INTERNATIONAL STANDARD

ISO 21360-5

First edition
2023-11

Vacuum technology — Standard methods for measuring vacuum-pump performance —

Part 5: Non-evaporable getter (NEG) vacuum pumps

Technique du vide — Méthodes normalisées pour mesurer les performances des pompes à vide —

Partie 5: Pompes à piégeur non évaporable (NEG)

Reference number
ISO 21360-5:2023(E)

Published in Switzerland

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Foreword

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This document was prepared by Technical Committee ISO/TC 112, *Vacuum technology.*

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Introduction

This document specifies methods for measuring the performance data of non-evaporable getters (NEGs) with the shape of pill, disk, ring, strip, module, cartridge, pump structures and coatings. This document complements ISO 21360-1, which provides a general description of the measurement of performance data of vacuum pumps.

The methods described here are well known from existing national and international standards. This document aims to show a collection of suitable methods for the measurement of performance data of NEGs. The method specified in this document takes precedence over the volume flow rate (pumping speed) measurement given in ISO 21360-1:2020, 5.1, 5.2 and 5.3.

Vacuum technology — Standard methods for measuring vacuum-pump performance —

Part 5: Non-evaporable getter (NEG) vacuum pumps

1 Scope

This document specifies methods for the measurement of pumping characteristics of non-evaporable getters (NEGs). It is applicable to all sizes and all types of NEGs, including those:

— with the shape of pill, disk, ring, strip, module, cartridge;

— with pump structures;

— and NEG coatings on inner surface of pipes and vacuum chamber.

A significant difference of pumping characteristics of the NEG and other vacuum pumps is that the pumping speed of the NEG depends on the sorption quantity. Furthermore, especially in the case of NEG coating, the sticking probability rather than the pumping speed is often the index of the pumping performance. Therefore, this document specifies the methods for measuring the pumping speed, the sorption quantity, and the sticking probability of NEGs.

WARNING — It is assumed that the user is familiar with the handling of combustible gases and poisonous ones and with ultra-high vacuum technology.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
non-evaporable getter
NEG
getter material to sorb gases in vacuum chambers without evaporation

Note 1 to entry: Sorbing gases mean the process of removing gases from vacuum chambers by adsorption or absorption phenomena. The adsorption is a kind of sorption in which the gas is retained at the surface of the getter material. Most of gas molecules are chemisorbed at the surface of the getter material. The absorption is also a kind of sorption in which the gas molecules diffuse into the bulk of the getter material. The term of “sorption”, “adsorption”, “chemisorption” and “absorption” are defined in ISO 3529 1:2019, 3.4.1, 3.4.2, 3.4.4 and 3.4.6, respectively.

Note 2 to entry: NEGs have a variety of forms, such as pellets (pills), bars, chips, powders, sheets, strips, washers, wires, module and cartridge.

3.2
non-evaporable getter vacuum pump
NEG vacuum pump
entrapment vacuum pump with a reactive porous alloy or powder mixtures getter material

Note 1 to entry: NEG vacuum pumps are typically mounted on a vacuum flange. The internal heaters and the controller for the activation may be included.

[SOURCE: ISO 3529-2:2020, 3.1.36]

3.3
non-evaporable getter coating
NEG coating
thin films made from non-evaporable getter, which is coated on inner surface of pipes and vacuum chamber

3.4
surface getter
getter where only the surface shows pumping action

Note 1 to entry: The pumping speed and sorption capacity are essentially proportional to the surface area.

Note 2 to entry: For example, Zr-Fe-V alloy acts as a surface getter for CO at room temperature.

3.5
volume getter
getter where the pumping speed and/or sorption capacity depends on the volume

Note 1 to entry: The dependence of the pumping speed and sorption capacity of the volume getters on the temperature and operation pressure is more significant compared with the *surface getter* (3.4).

Note 2 to entry: For example, Zr-Fe-V alloy acts as a volume getter for H_2 at room temperature. Zr-Fe-V alloy also acts as a volume getter for CO at high temperature.

3.6
activation
conditioning by thermal treatment of a getter to develop its gettering characteristics

Note 1 to entry: Hydrogen reversibly acts with non-evaporable getters (NEGs) and therefore allows to be released by activation.

Note 2 to entry: Other active gases such as CO, CO_2, N_2, and O_2 are chemisorbed irreversibly with NEGs. The activation promotes the diffusion of these gas atoms into the bulk.

3.7
getter pumping speed
S
volume of gas sorbed per unit time

Note 1 to entry: The pumping speed has the same meaning of the volume flow rate.

Note 2 to entry: Getter pumping speed depends on gas species and the amount of gas being sorbed.

3.8
initial pumping speed of NEG
instantaneous pumping speed 3 min after the start of the test at the chosen pressure and temperature

Note 1 to entry: This time delay is necessary to allow initial transient effects, until the pressure equilibrium has become negligible.

3.9
intrinsic sticking probability
sticking coefficient
ratio of the number of sorbed gas molecules to that of impinging ones at a unit area per unit time, where the surface is assumed to be flat.

3.10
sticking probability
α
ratio of the number of sorbed gas molecules to that of impinging ones at a unit apparent area per unit time

Note 1 to entry: Sticking probability depends on gas species, surface chemical composition, surface roughness and coverage.

Note 2 to entry: Sticking probability is typically measured as pumping characteristics of NEGs.

3.11
sorption quantity
C_q
quantity of gas sorbed by the getter

3.12
sorption capacity
C_C
quantity of gas sorbed by the getter until the getter pumping speed decrease to 10 % of the initial pumping speed

4 Symbols and abbreviated terms

Symbol	**Designation**	**Unit**
A	apparent surface area of getter material	m^2
C_0	conductance of orifice	m^3/s
C_q	sorption quantity	Pa m^3
C_C	sorption capacity	Pa m^3
F_1	correction factor of vacuum gauge 1, where $F_1 = 1/K_1$	
F_2	correction factor of vacuum gauge 2, where $F_2 = 1/K_2$	
K_1	sensitivity of vacuum gauge 1	
K_2	sensitivity of vacuum gauge 2	
p_{R1}	pressure reading of vacuum gauge 1, which is located at the upstream side of orifice	Pa
p_{R2}	pressure reading of vacuum gauge 2, which is located at the downstream side of orifice	Pa
p_{B1}	base pressure of vacuum gauge 1	Pa
p_{B2}	base pressure of vacuum gauge 2	Pa
Q_{pv}	gas flow rate	Pa m^3/s
Q_{mol}	molar flow rate	mol/s
R	ideal gas constant	8,134 J/(mol K)
S	getter pumping speed	m^3/s
T	temperature	K
α	sticking probability	
α_0	initial sticking probability	

5 Test methods

5.1 General

5.1.1 Test gases

H_2 and CO shall be used to test for NEG. CO can be replaced by N_2 or CO_2 from a safety perspective when an agreement is made between customer and testing laboratory. In addition, other gases such as O_2 can be required depending on the application. The purity of the test gas in the gas cylinder shall be higher than 99,99 % for H_2 and 99,95 % for CO, respectively. It is also recommended to measure the purity of the test gas by using quadrupole mass spectrometer (QMS) in the vacuum chamber because the test gas can be polluted during the transportation from the gas cylinder to the vacuum chamber.

