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

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

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 135, *Non-destructive testing*, Subcommittee SC 6, *Leak testing*.

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

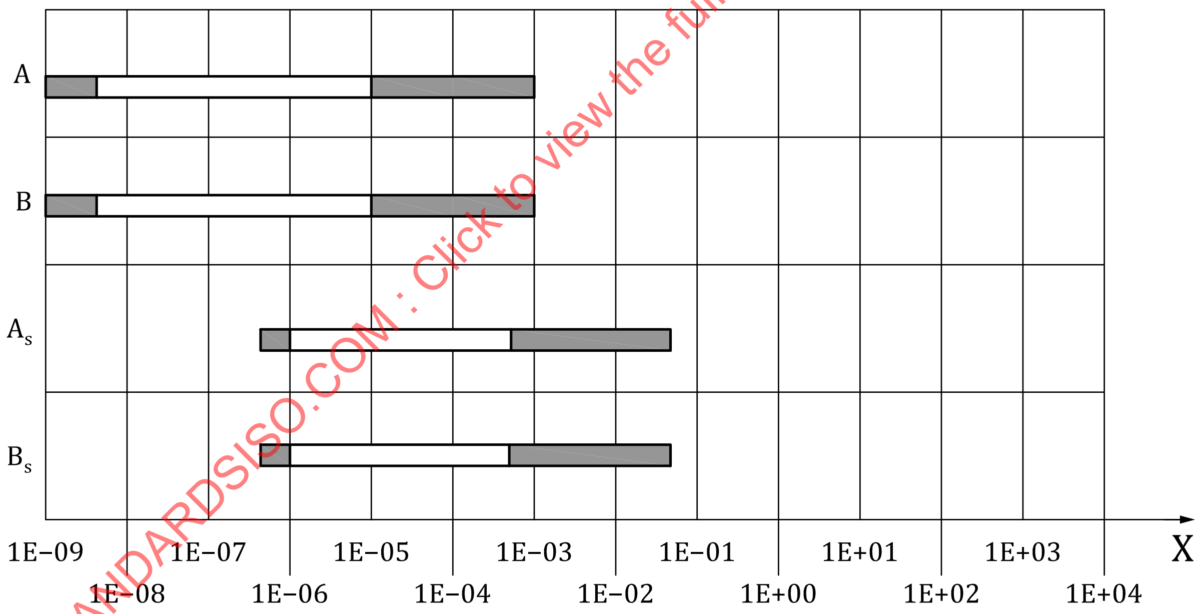
1 Scope

This document specifies the calibration of those leaks that are used for the adjustment of leak detectors for the determination of leakage rate in everyday use. One type of calibration method is a comparison with a reference leak. In this way, the leaks used for routine use become traceable to a primary standard. In other calibration methods, the value of vapour pressure was measured directly or calculated over a known volume.

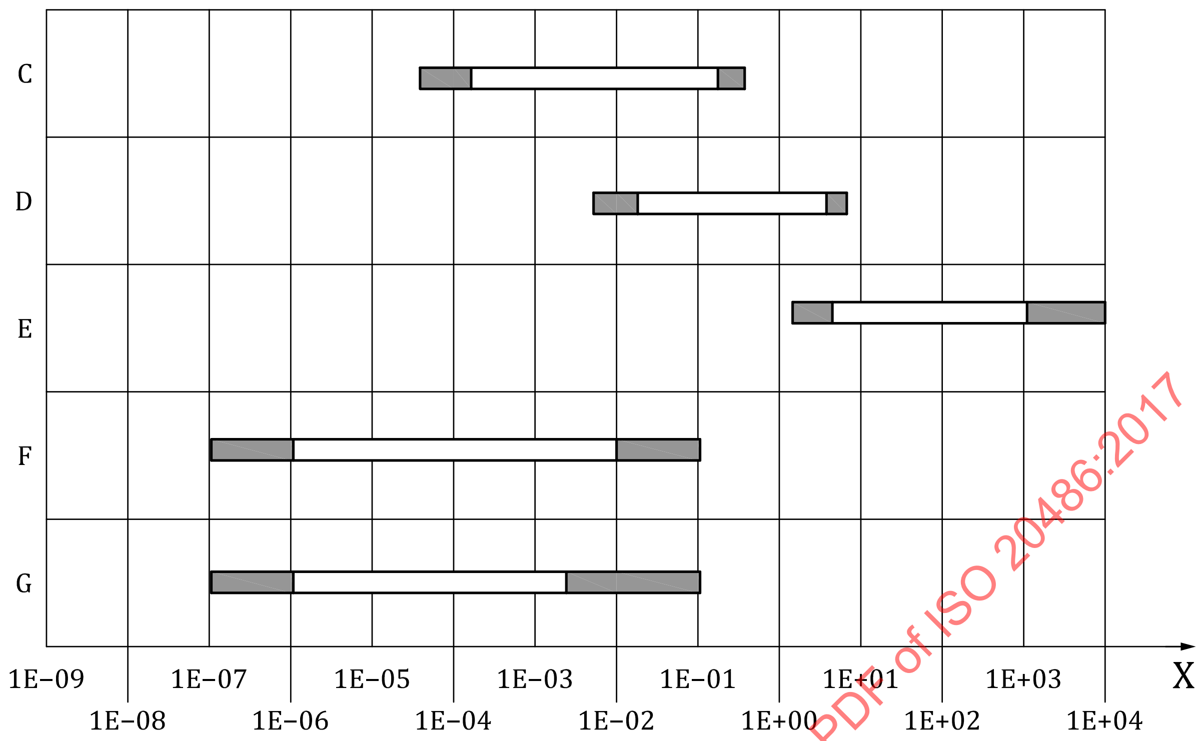
The comparison procedures are preferably applicable to helium leaks, because this test gas can be selectively measured by a mass spectrometer leak detector (MSLD) (the definition of MSLD is given in ISO 20484).

Calibration by comparison (see methods A, A_S, B and B_S below) with known reference leaks is easily possible for leaks with reservoir and leakage rates below 10^{-7} Pa·m³/s.

Figure 1 gives an overview of the different recommended calibration methods.



a) Calibration by comparison



b) Calibration by direct measurement

Key

X	leakage rate in Pa·m^3/s	C	Method C
A	Method A	D	Method D
B	Method B	E	Method E
A_S	Method A_S	F	Method F
B_S	Method B_S	G	Method G
(white box)	normal range	(grey box)	possible range

Figure 1 — Calibration ranges

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 20484, *Non-destructive testing — Leak testing — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20484 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

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
reference leak
calibrated leak which may be used to calibrate another leak

Note 1 to entry: The uncertainty of the reference leak is lower than the required uncertainty of the leak to be calibrated.

3.3
calibration
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

Note 1 to entry: In the case of calibration by comparison, the known values of the leakage rate are represented by a reference leak.

Note 2 to entry: Normally, the result of a calibration is given as the leakage rate value for the reference leak with a standard uncertainty.

3.4
nominal leakage rate
leakage rate of a leak calculated for specified reference conditions

Note 1 to entry: In leak detection, leakage rates are commonly given in units of pV-throughput (Pa·m^3/s, mbar l/s, Std cm^3/min). These are only a precise measure of gas flow if the temperature is given and kept constant. Flow units such as mass flow (g/y) or molar flow (mol/s) are sometimes used to overcome this problem.

