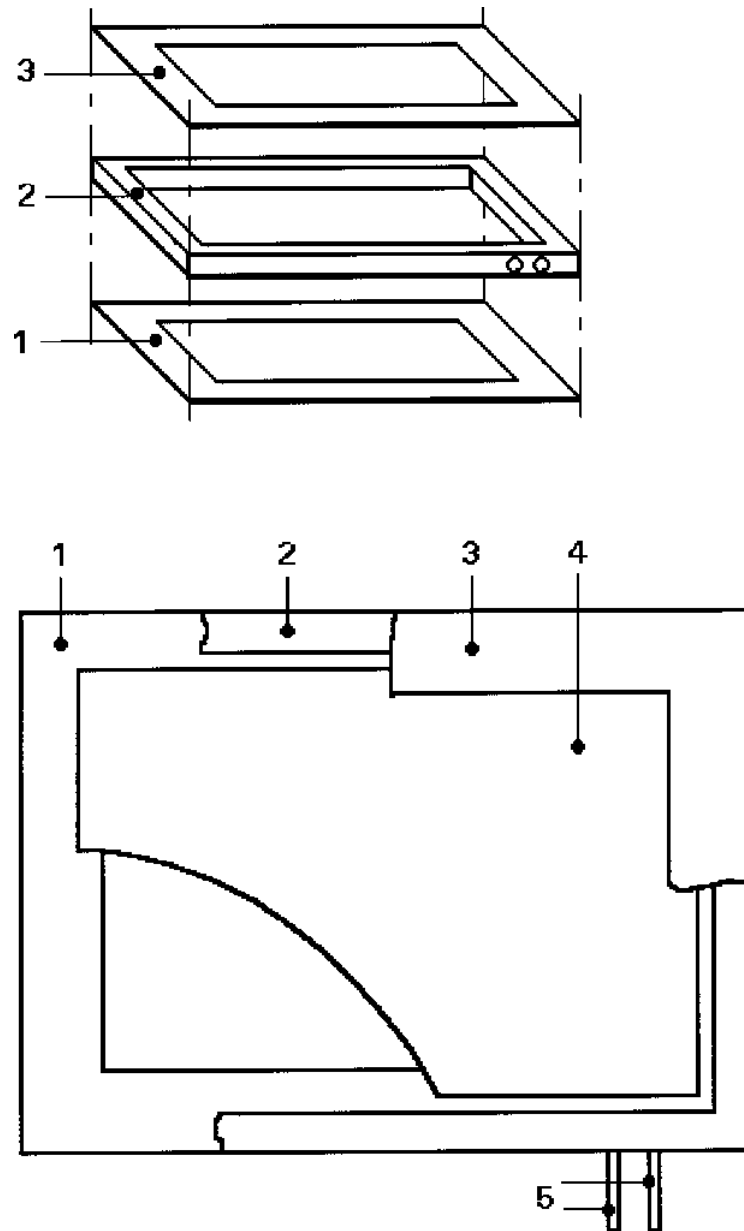
Key

- | | |
|---|--------------------------|
| 1 Lower part | 6 Purging gas in and out |
| 2 Metal foil | 7 Protective gas in |
| 3 Mat | 8 Protective gas out |
| 4 Cover | 9 Annular groove |
| 5 Elements for tightening (e.g. screws) | |

Figure C.1 – Full container

C.2.2 Ring container

The ring container (see Figure C.2) has a frame of metal and two masks, made of a self-adhesive metal foil or a sheet metal coated with a sealant. The frame has two bore holes, one for the supply pipe and the other for extraction of the purging gas. The dimensions of the ring container and test specimen have to be reconciled, so that after installation of the test specimen the residual volume is as small as possible.



Key

- 1 Lower mask
- 2 Frame
- 3 Upper mask
- 4 Test specimen
- 5 Purging gas in and out

Figure C.2 – Ring container

C.2.3 Cooling trap

The cooling trap consists of a metal tube, filled with an adsorbent, which is bent into either a U-shape or a spiral, and of two interchangeable containers, one with liquid nitrogen and one with water at $(95 \pm 5) ^\circ\text{C}$. The adsorbent is selected in such a way that the gas for measuring will be adsorbed at liquid nitrogen temperature and will be driven off abruptly at hot water temperature.