5.1.2 Vacuum chamber

The vacuum chamber shall consist of all-metal vacuum components with a baking system. When valves with elastomer sealing parts are used, they shall be bakeable and fabricated for the usage of UHV condition. The cleanliness shall be appropriate to obtain sufficiently low base pressure in the range of ultrahigh vacuum or extreme-high vacuum (XHV), depending on the application. The apparatus shall be capable of reaching a base pressure of less than 1×10^{-6} Pa without NEG sample installation or with uncoated tube. In addition, it is recommended to measure the residual gas by QMS to make sure that both the air leak and the outgassing of H_2O, CO, CO_2, and hydrocarbons are sufficiently small.

Note that H_2 should be the dominating gas species at the base pressure.

5.1.3 Orifice

An orifice is used to determine the gas flow rate for the throughput method as shown in 5.2 and 5.3. The molecular conductance of the orifice shall be calculated from the molecular mass, temperature of the gas, and the diameter and the thickness of the orifice. The calculation method is shown in Annex A. The conductance of the orifice C_0 is carefully selected from four points of view:

— the diameter of the orifice is smaller than the mean free path of the test gas;

— C_0 is sufficiently smaller than the system conductance which is obtained by combining conductances of the pipe and vacuum chamber. The ratio of system conductance to C_0 shall be larger than 100;

— C_0 shall be selected so that the pressure ratio of the upstream pressure p_1 of the orifice to the downstream pressure p_2, p_1/p_2, during the test is larger than their error measured by the vacuum gauges specified in 5.1.5. In this document, the p_1/p_2 value during test is recommended to be larger than 2;

— C_0 shall be selected so that the pressure during the test is in the linear response range of the vacuum gauge specified in 5.1.5. When C_0 is too small, the upstream pressure p_1 may be higher than the linear response range of the Bayert-Alpert gauge (BAG) (key reference 3 in Figure 1) because the downstream pressure p_2 is set to satisfy the requirements in 5.2.5.

5.1.4 Vacuum pumping system for rough pumping

A turbomolecular vacuum pump (TMP) shall be used to obtain the sufficiently low base pressure and to evacuate outgases during degassing and/or activation of test chamber and NEGs under test. In addition, an ion pump may be useful to obtain lower base pressure before measurements. A dry pump is recommended to be used as a roughing vacuum pump to avoid oil pollution, but it should be carefully chosen because gases released from fluorine elements such as F and Cl can also pollute the surface of the NEG.

Installing the valve between TMP and the vacuum chamber (for example, key reference 8 in Figure 1) is strongly recommended so as to keep the inside of vacuum chamber clean in order not to be contaminated by oil vapor and dust. In addition, it is useful to adjust the pressure p_1 or to keep the inside of vacuum chamber under vacuum while the system is not in operation.

5.1.5 Vacuum gauges

Bayert-Alpert vacuum gauges (BAGs), extractor gauges or ion analysing gauges shall be used to measure the getter pumping speed and sorption capacity of NEG. BAGs shall be calibrated in a traceable way to an applicable SI unit. In addition, using a quadrupole mass spectrometer (QMS) is strongly recommended to not only to measure the performance of NEGs but also for other purposes such as leak testing, checking a purity of test gas, evaluation of outgassing during activation. A spinning rotor gauge or an ionization gauge according to ISO/TS 6737 instead of BAGs can be used, but a Magnetron gauge is not recommended because the high pumping effect can cause overestimation of pumping performance of NEG.

There are two methods to calibrate the BAGs. One is that the BAGs are calibrated in a laboratory meeting the requirements of ISO/IEC 17025 or a national metrology institute. The other is that the BAGs are calibrated from the direct comparison with a reference gauge in situ.

A spinning rotor gauge (SRG) or a high accuracy capacitance diaphragm gauge (CDG) with a full scale of 133 Pa or lower shall be used as the reference gauge for in situ calibration. The position where the reference gauge is attached shall be the upstream side of gas inlet against to TMP. The calibration gas shall be the same as the one to be tested because BAG has gas species dependence. The nonlinearity of the sensitivity of BAGs is recommended to be evaluated in advance although BAGs has liner characteristics in principle. The QMS is similarly calibrated from the direct comparison with SRG, CDG, and/or BAG. For information on the calibration method of QMS, refer to ISO/TS 20175.

5.1.6 Temperature

The measurements shall be taken at an ambient temperature of (23 ± 7) °C and the temperature shall not change by more than 2K (peak-to-peak) during the measurement. The temperature of the vacuum chamber shall be recorded.

5.1.7 Activation method of NEG

NEGs shall be activated according to the method specified by manufacturer if available. Various methods are used to heat NEGs for activation such as induction heating, joule (resistance) heating, radiant heating, conductance heating and electron bombardment. The non-uniformity of temperature of NEG during heating shall be minimized. The temperature during the activation and activation time shall be measured and recorded.

5.1.8 Procedure of sample installation and activation

The procedures of sample installation and activation shall be followed to the operation manual provided by the manufacture if available. The general procedure is given in below.

a) A NEG sample to be tested is installed by using clean tools to the test chamber/dome.

b) The whole vacuum system is evacuated by the vacuum pumping system (see 5.1.4).

c) Bake-out the whole vacuum system including the test chamber/dome (e.g. 150 °C – 300 °C). The bake-out time is from several hours to several days depending on the condition of the vacuum system.

d) After cooling down the vacuum system, the NEG sample is heated up to the specified temperature and time to activate. This activation should be initiated under high vacuum conditions of approximately 1×10^{-4} Pa or lower.

e) After activation, cooling the sample down to the operating temperature.

f) Reactivation of NEG shall be performed before each test.

