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Sputter-ion pumps — Measurement of performance characteristics —

Part 1:
Pumps with a volume rate of flow greater than 10 l/s

Pompes ioniques à pulvérisation — Mesurage des caractéristiques de fonctionnement —
Partie 1: Pompes avec débit de pompage supérieur à 10 l/s

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Descriptors: vacuum technology, vacuum apparatus, pumps, vacuum pumps, ion pumps, tests, performance tests, measurement.

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 3556-1 was prepared by Technical Committee ISO/TC 112, *Vacuum technology*, Sub-Committee SC 3, *Measurement of the performance characteristics of vacuum pumps*.

ISO 3556 consists of the following parts, under the general title *Sputter-ion pumps — Measurement of performance characteristics*:

— *Part 1: Pumps with a volume rate of flow greater than 10 l/s*

Introduction

The purpose of this International Standard is to ensure that measurements of the performance characteristics of sputter-ion pumps are, as far as possible, carried out by uniform procedures and under uniform conditions. It is hoped that, as a result, measurements conducted by different manufacturers or in different laboratories, and statements of performance quoted in manufacturers' litterature, will be on a properly comparable basis to the benefit of both user and manufacturer.

It is envisaged that ISO 3556 will, in due course, deal comprehensively with the measurement of the performance of the main types of sputter-ion pumps. In order, however, that useful agreements of more restricted scope may be implemented with the least possible delay, it is intended to publish this International Standard in parts.

Sputter-ion pumps — Measurement of performance characteristics —

Part 1:
Pumps with a volume rate of flow greater than10 l/s

1 Scope

This part of ISO 3556 specifies methods of measuring the performance characteristics of sputter-ion pumps of which the volume rate of flow (pumping speed) is greater than 10 l/s. It concerns methods of measuring the following characteristics:

a) the ultimate total pressure obtained within a test dome of specified form and dimensions;

b) the relation between the volume rate of flow for nitrogen and the pressure, after stabilization of the pump with nitrogen;

c) the relation between the volume rate of flow and the pressure after subjecting the pump to a regenerative bake-out procedure.

The range of pressures for the determination of the volume rate of flow should be between at least $1,0 \times 10^{-3}$ Pa[1] and $1,0 \times 10^{-6}$ Pa.

It is to be expected that new pumps may deteriorate in time because during their life they may be subjected to unknown gas loads which will bring about irreversible changes in performance. The tests recommended are therefore for new pumps. In the case of used pumps, the tests will enable performance comparisons to be made provided that they are conducted under the vacuum conditions described below.

2 Definitions

For the purposes of this part of ISO 3556, the following definitions apply.

2.1 volume rate of flow (pumping speed), S: Under ideal conditions, the volume of gas which flows in unit time through the pump inlet. For practical purposes, however, the volume rate of flow of a given pump for a given gas is, by convention, taken to be the quotient of the throughput (Q) of that gas and the equilibrium pressure (p) at a specified position in a given test dome and under specified conditions of operation. Thus $S = Q/p$.

The unit adopted for the volume rate of flow is litre per second (l/s).

2.2 test dome (test header): A chamber of specified form and dimensions attached to the inlet of the pump through which a measured flow of gas may be admitted to the pump, and which is equipped with means of pressure measurement.

A test dome for the determination of ultimate total pressure is described in 3.4 and a test dome for the determination of volume rate of flow in 3.5.

2.3 ultimate pressure: The limiting pressure approached asymptotically in the dome, with the gas inlet valve closed and the pump in normal operation following the regenerative bake-out as described in 4.1.

3 Apparatus and test gas

3.1 General

All total pressures shall be quoted as equivalent nitrogen pressures. An adequate hot-cathode ionization gauge shall be used for the measurement of total pressure in the range $4,0 \times 10^{-2}$ Pa to

1) 133 Pa = 1 Torr = 1,33 mbar; 1 Pa = $7,5 \times 10^{-3}$ Torr = 1×10^{-2} mbar

1.0×10^{-8} Pa. The ionization gauges used shall be calibrated with nitrogen. The calibration of the ionization gauges is liable to change and so the calibration shall be checked periodically.