4 Nominal leakage rates

Calibrated leaks are only comparable under the same reference conditions. Nominal leakage rates shall be used for comparison. Recommended reference conditions are:

— Ambient temperature: 20 °C

— Atmospheric exhaust pressure: 1 000 mbar

— Vacuum exhaust pressure: < 100 mbar

The reference inlet pressure is given by the leak reservoir pressure or the application requirement.

5 Classification of leaks

5.1 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^3/s to 10^{-4} Pa·m^3/s.

5.2 Conductance leaks

5.2.1 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^3/s.

5.2.2 Aperture leak (orifice)

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

NOTE Critical flow orifices are a form of aperture leak that is commonly found in industry, but are out of the scope of this document.

5.2.3 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 calibrated leaks. Their suitability depends on how well controlled the storage and operating conditions are, and on the required uncertainty.

6 Calibration by comparison

6.1 Methods A, A_S, B and B_S

There are two ways of calibrating leaks by comparison with known reference 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 reference leaks, resulting in different uncertainties of measurement. In the following, the two methods are designated as A and B:

— Method A: Comparison to one reference leak normally with a leakage rate of the same order of magnitude, calibration with vacuum method.

— Method A_S: Comparison to one reference leak normally with a leakage rate of the same order of magnitude, calibration with sniffing method.

— Method B: Comparison to two reference leaks with leakage rates normally lying on either side of the unknown leakage rate, calibration with vacuum method.

— Method B_S: Comparison to two reference leaks with leakage rates normally lying on either side of the unknown leakage rate. Calibration with sniffing method.

Method A is most suitable for use on site as only one reference leak is used. It is generally applicable but is most reliable when the leakage rate of the unknown is close to that of the reference leak. This is because the measurement uncertainty is directly dependent on the linearity of the leak detector in use. As the linearity error cannot be measured independently, it needs 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 more reliable measure of uncertainty is required or if a reference 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.

6.2 Applicability of comparison methods

Since comparison of leaks is not a fundamental measurement method, it relies on the stability of the transfer device and cleanliness of the ambient gas atmosphere. Moreover, the temperature dependence of the reference and unknown leaks shall be taken into account.

The most stable and clean conditions are achieved for leaks with exhaust into vacuum and a mass spectrometer leak detector as transfer device measuring the partial pressure generated by the leaks in vacuum. Under these conditions, all interfering background gases are reduced to a minimum so that the zero point of the transfer device is defined and stable.

For leaks with exhaust into the atmosphere and measurement by sniffing gas, more conditions shall be controlled. These are:

— the background level of tracer gas shall be as low as possible and as stable as possible;

— the total gas flow rate of the sniffer shall be high enough to take up the total tracer gas flow out of the leak;

— the aspiration of the sniffer (the coupling to the leak exhaust) shall be of suitable geometry to make sure that the atmospheric gas flow across the leak exhaust takes up the whole tracer gas flow from the leak opening.

As a consequence, the measurement uncertainty is appreciably higher for sniffer leaks than for vacuum leaks.

Methods by comparison are therefore applicable but not preferable for the calibration of sniffer leaks (with exhaust to atmosphere).

6.3 Preparation of leaks and apparatus

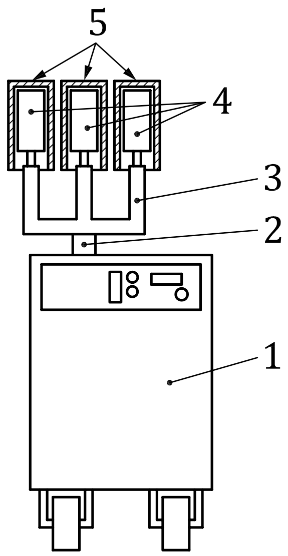
6.3.1 Leak detector

The leak detector (LD) 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.

6.3.2 Connection to the leak detector

The reference and unknown leaks are connected to the leak detector used as the transfer instrument. The connection shall be kept continuously until the measurement is completed. This includes thermal accommodation[1].

In the case of vacuum leaks, they are connected to the inlet flange and pumped with their valves (if any) open for at least 30 min to remove any tracer gas that can 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.

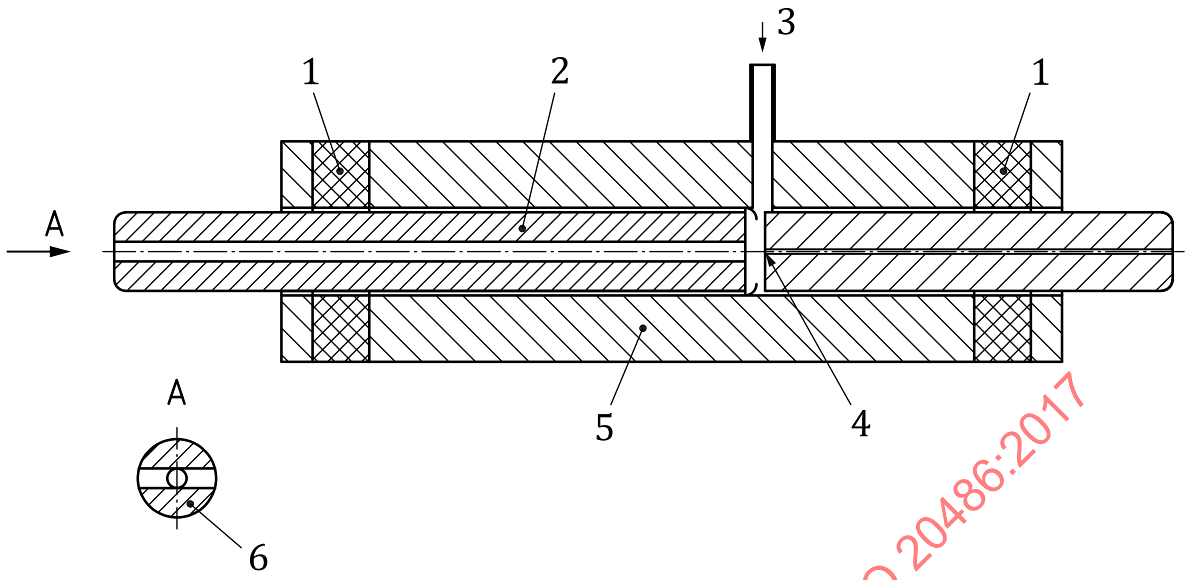


Key

1 transfer device (leak detector)
2 test port
3 rig
4 leaks to be calibrated and reference leak
5 hoods for thermal stability

Figure 2 — Coupling of leaks to the leak detector

In the case of sniffer leaks, the connection to the leak detector sniffing tip is made by an adapter which makes a tight connection to the leak outlet and enables atmospheric air to be continuously sucked across the leak exhaust via an air inlet (see Key item 3 in Figure 2), so that the whole leak gas flow is taken up by the sniffer tip. The air inlet opening shall not throttle the free flow of air to maintain atmospheric pressure in front of the sniffer tip. See Figure 3.



Key

1 gasket
2 sniffer tip
3 air inlet
4 test leak with leak opening
5 adapter body with gasket
6 sniffer opening with cross-wise slot

Figure 3 — Example for a coupling adapter for sniffer leaks

6.3.3 Temperature accommodation

The unknown leak and the reference 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). Vacuum leaks, connected to the LD shall be pumped 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.

6.4 Measurement

6.4.1 Set-up

It is important to ensure that the effective pumping speed at the leak detector inlet for vacuum leaks, respectively the sniffer gas flow for overpressure leaks, 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.