It should be avoided that the pressure during activation becomes too high to avoid the pollution of NEGs. NEG coatings should be especially taken care of the pollution during activation because their maximum sorbed amount is smaller than other getters. Also, a safety guide provided by the manufacturer should be kept for the handling of NEGs. In general, heating NEGs at low vacuum condition should be avoided.

BA gauges and QMS should be degassed between bake-out (procedure step c) and NEG activation (procedure step d) if necessary.

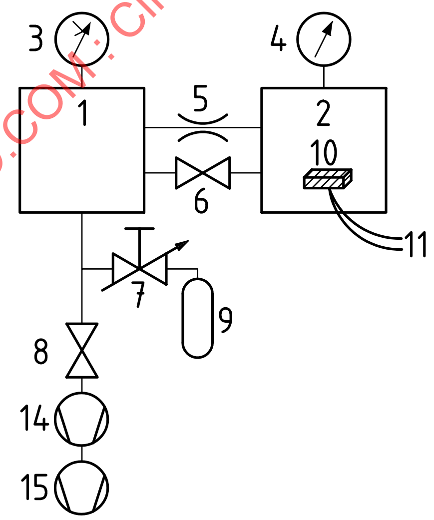
NOTE In the case of a CO test after a H_2 test, the influence of H_2 gas exposure on the pumping performance of CO is small. However, the opposite is not the case.

5.2 Throughput method for small NEG samples

5.2.1 Experimental setup

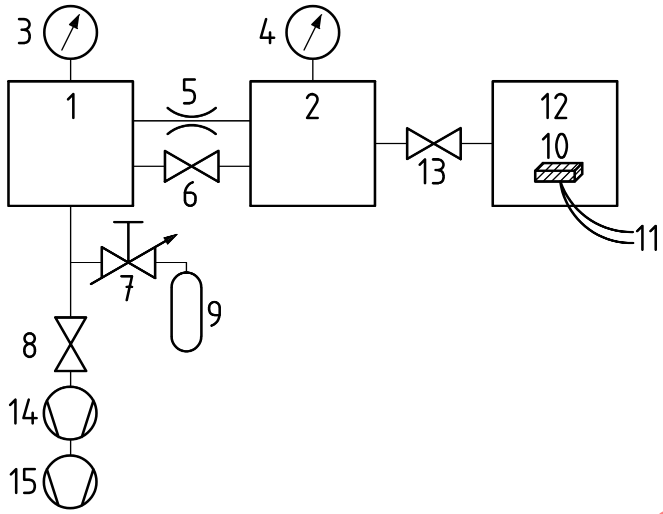
Figure 1 shows a schematic diagram of the measurement system of the throughput method for small NEG samples. This method is based on ASTM F798-97[1].[14] The system consists of two chambers, a gas manifold and a test chamber. The gas manifold includes a variable leak valve to introduce test gases, a BA gauge (BAG-1), and a turbomolecular vacuum pump. An isolation valve may be located between the manifold and the turbomolecular vacuum pump. An ion pump can also be added between the turbomolecular vacuum pump and the isolation valve to obtain lower base pressure. The test chamber shall include a test sample (NEGs or NEG pumps) including a heater to activate the sample, a thermometer to measure the sample temperature, and another BA gauge (BAG-2). The test chamber and the gas manifold are connected by the orifice with known conductance and a bypass valve. The conductance of the orifice C_0 is carefully selected to satisfy the requirement of the 5.1.3. Equipping a sample chamber with the test chamber can be useful as shown in Figure 1 b) to increase the efficiency of testing, but special care shall be taken so that the conductance between the test chamber and the sample chamber is sufficiently large.

The controller of BA gauges shall be set to be the constant emission current against the changing in the pressure. The lower emission current such as from 0,01 mA to 0,1 mA is recommended to decrease the influence of outgassing and pumping effect of the BA gauge.



a) Method without sample chamber

1) Withdrawn.



b) Method with sample chamber

Key

1 manifold
2 test chamber
3 BA gauge (BAG-1)
4 BA gauge (BAG-2)
5 orifice, C_0
6 bypass valve
7 variable leak valve
8 valve
9 gas cylinder
10 sample (NEG)
11 thermometer
12 sample chamber
13 gate valve
14 turbomolecular vacuum pump
15 roughing vacuum pump

Figure 1 — Throughput method for small NEG sample

5.2.2 Sample

Relatively small size of NEGs with the type of pill, disk, ring, strip, module, cartridge, NEG coating disk shall be tested by using either system shown in Figure 1a) or Figure 1b). NEG pumps with a small diameter of connection flange can also be tested by this system.

5.2.3 Determination of getter pumping speed, S, and sorption quantity, C_q

The flow rate, Q, passing through the orifice equals to that pumped by NEGs or NEG pumps with the pumping speed of S at the quasi-equilibrium condition. Then, the following relation is established as shown in Formula (1).

$$Q = C_0 (p_1 - p_2) = S \cdot p_2 \tag{1}$$

where C_0 is the conductance of orifice and should be noted that C_0 depends on gas species and temperature. This document strongly recommends selecting parameters such as the measurement pressure and the inner diameter of orifice so that the molecular flow condition is realized for gases passing through the orifice. The data analysis becomes much simple because the conductance C_0 becomes constant and is obtained analytically.

The pressure p_1 and p_2, which are the pressures in the gas manifold and test chamber, respectively, are obtained by Formulae (2) and (3):

$$p_1 = \frac{p_{R1} - p_{B1}}{K_1} = F_1 (p_{R1} - p_{B1}), \tag{2}$$

$$p_2 = \frac{p_{R2} - p_{B2}}{K_2} = F_2 (p_{R1} - p_{B2}), \tag{3}$$

where

p_{R1} is the pressure reading of BAG-1;

p_{B1} is the base pressure of BAG-1;

K_1 is the sensitivity of BAG-1;

F_1 is the correction factor of BAG-1;

p_{R2} is the pressure reading of BAG-2;

p_{B2} is the base pressure of BAG-2;

K_2 is the sensitivity of BAG-2;

F_2 is the correction factor of BAG-2;

The sensitivity K is equal to the inverse of the correction factor F.

