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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 TC 112, *Vacuum technology*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Outgassing from the inner wall of a vacuum chamber and from components in a vacuum chamber limit the achievable lowest pressure in a vacuum system and its cleanliness. The lowest achievable pressure is usually important in research facilities as accelerators, in facilities that need to ensure a clean surface, e.g. molecular beam epitaxy, or in devices that need to ensure high vacuum without pump for long times, such as transmitters or X-ray tubes, medical instruments, surface analytical instrumentation or insulation panels. Cleanliness of a vacuum, i.e. the absence or sufficiently low partial pressure of specific gas species or vapours, is important in many different industrial applications such as coating, EUV lithography, catalysis, drying processes in the pharmaceutical or food industry but also in accelerators, fusion reactors, etc. The measurement of outgassing rates is therefore an important tool of quality assurance in vacuum technology. This document recommends well-defined procedures with the possibility of getting traceability of the results of an outgassing rate measurement.

Annex A lists schemes of principles of measurement systems.

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Vacuum technology — Vacuum gauges — Procedures to measure and report outgassing rates

1 Scope

This document describes procedures to measure outgassing rates from components designed for vacuum chambers and of vacuum chambers as a whole. The outgassing rates are expected to be lower than $10^{-5} \text{ Pa m}^3 \text{ s}^{-1}$ ($10^{-2} \text{ Pa L s}^{-1}$) at 23 °C and ~~that they~~ emerge from devices that are suitable for high or ultra-high vacuum applications. The molecular mass of the outgassing species or vapour is below 300 u.

The upper limit $10^{-5} \text{ Pa m}^3 \text{ s}^{-1}$ of total outgassing rate is specified independent of the size, the total surface area and texture or state of the outgassing material. If a specific outgassing rate (outgassing rate per area) is determined, the area is not a specific surface area including the surface roughness, but the nominal geometrical one. When it is difficult to determine the nominal geometrical surface area of the sample, such as powders, porous materials, very rough surfaces, or complex devices, mass specific outgassing rate (e.g. outgassing rate per gram) is used.

For many practical applications, it is sufficient to determine the total outgassing rate. If a measuring instrument, which sensitivity is gas species dependent, is used, the total outgassing rate are given in nitrogen equivalent. In cases, however, where the total outgassing rate is too high, the disturbing gas species is identified, and its outgassing rate is measured in order to improve the sample material. This document covers both cases.

Some outgassing molecules can adsorb on a surface with a residence time that is much longer than the total time of measurement. Such molecules cannot be detected by a detecting instrument when there is no direct line of sight. This is considered as a surface effect and surface analytical investigations are more useful than general outgassing rate measurements considered here. Also, molecules that are released from the surface by irradiation of UV light or X-rays, are out of the scope of this document.

This document is written to standardize the measurement of outgassing rates in such a way that values obtained at different laboratories and by different methods are comparable. To this end, for any of the described methods, traceability is provided to the System International (SI) for the most important parameters of each method and according to the metrological level.

Outgassing rate measurements by mass loss, which were mainly developed for testing of spacecraft and satellite materials, are not gas specific. For acceptable measurement times, mass loss measurements require significantly higher outgassing rates ($>10^{-5} \text{ Pa m}^3 \text{ s}^{-1}$) than typical for high and ultrahigh vacuum components. Also, it is not possible to measure the sample *in situ* due to the weight of the vacuum chamber, since the balances are not vacuum compatible. For these reasons, mass loss measurements are not considered in this document.

It is assumed that the user of this document is familiar with high and ultra-high vacuum technology and the corresponding measuring instrumentation such as ionization gauges and quadrupole mass spectrometers.

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/TS 20177:2018(E)

ISO 3529 (all parts), *Vacuum technology — Vocabulary*

ISO 14291, *Vacuum gauges — Definitions and specifications for quadrupole mass spectrometers*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO/TS 20175, *Vacuum technology — Vacuum gauges — Characterization of quadrupole mass spectrometers for partial pressure measurement*

ISO 27894, *Vacuum technology — Vacuum gauges — Specifications for hot cathode ionization gauges*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3529-1, ISO 3529-2, ISO 3529-3, ISO 14291, ISO 27894 and the following apply.

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

~~— IEC Electropedia: available at~~ — ISO Inline browsing platform: available at <https://www.iso.org/obp>

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

3.1 outgassing rate

rate of molecules that leave a surface in vacuum, in units of throughput, measured at 23 °C or calculated for a gas temperature $T = 23$ °C

Note 1 to entry: For scientific investigations, it is distinguished between desorption and outgassing. The first is a surface effect, the second a bulk (diffusion) effect. For many measurement procedures, however, it is not possible to distinguish between the two effects. To measure the true outgassing rate, all molecules from a surface are removed by a bake-out or a similar procedure. A bake-out, however, in particular in industrial applications, is not possible or not desirable. For this reason, in this document, the term "outgassing rate" includes both desorption and outgassing rate.

