

# Non-destructive testing — Leak testing — Calibration of reference leaks for gases

The European Standard EN 13192:2001 has the status of a  
British Standard

ICS 19.100

## National foreword

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The UK participation in its preparation was entrusted to Technical Committee WEE/46, Non-destructive testing, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
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English version

## Non-destructive testing — Leak testing — Calibration of reference leaks for gases

Essais non-destructifs — Contrôle d'étanchéité —  
Étalonnage des fuites de référence des gaz

Zerstörungsfreie Prüfung — Dichtheitsprüfung —  
Kalibrieren von Referenzlecks für Gase

This European Standard was approved by CEN on 10 October 2001.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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## Foreword

This European Standard has been prepared by Technical Committee CEN/TC 138, Non-destructive testing, the Secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by May 2002, and conflicting national standards shall be withdrawn at the latest by May 2002.

This European Standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association. This European Standard is considered to be a supporting standard to those application and product standards which in themselves support an essential safety requirement of a New Approach Directive and which make normative reference to this European Standard.

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, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

## 1 Scope

This draft European Standard specifies the calibration of those leaks that are used for the adjustment of leak detectors for the determination of leakage rate in everyday use. The preferred calibration method in this case is a comparison with a standard leak. In this way the leaks used for routine use become traceable to a primary standard as the ISO 9000 series of standards require.

The comparison procedures are preferably applicable to helium leaks, because this test gas can be selectively measured by a mass spectrometer leak detector (MSLD) (the definition of MLSD is given in EN 1330-8).

Calibration by comparison (see methods A and B below) with known standard leaks is easily possible for leaks with reservoir and leakage rates below  $10^{-7} \text{ Pa}\cdot\text{m}^3/\text{s}$ .

From  $10^{-7} \text{ Pa}\cdot\text{m}^3/\text{s}$  to  $10^{-4} \text{ Pa}\cdot\text{m}^3/\text{s}$  no leaks reliable enough to be used as transfer standard exist. Leaks in this range can only be calibrated by measurement of flow in a calibrated capillary tube (see method C below).

Leakage rates greater than  $10^{-4} \text{ Pa}\cdot\text{m}^3/\text{s}$  can be measured by flow meters calibrated against primary national standards.

## 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 in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1330-8, *Non-destructive testing — Terminology — Part 8: Terms used in leak tightness testing*.

EN 13625, *Non-destructive testing — Leak test — Guide to the selection of instrumentation for the measurement of gas leakage*.

## 3 Terms and definitions

For the purposes of this European Standard, the terms and definitions given in EN 1330-8 and the following apply.

### 3.1

#### **unknown leak**

leak having a stable and repeatable leakage rate of known order of magnitude that can be determined by calibration

### 3.2

#### **calibration of a reference leak**

set of operations which establish, under specified conditions, the relationship between leakage rate values represented by an unknown leak and the corresponding known values of the leakage rate by general definition in: "International vocabulary of basic and general terms in metrology"

NOTE 1 In the case of calibration by comparison, the known values of the leakage rate are represented by a standard leak.

NOTE 2 Normally, the result of a calibration is given as the leakage rate value for the reference leak.

For proper usage of the different definitions of leakage rate, the following should be carefully considered:

In leak detection, leakage rates are commonly given in units of pV-throughput ( $\text{Pa}\cdot\text{m}^3/\text{s}$ ,  $\text{mbar l/s}$ ). These are only a precise measure of gas flow if the temperature is given and kept constant.

Flow units such as mass flow ( $\text{g/y}$ ) or molar flow ( $\text{mol/s}$ ) are sometimes used to overcome this problem.

## 4 Classification of leaks

### 4.1 Permeation leak

This type of leak is normally made with a tracer gas reservoir. It has the best long-term stability but an appreciable temperature coefficient (approximately 3,5 %/K). Typical leakage rates are in the range from  $10^{-10}$  Pa·m<sup>3</sup>/s to  $10^{-4}$  Pa·m<sup>3</sup>/s.

### 4.2 Conductance leaks

#### 4.2.1 Capillary leak

This type of leak is available with or without a tracer gas reservoir. It has a low temperature coefficient (approximately 0,3 %/K) but easily blocks if not handled with care. Typical leakage rates are greater than  $10^{-7}$  Pa·m<sup>3</sup>/s.