Following the above relations, the getter pumping speed, S, and the sorption quantity, C_q, are obtained by Formulae (4) and (5):

$$S = \frac{C_0 (p_1 - p_2)}{p_2}, \tag{4}$$

$$C_q = \int_0^t Q_{pV} dt = C_0 \int_0^t (p_1 - p_2) dt, \tag{5}$$

where

Q_{pV} is the gas flow rate

t is the elapsed time

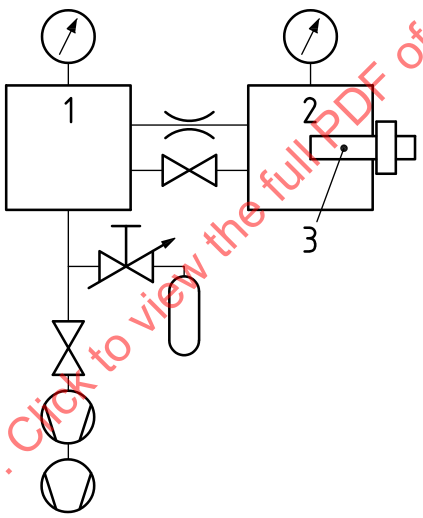
When the sample chamber is employed (see Figure 1b), the pressure in the test chamber may be different with that in the sample chamber. Then, the Formula (4) shall be corrected as follows[15].

$$S = \frac{C_0 (p_1 - p_2)}{p_2 - \frac{C_0}{C_p}(p_1 - p_2)}, \tag{6}$$

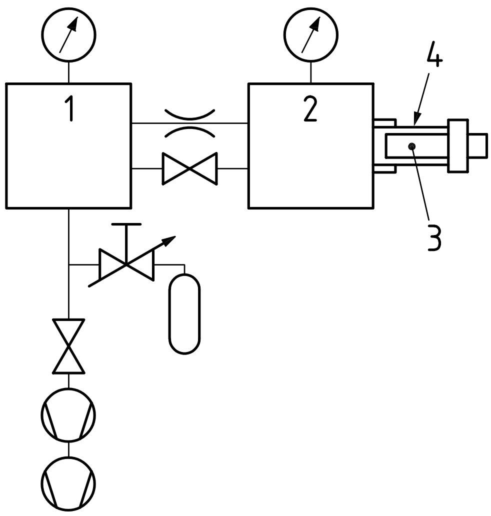
where C_p is the conductance of the pipe and the valve (key reference 13 in Figure 1 b)) between the test chamber and the sample chamber. Monte Carlo simulation is also useful to correct the pressure distribution in the test chamber and sample chamber[16-18].

NOTE 1 Commercially available BA gauges are calibrated for N_2 in general. Relative sensitivities of H_2, K_{H2}/K_{N2}, and CO, K_{CO}/K_{N2}, for N_2 are about 0,40 and 1,03, respectively, although those depend on the type and the operational parameter of BA gauge[19].

NOTE 2 When NEG pumps are tested by this method, the measurement result depends on the connection method to the test chamber. For example, the pumping speed when the NEG pump is inserted into the test chamber [see Figure 2a)] becomes larger than that when NEG pump is connected to the test chamber by using a nipple [see Figure 2b)]. Since the latter case is close to the method shown in 5.3, refer to the requirements specified in 5.3.



a) NEG pump is inserted into the test chamber



b) NEG pump is connected by using a nipple

Key

1 manifold
2 test chamber
3 NEG pump
4 nipple

Figure 2 — The connection method of NEG pump to the test chamber.

5.2.4 Determination of sticking probability, α

When the pressure distribution in the test chamber is homogeneous, in other words, the gas molecules in the test chamber hit on the NEG surface with the same probability independent on the position, the sticking probability, α, on the NEG surface is obtained by Formula (7). The sticking probability, α, corresponds to the intrinsic sticking probability or sticking coefficient, α_0, when the NEG surface is flat.

$$\alpha = \frac{4S}{A\bar{v}} \tag{7}$$

where

S is the getter pumping speed S obtained by Formulae (4) or (6) (m^3/s);

A is the apparent surface area of NEG (m^2);

$\bar{v}$ is the arithmetic mean velocity of gas molecule for the test gas ($=\sqrt{8RT/\pi M}$) (m/s);

R is ideal gas constant (=8,134 J/(mol K)) ;

T is the temperature (K) ;

M is the mass of gas molecule (kg) ;

NOTE 1 The assumption that the pressure distribution in the test chamber is negligible is satisfied only when the size of NEG sample is sufficiently small compared with that of the test chamber. If not, for example, when the NEG sample is located in the nipple as shown in Figure 2b), the α estimated by Formula (7) gives an incorrect value.

NOTE 2 The accuracy of α can be improved by calculating the pressure distribution in the test chamber by Monte Carlo simulation[16-18].

NOTE 3 The typical values of initial sticking probability measured by the method of 5.2 are listed in Table C.1 of Annex C.

5.2.5 Measurement procedure

a) Base pressures of both the test chamber and gas manifold shall be less than 1×10^{-6} Pa.

b) The bypass valve is closed. No significant increase of the pressure p_2 shall be observed.

c) The test gas is introduced into the gas manifold and adjusted by leak valve so that the pressure of p_2 becomes constant. The pressure ratio of p_1/p_2 shall be kept higher than 2.

d) Record the pressure p_1, p_2, and temperature of NEG at least with a suitable time interval. The several ten seconds of time interval should be suitable at the beginning of the measurement because of rapid change in getter pumping speed. It can be extended as the sorption quantity of NEG increases because the change becomes slow.

In the absence of specified request, p_2 should be set between 1×10^{-4} Pa and 1×10^{-3} Pa, preferably 4×10^{-4} Pa for applications in medium and high vacuum conditions. This test pressure is more than 100 times higher than the base pressure before the gas admitting at procedure step b) and is sufficiently low to realize the molecular flow condition in typical. In addition, it is within the linear response range of BA gauges and close to the calibration pressure range in typical.

For UHV applications the lower base and test pressures at p_2 may be required. For example, setting the test pressure from 10^{-8} Pa to 10^{-10} Pa is required for high performance surface analysis devices and electron microscopy systems.

The dependence of S, C_q, *and* C_c on NEG temperature may be measured. The S, C_q, *and* C_c of CO, for example, depend on the NEG temperature in general[7,8,12,13].

When the volume getter is dominant, for example H_2 pumping, note that the pumping speed may depend on the test pressure because it is competing reaction between gas injection onto the surface and diffusion into the bulk.

5.2.6 Measurement uncertainty

The measurement uncertainties of S and C_q are estimated from the uncertainty to measure p_1 and p_2 by BA gauges, the uncertainty of the orifice conductance, and the uncertainty due to the pressure distribution in the test chamber. In the case of α, the uncertainties of the apparent surface area of NEG, A, and the arithmetic mean velocity of gas molecule for the test gas, $\bar{v}$, are also added. A total uncertainty of several tens of percentage is sufficient for general purposes.