Note 2 to entry: The temperature of the sample or the measurement chamber may be different from 23 °C, but the measured outgassing value in units of throughput shall be calculated as if released gas would have temperature 23 °C. In this way, it is possible, to state a measured throughput (at 23 °C) for a sample at 150 °C.

Note 3 to entry: In vacuum applications, the unit of throughput is commonly used for outgassing rates, but for known outgassing gas species it is also possible to use the unit of mass flow, e.g. g/s.

Note 4 to entry: In some cases, when outgassing gas molecules of a certain species easily adsorb on the surrounding surfaces of the measurement system, it is distinguished between intrinsic and measured outgassing rate. The intrinsic outgassing rate is the outgassing rate that leave the sample surface. It is higher than the measured one, when no equilibrium of ad- and desorption on the surfaces of the walls has been reached. In this document, it is assumed that equilibrium is established, but this may not be true for absorbing gases like water vapour.

3.2 geometrical surface area

area of a surface determined from its geometrical dimensions, not including surface roughness

3.3 mass range of outgassing

mass range of QMS from 1 to the highest m/z of all measured or expected outgassing gas species

Note 1 to entry: For the scope of this document, the mass range of outgassing is 300.

3.4 nitrogen equivalent outgassing rate

outgassing rate (3.1) when all gases released from the sample are assumed to be nitrogen molecules

Note 1 to entry: To be consistent, all quantities involved in a physical equation (e.g conductance and pumping speed) need to be expressed for nitrogen, if pressure is measured in nitrogen equivalent. Otherwise, the same nitrogen reading of a vacuum gauge could lead to different quantities dependent on gas species (see the following example).

EXAMPLE If the throughput of hydrogen is measured by $p_{N_2}C$, where pressure p_{N_2} is measured in nitrogen equivalent and C is the effective conductance (pumping speed), C shall be the value calculated or determined for nitrogen, and not hydrogen.

3.5 bake-out time

time for which a sample or chamber is maintained at a specified (bake-out) temperature

Note 1 to entry: Bake-out time does not include the warm-up and cool-down phase.

4 Symbols and abbreviated terms

Symbol	Designation	Unit
ψ	combined calibration factor for sensitivity and effective pumping speed	Pa m ³ s ⁻¹ A ⁻¹
$C_{\text{eff},i}$	effective conductance or pumping speed of gas species, i	m ³ s ⁻¹ or L s ⁻¹
D	diameter of cylindrical chamber	m
f	fragmentation factor	1
i	gas species	
I	ion current at partial pressure, p	A
I_0	ion current at residual pressure, p_0	A
l	length of cylindrical chamber	m
m	mass	kg
M	molecular weight	kg mol ⁻¹
MDPP p_{MDPP}	minimum detectable partial pressure	Pa
p	pressure or partial pressure	Pa
p_0	residual pressure or residual partial pressure	Pa
p_{N_2}	pressure in nitrogen equivalent	Pa
q_{pV}	throughput	Pa m ³ s ⁻¹ or Pa L s ⁻¹
q_{out}	outgassing rate	Pa m ³ s ⁻¹ or Pa L s ⁻¹
q_{out,N_2}	outgassing rate in nitrogen equivalent	Pa m ³ s ⁻¹ or Pa L s ⁻¹
r_x	relative sensitivity for a specified gas species "x" divided by sensitivity SN ₂ for nitrogen	1

R	universal gas constant	$\text{J mol}^{-1} \text{K}^{-1}$
S	sensitivity (coefficient)	A/Pa
S_{N_2}	sensitivity for nitrogen	A/Pa
t	time	s
T	temperature	K
V	volume	m^3 or L (Liter)
z	charge state after ionization	1
CDG	capacitance diaphragm gauge	
QMS	quadrupole mass spectrometer	
SEM	secondary electron multiplier	
SI	System International	
SRG	spinning rotor gauge	

5 Measurement systems

5.1 General

5.1.1 Overview

This document intentionally leaves a choice of measurement systems for outgassing measurements. The reason is that, at the present stage, there are no scientific reasons to prefer one method over the other, as long as traceability to the SI is ensured in some way. In the past, lack of traceability was a great deficit of systems and procedures. All measurement systems listed in this document ensure some kind of traceability to the SI. The higher the metrological level, the more direct the traceability is, and the lower the uncertainties of the traceable quantities are, which are significant. The traceable quantities of each kind of system are given in Annex C.