C.2.4 Gas chromatograph

A commercial gas chromatograph (GC) with a thermal conductivity or electron capture detector, and an integrator with recorder, is required. A calibrating loop of approximately 1 ml is connected to the gas chromatograph. The carrier gas is helium.

C.2.5 Connecting pieces

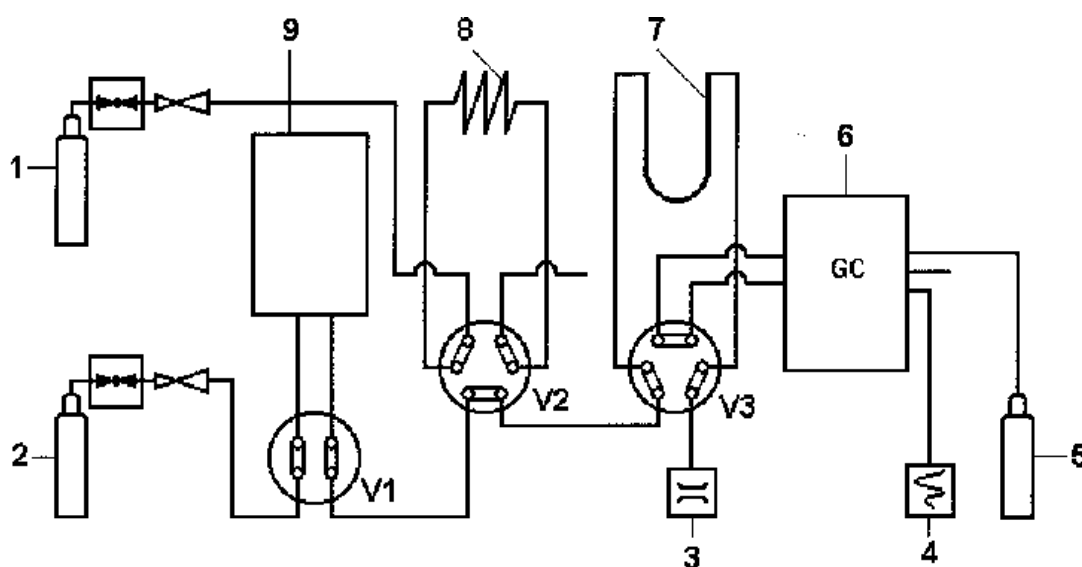
The pipes, valves and adapters as illustrated in Figure C.3, should be so tight that the leakage rate during testing with helium is not more than $0,0001 \text{ l} \cdot \text{Pa} \cdot \text{s}^{-1}$.

C.2.6 Solvents

For cleaning the test specimen surface, ethanol and isopropanol are recommended.

C.2.7 Purging and carrier gas

Helium with a purity of 99,999 6 % parts by volume.



Key

1 Calibrating gas	4 Integrator	7 Cooling trap
2 Purging gas	5 Carrier gas	8 Calibrating loop
3 Flow meter	6 Gas chromatograph	9 Container

V1, V2 and V3: valves, for valve positions, see Table C.1.

Figure C.3 — Gas pipe plan

Table C.1 — Overview of valve positions

Valve position	Valves			Function
	V1	V2	V3	
a)				purging
b)				filling the calibrating loop with calibrating gas
c)				concentration of calibrating gas by cooling
d)				measuring
e)				concentration of contamination by cooling
f)				concentration of gas specimen by standing time
g)				concentration of gas specimen by cooling

C.2.8 Calibrating gas

Helium using a volume fraction of 1 % of the relevant gases, for example nitrogen, oxygen, argon and sulphurhexafluoride, is recommended if the calibrating loop has a volume of approximately 1 ml.

C.3 Preparation of test specimens

Test specimens according to 5.3. All traces of sealant, labels and other contaminations which might influence the gas exchange, are removed. The test specimen surfaces are cleaned with the solvent avoiding all contact with the sealant.

C.4 Procedure

C.4.1 Connection of the apparatus

The apparatus is connected as shown in the gas pipe plan (see Figure C.3).