All the measurement instruments should be adjusted in such a way that they give nearly full-scale deflections for the biggest leak.

6.4.2 General measurement sequence

Generally, each reading shall be obtained only after the signal of the transfer instrument has stabilized. A sufficient number of readings shall be taken to achieve the lowest possible statistical uncertainty.

This way, a measure of statistical deviation can also be found. The general sequence of measurements is as follows:

a) zero signal determination: all valves closed for vacuum leaks, respectively sniffer tip in pure ambient air for sniffing leaks;

b) connect reference leak No. 1, wait for steady flow and measure the resulting output signal (Method A, A_S, B and B_S);

c) disconnect reference leak No.1;

d) connect reference leak No. 2, wait for steady flow and measure the resulting output signal (only Method B and B_S);

e) disconnect reference leak No. 2;

f) connect unknown leaks, wait for steady flow and measure the resulting output signal;

g) repeat steps a) to f) at least three times.

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

6.5 Evaluation for methods A, A_S, B and B_S (Comparison)

6.5.1 Determination of leakage rate

6.5.1.1 Method A and A_S: Result of comparison to one reference leak

Formula (1a) is used to calculate the unknown leakage rate, Q_u, from the reading, R_{ref}, of the reference leak with leakage rate, Q_{ref}, and the reading, R_u, of the unknown leak:

$$Q_u = Q_{ref} \frac{R_u}{R_{ref}} \tag{1a}$$

This formula is only valid, if the temperature coefficients and the temperatures of all leaks are equal. Otherwise, Formula (1b) shall be used:

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

where

Q_{ref}, Q_u are the leakage rates of the reference and unknown leak, respectively;

R_{ref}, Ru the readings of the reference and unknown leak, respectively;

α_{ref}, α_u are the temperature coefficients of the reference and unknown leak, respectively;

ΔT_{ref}, ΔT_u are the departures of the temperature of the leaks from the reference temperature of the reference and unknown leak, respectively.

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

NOTE 1 The readings (R_{ref} and R_u) are obtained from the leak detector display as the difference of the output signals with leak connected and disconnected (respectively, valve opened and closed).

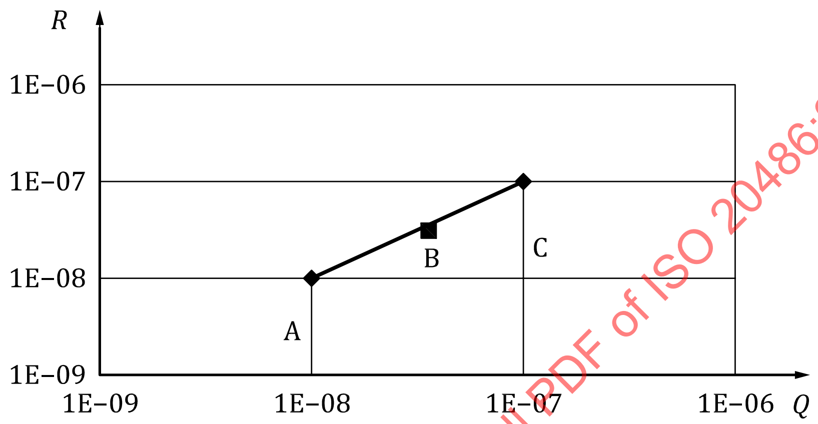
NOTE 2 The temperature coefficient of the reference leak is normally stated. If the temperature coefficient of the unknown leak is not given, it can be assumed that it is approximately 3,5 %/K for a quartz permeation leak, and 0,3 %/K for conductance type leaks in viscous flow mode.

6.5.1.2 Method B and B_S: Result of comparison to two reference leaks

To keep this procedure practical, only the case of equal temperature coefficients and the same temperature difference between each reference leak and its calibration reference temperature is considered. In this case, the simplified Formula (2) holds. See Figure 4.

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

where



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

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

Key

A reference leak 1

B unknown leak

C reference leak 2

R reading

Q leakage rate

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

Figure 4 — Two-point calibration of a leak

6.5.2 Influence factors to measurement uncertainty

The calibration is mainly influenced by the following factors:

— uncertainty of the reference leak(s);

— ambient temperature (all leaks at the same temperature level);

— linearity of the leak detector.

The uncertainty shall be calculated according to common guidelines (see [2]).

7 Volumetric calibration

7.1 Direct flow (Method C)

7.1.1 General

This method is applicable to conductance leaks in the range of 10^{-5} Pa·m^3/s (~0,006 Std cm^3/min) to 0,2 Pa·m^3/s (~100 Std cm^3/min). Leaks from 10^{-6} Pa·m^3/s (~0,000 6 Std cm^3/min) to 10^{-5} Pa·m^3/s (~0,006 Std cm^3/min) 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.

Two types of pressure and flow conditions shall be considered:

— flow from pressure to atmosphere (see 7.1.4.1);

— flow from atmosphere to vacuum (see 7.1.4.2).

The third possible flow condition, pressure to vacuum, cannot be measured with Method C (if this is required, a calibration with tracer gas according Method A or B with a suitable MSLD or a calibration according to Method F or G needs to be made.)

7.1.2 Equipment

To calibrate a leak by measurement of capillary flow according to Method C described in 7.1.4, a calibrated glass tube (preferably with a suitable vent valve at one end, see Figures 5 and 6) 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 is needed. Instruments based on the timed movements of a film in a tube are also available, e.g. a bubble flow meter.

As conductance leak elements normally have no tracer gas reservoir, a separate tracer gas supply is needed or calibration may be performed with filtered, oil free and dry atmospheric air.

7.1.3 Preparation of leaks and apparatus

7.1.3.1 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).

7.1.3.2 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 can 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 it is to keep all dead volumes as small as possible to reduce measurement errors.

Pressure to atmosphere

In this case, the leak inlet is connected to the tracer gas supply and the outlet to the capillary tube. The capillary tube is open to atmosphere at the other end (see Figure 5).

Atmosphere to vacuum

In this case, the leak outlet is connected to a vacuum and the inlet to the capillary tube. The capillary inlet is open to atmospheric pressure (see Figure 6).

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

7.1.4 Measurement

7.1.4.1 Pressure to atmosphere

The measurement is performed according to the following procedure:

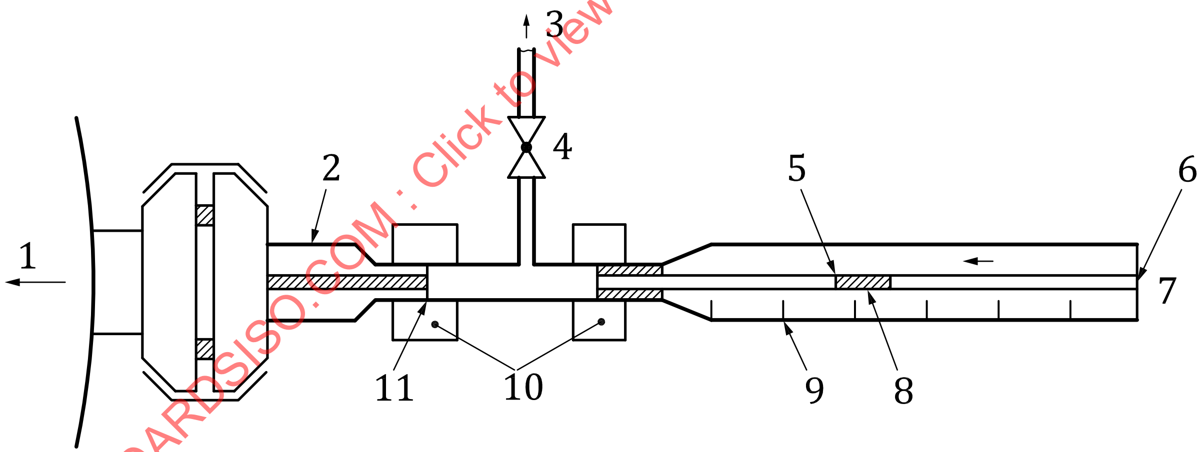
a) maintain the gas flow through the leak to be calibrated for a minimum of 1 h at nominal pressure. All connectors shall be dry and clean;

b) dip the open end of the capillary into the indicator fluid to produce a liquid slug;

c) 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 the slug);

d) close the vent valve (if present) and time the movement of the trailing edge of the slug for a convenient distance (minimum 1,5 × expected flow rate);

e) reposition the slug (see above) and repeat steps a) to d) at least three times. The repeatability of the measurements should be within ±2 %.