#### 4.2.2 Aperture leak (orifice)

Orifice leaks are seldom used in practice, as they are difficult to manufacture and even more prone to blocking than capillaries.

#### 4.2.3 Compressed powder leak

This type of leak uses metal powder compressed into a tube. They are usually offered without reservoir. They are used for routine check of the sensitivity of leak detectors but they are not stable enough to be used as calibration leaks.

## 5 Apparatus

### 5.1 Mass Spectrometer Leak Detector (MSLD), for methods A and B (see clause 6)

To calibrate a leak by comparison to a known standard according to methods A and B described in clause 6, a mass spectrometer leak detector is necessary as the transfer device. Such a leak detector shall fulfil the minimum requirements for the measurement of leakage rate, laid down in EN 13625.

The test port of the leak detector shall be equipped with an inlet system consisting of a set of ports with valves (preferably all metal) to couple the standard leak and the unknown leaks to the detection system and to shut off the leaks individually.

The leak tightness of the inlet system shall be checked to a suitable level before a calibration is performed so that ambient tracer (e.g. helium or ambient atmospheric air) will not affect the measurement.

### 5.2 Capillary measurement tube equipment for method C (see clause 7)

To calibrate a leak by measurement of capillary flow according to method C described in clause 7, a calibrated glass capillary tube (preferably with a suitable vent valve at one end, see Figure 1) is necessary.

An indicator fluid (normally water with some surfactant added or special oils) is used to produce the measurement slug in the capillary.

To measure the time of slug movement, a timer or stopwatch will be needed. Instruments based on the timed movements of a film in a tube are also available, e.g. a bubble flow meter.

As conductance leaks normally have no tracer gas reservoir, a separate tracer gas supply is needed or calibration may be performed with filtered atmospheric air.

## 6 Calibration by comparison (methods A and B)

There are two ways of calibrating leaks by comparison with known standard leaks. Both methods require the knowledge of the order of magnitude of the leakage rate to be measured. The methods differ in using one or two standard leaks, resulting in different uncertainties of measurement. In the following, the two methods are designated as A and B:

Method A: Comparison to one standard leak normally with a leakage rate of the same order of magnitude

Method B: Comparison to two standard leaks with leakage rates normally lying on either side of the unknown leakage rate

Method A is most suitable for use on site as only one standard leak is used. It is generally applicable but is most reliable when the leakage rate of the unknown is close to that of the standard leak. This is because the measurement uncertainty is directly dependent on the linearity of the leak detector in use. (See 8.1.2.2). As the linearity error cannot be measured independently, it has to be estimated. To keep the linearity error small, the operating characteristics of leak detector should not change during calibration (e.g. automatic ranging should be disabled).

For more precise calibrations, where a definite measure of uncertainty is required or if a standard leak with a leakage rate close to the unknown is not available method B should be used. By the use of two reference leaks, the non-linearity of the leak detector is accounted for (see 8.1.2.3).

### 6.1 Preparation of leaks and apparatus

#### 6.1.1 Warm-up of leak detector

The leak detector used as a transfer device shall be set up according to the manufacturer's manual. The warm-up time shall be at least 2 h.

#### 6.1.2 Temperature accommodation

The unknown leak and the standard leak(s) for the comparison shall be stored in the same room where the test is to be carried out for at least 12 h to allow for temperature equilibration (an air-conditioned room is not necessary if there are no rapid temperature changes. Because of temperature fluctuations, an air-conditioning system can even increase the measurement uncertainty). The leaks shall be pumped out during the phase of thermal accommodation. After temperature accommodation, to prevent any temperature changes during measurement, thermally insulating hoods (made of plastic foam or similar material) should be put over the leaks.

#### 6.1.3 Connection to the leak detector

The standard and unknown leaks are connected to the inlet system of the MSLD after temperature accommodation and pumped with their valves (if any) open for at least 30 min to remove any tracer gas that may have accumulated in seals or valves. For the calibration of more than one leak, a separate pumping system and set of valves is useful to keep all the leaks pumped until they are measured.