In the future, when this document is established and comparisons between different systems have been carried out, it may turn out that some systems should be preferred against others, because the kind of traceability is insufficient or data are less reliable for other reasons. This will lead to new recommendations.

5.1.2 Recommendations for systems

The criteria for which system is best to use are as follows:

- a) need to measure time dependence;
- b) need to identify outgassing species;
- c) outgassing of vapour (e.g. water vapour) in a significant magnitude;
- d) expected outgassing rate;
- e) needed accuracy or uncertainty of measurement;
- f) effort, budget and experience.

Annex B gives an overview on which system is suitable or not in specific cases.

If the time dependence of outgassing shall be measured, one of the throughput method systems described in 5.2.2 to 5.2.4 should be applied. The throughput system described in 5.2.5, however, is not useful for measuring time dependence.

Accumulation systems are not suitable for gases or vapours (e.g. water vapour) that adsorb on the inner walls, since this will lead to an underestimation of the outgassing rates.

If a significant amount of vapour, in particular water vapour, is outgassing from the sample, one of the throughput methods described in 5.2 should be applied.

If a UHV-compatible sample after a bake-out has to be measured, one of the accumulation methods described in 5.3 should be applied. This method is usually more accurate than the throughput method and at the same time associated with less effort. For very low outgassing rates ($<10^{-12}$ Pa m³ s⁻¹), the accumulation method is also applicable, but will need long measurement times. If this shall be avoided, the throughput method systems described in 5.2.2, 5.2.3 or 5.2.5 should be applied.

If the outgassing species have to be identified, the system described in 5.3.2 cannot be applied. This system is, however, well-suited for measuring total outgassing rates.

All systems can be used for outgassing rates $>10^{-9}$ Pa m³ s⁻¹, however, the effort for the systems described in 5.2.3 with two path design and 5.2.5 is not adapted to these rates. For lower outgassing rates $<10^{-9}$ Pa m³ s⁻¹, the two designs described in 5.2.3 and 5.2.5 or the accumulation systems described in 5.3 should be used.

A great deal of experience is required when systems described in 5.2.2, 5.2.3, 5.2.5 and 5.3.3 are established and operated.

The budgetary investments as well the effort to establish and validate the systems roughly rises in the following order:

- basic accumulation system (5.3.2);
- throughput system with measured effective pumping speed (5.2.4);
- throughput system with calculated conductance element, two path design is more expensive (5.2.3);
- accumulation system with gas analysis system (extended accumulation system) (5.3.3);
- throughput system with modulated conductance (5.2.5);
- continuous expansion system as flow comparator (5.2.2).

The lowest uncertainties in terms of nitrogen equivalent of total outgassing rate can be achieved with the basic accumulation system. The measurement uncertainties typically rise in the following order:

- basic accumulation system (5.3.2);
- continuous expansion system as flow comparator (5.2.2);
- throughput system with modulated conductance (5.2.5);
- throughput system with calculated conductance element; two path design is more accurate at lower rates (5.2.3);
- accumulation system with gas analysis system (extended accumulation system) (5.3.3);
- throughput system with measured effective pumping speed (5.2.4).

The so-called mass change method, where the change of mass of an outgassing sample or the mass of detected outgassing species from a sample is measured, was introduced for testing of spacecraft and satellite materials^[3]. It cannot be recommended for outgassing rate measurements $<10^{-5}$ Pa m³ s⁻¹ which are within the scope of this document, because the method is not sensitive enough for high and ultrahigh vacuum applications. Measurement times of more than 1 000 h are needed for typical outgassing rates of 10^{-7} Pa m³ s⁻¹.

5.1.3 Vacuum chambers and pumps

The vacuum chambers should be prepared such that an increase of the signal ($p-p_0$ or $I-I_0$) due to the lowest sample outgassing rate during the measurement time is at least the same as the background signal (p_0 or I_0). As a minimum, the system shall at least contain a measurement chamber with a pump system and appropriate vacuum gauges. In addition, a separate sample chamber, a load-lock system and a pump chamber may be used. If a separate sample chamber is used, it should have a separate pump system with a valve in between. The conductance from the sample chamber to the measurement chamber should be at least 2 L/s for nitrogen.

NOTE 1 A separate sample chamber to load the sample allows to keep the measurement chamber under background conditions all the time and also independent bake-outs of sample and measurement chamber.