C.4.2 Installation of the test specimen

Install the test specimen in such a way that the periphery can be purged totally with the exception of the interruption between the two purging holes. The gas stream is interrupted between the two purging bore-holes by a suitable sealant e.g. butyl.

- a) Full container: grease the contact surfaces of the lower part and the metal foil with a high vacuum grease. Place a fault-free metal foil on the test specimen and the sealing face. Place the mat on the metal foil in the middle, so that the metal foil will be flattened by the cover during tightening of the full container, and the residual volume will be decreased;
- b) Ring container: Cover the space between test specimen and frame with masks. The overlap is at least 15 mm. Press the upper and lower mask and the frame together.

C.4.3 Temperature

Maintain the temperature for measuring the gas leakage rate at (20 ± 1) °C either by controlling the temperature of the room or by storage of the container with the test specimen in a water bath. Store the ring container in a water bath and cover it totally.

C.4.4 Calibration

Control daily the retention time, the separating performance and the sensitivity of the gas chromatograph, to obtain reliable quantitative and qualitative results. Direct the calibrating gas to the analysis system by using a calibrating loop of a defined volume, e.g. 1 ml. The evaluation of the signals in the detector by the integrator requires sufficient resolving power of the peaks.

Adjust the parameters of the gas chromatograph in accordance with this operation. Because of the volume of the gases, e.g. nitrogen, oxygen, argon and sulphurhexafluoride, in the calibrating gas is of the same order as the volume of the gases to be detected, so that the parameters can be controlled during calibration.

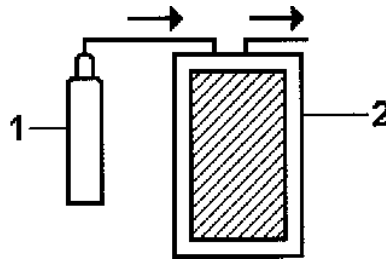
C.4.5 Measurement of the gas leakage

The sensitivity of a thermal conductivity detector is normally insufficient to analyse the gas specimen quantitatively in a direct way, so the following steps for concentration should be performed:

- concentration in the container by standing time: interruption of purging and closing of the container;
- concentration in the cooling trap by cooling: adsorption of the gas specimen, or of the calibrating gas, or of the contaminants in the purging gas, with suitable adsorbents at liquid nitrogen temperature.

The measurement consists of six steps:

Step 1: Purge (see Figure C.4) the volume between test specimen and container with purging gas.



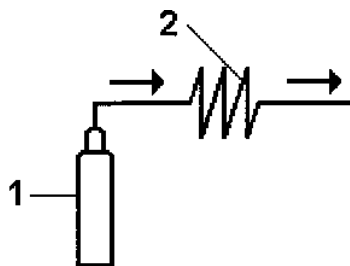
Key

- 1 Purging gas (no. 2 in Figure C.3)
- 2 Container with test specimen (no. 9 in Figure C.3)

Figure C.4 – Purging - Valve position a) (see Figure C.3 and Table C.1)

Step 2: Calibrate the detector system by the calibrating gas:

- take a defined amount, e.g. 1 ml, of gas by the calibrating loop (see Figure C.5);
- turn the valves to position (c) of Table C.1;
- enrich the purging gas by cooling (see Figure C.6);
- turn the valves to position (d) of Table C.1;
- drive off, separate and analyse the calibrating gas with the help of the carrier gas (see Figure C.7);
- control the resolving power and peak shape and determine the calibrating factor for each gas.

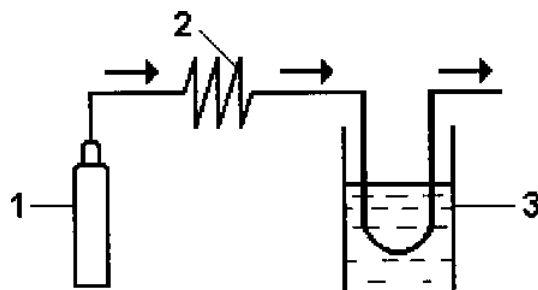


Key

- 1 Calibrating gas (no. 1 in Figure C.3)
- 2 Calibrating loop (no. 8 in Figure C.3)

Figure C.5 – Filling the calibrating loop with calibrating gas C

Valve position b) (see Figure C.3 and Table C.1).