Key

1 gas pressure from reservoir
2 unknown leak
3 optional pump connection (to initialize the slug position)
4 optional vent valve
5 trailing edge of slug
6 inside diameter capillary tube
7 atmosphere
8 indicating slug
9 calibration marks
10 connecting tubes
11 zero space

Figure 5 — Set-up for Method C measurement: pressure to atmosphere

7.1.4.2 Atmosphere to vacuum

The measurement is performed according to the following procedure:

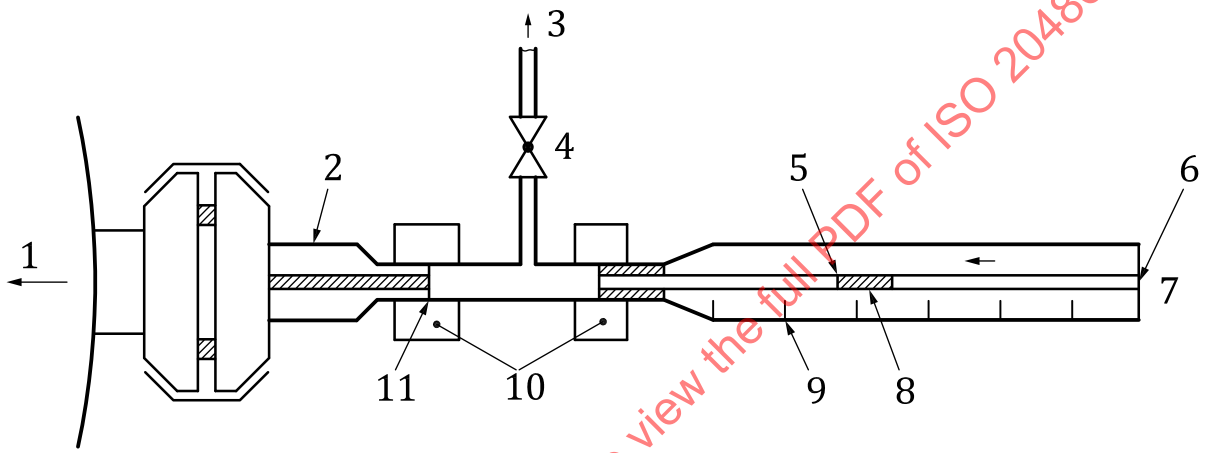
a) maintain the gas flow through the leak to be calibrated for a minimum of 1 hour at nominal pressure. All connectors shall be dry and clean;

b) dip the open end of the capillary into the indicator fluid to produce a liquid slug;

c) close the vent valve and time the movement of the leading edge of the slug over a convenient distance (minimum 1,5 × expected flow rate).

d) reposition the liquid slug to the end of the capillary again, using gas pressure via the vent valve;

e) repeat steps a) to d) at least three times. The repeatability of the measurements should be within ±2 %.



Key

1 vacuum
2 unknown leak
3 gas fill connection
4 filling valve
5 leading edge of slug
6 inside diameter capillary tube
7 atmosphere
8 indicating slug
9 calibration marks
10 connecting tubes
11 zero space

Figure 6 — Set-up for Method C measurement: atmosphere to vacuum

7.1.5 Evaluation for Method C (direct flow measurement)

7.1.5.1 Determination of leakage rate

The unknown leakage rate is given by:

$$Q_{pV} = p_{TA} \times \frac{\Delta V}{\Delta t} \tag{3}$$

where

Q_{pV} is the pV-throughput at test condition;

p_{TA} is the test pressure at leak exit;

ΔV is the collected volume;

Δt is the collecting time.

Pressure-to-atmosphere measurements shall be corrected for the vapour pressure of the indicating liquid, for the viscosity of the tracer gas and also for the influence of pressure and temperature. For the nominal leakage rate, reference pressure and temperature are given in Clause 4. Formula (4) assumes laminar flow in a round pipe (Hagen-Poiseuille). For each type of leak, it shall be checked whether this assumption is applicable. Otherwise, it is necessary to calculate with an adequate formula or an empirical correction.

The nominal leakage rate is calculated by Formula (4):

$$Q_N = Q_{pV} \times \frac{\eta_{ref}}{\eta_{test}} \times \frac{p_{ref,in}^2 - p_{ref,out}^2}{p_{test,in}^2 - p_{test,out}^2} \times \frac{p_{test,out} \times T_{ref}}{p_{ref,out} \times T_{test}} \times \frac{p_{test,out}}{p_{test,out} - \left(p_{v,ref} - p_{v,test}\right)} \tag{4}$$

where

Q_N is the nominal leakage rate;

Q_{pV} is the pV-throughput at test condition according to Formula (3);

η_{ref} is the viscosity of gas at reference conditions;

η_{test} is the viscosity of gas at test conditions;

$p_{ref,in}$ is the reference pressure at leak entrance;

$p_{ref,out}$ is the reference pressure at leak exit;

$p_{test,in}$ is the test pressure at leak entrance;

$p_{test,out}$ is the test pressure at leak exit;

T_{ref} is the reference temperature;

T_{test} is the temperature at test conditions;

$p_{v,ref}$ is the vapour pressure at reference conditions;

$p_{v,test}$ is the vapour pressure at test condition.

Atmosphere to vacuum measurement need not to be corrected for vapour pressure because, in this situation, gas with high humidity is passing the leak.

The nominal leakage rate is given by:

$$Q_N = Q_{pV} \times \frac{\eta_{ref}}{\eta_{test}} \times \frac{p_{ref,in}{}^2 - p_{ref,out}{}^2}{p_{test,in}{}^2 - p_{test,out}{}^2} \times \frac{p_{test,out} \times T_{ref}}{p_{ref,out} \times T_{test}} \quad (5)$$

7.1.5.2 Influence factors to measurement uncertainty

The calibration is mainly influenced by the following factors:

— test time;

— test volume;

— ambient pressure;

— ambient temperature;

— vapour pressure;

— test pressure.

The uncertainty shall be calculated according to common guidelines (see [2]).

7.2 Leak measurement under water (Method D)

7.2.1 General

This method is applicable to conductance leaks in the range of 0,2 Pa·m^3/s (~100 Std cm^3/min) up to around 8 Pa·m^3/s (~5 000 Std cm^3/min).

Calibration from atmosphere to vacuum is not possible with this method.

7.2.2 Equipment

Leaks, which are too large to be calibrated by Method C can be calibrated by catching the escaping gas under water in a calibrated volume.