3.2 Hot-cathode ionization gauges

Two hot-cathode ionization gauges are required. In the region of pressure concerned, these gauges shall have a known calibration curve and shall not have an error in pressure reading exceeding ± 10 %. The magnetic flux density in the vicinity of these gauges due to stray flux from the magnet of the sputter-ion pump shall be at a sufficiently low level to ensure that the sensitivity of the gauges is not affected by more than ± 3 %.

3.3 Residual gas analyser

A mass spectrometer used as a residual gas analyser (RGA) may be fitted to the test dome used for the measurement of the volume rate of flow of the pump. Whilst this RGA, is not used in arriving at the specification of performance, it is a valuable facility for leak detection of the test system and for the study of inert gas partial pressures, hydrogen desorption and possible hydrocarbon contamination. The use of an RGA is not recommended in the ultimate pressure measurements, however, because it may give a sufficient outgassing load to limit the achievable lowest ultimate pressure.

3.4 Test dome for determination of ultimate total pressure (see figure 1)

The test dome shall be of circular cross-section and of inside diameter D. For large sputter-ion pumps of inlet diameter of 100 mm or greater, the inside diameter D shall be equal to that of the inlet mouth of the pump to within ± 1,5 mm for pumps of up to 300 mm inlet diameter and to within ± 3,0 mm for pumps of inlet diameter greater than 300 mm. For sputter-ion pumps of inlet diameter less than 100 mm, the inside diameter of the test dome shall be 100 mm, irrespective of the pump size. The height of this cylindrical test dome shall be 3 $D/2$.

The top of the test dome shall be flat. Gaskets between the dome and the flange at the inlet of the sputter-ion pump shall be as recommended by the manufacturer. The dome shall be able to withstand baking under vacuum to 400 °C, and is to be supplied with piping to a bakeable closure valve for connection to a roughing pump.

The axis of the piping to the ionization gauge attached to the test dome shall project perpendicularly outwards from the wall of the dome and be at height $D/2$ above the base of the dome flange. This piping shall contain a right-angle bend upwards to the vertically mounted gauge and shall have a conductance for nitrogen of more than 10 l/s.

3.5 Test dome for determination of volume rate of flow (see figure 2)

The test dome shall be of circular cross-section and of inside diameter D. For large sputter-ion pumps of inlet diameter of 100 mm or greater, the inside diameter D shall be equal to that of the inlet mouth of the pump to within ± 1,5 mm for pumps of up to 300 mm inlet diameter and to within ± 3,0 mm for pumps of inlet diameter greater than 300 mm. For sputter-ion pumps of inlet diameter less than 100 mm, the inside diameter of the test dome shall be 100 mm, irrespective of the pump size.

The total height of this cylindrical test dome shall be 3 D, and it shall have at a height of 3 $D/2$ above the bottom flange of the dome, a thin flat metal plate furnished with a central circular orifice O of diameter d and thickness a. The relation a/d shall be less than 0,1 D for sputter-ion pumps of inlet diameter equal to or greater than 100 mm.

For pumps with an internal diameter of less than 100 mm, the diameter of the orifice is selected in relation to the expected nominal pump volume rate of flow of such that the indicated pressure ratio in the two sections X and Y of the test dome lies in the range between 5 and 100.

The upper section Y of this dome shall have two cylindrical tubes each projecting perpendicularly outwards from the dome wall. One of these tubes has its axis at a height $D/2$ above the plane of the plate containing the central orifice and contains a right-angle bend upwards to the vertically mounted gauge at P_1 and shall have a conductance for nitrogen of more than 10 l/s.

The other tube has a gas inlet containing a right-angle bend and which terminates along the central axis of the dome with its exit at a distance $D/2$ below the flat top of the dome. This gas inlet tube is furnished at its entrance with a bakeable control valve.

The lower section X of the test dome has three cylindrical tubes, each projecting perpendicularly outwards from the dome wall and each with its axis at a height $D/2$ above the bottom flange of the dome. One of these tubes is connected to the residual gas analyser (RGA) and the second to a bakeable valve; the third contains a right-angle bend upwards to the vertically mounted gauge at P_2 and shall have a conductance of more than 10 l/s.

3.6 Test gas

The test gas shall be nitrogen of purity 99,6 % or higher.

4 Procedure

4.1 Measurement of ultimate total pressure

First undertake a regenerative bake-out procedure.