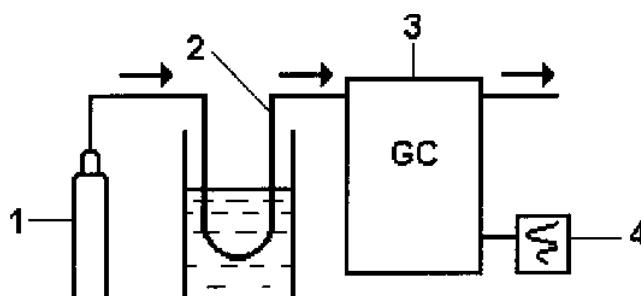


Key

- 1 Purging gas (no. 2 in Figure C.3)
- 2 Calibrating loop (no. 8 in Figure C.3)
- 3 Calibrating gas in cooling trap; cooling trap with liquid nitrogen (no. 7 in Figure C.3)

Figure C.6 – Concentration of calibrating gas by cooling

Valve position c) (see Figure C.3 and Table C.1).



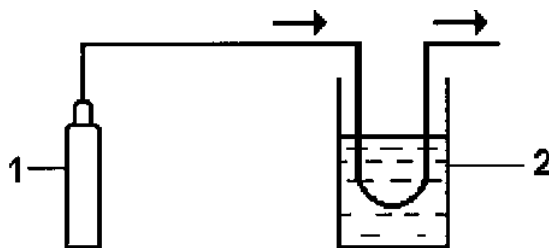
Key

- 1 Carrier gas (no. 5 in Figure C.3)
- 2 Cooling trap with water of (95 ± 5) °C (no. 7 in Figure C.3)
- 3 Gas chromatograph (no. 6 in Figure C.3)
- 4 Integrator (no. 4 in Figure C.3)

Figure C.7 – Driving off, separation and detection of the adsorbed gas

Valve position d) (see Figure C.3 and Table C.1).

Step 3: Inspect the purging gas and the pipe system by cooling, enriching, driving off, separating and detection of the contaminants in the purging gas (see Figure C.8). Gas flow duration during this inspection is similar as for gas leakage measurement.



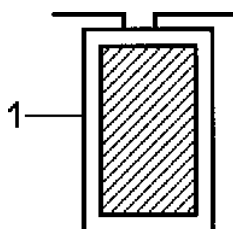
Key

- 1 Purging gas (no. 2 in Figure C.3)
- 2 Cooling trap with liquid nitrogen (no. 7 in Figure C.3)

Figure C.8 – Concentration of contaminants by cooling

Valve position e) (see Figure C.3 and Table C.1).

Step 4: Concentrate the gas specimen by standing time (see Figure C.9), purge the gas specimen with purging gas and concentrate it by cooling (see Figure C.10), followed again by purging (see Figure C.4).

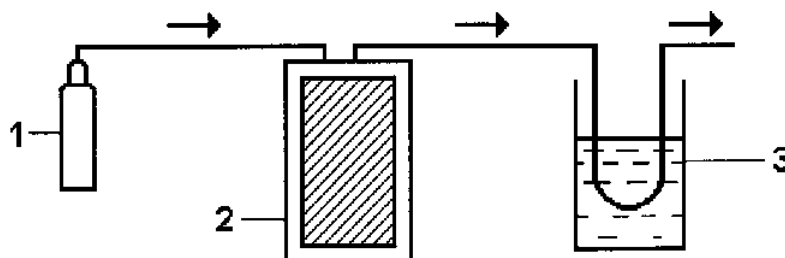


Key

- 1 Container with test specimen (no. 9 in Figure C.3)

Figure 9 – Concentration of gas specimen by standing time

Valve position f) (see Figure C.3 and Table C.1).



Key

- 1 Purging gas (no. 2 in Figure C.3)
- 2 Container with test specimen (no. 9 in Figure C.3)
- 3 Cooling trap with liquid nitrogen (no. 7 in Figure C.3)

Figure C.10 – Concentration of gas specimen by cooling

Valve position g) (see Figure C.3 and Table C.1)

Step 5: Drive out the gas specimen by warming the cooling trap, separation and detection (see Figure C.7). Evaluate quantitatively the gas leakage rate by following steps 2 and 3.