## 6.2 Measurement

### 6.2.1 Set-up

The leak detector is adjusted so that the major of the used leaks gives approximately a full-scale indication. It is important to ensure that the effective pumping speed is not changed during the measurements. If possible, either with the leak detector or in an auxiliary device a long averaging time may be used to decrease the statistical measurement uncertainty. Further a recorder taking data over some time can be used for that purpose. All those measurement instruments should be used in such a way that they give nearly full-scale deflections for the biggest leak.



## 6.2.2 General measurement sequence

Generally, each reading shall be obtained under steady flow conditions constant signal from the MLSD. A sufficient number of readings have to be taken to achieve the lowest possible statistical uncertainty. For this purpose, digital meters may be used and a large number of measurements obtained. In this way a measure of statistic deviation can also be found. The general sequence of measurements is as follows:

- a) zero signal determination: all valves closed;
- b) open standard leak no. 1, wait for steady flow and measure the resulting output signal (method A and B);
- c) close standard leak no. 1;
- d) open standard leak no. 2, wait for steady flow and measure the resulting output signal (only method B);
- e) close standard leak no. 2;
- f) open unknown leaks, wait for steady flow and measure the resulting output signal;
- g) repeat a) to f) at least three times.

NOTE The leak valves should be kept closed for as short a time as possible to prevent extensive helium accumulation resulting in long equilibration time.

## 7 Calibration by direct flow measurement (method C)

This method is only applicable to conductance leaks in the range of  $10^{-6}$  Pa·m<sup>3</sup>/s and greater. Leaks from  $10^{-7}$  Pa·m<sup>3</sup>/s to  $10^{-6}$  Pa·m<sup>3</sup>/s can be calibrated, but with a rather large uncertainty. In that range, if a suitable reference leak is available, methods A or B should be employed to give lower uncertainty. Leakage rates greater than  $10^{-4}$  Pa m<sup>3</sup>/s should be measured with flow meters traceable to a National Standard.

Two types of flow conditions that may be considered are those established by gas flow:

- from over-pressure to atmosphere (see 7.2.1);
- from atmosphere to vacuum (see 7.2.2).

In both cases one has to consider whether air or a specific tracer gas has to be used. The third possible flow condition, pressure to vacuum, cannot be measured with method C (if this is required, a calibration with tracer gas and atmospheric pressure against vacuum has to be made initially and afterwards the pressure dependence shall be measured with a suitable MSLD).

### 7.1 Preparation of leaks and apparatus

#### 7.1.1 Temperature accommodation

The unknown leak and the calibrated capillary shall be stored in the room where the test is to be carried out for at least 12 h to allow for temperature equilibration (an air-conditioned room is not necessary if there are no rapid temperature changes. Because of temperature oscillations, an air-conditioning system can even increase the measurement uncertainty).

#### 7.1.2 Connection of leak to capillary tube

The capillary tube and vent valve shall be cleaned with alcohol and purged with pressurized air to remove any dirt from the surfaces that might disturb the free movement of the liquid slug during measurement. The connection between the leak and the capillary tube shall be made with a thick elastomer connecting hose fitting tightly on both the leak outlet and the vent valve of the capillary. The smaller the unknown leak the more important is it to keep all dead volumes as small as possible to reduce measurement errors.

**7.1.2.1 Pressure to atmosphere**

In this case, the leak inlet is connected to the tracer gas supply and the outlet to that end of the capillary tube where the vent valve is. The capillary tube is open to atmosphere at the other end (see Figure 1).

**7.1.2.2 Atmosphere to vacuum**

In this case, the leak outlet is connected to vacuum side and its inlet to the capillary end with the vent valve. The capillary inlet is left open to atmospheric pressure (see Figure 2).