With the test dome (3.4) mounted on its inlet flange, evacuate the sputter-ion pump by means of a suitable roughing pump to below the recommended starting pressure and check for leaks. Where necessary, precautions shall be taken by suitable trapping to prevent the ingress into the sputter-ion pump of fluid vapours from the roughing pump.

With the roughing pump in operation, bake the sputter-ion pump and the test dome for at least 4 h at a temperature of 300 °C.

Operate the sputter-ion pump with its normal power supply unit. Isolate the roughing pump by closure of the bakeable valve, and bake the sputter-ion pump and test dome for an additional period of 10 h at a temperature of 300 °C. If the pressure during this bake-out rises above 5×10^{-2} Pa due to a large degassing load, switch off the oven heater until the pressure falls again to about 1×10^{-3} Pa and then switch on again.

Degas the electrodes of the ionization gauge by electron bombardment or another heating process following the procedure recommended by the manufacturer of the gauge. Undertake this degassing during the bake-out, again at the end of the bake-out period, and additionally at least after every 24 h period.

Stop the bake-out but leave the pump in operation. Allow the test dome to cool until it is at a temperature between 15 °C and 25 °C. This temperature shall be maintained within ± 3 °C for a subsequent period of 48 h. The pressure measured at this time is the ultimate pressure provided that there are no significant variations in its value.

Note the values of the voltage applied to the pump and the current through the pump at the specific pressure. The maximum and minimum ambient temperature prevailing should also be recorded.

4.2 Measurement of rate of flow of gas during determination of volume rate of flow

Install the test dome for the volume rate of flow determination (3.5) above the sputter-ion pump.

The gas inlet to the upper section Y of the test dome (see figure 2) is via a bakeable control valve to establish within Y a pressure p_1 as measured by the ionization gauge in position P_1, whilst the pressure p_2 obtained within the lower section X is measured by an identical gauge in position P_2.

The throughout, Q, of the pump into the section Y of the test dome is determined (taking into account the appropriate Clausing factor correction for the thickness-to-diameter ratio of the orifice used) by the following equation:

$$Q = C(p_1 - p_2) \qquad \ldots (1)$$

where C is the calculated conductance for nitrogen at the orifice O.

The maximum value of the pressure p_1, in pascals, is

$$p_{1,\,max} = \left(\frac{5 \times 10^{-1}}{d} \right)$$

where d is the diameter, in millimetres, of the orifice O, to ensure that C is a true molecular conductance.

From the definition of the volume rate of flow of the pump, S, it follows that

$$S = \frac{Q}{p_2} \qquad \ldots (2)$$

Substitution from equation (1) into equation (2) gives

$$S = C\left(\frac{p_1 - p_2}{p_2} \right) = C\left(\frac{p_1}{p_2} - 1 \right) \qquad \ldots (3)$$

The orifice conductance may be calculated using the formula

$$C = \sqrt{\frac{R\,T\,\pi}{32M}} \times \frac{1}{1 + \frac{a}{d}}\, d^2 \qquad \ldots (4)$$

where

- R is the ideal gas constant, in joules per mole kelvin;
- T is the thermodynamic temperature, in kelvins;
- M is the molar mass of the gas, in kilograms per mole;
- a is the thickness of orifice plate, in metres;
- d is the orifice diameter, in metres;
- C is the conductance, in litres per second ($C = 10^3$ l/s).

In equation (4), $R = 8{,}314$ J/(mol K), $T = 299$ K(20 °C) and $M_{N_2} = 28 \times 10^{-3}$ kg/mol.

Therefore, for nitrogen at 20 °C

$$C_{N_2} = 92\,368 \frac{d^2}{1 + \frac{a}{d}} \text{ l/s}$$

It is recommended to accept that

$$C = \frac{S}{10} \qquad \ldots (5)$$

The orifice conductance may be calculated using equations (4) and (5) and the values given for R, T and M.

4.3 Determination of rate of flow of the pump after stabilization

Install the test dome for determination of the pumping volume rate of flow (3.5) above the sputter-ion pump. First undertake the regenerative bake-out procedure as required for the measurement of ultimate pressure (see 3.1).

Establish the equilibrium total pressure in the test dome and measure it at position P_2.