A measuring glass with calibrated volume, a stopwatch, temperature measuring equipment for ambient temperature and water temperature, a test pressure sensor and a sensor for ambient pressure are necessary.

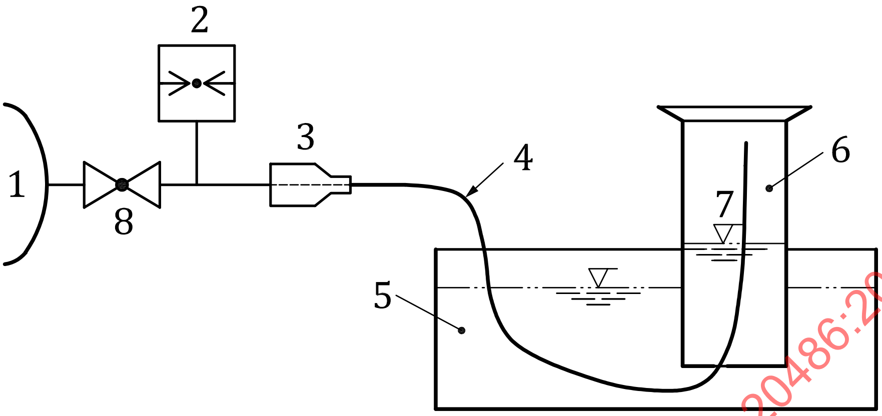
As conductance leaks normally have no tracer gas reservoir, a separate tracer gas supply is needed for calibration.

7.2.3 Preparation of leaks and apparatus

The measuring glass is filled with water (see Figure 7). The water shall have the same temperature as the ambient temperature. The leak is connected to the gas supply and the flow of the leak is conducted via a hose into the measuring glass. The time to fill a certain volume with gas shall be recorded.

If the inlet pressure of the leak is low (<50 kPa), the hydrostatic pressure is not negligible and the average pressure shall be considered and checked at the beginning of the calibration. This is achieved by replacing some of the water in the measuring glass with gas via the unknown leak. When the water in the measuring glass has been replaced by half of the planned measuring gas volume, the gas supply valve shall be closed. After a few seconds the pressure will equilibrate and the pressure gauge at the leak inlet shows the difference between ambient pressure and the average exhaust pressure at the unknown leak. This pressure plus the ambient pressure is the outlet pressure, $p_{test,out}$, of the leak and shall be used in the calculation by Formula (6).

Furthermore, satisfy the requirements for the preparation according to Method C.



Key

1	test pressure	5	water basin
2	pressure gauge	6	measuring glass
3	unknown leak	7	water level
4	connecting hose	8	shut-off valve

Figure 7 — Set-up for Method D measurement

7.2.4 Measurement

a) The pressure sensor shall be placed directly at the entrance of the reference leak (see Figure 8);

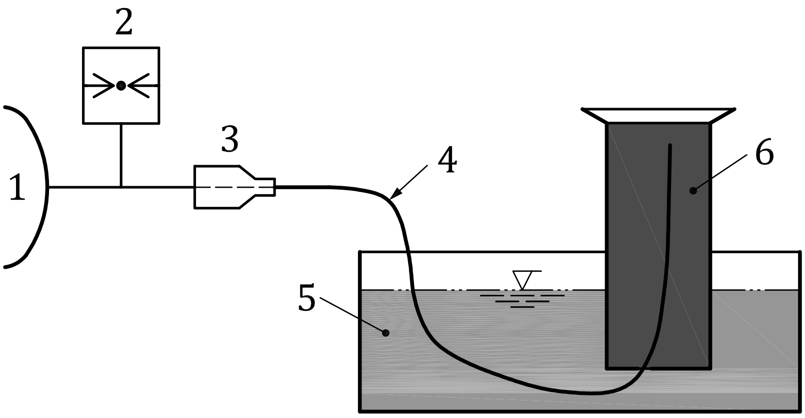
b) the inlet pressure at the reference leak shall be adjusted with exhaust to ambient;

c) water temperature and ambient temperature shall be the same within a tolerance of ±1 K;

d) the measuring glass shall completely be filled with water;

e) the hose shall be inserted into the measuring glass and the time shall be measured until a certain volume of gas is accumulated inside the measuring glass (see Figure 9);

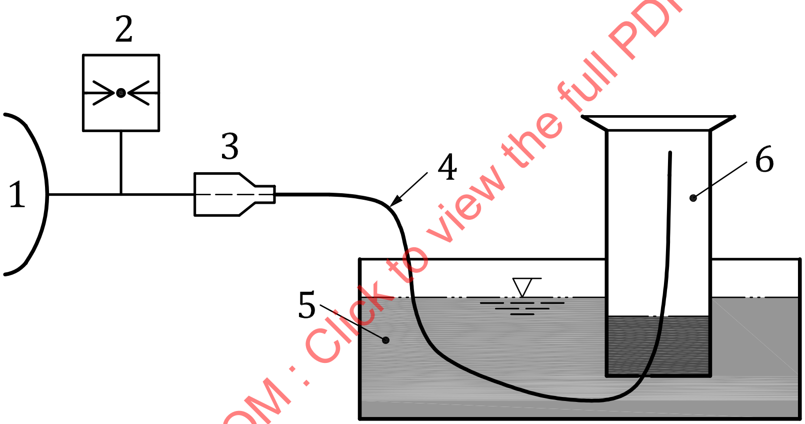
f) repeat steps b) to e) at least three times. The repeatability of the measurements shall be within ±2 %.



Key

1 test pressure
2 pressure gauge
3 unknown leak
4 connecting hose
5 water basin
6 measuring glass

Figure 8 — Method D: Start condition of measurement



Key

1 test pressure
2 pressure gauge
3 unknown leak
4 connecting hose
5 water basin
6 measuring glass

Figure 9 — Method D: End condition of measurement

7.2.5 Evaluation for Method D

The pressure condition in Formula (6) is based on a laminar flow in a round pipe (Hagen-Poiseuille). For each type of reference leak, it shall be checked whether this calculation is permitted. Otherwise, it is necessary to calculate with an empirical correction factor.

The unknown leakage rate is given by:

$$Q_N = Q_{pV} \times \frac{\eta_{ref}}{\eta_{test}} \times \frac{p_{ref,in}^2 - p_{ref,out}^2}{p_{test,in}^2 - p_{test,out}^2} \times \frac{p_{test,out} \times T_{ref}}{p_{ref,out} \times T_{test}} \times \frac{p_{test,out}}{p_{test,out} - \left(p_{v,ref} - p_{v,test}\right)} \quad (6)$$

where

Q_N is the nominal leakage rate;

Q_{pV} is the pV-throughput at test condition according to Formula (3);

η_{ref} is the viscosity of gas at reference conditions;

η_{test} is the viscosity of gas at test conditions;

$p_{ref,in}$ is the reference pressure at leak entrance;

$p_{ref,out}$ is the reference pressure at leak exit;

$p_{test,in}$ is the test pressure at leak entrance;

$p_{test,out}$ is the test pressure at leak exit (in the measuring glass);

T_{ref} is the reference temperature;

T_{test} is the temperature at test conditions;

$p_{v,ref}$ is the vapour pressure at reference conditions;

$p_{v,test}$ is the vapour pressure at test condition.

7.2.6 Influence factors to measurement uncertainty

The calibration is influenced by the following factors:

— test time;

— test volume;

— ambient pressure;

— temperature of the water;

— ambient temperature;

— vapour pressure;

— test pressure;

— solubility of the gas into the liquid.