When the testing devise has a sample chamber as shown in Figure 1b), it is recommended that the sensitivity of two BA gauges are cross-checked by admitting the test gas after opening the bypass valve (key reference 6 in Figure 1) and closing the gate valve (key reference 13 in Figure 1).

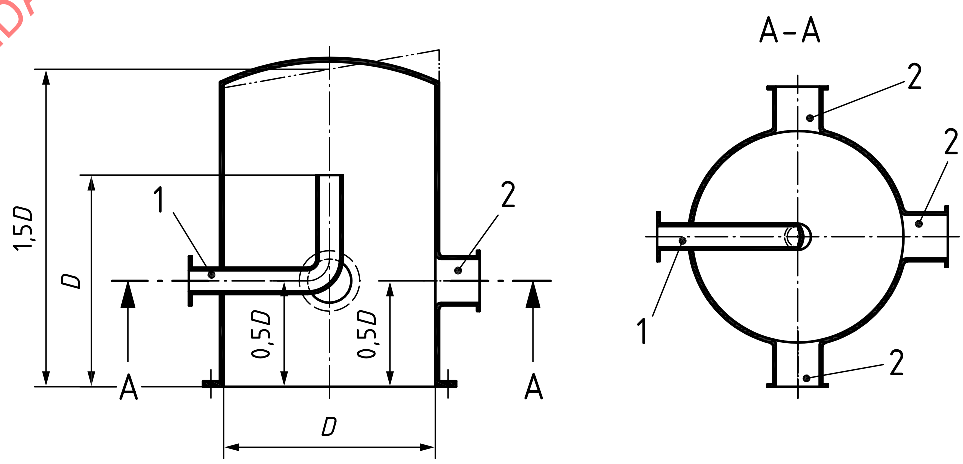
EXAMPLE References [15] and [18] include examples of the evaluation of measurement uncertainty.

5.3 Throughput method with test dome

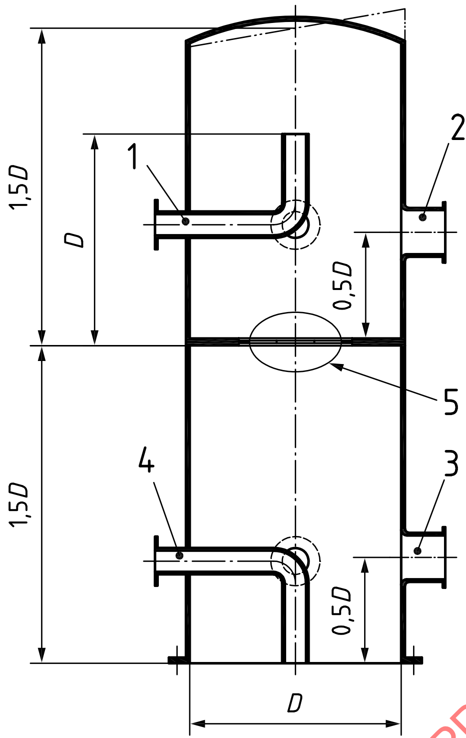
5.3.1 Experimental setup

Three types of test domes are shown in Figure 3. The test dome shown in Figure 3a) is allowed when a suitable gas flow measuring instrument is available. Leak elements such as small orifice, capillary, and sintered filter are used[20-25].

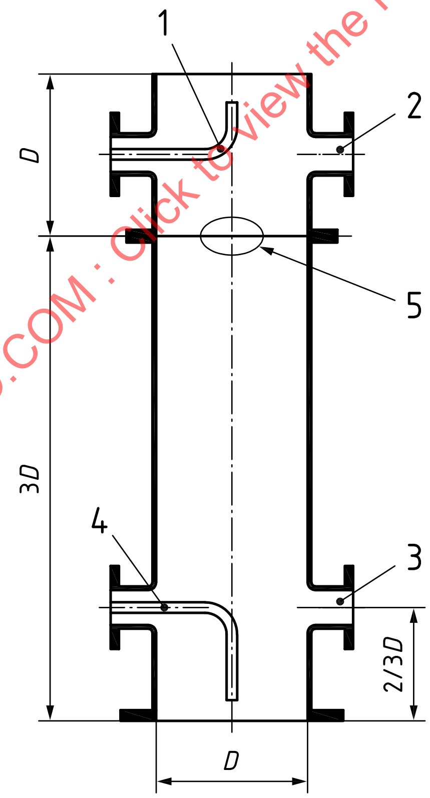
The test domes of Figure 3b) and Figure 3c) are used for the orifice flow method. The test dome Figure 3c) is the so-called Fischer- Mommsen dome.[26-30] The diameter and the thickness of the orifice shall be measured for the calculation of the conductance in advance. The calculation method of the molecular conductance is shown in Annex A. The conductance of the orifice C_0 is carefully selected so as to satisfy the requirements of the 5.1.3.



a) Test dome



b) Test dome



c) Fischer – Mommsen dome

Key

1 gas inlet

2 connections for BA gauge, mass spectrometer, and high vacuum pumping system

3 connections for BA gauge, mass spectrometer

4 gas inlet for vacuum gauge calibration and temperature measuring point, T_D

5 orifice

D inner diameter of test dome

a) SOURCE: Reproduced with permission from ISO 21360-1:2020, Figures 1.

b) SOURCE: Reproduced with permission from ISO 21360-1:2020, Figures 3.

c) SOURCE: Based on references [20-24].

Figure 3 — Three types of test domes for throughput method

NEG pumps or NEG coated chambers under test shall be located at the bottom of each test dome. Another high pumping system for rough pumping (5.1.4) shall be located at the connection flange labelled as key reference 2 in Figure 3 via an isolation valve.

The main problem in using the test dome is its pressure distribution. In the case of the test dome shown in Figure 3 a), the gauge position (key reference 2 in Figure 3) has been determined so that the pressure at the gauge position is comparable with that at the inlet of the NEG pumps or NEG coated chambers under test. In the case of the test dome shown in Figures 3b) and 3 c), the gauge position of downstream side of the orifice (key reference 3 in Figure 3) has been similarly determined. On the other hand, the gauge position of upstream side of the orifice (key reference 2 in Figure 3) has been selected so that the pressure at the gauge position is comparable with that at the inlet of the orifice. A different shape of test dome can be used when the pressure measured by vacuum gauges is compensated by considering the pressure distribution in the test dome.

5.3.2 Sample

Relatively large size of NEGs such as strip, module, cartridge, NEG pumps, and NEG coated tubes or chambers shall be tested by using this method.