NOTE 2 A much smaller conductance than 2 L/s does not affect measurement results, but can lead to long relaxation times and inhibit the measurement of time dependent outgassing rates.

All systems for outgassing rate measurements should be all metal systems. Large measurement chambers that can host large samples, however, will need large doors which require elastomer sealing. Systems with elastomer sealing will also be suitable, if the expected outgassing rates from the sample are relatively high, typically $>10^{-8}$ Pa m³ s⁻¹. Double sealing techniques can be applied, if the permeation through elastomer seals is too high. Also, valve seat sealings which are not exposed to the air from outside, may consist of elastomers.

All systems shall be equipped with high vacuum pumps that are without the risk of changing pumping speed and emitting previously pumped gases. To this end, turbomolecular pumps and cryopumps are recommended. Diffusion pumps are also acceptable. Ion getter pumps, sublimation pumps or passive getter pumps, however, are only acceptable, if it can be ensured at all times that the change of pumping speed or ejection of previously pumped gases does not influence the results. Dry pump systems should be used when low hydrocarbon outgassing rates are expected or when the sample requires an oil-free environment.

NOTE 3 Also cryopumps can change pumping speed to some extent, depending on the gas amount already pumped.

5.1.4 Vacuum gauges

Vacuum gauges used for the measurement of outgassing rate or to calibrate the sensitivity of the QMS shall have a certificate according to ISO/IEC 17025.

QMS are needed to identify species and measure their outgassing rates. The QMS shall be calibrated according to ISO/TS 20175, either in a separate system or *in situ* in the outgassing measurement system. If calibration was performed in a separate system, an *in situ* calibration check has to follow.

If a procedure requires *in situ* calibration of vacuum gauges, then the gauges to be compared should have symmetrical positions to optional gas inlets and pump outlet and both ionization gauges and QMS should have no line of sight to any other gauge.

5.1.5 Purity of gases

For the purpose of this document, a pure gas specifies a purity of $>99,9$ %.

5.2 Systems applying the throughput method

5.2.1 General

These methods are based on measuring a steady flow through the measuring system.

5.2.2 Continuous expansion system as flow comparator

This method is considered as of high metrological level.

The continuous expansion systems are based on the principle that well-known pressures or partial pressures can be established in a vacuum chamber by the injection of gas of known flow rate (here: throughput $q_{pV,i}$) and pumped out of the vacuum chamber via a duct of known conductance $C_{eff,i}$ ^[3]. The known throughput is generated by a flowmeter or a gas flow out of a reservoir volume V_R (see Figure A.1) filled with gas of pressure $p_{R,i}$ through a known conductance $C_{R,i}$, as shown in Formula (1):

$$q_{pV,i} = p_{R,i}C_{R,i} \quad (1)$$

The flow injection can also be provided by a flow divider method where the pressure ratio across a flow restricting element is independent of pressure in molecular flow regime.

NOTE Generating the known flow traceable to the SI is the part of the system which requires a great deal of experience in vacuum metrology and a comprehensive equipment.

This flow $q_{pV,i}$ generates a partial pressure p_i for species i in the continuous expansion chamber by Formula (2):

$$q_{pV,i} = p_i C_{eff,i} \quad (2)$$

where $C_{eff,i}$ is the effective conductance of species i to the pump system.

In the same manner, an outgassing sample with rate $q_{out,i} = q_{pV,i}$ will generate the same partial pressure, p_i , in the measurement chamber and can be recorded by a QMS. By this recording, the continuous expansion system acts as a flow comparator between the unknown $q_{out,i}$ and the known $q_{pV,i}$. If the rates are not the same, $q_{out,i}$ will generate the partial pressure p'_i according to Formula (3):

$$q_{out,i} = p'_i C_{eff,i} \quad (3)$$

Finally, $q_{out,i}$ is determined by Formula (4):

$$q_{out,i} = \frac{p'_i}{p_i} q_{pV,i} \quad (4)$$

The partial pressures shall be measured by a QMS and the total pressure additionally by a total pressure vacuum gauge. The outgassing can be analysed in terms of gas species as shown in Formula (4). Since the signal of a QMS is normally a current I_i , for the purpose of this document, it makes sense to define a calibration factor ψ_i as given in Formula (5):

$$\psi_i = \frac{q_{pV,i}}{(I_i - I_{i,0})} = \frac{C_{eff,i}}{S(p_i)} \quad (5)$$

where

$S(p_i)$ is the sensitivity as defined in ISO 14291;

I_i is the QMS signal at p_i ;

$I_{i,0}$ is residual pressure conditions.