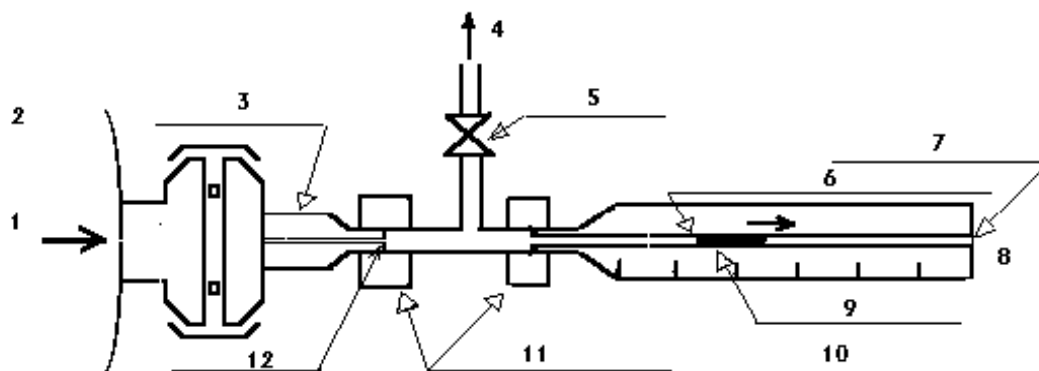
If normalized leakage rates are required, the outlet absolute pressure shall be less than 100 Pa.

**7.2 Measurement**

**7.2.1 Over-pressure to atmosphere**

The measurement is performed according to the following procedure:

- a) dip the open end of the capillary into the indicator fluid to produce a liquid slug with a length of about 1 cm;
- b) draw this slug slowly up the capillary to the other end by carefully pumping via the vent valve (if there is no vent valve, disconnect the capillary, dip the tip into the indicator fluid and replace it);
- c) close the vent valve (if present) and time the movement of the trailing edge of the slug for a convenient distance;
- d) reposition the slug (see above) and repeat step (a) to (c) at least three times. The repeatability of the measurements should be within  $\pm 5\%$ .



### Key

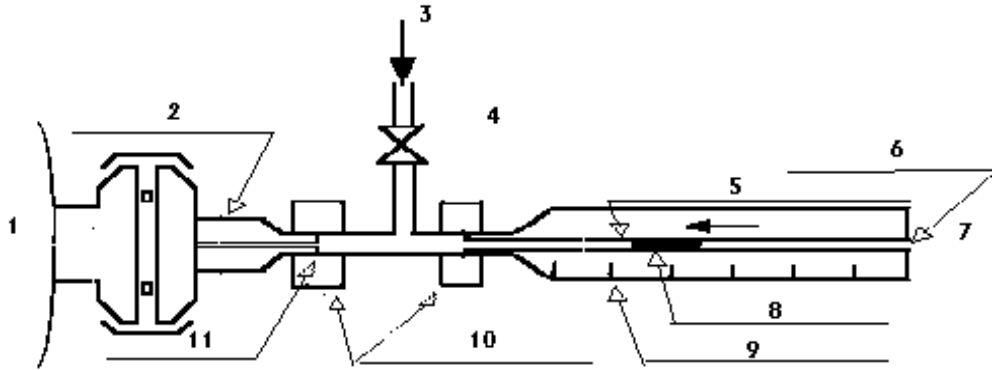
- 1 Pressure
- 2 Tracer gas
- 3 Unknown leak
- 4 Pump to initialize the slug position
- 5 Vent valve
- 6 Read the trailing edge of slug
- 7  $d = 0,1 \text{ mm to } 0,5 \text{ mm}$
- 8 Atmosphere
- 9 Indicating slug
- 10 Calibration marks
- 11 Connecting tubes
- 12 Zero space

**Figure 1 — Set-up for method C measurement: over-pressure to atmosphere**

### 7.2.2 Atmosphere to vacuum

The measurement is performed according to the following procedure:

- a) purge tracer gas through the vent valve, leak and capillary for a minimum of 30 s;
- b) dip the open end of the capillary into the indicator fluid to produce a liquid slug with a length of about 1 cm;
- c) close the vent valve and time the movement of the leading edge of the slug over a convenient distance. It is essential that the vent valve is opened before the slug is drawn into the leak;
- d) reposition the slug to the end of the capillary again, using tracer gas pressure via the vent valve;
- e) repeat step (a) to (c) at least three times. The repeatability of the measurements should be within  $\pm 5 \%$ .