Step 6: Repeat steps 4 and 5 until a sufficiently constant values, is reached. In addition repeat steps 2 and 3 daily.

Sufficient constancy is reached when the standard deviation over the last four measurements, which in the case of natural gases such as the components of air are made with at least one day between them, is less than $0,25 \mu\text{g/h}$, and with at least one measurement being higher than the one immediately preceding it (see Figure C.11).

Conditions for measuring gas leakage rate:

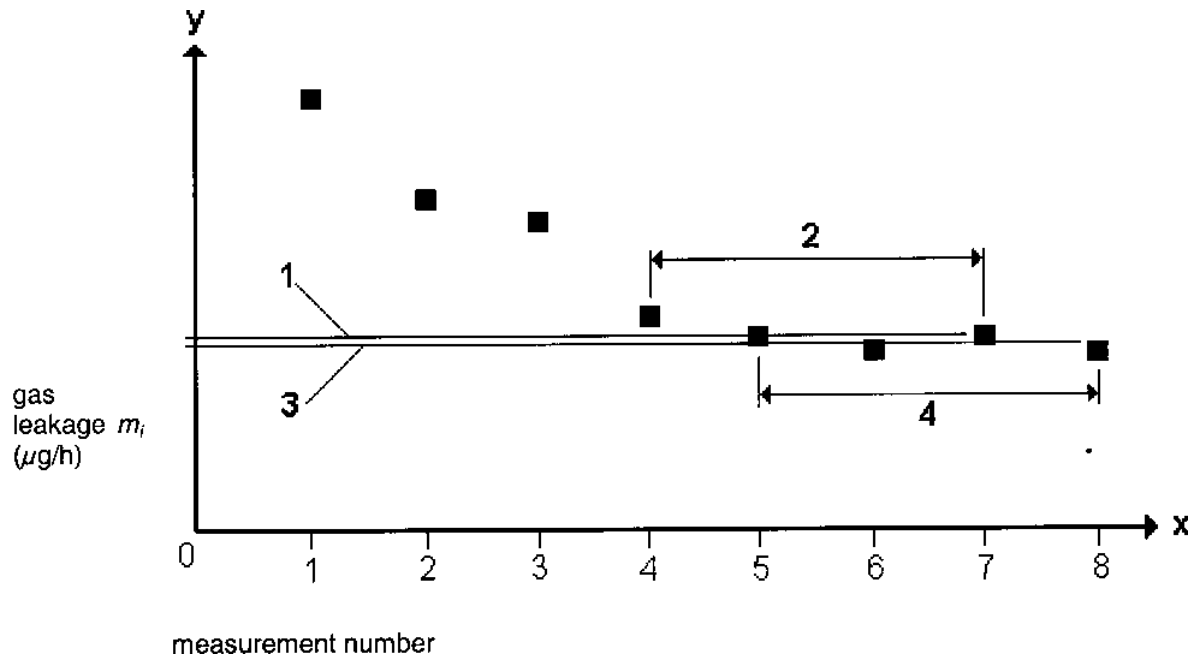
- standing time: up to 20 h;
- cooling time: 30 min;
- purging time: 5 h up to 3 days;
- purging gas flow: about 50 ml/min for purging; 100 ml/min for enriching by cooling.

C.4.6 Blank test

Control periodically the tightness of the system by a blank test. Install a plate of glass with approximately the same dimensions as the test specimen in the container, and measure as described under C.4.5.

C.4.7 Result

Evaluate the gas leakage rate m_i , in $\mu\text{g/h}$, from the measured volume, $\mu\text{l/h}$, in relation to temperature and pressure.



Key

- 1 Mean value of measurements 4 to 7; standard deviation less than $0,25 \mu\text{g/h}$
- 2 Measurements 4 to 7
- 3 mean value of measurements 5 to 8; standard deviation less than $0,25 \mu\text{g/h}$
- 4 measurement 5 to 8

Figure C.11 – Either the measurements 4 to 7 and measurements 5 to 8 is acceptable

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- [2] prEN 13022, *Glass in Building - Structural sealant glazing.*
- [3] ETAG 002, *Guideline for European Technical Approvals for Structural sealant glazing systems.*

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