5.3.3 Determination of getter pumping speed S and sorption quantity, C_q

The method to determine the pumping speed S is specified in ISO 21360-1:2020, 5.1 and 5.2. When the dome shown in Figure 3a) is used, the getter pumping speed S is obtained by the flow rate Q, which is determined by the flow meter, divided by the pressure p in the test dome ($S = Q/p$). This p is the value after subtracting the base pressure. When the dome shown in Figure 3b) or the Fischer-Mommsen dome shown in Figure 3c) is used, the pumping speed S is obtained by Formula (4) by using p_1 and p_2 to the upstream pressure for the orifice and the downstream one, respectively. The sorption quantity C_q is also obtained by the Formula (5).

5.3.4 Determination of sticking probability, α

It is impossible to determine the α by using Formula (7) except for strip, module, and NEG coating chambers with the small length to diameter ratio, because the pressure distribution in the NEG pump or NEG coating chamber under test is significant. However, the α can be obtained by comparing the calculation result by a Monte Carlo simulation.

NOTE 1 The typical values of initial sticking probability measured by the method of 5.3 is listed in Table C.1 of Annex C.

5.3.5 Measurement procedure

a) It is confirmed that the base pressure of the test dome is less than 1×10^{-6} Pa.

b) In the case of test dome Figure 3a), the isolation valve, which is located at key reference 2 in Figure 3 between the test dome and the high vacuum pumping system, is closed. No significant increase of the pressure p_2 is confirmed. In the case of test dome Figure 3b) or c), it is not necessary to close the valve located at the connection to TMP (key reference 2) but it can be throttled.

c) Test gas is introduced into the test dome through the gas inlet (key reference 1 in Figure 3). The test pressure p in the test dome Figure 3a) should be set more than twice as large as the base pressure before the gas admitting. The pressures p_1 and p_2 in the dome Figure 3b) and Figure 3c) is similar.

d) Record the flow rate Q and the pressure p for the test dome Figure 3a) or the pressures p_1 and p_2 for the test dome Figure 3b) or Figure 3c), and temperature of NEG at least with a suitable time interval.

NOTE 1 The test pressure p_2 is similarly chosen in 5.2.5.

NOTE 2 The dependence of S and the sorption capacity C_C on NEG temperature can be measured. The S and C of CO, for example, depend on the NEG temperature in general.

NOTE 3 The pumping speed can depend on the gas flow rate when volume getter acts, for example H_2 pumping, because it is a competing reaction between gas injection onto the surface and diffusion into the bulk.

5.3.6 Measurement uncertainty

When the dome shown in Figure 3a) is used, the measurement uncertainties of S and C_q are estimated from the uncertainty to measure the flow rate Q and p, and that due to the pressure distribution in the test chamber. When the dome shown in Figure 3b) or Figure 3 c) is used, the measurement uncertainty is estimated from the uncertainty to measure p_1 and p_2 by BA gauges, the orifice conductance, and the pressure distribution in the test chamber. In the case of α, the uncertainties of the apparent surface area of NEG, A, and the arithmetic mean velocity of gas molecule for the test gas, $\bar{v}$, are also added. The total uncertainty of several tens of percentage is sufficient for general purposes.

When the domes shown in Figure 3b) 3c) are used, it is recommended that the sensitivities of two BA gauges are cross-checked by admitting the test gas from the gas inlet (labelled as key reference 4) in Figure 3.

5.4 Transmission method for NEG coatings

5.4.1 Experimental setup

Schematic diagrams of the measurement system of transmission method for NEG coating are shown in Figure 4.[31,32] In the case of the closed design illustrated in Figure 4a), the test gas is admitted at right-hand end of the coated pipe by the variable leak valve. Although leak elements such as small orifice, capillary, and sintered filter may be also used instead of the variable leak valves, special care is necessary to select their conductances to avoid the rapid saturation of sorption capacity. The gas passes through the coated tube and is absorbed by the NEG coated wall. The pressure p_1 at the right-hand end of the coated pipe and the p_2 at the left-hand end are measured by BAG-1 and BAG-2, respectively. Using the extractor gauge or similar is recommended when the α is expected to be close 1 and/or the ratio of length to diameter of the coated pipe is large because the increase of p_2 becomes small at that time. Also, the accumulation of CH_4 in the apparatus during H_2 measurement may disturb the accurate measurement of p_2 because the pumping performance of NEG coating for CH_4 is poor at room temperature. Then, using QMS is recommended to measure the p_2. A cold trap by liquid nitrogen around p_2 is also useful to decrease the partial pressure of CH_4.

In the case of the open design Figure 4b), the test gas is admitted at left-hand end of the coated pipe and measured the inlet pressure p_1 by BAG-1. The outlet pressure p_2 at the right-hand end of the coated pipe is measured by BAG-2. The orifice located between the outlet of the pipe and TMP is necessary to estimate the pumping efficiency of the test gas. The conductance of orifice (key reference 7 in Figure 4 b)) is recommended to be around 100 smaller than the effective pumping speed of TMP (key reference 8 in Figure 4 b). This is because gas molecules flowing back through the orifice are negligible, making Monte Carlo simulations easy to perform. Using the extractor gauge, QMS, or similar is also recommended to measure p_2. When QMSs are used to measure the p_1 and p_2, they should be calibrated carefully before and/or after test with the test gas (see 5.1.5).

The transmission method requires obtaining the relation between the pressure ratio of p_1 to p_2 and the sticking probability α by Monte Carlo simulation, which depends on the pipe shape. An example in the case of the length to the radius ratio (L/R) equals to 40 is shown in Figure 5. Here, the α is assumed to be uniform along the NEG coated pipe in general. The sticking probability α is obtained from the measurement result of p_1/p_2 ratio and this relation. Since the gas is injected from the one end of NEG coated pipe, the sorption amount of gas molecules depends on the position of pipe. Therefore, the change in the α cannot evaluate as a function of the sorption quantity, C_q, as well as the throughput method, although the overall sorption amount in the coated pipe is available. Instead, the p_1/p_2 ratio is plotted as a function of the sorption quantity C_q, the elapsed time, or similar.

There are no strict requirements for the chamber shape and the gauge position in this method. Instead, they shall be accurately modelled on the Monte Carlo simulation as possible.