**Key**

- 1 Vacuum
- 2 Unknown leak
- 3 Purge with tracer gas (before measurement)
- 4 Vent valve
- 5 Read the leading edge of slug
- 6  $d = 0,1 \text{ mm to } 0,5 \text{ mm}$
- 7 Atmosphere
- 8 Indicating slug
- 9 Calibration marks
- 10 Connecting tubes
- 11 Zero space

**Figure 2 — Set-up for method C measurement: atmosphere to vacuum**

## 8 Results

### 8.1 Evaluation for methods A and B (comparison)

#### 8.1.1 Determination of leakage rate

##### 8.1.1.1 Method A: Result of comparison to one standard leak

The following formula is used to calculate the unknown leakage rate  $Q_u$  from the reading  $R_{std}$  of the standard leak with leakage rate  $Q_{std}$  and the reading  $R_u$  of the unknown leak:

$$Q_u = Q_{std} \cdot \left[ \frac{R_u (1 + \alpha_{std} \cdot \Delta T_{std})}{R_{std} (1 + \alpha_u \cdot \Delta T_u)} \right] \quad (1a)$$

where

$Q_{std}$ ,  $Q_u$ , are the leakage rates of the standard and unknown leak respectively;

$R_{std}$ ,  $R_u$  the readings of the standard and unknown leak respectively;

$\alpha_{std}$ ,  $\alpha_u$  are the temperature coefficients of the standard and unknown leak respectively;

$\Delta T_{std}$ ,  $\Delta T_u$  are the departures of the temperature of the leaks from the reference temperature of the standard and unknown leak respectively.

NOTE 1 The readings ( $R_{std}$  and  $R_u$ ) are obtained from the leak detector display as the difference of the output signals with opened and closed leak.

NOTE 2 The temperature coefficient of the standard leak will normally be stated. If the temperature coefficient of the unknown leak is not given it can be assumed that for a quartz permeation leak it is approximately 3,5 %/K and for conductance type leaks 0,3 %/K.

This formula simplifies considerably, if the temperature coefficients and the temperatures of all leaks are equal:

$$Q_u = Q_{std} \frac{R_u}{R_{std}} \quad (1b)$$

The readings can be in any consistent units, as only ratios are considered.

##### 8.1.1.2 Method B: Result of comparison to two standard leaks

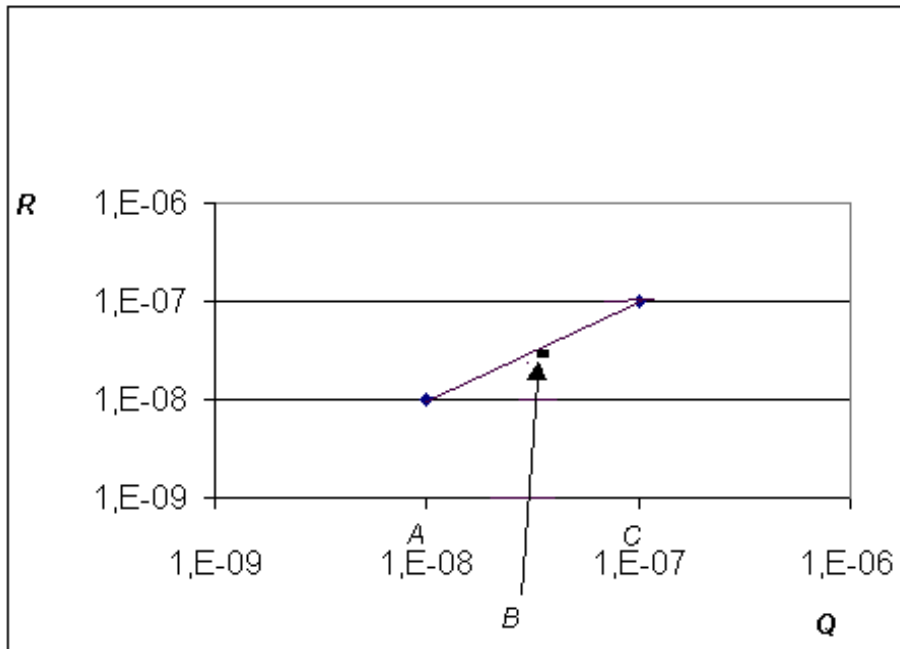
To keep this procedure practical, only the case of equal temperature coefficients and temperatures of all leaks is considered. In this case the following simplified formula holds:

$$Q_u = (Q_2 - Q_1) \left( \frac{R_u - R_1}{R_2 - R_1} \right) + Q_1 \quad (2)$$

where

$Q_u$ ,  $Q_1$ ,  $Q_2$  are leakage rates of the unknown leak and the standard leaks 1 and 2 respectively;

$R_u$ ,  $R_1$ ,  $R_2$  are readings for the unknown leak and the standard leaks 1 and 2 respectively.