Then pump a total quantity of nitrogen which is in excess of $4S$ l/s, where S is the nominal volume rate of flow in litres per second. This quantity of nitrogen is needed to stabilize the pump.

After pumping this quantity of nitrogen, allow the pump to reach its equilibrium pressure without bake-out. This pressure shall not exceed 0,2 of the lowest pressure at which the volume rate of flow shall be measured.

Then increase the pressure in steps (three per decade) by admitting nitrogen to the test dome to establish the measured pressure p_2 over the range from the lowest value up to 1×10^{-3} Pa. The pressure p_1 in the section Y of the test dome above the orifice (see figure 2) shall not exceed $p_{1,\ max}$, otherwise molecular flow through the orifice cannot be assumed.

The pressure should then be decreased in steps to check for any variation in speed measurements for at least a third of the number of previously measured points. It is necessary to wait for a considerable period of time (sometimes a few hours) between pressure settings to ensure that equilibrium conditions prevail. For this purpose, equilibrium conditions are said to have been established when any increasing or decreasing trend of the measured volume rate of flow or of the indicated pressure does not exceed ± 5 % in a period of time of the order of 1 h.

4.4 Test for reversibility of the regenerative procedure following stabilization

Repeat the regenerative bake-out after measurement of the volume rate of flow and measure the equilibrium pressure. This value shall not be more than 2 to 3 times that found just prior to the regenerated volume rate of flow measurements. This procedure is to establish that the performance of the pump has not been significantly modified by the stabilization process.

4.5 Determination of the regenerated volume rate of flow for nitrogen

Carry out the regenerative bake-out procedure as required for the measurement of ultimate pressure with the test dome for the volume rate of flow determination installed above the sputter-ion pump.

Then repeat the procedure described in 4.3. The volume rate of flow measurements should be carried out for at least a third of the number of the previously measured points.

5 Test results

The ultimate pressure shall be recorded in pascals.

The saturated volume rate of flow as a function pressure shall be recorded as a graph of volume rate of flow (linear or logarithmic scale) in litres per second against pressure (logarithmic scale).

The nominal volume rate of flow is quoted as the maximum volume rate of flow obtained on the stabilized volume rate of flow characteristics; it is used as a brief specification of the pump. The pressure at which the nominal volume rate of flow is quoted shall be stated.

The regenerated volume rate of flow against pressure shall be recorded as a graph of volume rate of flow (linear or logarithmic scale) in litres per second against pressure (logarithmic scale).

6 Test report

The reported results of ultimate total pressure and of volume rate of flow against pressure characteristics shall include a statement of the maximum and minimum ambient temperature in the immediate vicinity of the test dome.

If a residual gas analyser is used, the mass spectrum of the residual gases during the test shall be given.