5.4.2 Sample

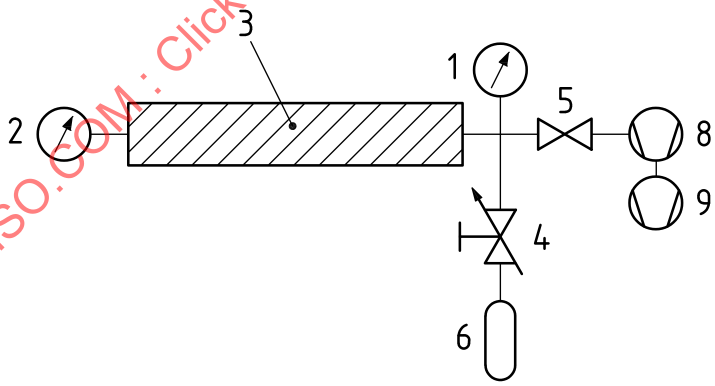
NEG coated pipes shall be tested by using this system.

5.4.3 Determination of average getter pumping speed per unit area, S_A, and sorption quantity C_q

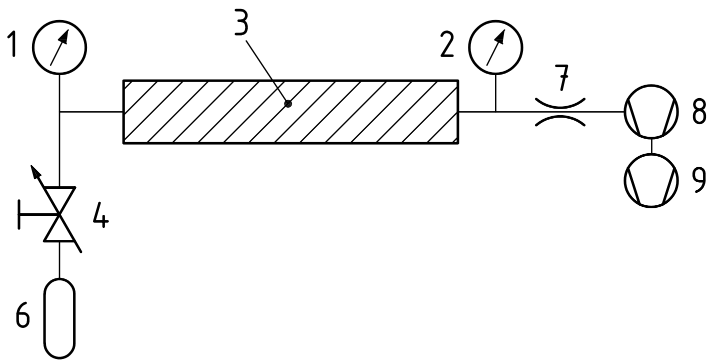
The average getter pumping speed per unit area of NEG coating, S_A, is obtained by Formula (8),

$$S_A = \frac{1}{4}\bar{v}\alpha, \tag{8}$$

where α is the sticking probability determined by the procedure shown in 5.4.4. The sorption quantity C_q cannot be measured by this method.



a) closed design

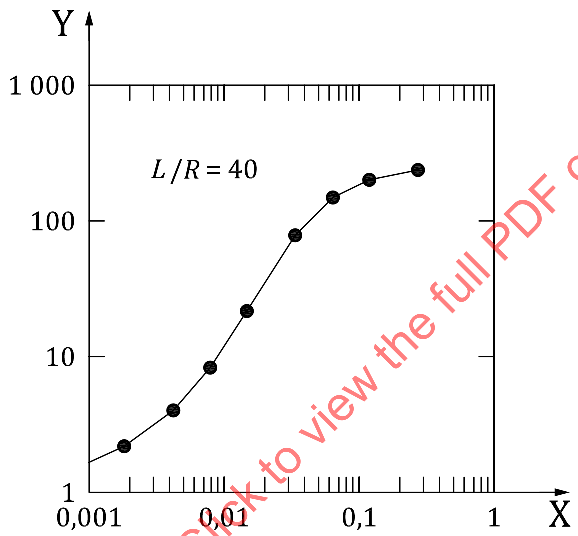


b) open design

Key

1 BA gauge (BAG-1)
2 BA gauge (BAG-2) and/or QMS
3 sample (NEG coated pipe)
4 variable leak valve
5 isolation valve
6 gas cylinder
7 orifice
8 turbomolecular vacuum pump
9 roughing vacuum pump

Figure 4 — Experimental setup for transmission method



Key

X sticking probability
Y p_1/p_2

NOTE The length to the radius ratio, L/R, of the NEG coated pipe is 40.

Figure 5 — Example of the relation between the p_1/p_2 ratio and the sticking probability calculated by Monte Carlo simulation

5.4.4 Determination of sticking probability, α

The sticking probability, α, is determined from the measurement result of the p_1/p_2 ratio during test gas admission and the result of Monte Carlo simulation as shown in Figure 5. The pressure p_1 and p_2 are similarly determined by Formulae (2) and (3).

NOTE 1 The typical values of initial sticking probability measured by the method of 5.4 are listed in Table C.1 of Annex C.

5.4.5 Measurement procedure

a) It is confirmed that the base pressure of the test chamber is less than 1×10^{-6} Pa.

b) In the case of Figure 4a closed design, it is not necessary to close the isolation valve [key reference 5 in Figure 4a)] but it may be throttled. In the case of Figure 4b) open design, the isolation valve between the sample [key reference 3 in Figure 4 b)] and the roughing vacuum pump [not shown in Figure 4b)] is closed.

c) Test gas is introduced into the test chamber through the variable leak valve (key reference 4 in Figure 4). The downstream pressure of p_2 during the test is recommended to set twice as large as the base pressure before the gas admitting.

d) Record the pressure p_1, p_2, and temperature of NEG at least with a suitable time interval.

The conductance of orifice [key reference 7 in Figure 4b)] is recommended to be around 100 smaller than the effective pumping speed of TMP in Figure 4b). This is because gas molecules flowing back through the orifice are negligible, making Monte Carlo simulations easy to run.

NOTE The test pressure p_2 is similarly chosen in 5.2.5.

5.4.6 Measurement uncertainty

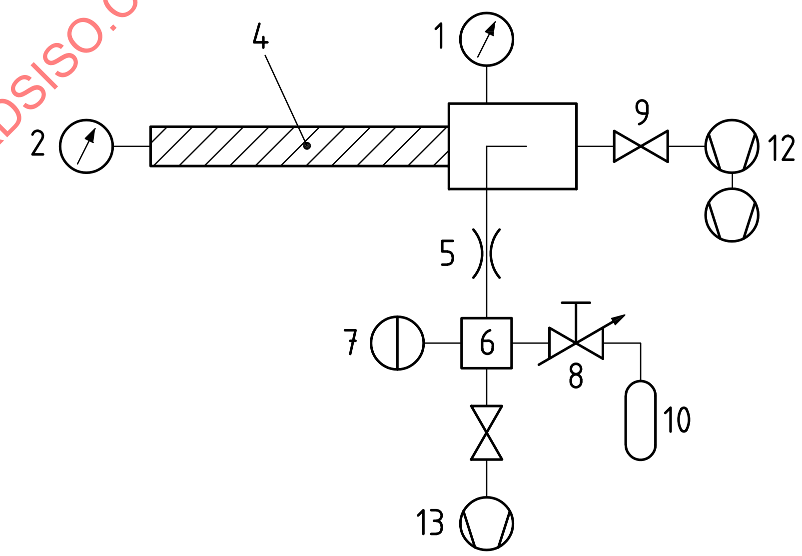
The measurement uncertainty of α is estimated from the uncertainty to measure p_1 and p_2 by BA gauges or QMS, and that due to the Monte Carlo calculation including the interpolation error between calculation points.