The uncertainty shall be calculated according the common guidelines (see [2]).

7.3 Calibration by (volumetric) gas meter (Method E)

7.3.1 General

This method is applicable to conductance leaks in the range of 2 000 Std cm^3/min up to around 100 000 Std cm^3/min.

7.3.2 Equipment

If the leaks are so large that it is not possible to calibrate them using Methods C or D, it is possible to use a calibrated gas meter. The gas flow from the reference leak (outlet) shall be led into the gas meter. The test time shall be minimum 1,5 min.

As equipment, a calibrated gas meter, a stopwatch, temperature measuring equipment for ambient temperature and a sensor for ambient pressure are necessary.

NOTE As a gas meter measures volume flow (e.g. m^3/h) the total pressure is needed to obtain pV-throughput.

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

7.3.3 Preparation of leaks and apparatus

The reference leak shall be fitted directly at the entrance of the gas meter. The pressure sensor shall be close to the entrance of the reference leak.

Furthermore, the requirements for the preparation according to Method C shall be observed.

7.3.4 Measurement

a) The pressure sensor shall be directly at the entrance of the reference leak;

b) the reference leak shall be mounted directly at the inlet of the gas meter;

c) the time shall be measured until a certain volume has passed the gas meter;

d) the measured volume should be larger than 1,5 × of the nominal volume of the reference leak;

e) repeat step c) at least three times. The repeatability of the measurements should be within ±2 %.

7.3.5 Evaluation for Method E (gas meter)

The pressure condition in Formula (7) is based on a laminar flow in a round pipe (Hagen-Poiseuille). For each type of reference leak, it shall be checked whether this calculation is permitted. Otherwise, it is necessary to calculate with an empirical correction factor.

The unknown leakage rate is given by:

$$Q_N = Q_{pV} \times \frac{\eta_{ref}}{\eta_{test}} \times \frac{p_{ref,in}^2 - p_{ref,out}^2}{p_{test,in}^2 - p_{test,out}^2} \times \frac{p_{test,out} \times T_{ref}}{p_{ref,out} \times T_{test}} \quad (7)$$

where

Q_N is the nominal leakage rate;

Q_{pV} is the pV-throughput at test condition according to Formula (3);

η_{ref} is the viscosity of gas at reference conditions;

η_{test} is the viscosity of gas at test conditions;

$p_{ref,in}$ is the reference pressure at leak entrance;

$p_{ref,out}$ is the reference pressure at leak exit;

$p_{test,in}$ is the test pressure at leak entrance;

$p_{test,out}$ is the test pressure at leak exit;

T_{ref} is the reference temperature;

T_{test} is the temperature at test conditions;

Because the gas counter is a real volumetric unit, there is no vapour pressure influence taken into account.

7.3.6 Influence factors to measurement uncertainty

The calibration is mainly influenced by the following factors:

- test time;
- test volume;
- ambient pressure;
- ambient temperature;
- test pressure;
- internal flow resistance of the gas meter;
- uncertainty of the gas meter.

The uncertainty shall be calculated according the common guidelines (see [2]).

7.4 Calibration by pressure change in a known volume (Method F)

7.4.1 General

This method is an alternative to Methods A and B and it can be used for most types of non-condensing, non-corrosive gases.

The leakage rate is calculated by the pressure rise in a known volume over time. By using different volumes and different times, it is possible to adapt to different leakage rates. This method is usable for leaks with or without a gas reservoir. It is also possible to measure a gas flow from over-pressure to ambient pressure, from ambient pressure to vacuum, from over-pressure to vacuum or from over-pressure to another over-pressure.

As equipment, a pressure sensor, a stopwatch, a chamber with a known volume and a gas supply for all reference leaks without own gas reservoir is necessary. If it is necessary to use other pressures inside the known volume, a vacuum pump or a separate gas supply with dry and clean gas is also necessary.

The leak unit of 1 mbar·l/s means a pressure change of 1 mbar in 1 l volume per second. This is the same as a leakage rate of 1 bar cm^3/s = 0,987 Std cm^3/s.

The leakage rate for those leaks can be specified in Std cm^3/min, in mbar·l/s or in Pa·m^3/s.

Using this method, it is possible to determine a leakage rate by a pressure change in a known volume. There are three options for calibration with this method:

1) gas flow through the calibrated leak from pressure (absolute pressure) to vacuum in the test chamber;

2) gas flow through the calibrated leak from positive pressure to ambient pressure in the test chamber;

3) gas flow through the calibrated leak from pressure (absolute pressure) to a lower pressure in the test chamber.

The accuracy of the calibration depends on the test volume, on the test time and under certain conditions also on physical effects (e.g. desorption from the chamber walls). It can be a time-consuming calibration and uncertainty decreases with the test time. This type of calibration is preferred for leaks in the range between 10^{-7} Pa·m^3/s to 10^{-3} Pa·m^3/s.

The advantage of this method is that it can be used for nearly all gases and a mass spectrometer is not needed.

NOTE A deeper evaluation of this method can be found in [3].

7.4.2 Preparation of leaks and apparatus

Independent from the calibration method, the first step is to determine the exact inside volume of the test chamber. This can be done by two methods:

1) connection of the test chamber to a known volume;

2) injecting a known gas volume to the test chamber.

It is important that the valve between the chamber and the known volume does not have an internal volume itself. A change of the volume, which is generated by the valve, shall be taken into account. During the volume check, the valve to the calibrated leak shall be open so that the dead volume between leak and valve of the reference leak is included in the calculation. There should be no flow through the calibrated leak during the volume check.

The volume check shall be done 3 times and shall be used in the calculation of measurement uncertainty.

In a test with vacuum chambers, there is the effect of pressure rise by outgassing from the surfaces. Before a calibration, this effect of outgassing shall be determined and be subtracted from the result.

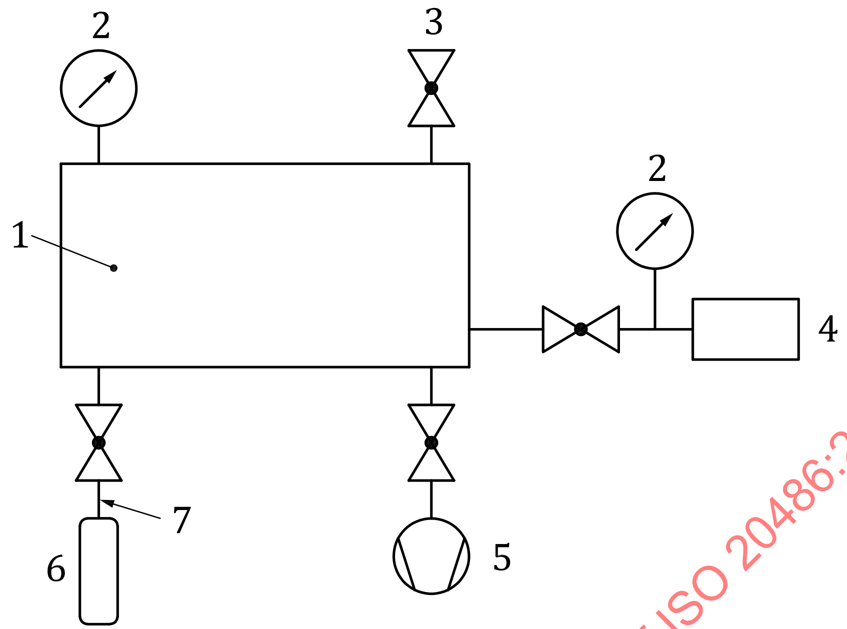
The system shall be thermally insulated against rapid ambient temperature changes.