With this definition, the outgassing rate is calculated from the QMS signal as Formula (6):

$$q_{\text{out},i} = \psi_i (I'_i - I_{i,0}) = q_{pV,i} \frac{(I'_i - I_{i,0})}{(I_i - I_{i,0})} \quad (6)$$

where $I'_{i,0}$ as the QMS signal at p'_i . If only a total pressure vacuum gauge is used, the total outgassing rate in nitrogen equivalent can be determined by using Formula (7):

$$q_{\text{out},N2} = \frac{p'_{N2}}{p_{N2}} q_{pV,N2} \quad (7)$$

The position of the calibration gas inlets shall be at such positions that equal flows from them and the outgassing sample generate the same signal on the QMS. This can be accomplished by a cylindrical chamber with the conductance at one endface and all gas inlets and the inlet from the outgassing sample located on the same equatorial plane perpendicular to the cylindrical axis.

For the purpose of a primary outgassing rate measurement system, it shall be possible to establish at least three calculable partial pressures at the same time in the chamber. The three gases shall correspond to the three major outgassing species (if applicable) from the outgassing sample. The QMS is calibrated *in situ* for these one to three major gas species and for other outgassing species of interest as described by Formula (5).

The measurement chamber should have a volume of at least four times the total volume of all the gauges and associated pipe work connecting the chamber and the gauges (e.g. elbows shall be considered as part of the gauge volume). Another chamber, separated by a valve from the measurement chamber and to host the sample, shall be available. The injected $q_{pV,i}$ shall not change by more than 1 % within 30 min. The system shall be bakeable.

A load-lock for the sample introduction shall be available, when small outgassing rates shall be measured, but the sample stay in non-baked-out condition. In all other cases, a load-lock is strongly recommended.

5.2.3 Throughput system with calculated conductance element

5.2.3.1 General

This method is considered as of medium or high metrological level, depending on effort (see below). The system can be also named pressure difference system both for one (5.2.3.2) and two path design (5.2.3.3).

5.2.3.2 One path design

The idea of the throughput system with calculated conductance element is to measure the pressure difference Δp across an orifice, short duct (length lower or equal to the diameter) or other element of known conductance, C , located between two chambers. The upstream pressure p_1 shall be measured, the downstream pressure p_2 can be either measured or known from an estimation. In particular, if the pumping speed behind the duct is much higher than the conductance of the duct (>20 times), the downstream pressure can be neglected. In this case, the downstream chamber is optional.

Whenever size and shape of the sample will allow, the upper measurement chamber should have a cylindrical volume of diameter, D , and length, l , and its size of at least four times the total volume of all the gauges and associated pipe work connecting the chamber and the gauges (e.g. elbows shall be considered as part of the gauge volume). The ratio l/D shall be $\geq 1,5$ and ≤ 3 . The system should be bakeable. The flanges for vacuum measurement in the upstream vessel should be located at a height of $D/2$ above the orifice plane, where D is the diameter of the cylinder. The lower measurement chamber (pump chamber) may have a cylindrical volume of the same diameter as the upper one. For reason of

symmetry, the flanges for vacuum measurement in the downstream vessel should be located at a height of $D/2$ below the orifice plane.

NOTE 1 The cylindrical design of the chambers with the positioning of the gauge inlet ports at $D/2$ allows the use of the same domes as described in ISO 21360-1:2012, 5.2, if l/D is as required by ISO 21360-1. The downstream vessel, however, can be put upside down. An additional flange at half of the length of the cylinder and a sample holder inside can be required.

If the downstream pressure p_2 has to be measured, a bypass with two valves (all metal valves are recommended) and the measuring instrument should be installed (QMS and/or total pressure gauge) in between. With the corresponding valve open, either the upstream or downstream pressure can be measured with the same instrument.

The upstream pressure p_1 shall be measured by a calibrated total pressure gauge and optionally in addition by a calibrated QMS. The calibration certificates shall state the sensitivities for nitrogen as a minimum.

The sample may be placed in the upstream pressure vessel. A separate vessel, however, has the advantage because the background from the outgassing of the vessels can be measured just before and after the measurement of the sample. The additional background from the sample chamber shall be determined in a separate experiment. If a separate vessel is used, its connecting flange should be located at $l/2$ where l is the maximum length of the cylinder. In addition, any holder of a sample should be at this same height close to $l/2$. If the sample is placed inside the upstream vessel, it should be positioned at a height of at least $D/2$ above the orifice plane. A height of $l/2$ is recommended.

NOTE 2 A lower position of the sample holder can reduce the calculated conductance. This can result to a significant part of the molecules leaving the sample to not reach the QMS or gauges.

See Figure 1.