**Key**

- R Reading
- Q Leakage rate
- A Reference leak 1
- B Unknown leak
- C Reference leak 2

NOTE  $Q_1$  is the leak with the smaller rate,  $Q_2$  the leak with the larger rate.  $Q_w$ , the leakage rate of the unknown leak to be calibrated lies between these two known leaks.

**Figure 3 — Two points calibration of a leak**

**8.1.2 Calculation of measurement uncertainty**

**8.1.2.1 General**

The following formulae give the total uncertainty of measurement if the uncertainties of the readings and of the standard leaks are given. All uncertainties are derived for equal and constant temperatures of the leaks in order to keep the formula practical and following the EA-4/02.

### 8.1.2.2 Method A: Uncertainty of comparison to one standard leak

The standard leak, being a reference, shall be provided with a calibration certificate in which in addition to the leak value the “expanded uncertainty”  $U(Q_{\text{std}})$  and the coverage factor are given. The standard deviation of the readings  $s(R_{\text{std}})$  and  $s(R_{\text{u}})$  are calculated in the usual manner.

The standard uncertainty associated with the leak detector is given by the linear sum of the relative standard deviation of the two readings (of standard leak and unknown leak respectively) and a term to account for any non-linearity:

$$\frac{u(R)}{R} = \frac{s(R_{\text{std}})}{R_{\text{st}}} + \frac{s(R_{\text{u}})}{R_{\text{u}}} + \frac{s(L)}{L} \quad (3a)$$

where

$s(R_{\text{std}})$  is the standard deviation of readings of the standard leak;

$s(R_{\text{u}})$  is the standard deviation of readings of the unknown leak;

$s(L)/L$  is a relative measure of non-linearity (which has to be estimated) of the leak detector (= mean percentage deviation from a straight line through the calibration points of the leakage rates for the standard and unknown leaks).

NOTE The above formula takes into account that the readings of the same leak detectors are correlated.

In the case of equal  $\alpha$ s and equal constant temperature of all leaks the relative standard uncertainty of measurement of the unknown leakage rate  $Q_{\text{u}}$  is then given by:

$$\frac{u(Q_{\text{u}})}{Q_{\text{u}}} = \sqrt{\left(\frac{u(Q_{\text{std}})}{Q_{\text{std}}}\right)^2 + \left(\frac{u(R)}{R}\right)^2} \quad (3b)$$

where

$u(Q_{\text{std}})$  is the standard uncertainty of the (known) standard leak;

$u(R)$  is the standard uncertainty of the readings from equation (1a).

NOTE In this formula, the standard uncertainty of the known standard leak  $u(Q_{\text{std}})$  has to be calculated from the calibration certificate by dividing the given expanded uncertainty  $U(Q_{\text{std}})$  by the coverage factor  $k$  (which normally should be equal to 2).

$$u(Q_{\text{std}}) = \frac{U(Q_{\text{std}})}{k}$$

In the report (see clause 9) the expanded uncertainty  $U(Q_{\text{u}})/Q_{\text{u}}$  (with coverage factor 2) has to be stated. The expanded uncertainty is given by:

$$\frac{U(Q_{\text{u}})}{Q_{\text{u}}} = 2 \sqrt{\left(\frac{u(Q_{\text{std}})}{Q_{\text{std}}}\right)^2 + \left(\frac{u(R)}{R}\right)^2} \quad (3c)$$

**8.1.2.3 Method B: Uncertainty of comparison to two standard leaks**

The standard leaks, being references, shall be provided with calibration certificates in which in addition to the leak value the “expanded uncertainty”  $U(Q_{std})$  and the coverage factor are given. The standard uncertainties of the known standard leaks  $u(Q_{std1})$  and  $u(Q_{std2})$  then have to be calculated by dividing the given expanded uncertainties  $U(Q_{std})$  by the coverage factor  $k$  (which normally should be equal 2).