It shall be ensured that the test chamber is free of leaks.

7.4.2.1 Volume determination of test chamber by connecting a known volume

A known gas volume, V_2, with pressure, p_2, is connected to the test chamber of unknown volume, V_1, and pressure, p_1, by a valve (which has no internal volume). Opening the valve generates a total volume, V_3, with a uniform pressure, p_3.

The leak tightness of the overall system shall be ensured. See Figure 10.



Key

1 test chamber V_1 with pressure p_1
2 pressure sensor
3 venting valve
4 known gas volume V_2 with pressure p_2
5 vacuum pump
6 reference leak
7 dead volume V_D

Figure 10 — Set-up for Method F measurement by connection of the test chamber to a known volume

The chamber volume, V_1, can be calculated according to Formula (8):

$$V_1 = \frac{V_2 \times (p_2 - p_3)}{(p_3 - p_1)} \quad (8)$$

where

V_1 is the unknown volume;

V_2 is the known gas volume;

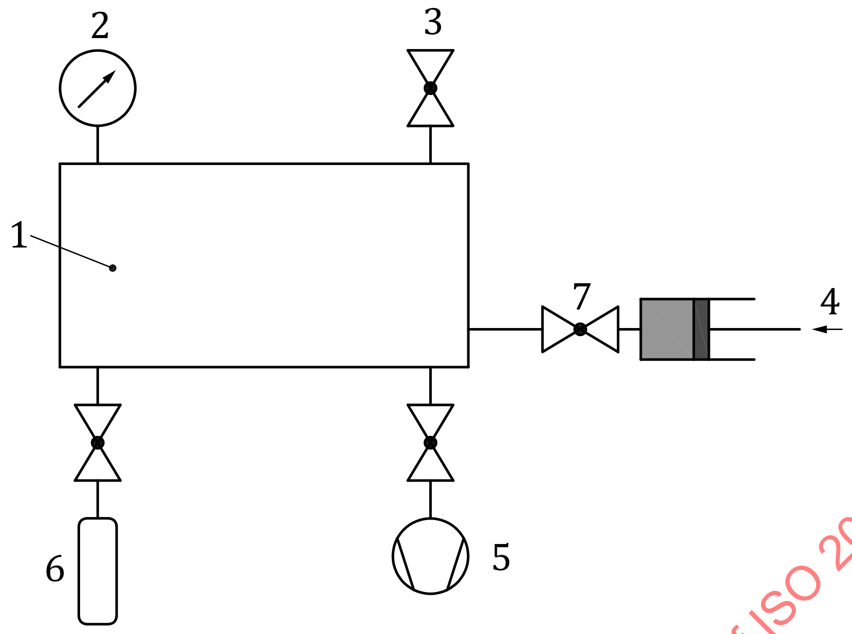
p_1 is the pressure of unknown volume;

p_2 is the pressure of known gas volume;

p_3 is the pressure after valve was open.

7.4.2.2 Volume determination of test chamber by injecting a known gas volume

Alternatively, the volume of the unknown test chamber can be determined by injecting a known gas volume, V_2, into the unknown volume, V_1 , via the connecting valve. By injecting the gas volume, V_2, the pressure inside the test chamber rises from p_1 to p_2. See Figure 11.



Key

1 test chamber V_1 with pressure p_1
2 pressure sensor
3 venting valve
4 known gas volume V_2 with pressure p_2
5 vacuum pump
6 reference leak
7 connecting valve

Figure 11 — Set-up for Method F measurement by injecting a known gas volume to the test chamber

The volume, V_1, can be calculated according to Formula (9):

$$V_1 = \frac{V_2 \times p_2}{(p_2 - p_1)} \tag{9}$$

where

V_1 is the unknown volume of the test chamber;

V_2 is the injecting gas volume;

p_1 is the pressure before injection;

p_2 is the pressure after injection.

7.4.3 Measurement

After the volume of the test chamber has been determined, the leak to be calibrated shall be connected to the test chamber via the connecting valve. The gas from the unknown leak causes an increase of pressure in the test chamber over time. The pressure increase in the test chamber can be calculated to yield a leakage rate.

During the test, the ambient temperature shall be constant.

The pressure condition in Formula (10) is based on laminar flow in a round pipe (Hagen-Poiseuille). For each type of leak, it shall be checked whether this calculation is permitted. Otherwise, it is necessary to calculate with an empirical correction factor.

The leakage rate of the leak to be calibrated at atmospheric pressure is given by Formula (10):

$$Q_N = \frac{\Delta p \times V_1}{t} \times \frac{\eta_{ref}}{\eta_{test}} \times \frac{p_{ref,in}{}^2 - p_{ref,out}{}^2}{p_{test,in}{}^2 - p_{test,out}{}^2} \times \frac{p_{test,out} \times T_{ref}}{p_{ref,out} \times T_{test}} \quad (10)$$

where

- Q_N is the nominal leakage rate;
- Δp is the pressure change in the chamber during test time;
- V_1 is the volume of the test chamber;
- t is the test time;
- η_{ref} is the viscosity of gas at reference conditions;
- η_{test} is the viscosity of gas at test conditions;
- $p_{ref,in}$ is the reference pressure at leak entrance;
- $p_{ref,out}$ is the reference pressure at leak exit;
- $p_{test,in}$ is the test pressure at leak entrance;
- $p_{test,out}$ is the test pressure at leak exit;
- T_{ref} is the reference temperature;
- T_{test} is the temperature at test conditions.

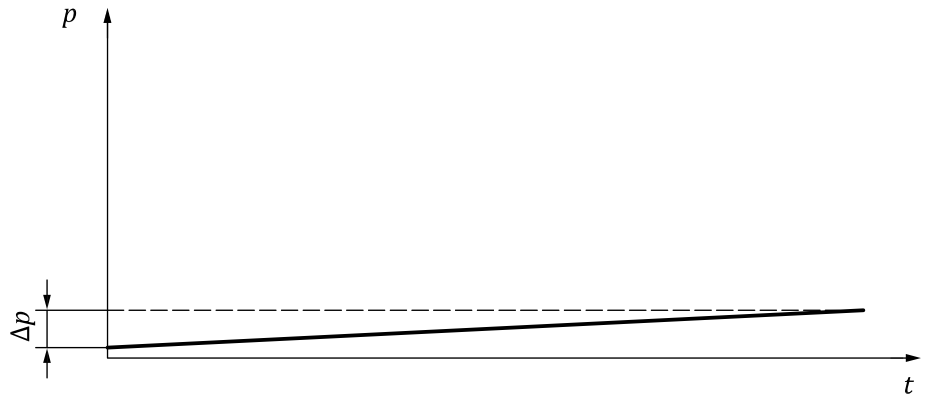
7.4.4 Special situation in vacuum chambers

7.4.4.1 General

If the chamber is under vacuum, it is possible that the inner surfaces are outgassing. This outgassing influences the test result and it is therefore necessary to determine the amount of desorbing gas before the calibration is performed.

To obtain the rate of desorbing gas, the test volume needs to be evacuated to the start vacuum pressure (for exhaust into vacuum, less than 1 mbar absolute). Wait the expected test time (without connecting the leak to be calibrated) and measure the pressure rise during this time. The pressure rise during this time originates from outgassing of the surface. See Figure 12.