5.5 Combination of transmission method and throughput method with test dome

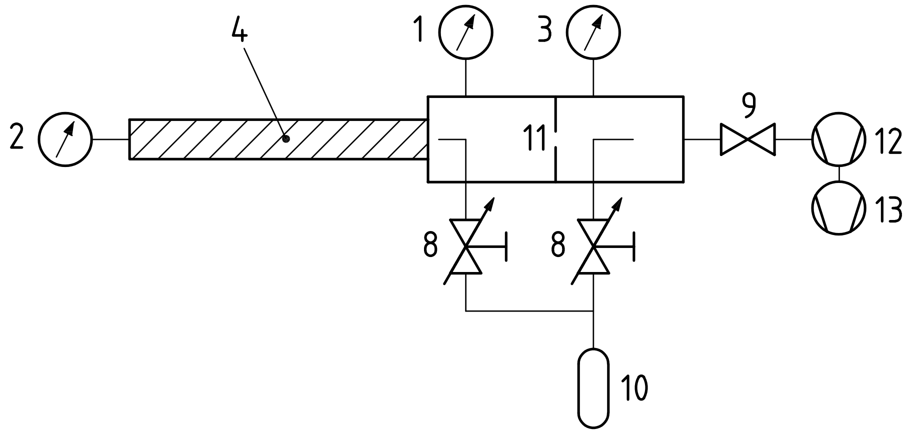
The transmission method (see 5.4) is often combined with the throughput method with test dome (see 5.3) to measure the sorption quantity C_q of NEG coated pipes. Thus, the sticking probability α is determined by the transmission method. On the other hand, the C_q and the overall pumping speed of the NEG coated pipe are determined by the throughput method. The typical experimental setup is shown in Figure 6. Detail experimental setups and procedures are referred by those of each method.

NOTE The α determined by the transmission method is often larger than the value obtained by the throughput method by Formula (7). This is because the NEG coating around the inlet saturates by test gas first and then it acts as a connection pipe when the throughput method is applied.

When the α is also determined by the transmission method only, the chamber shape and the position of BAG-2 in Figure 6 is flexibly designed. Instead, they shall be accurately modelled on the Monte Carlo simulation as possible. On the other hand, the gauge position of BAG-3 in Figure 6b) is carefully selected so that the pressure at the gauge position is comparable with that at the inlet of the orifice.



a) Method using leak element



b) Method using orifice

Key

1 BA gauge (BAG-1) and/or QMS
2 BA gauge (BAG-2) and/or QMS
3 BA gauge (BAG-3)
4 sample (NEG coated chamber)
5 leak element with known conductance
6 gas reservoir
7 capacitance diaphragm gauge
8 variable leak valve
9 isolation valve
10 gas cylinder
11 orifice
12 turbomolecular vacuum pump
13 roughing vacuum pump

Figure 6 — Two examples of combination of transmission method and throughput method with test dome

6 Reporting

6.1 General

The following elements shall be included in the report. Regarding chemical composition, there are three types of expressions: mass percentage (mass %), weight percentage (wt %), and atomic percentage (at %). The mass percentage and the weight percentage are comparable in practice. But the atomic percentage is different from the others because the atomic mass is taken it into account.

6.2 Small size of NEGs with the structure of pill, disk, ring, strip, module and cartridge

— type, manufacture's serial number and/or lot number of manufacturer

— chemical composition of NEG (mass % or wt %)

— geometric shape such as diameter or height

— geometric surface area

— mass of NEG

— test method of pumping performance
— activation temperature and duration
— temperature of sample and vacuum chamber during gas injection
— test gas
— test pressure and base pressure before gas admission
— diagram: pumping speed vs sorption quantity (see Figure B.1)
— initial pumping speed of NEG
— sorption capacity (optional)

6.3 NEG pumps

— type, manufacture's serial number and/or lot number of manufacturer
— chemical composition of NEG (mass % or wt%)
— flange type and size
— geometric surface area of NEG
— mass of NEG
— test method of pumping performance
— installing method of NEG pump to the test chamber (e.g. with or without nipple)
— activation temperature and duration
— temperature of sample and vacuum chamber
— test gas
— test pressure and base pressure before gas admission
— diagram: pumping speed vs sorption quantity (see Figure B.1)
— initial pumping speed of NEG
— sorption capacity
— reproducibility of pumping speed after repeating activation [optional]

6.4 NEG coatings

— type, manufacture's serial number and/or lot number of manufacturer
— chemical composition of NEG [mass %, wt % or atomic %]
— material of coated chamber or pipe
— geometric dimensions of the coated chamber or pipe such as diameter or height
— geometric surface area of NEG coating
— thickness of NEG coating
— test method of pumping performance

— base pressure at the test

— activation temperature and duration

— temperature of sample and vacuum chamber

— test gas

— test pressure and base pressure before gas admission

— initial sticking probability

— initial average pumping speed per unit area

— coating method (sputtering, evaporation, etc)

— diagram: pumping speed vs sorption quantity when throughput method is used (see Annex B)

— diagram: sticking probability vs p_1/p_2 ratio vs time calculated by Monte Carlo simulation when transmission method is used (see Figure 5)

— diagram: sticking probability vs CO sorption quantity (see Figure B.2) (optional)

— SEM image of NEG coating (optional)

— temperature dependence of surface chemical composition measured by XPS (optional)

Annex A
(informative)

Calculation method of the molecular conductance of the orifice

The molecular conductance of the orifice is calculated by Formula (A.1):

$$C_0 = \frac{1}{4}\bar{v}A_0 W, \tag{A.1}$$

where

$\bar{v}$ is the arithmetic mean velocity of gas molecule for the test gas $(=\sqrt{8RT/\pi M}$ (m/s));

R is ideal gas constant (=8,134 J/(mol K)) ;

T is the temperature (K) ;

M is the mass of gas molecule (kg) ;

A_0 is the cross-sectional area of the orifice $(=\pi(d/2)^2$ (m²));

d is the diameter of the orifice (m);

W is the transmission probability of the orifice;

The W is obtained by the following approximation Formula (A.2) with the relative error of less than 0,6 %[33]

$$W = \frac{14+4(l/d)}{14+18(l/d)+3(l/d)^2}, \tag{A.2}$$

where l is the thickness of the orifice.