The standard uncertainties of the readings  $u(R_{std1})$ ,  $u(R_{std2})$  and  $u(R_u)$  are calculated as the respective standard deviation of several readings for each leak.

The combined expanded uncertainty (with coverage factor  $k = 2$ ) is then given by linear addition of the individual standard uncertainties multiplied by the coverage factor of 2. The following formula gives the result for the total relative expanded uncertainty  $U(Q_u)/Q_u$  of a leak calibration with two standard leaks:

$$\frac{U(Q_u)}{Q_u} = \frac{2}{Q_u} \left\{ \left[ \left( Q_1 - Q_2 \right) \frac{(R_u - R_2)}{(R_2 - R_1)^2} \right] u(R_1) + \left[ \left( Q_1 - Q_2 \right) \frac{(R_u - R_1)}{(R_2 - R_1)^2} \right] u(R_2) \right. \\ \left. + \left[ \frac{(Q_1 - Q_2)}{(R_2 - R_1)} \right] u(R_u) + \left[ \frac{(R_u - R_1)}{(R_2 - R_1)} + 1 \right] u(Q_1) + \left[ \frac{(R_u - R_1)}{(R_2 - R_1)} \right] u(Q_2) \right\} \quad (4)$$

where

$Q_u$  is the leakage rate of the unknown leak according to equation (2);

$Q_1, Q_2$  are the leakage rates of the (known) standard leaks  $Q_1, Q_2$ ;

$R_1, R_2$  are the readings of the two standard leaks;

$u(Q_1), u(Q_2)$  are the standard uncertainties of the two standard leaks;

$u(R_1), u(R_2)$  are the standard uncertainties of the readings of the two standard leaks.

NOTE Linear instead of quadratic addition of terms is chosen since both the leak detector readings and the standard leakage rates are correlated quantities. This is a conservative estimation of uncertainty and may lead to slightly greater uncertainty statements as if the complete covariance matrix had been evaluated.



## 8.2 Evaluation for Method C (direct flow measurement)

### 8.2.1 Determination of leakage rate

Over-pressure to atmosphere measurements using the capillary method has to be corrected for the vapour pressure of the indicating liquid. With water as the liquid (vapour pressure approximately 3 kPa at the ambient temperature) the systematic error means a leak indication that is approximately 3 % too high because of evaporating liquid.

The unknown leakage rate is given by:

$$Q_{pV} = 0,97 \cdot 10^{-6} \frac{p_{\text{atm}} \cdot l \cdot k}{t_{\text{average}}} \quad (\text{over-pressure to atmosphere}) \quad (5a)$$

$$Q_{pV} = 1,03 \cdot 10^{-6} \frac{p_{\text{atm}} \cdot l \cdot k}{t_{\text{average}}} \quad (\text{atmosphere to vacuum}) \quad (5b)$$

where

$Q_{pV}$  is the leakage rate in pascals cubic metres per second;

$l$  is the distance the slug has moved in the capillary in centimetres;

$k$  is the capillary constant (volume per length) in cubic centimetres per centimetre;

$t_{\text{average}}$  is the average time for travelling the distance  $l$  in seconds;

$p_{\text{atm}}$  is the actual atmospheric pressure in pascals.

### 8.2.2 Calculation of measurement uncertainty

In the first step, the variances of distance  $l$ , capillary constant  $k$  and time measurement  $t$  have to be determined. The variances of  $l$  and  $k$  are given by:

$$(S_l)^2 = 1/3 (\delta l)^2 \quad (S_k)^2 = 1/3 (\delta k)^2 \quad (6a)$$

where the uncertainty of the distance  $\delta l$  is given by the parallax error of readings, the uncertainty of the capillary constant  $\delta k$  is given by the manufacturer. The variance of the time measurement is determined directly from the number of measurements.