The leak tightness of the overall system shall be ensured. It shall be also ensured, by trials, that the outgassing is repeatable.



Key

Δp pressure increase by outgassing

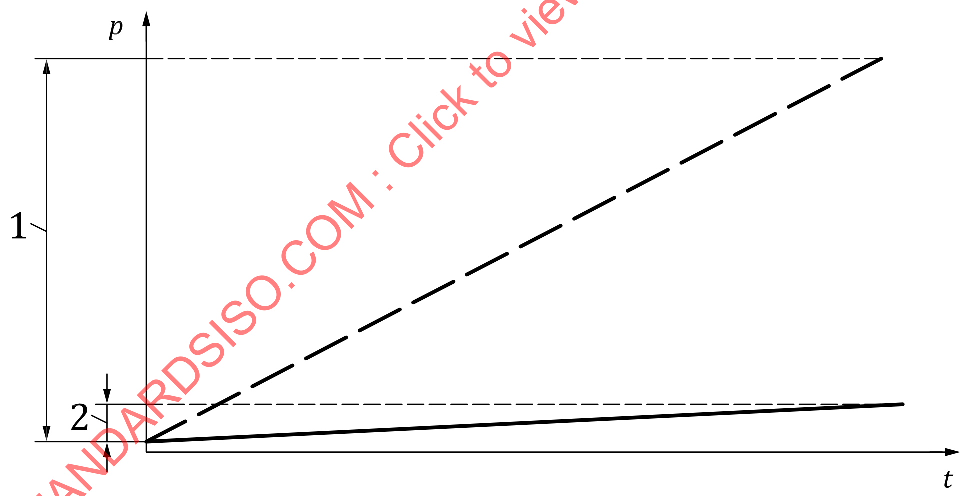
p total pressure

t measurement time

Figure 12 — Method F: pressure increase by outgassing process

After the volume of the test chamber has been determined, the test system shall be set to start conditions and the leak to be calibrated shall be connected to the test chamber.

The leakage rate of the unknown leak causes a pressure rise inside the test chamber volume, V_1. The leakage rate of the unknown leak is calculated by this pressure rise in the test chamber volume, V_1, corrected for the outgassing of the test chamber. See Figure 13.



Key

1 pressure increase by outgassing + leakage rate

2 pressure increase by outgassing

p total pressure

t measurement time

Figure 13 — Method F: pressure increase by outgassing process and leakage rate

7.4.4.2 Measurement in a vacuum chamber

Test time, test volume and pressure sensor shall be selected in a way that the pressure rise in the chamber during the measurement is lower than 1 % of the inlet pressure of the leak to be calibrated. Otherwise, the influence of the exit pressure on the leakage rate of the unknown leak would be too high.

If a leak without a reservoir is calibrated, then it is necessary to use a calibrated manometer at the inlet of the leak to be sure that there is the correct pressure. The uncertainty of this manometer shall be also taken into account in the calculation of the uncertainty of the unknown leak.

7.4.5 Evaluation for Method F (pressure change)

The leakage rate of the unknown leak is:

$$Q_N = \frac{(\Delta p - \Delta p_{des}) \times V_1}{t} \times \frac{\eta_{ref}}{\eta_{test}} \times \frac{p_{ref,in}^2 - p_{ref,out}^2}{p_{test,in}^2 - p_{test,out}^2} \times \frac{p_{test,out} \times T_{ref}}{p_{ref,out} \times T_{test}} \qquad (11)$$

where

Q_N is the nominal leakage rate;

Δp is the pressure change in the chamber during test time;

Δp_{des} is the pressure change by outgassing (desorption) during test time;

V_1 is the volume of the test chamber;

t is the test time;

η_{ref} is the viscosity of gas at reference conditions;

η_{test} is the viscosity of gas at test conditions;

$p_{ref,in}$ is the reference pressure at leak entrance;

$p_{ref,out}$ is the reference pressure at leak exit;

$p_{test,in}$ is the test pressure at leak entrance;

$p_{test,out}$ is the test pressure at leak exit;

T_{ref} is the reference temperature;

T_{test} is the temperature at test conditions.

7.4.6 Influence factors to measurement uncertainty

The calibration is mainly influenced by the following factors:

— test time;

— volume of the test chamber;

— ambient temperature;

— test pressure at reference leak;

— uncertainty of pressure sensor (if present) at unknown leak;

— uncertainty of pressure sensor at test chamber;

— pressure change by outgassing.

The uncertainty shall be calculated according the common guidelines (see [2]).

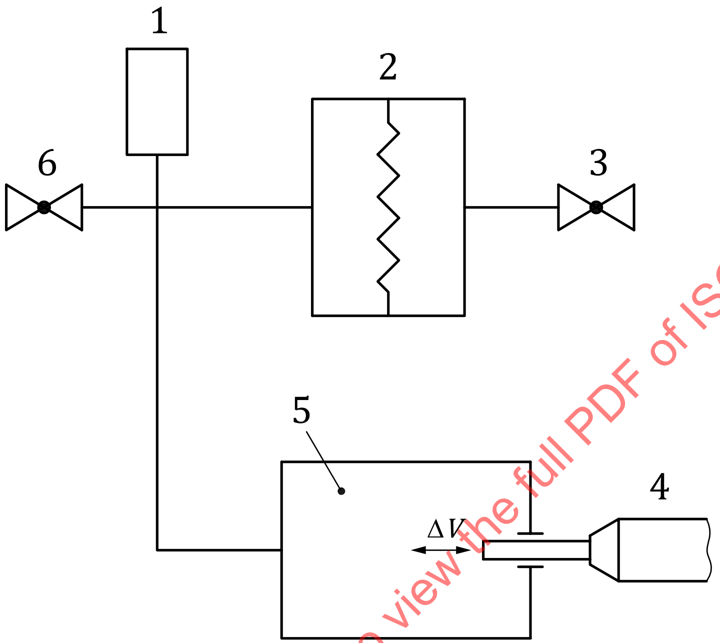
To calculate the uncertainty of the volume of the test chamber all dead volumes, especially those between the valves, shall be taken into account.

7.5 Calibration by volume change at constant pressure (Method G)

7.5.1 Equipment

The system setup is shown in Figure 14. There is a connection flange for the leak to be calibrated and two valves for venting to atmospheric pressure. A diaphragm gauge measures the differential pressure.

The change in volume ΔV is made with a calibrated drive to which a piston is connected. The piston has a vacuum tight seal to the chamber of calibration.



Key

1 leak to be calibrated

2 differtial pressur sensor

3 valve V_1

4 piston drive

5 volume V

6 valve V_2

Figure 14 — Schematic of the $p\Delta V$ calibration system

7.5.2 Preparation of leaks and apparatus

The system is thermally insulated against rapid ambient temperature changes. Before a calibration can be performed, the unknown leak needs to reach equilibrium with the temperature of the room in which the calibration system resides.

7.5.3 Measurement

Once the leak is connected and the valves shut, the pressure in the calibration volume starts to increase. As soon as the differential pressure rises above a certain level, a change of volume, ΔV, is made by the piston. As a consequence of the increase in volume, the pressure drops below the zero differential pressure level but starts to increase again due to the gas emitted from the leak. When the pressure reaches the original level, another increase in volume is performed. This procedure of the volume increment is repeated at least 20 times. The pressure data shall be recorded. See Figure 15.