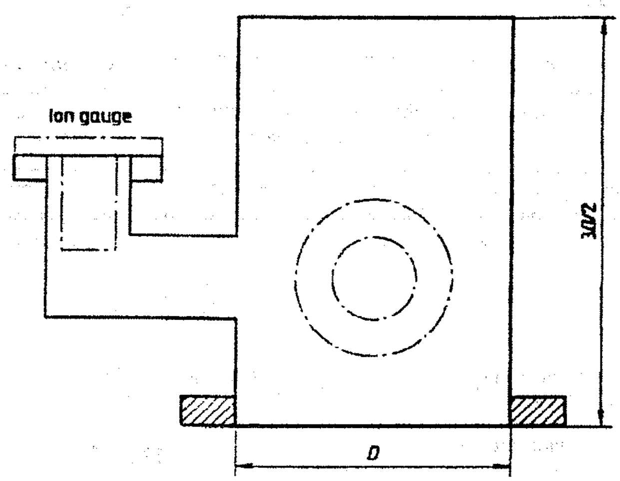

Figure 1 — Test dome for measurement of ultimate pressure

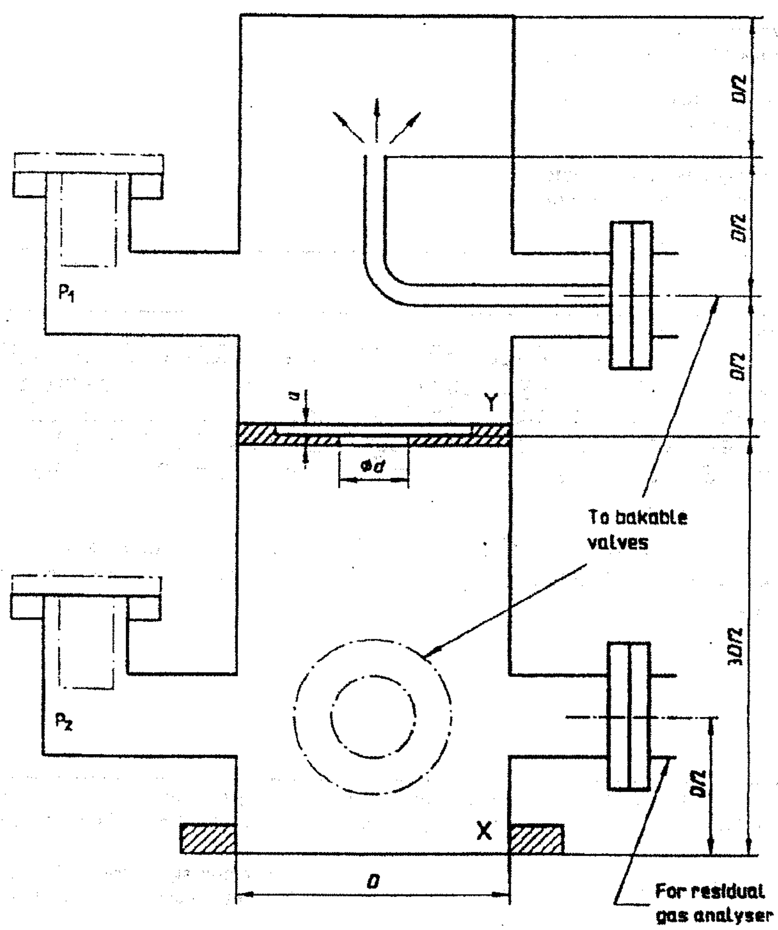

Figure 2 — Test dome for measurement of volume rate of flow

EXPLANATORY REPORT **RAPPORT EXPLICATIF**	ISO/DIS 3556/1.2
will supersede: remplacera: ISO/CD 3556/1	
ISO/TC 112 SC 3	Secretariat GOST

This form should be sent to the ISO Central Secretariat, together with the English and French versions of the committee draft, by the secretariat of the technical committee or sub-committee concerned (see 2.4.6 of part 1 of the IEC/ISO Directives)

Ce formulaire doit être envoyé au Secrétariat central de l'ISO en même temps que les versions anglaise et française du projet de comité, par le secrétariat du comité technique ou du sous-comité concerné (voir 2.4.6 de la partie 1 des Directives CEI/ISO)

The accompanying document is submitted for circulation to member body vote as a DIS, following consensus of the P-members of the committee obtained

Le document ci-joint est soumis, pour diffusion comme DIS, au vote comité membre, suite au consensus des membres (P) du comité obtenu

on / le 1975-05-21

☐ at the meeting of / à la réunion du TC...... /SC......: see / voir resolution No. / nº .... in / dans le document ....

☒ by postal ballot initiated on / par un vote par correspondance démarré le 1974-11-21

P-members in favour:
Membres (P) approuvant le projet:

P-members voting against:
Membres (P) désapprouvant:

P-members abstaining:
Membres (P) s'abstenant:

P-members who did not vote:
Membres (P) n'ayant pas voté:

Remarks/Remarques

ISO/DIS 3556/1 was balloted in 1975 and approved by the required majority. However, it was not returned to the Central Secretariat in due time evidently because of the intention of the BSI Secretariat to relinguish its duties which indeed occurred in 1976. Since then the development of DIS 3556/1 was actually discontinued.

The SC 3 Secretariat (GOST) has reexamined the document and now submits the revised English and French versions for the second combined vote.

I hereby confirm that this draft meets the requirements of part 3 of the IEC/ISO Directives
Je confirme que ce projet satisfait aux prescriptions de la partie 3 des Directives CEI/ISO

Date 91 - 11 - 22

Name and signature of the secretary
Nom et signature du secrétaire

(N. Levina)