The leakage rate measurement uncertainty for both kinds of capillary measurements is given by the sum of the variances of the measured or given quantities multiplied by 2 according to EA-4/02 rules. This leads to the formula:

$$U_{Q_{pV}} / Q_{pV} = 2 \sqrt{(S_l)^2 + (S_k)^2 + (S_t)^2} \quad (6b)$$

## 9 Report

The calibration report of a reference leak shall contain the following information:

- type of leak (permeation or conductance);
- type of tracer gas;
- measured leakage rate ( $\text{Pa}^{-3}/\text{s}$ );
- inlet and outlet pressure;
- uncertainty of measurement (result of calculation);
- calculation of uncertainty of measurement (formulae 3b, 4, 6b, inclusive of values);
- linear temperature coefficient of leakage rate ( $\%/K$ );
- reference temperature ( $^{\circ}\text{C}$ );
- expected depletion of tracer gas reservoir, if any ( $\%/y$ ) (see annex A);
- date of calibration;
- method used, according to this standard (A, B or C);
- unique identification for the reference leak;
- signature of the tester;
- name and location of the calibration site;
- leak detector reference, if applicable.

## 10 Labelling of reference leaks

The label of a calibrating leak shall contain all the information stated above under clause 9 and marked. The operation and storage temperature ranges should be stated to assure proper handling of the leak.

## 11 Handling of reference leaks

To prevent breakage of the leak element, which is often made from glass or quartz, calibrated leaks should always be handled with care. If damage is suspected, the leak shall be re-checked for correct leakage rate.

### 11.1 Permeation leaks (normally with reservoir fitted the leak outlet)

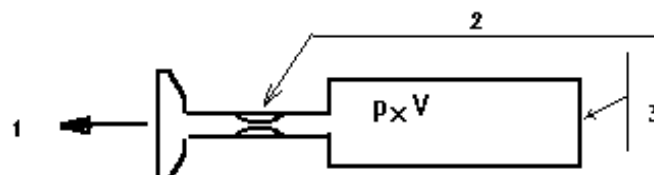
For permeation leaks, which normally have very small leakage rates, it is most important during storage to keep open any valve in front of the leak. This prevents any accumulation of helium either in a dead space in front of the closed valve or in the elastomeric seal of the valve.

### 11.2 Conductance leaks (normally without reservoir)

Conductance leaks have to be kept in an environment free of dust particle and moisture which is best achieved by closing their outlet by either a flange or all-metal valve. However this will not prevent tracer gas loss over extended periods of time but will keep away dirt particles and moisture during storage.

## Annex A (informative)

### Calculation of leakage rate decrease due to tracer gas depletion in the reservoir



#### Key

- 1 Tracer gas flow  $Q_x$
- 2 Leak with conductance  $C_x$
- 3 Reservoir with volume  $V$ , partial pressure of tracer gas  $P_x$

$$Q_x = V \frac{dp_x / P_x}{dt} \quad (\text{A.1})$$

**Figure A.1 — Relevant parameters for the calculation of tracer gas depletion**

The relative pressure decrease with time  $D_{qx}$  is given by the following equation:

$$D_{qx} = \frac{dp_x / p_x}{dt} = \frac{Q_{0,x}}{P_{0,x} V} \quad (\text{A.2})$$

where:

- $D_{qx}$  is the pressure decrease, in reciprocal seconds;
- $Q_{0,x}$  is the nominal leakage rate (with pressure  $p_{0,x}$ ), in pascals cubic metres per second;
- $P_{0,x}$  is the filling (initial) pressure of the tracer gas in the reservoir, in pascals;
- $V$  is the volume of reservoir, in cubic metres;
- $p_x$  is the actual pressure of the tracer gas in the reservoir, in pascals;
- $t$  is the time, in seconds.

NOTE To obtain  $D_{qx}$  in % per year, multiply by  $3,15 \times 10^9$ .

## Bibliography

- [1] EA-4/02 <sup>1)</sup> *Guidelines for the Expression of the Uncertainty of Measurement in Calibration (previously EAL-R2)*

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<sup>1)</sup> The document EA-4/02 can be found on the web page of the European Accreditation Organization with the address <http://www.european-accreditation.org/documents.html>



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