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

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

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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

A list of all parts in the ISO 21360 series can be found on the ISO website.

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

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 NEG with other vacuum pumps is that the pumping speed of 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 at 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

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

Note 1 to entry: non-evaporable getter vacuum pumps are mounted on a vacuum flange in typical. The internal heaters and the controller for the activation may be included.

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 is 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 getter (or 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 the valves of 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). 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) to keep the downstream pressure p_2 the specified value 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 not to pollute by such as

oil vapor and dust. In addition, it is useful to adjust the pressure p_1 or to keep the inside of vacuum chamber in vacuum while the system is stopped.

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.