
Methods of Calibrating leak-detectors of the mass-spectrometer-type used in the field of vacuum technology

[ISO title : Vacuum technology — Mass spectrometer-type leak-detector calibration]

Méthodes pour l'étalonnage des spectromètres de masse détecteurs de fuites utilisés dans la technique de vide

Methoden für das Eichen von Lecksuchern der Massenspektrometerausführung für Anwendung auf dem Gebiet der Vakuum-Technologie

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National foreword

This British Standard is identical with ISO 3530-1979 'Vacuum technology — Mass spectrometer-type leak-detector calibration', and has been prepared under the direction of the Chemical Engineering Standards Committee. The United Kingdom has taken part in the discussions in Technical Committee TC 112 of the International Organization for Standardization (ISO) that resulted in the preparation of ISO 3530.

Terminology and conventions. The text of the international standard has been accepted as suitable for publication, without deviation, as a British Standard.

Some terminology and certain conventions are not identical with those used in British Standards; attention is especially drawn to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'British Standard'.

British Standard Methods of

Calibrating leak-detectors of the mass-spectrometer-type used in the field of vacuum technology

0 Introduction

This International Standard specifies procedures to be used for calibrating leak detectors of the mass-spectrometer type; that is, for determining a sensitivity figure for such leak detectors. The procedures require the use of a calibrated leak and a standard gas mixture; the preparation and standardization of these are outside the scope of this International Standard. Hereafter, the designation "leak detector" will be used to refer to a detector of the mass-spectrometer type.

A leak detector permits detection of leakage due to mechanical openings, such as pinholes, and of leakage due to permeation, such as occurs through many polymeric materials. Virtual leaks, such as those due to surface desorption, vaporization, and gas pockets, cannot, in general, be detected by a leak detector.

The range of leakage-rate calibration is limited to a specified level since factors that are unimportant for larger leaks may become significant for leak rates that are substantially smaller than $10^{-12} \text{ Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$.

Objects being tested by a leak detector may be under high vacuum, or, at the other extreme, under pressure greater than atmospheric. The leak-detection techniques will, in general, differ in the two situations. In the first case, the leak detector usually will be operating near its ultimate low pressure; in the second case, the detector is frequently used at or near its maximum operating pressure. Corresponding to these two conditions of operation, two sensitivity terms are defined, "minimum detectable leak rate" and "minimum detectable concentration ratio" (see clause 2).

The two quantities thus defined are related, but in practice it is not feasible to obtain either figure from the other by calculation. Methods are therefore specified for determining both.

This International Standard is one of a series standardizing leak-testing procedures and apparatus, prepared for use in the field of vacuum technology.

Applications fall into the categories: leak tightness, leak-detector calibration, calibration of leaks, gas mixtures, acceptance specifications for leak-detection instruments and general procedures for tightness-proving of vacuum plant.

The above-mentioned requirements will form the subjects of future International Standards.

1 Scope and field of application

This International Standard specifies procedures to be used for the calibration of mass-spectrometer-type leak detectors.

It deals only with leak detectors which have an integral high vacuum system to maintain the sensing element (mass spectrometer tube) at a low pressure. Specifically excepted from treatment are sensing elements without such a vacuum system. It is also to be understood that the procedures are not intended to constitute a complete acceptance test; such tests will be the subject of a future International Standard.

This International Standard concerns the use of helium-4. Nevertheless, the procedures described may be used for other search gases such as argon-40, subject to appropriate precautions.

The application of this International Standard is restricted to leak detectors not capable of detecting leaks smaller than $10^{-12} \text{ Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$.

Two procedures are outlined, one for determining the minimum detectable leak rate and the other for determining the minimum detectable concentration ratio. These are applicable to the use of the leak detector for high vacuum and for pressures greater than atmospheric, respectively.

2 Definitions

NOTES

1 An ISO glossary of terms used in vacuum technology is not yet available. In view of this, the following list of definitions has been prepared; usage in this International Standard will conform to these definitions.

2 Where a word may be either a noun or a verb, the letters "n" or "v", in parentheses, indicate which usage is involved.

2.1 Background (or residual signal)

2.1.1 background : In general, the total spurious indication given by the leak detector without injected search gas. Background can originate in either the mass spectrometer tube (see below) or the associated electric and electronic circuitry, or both. (Frequently, the term is used to refer specifically to the indication due to ions other than those produced from injected search gas.)

2.1.2 drift : The relatively slow change in the background. The significant parameter is the maximum drift measured in a specified period of time.

2.1.3 noise : The relatively rapid changes in the background. The significant parameter is the noise measured in a specified period of time.

2.1.4 helium background : Background due to helium released from the walls of the leak detector or leak-detection system.

2.2 Components

2.2.1 inlet line or sample inlet line : The line through which the search gas passes from the object under test to the leak detector.

2.2.2 inlet valve : A valve which is placed at the end of the sample inlet line and adjacent to the leak detector. (See figure 1.) Almost invariably the inlet valve is an integral part of the leak detector.

2.2.3 leak isolation valve : A valve placed between a leak which is to be used for testing the leak detector and the sample inlet line. (See figure 1.)

2.2.4 pump valve : A valve placed between the roughing pump used for evacuating the sample inlet line and that line. (See figure 1.)

2.2.5 vent valve : A valve used to admit air or other gas into an evacuated space so as to increase the pressure therein to atmospheric pressure. (See figure 1.)

2.2.6 backing-off control; zero control : An electrical control, present on most leak detectors, which may be used to shift the output indication of the device. Frequently, the backing-off control is used to return the output indication to zero of the scale, whence the alternative name.

2.2.7 filament : The source of the (thermal) electrons which ionize the gases in the mass spectrometer tube; the filament is located in this tube.

2.2.8 mass spectrometer tube : That element of a leak detector in which the search gas is ionized and detected.

2.3 Search gas

search gas : A gas applied to the outer surface of equipment under leak test and detected, after entry into the equipment through the leak, in a vacuum test, or introduced into the

equipment under test and detected after it is emitted from the leak, in a pressure test.

2.4 Leaks

2.4.1 leak (*n*) : In vacuum technology, a hole, porosity, permeable element, or other structure in the wall of an enclosure capable of passing gas from one side of the wall to the other under action of a pressure or concentration difference existing across the wall.

Also, a device which can be used to introduce gas into an evacuated system.

2.4.2 channel leak : A leak which consists of one or more discrete passages that may be ideally treated as long capillaries.

2.4.3 membrane leak : A leak which permits gas flow by permeation of the gas through a non-porous wall. For helium, this wall may be of glass, quartz, or other suitable material.

2.4.4 molecular leak : A leak through which the mass rate of flow is substantially proportional to the reciprocal of the square root of the molecular mass of the flowing gas.

2.4.5 viscous leak : A leak through which the mass rate of flow is substantially proportional to the reciprocal of the viscosity of the flowing gas.

2.4.6 calibrated leak : A leak device which provides a known mass rate of flow for a specific gas under specific conditions.

2.4.7 standard leak : A calibrated leak for which the rate of leakage is known under standard conditions; namely, $23 \pm 7^\circ\text{C}$, a pressure of $100\text{ kPa} \pm 5\%$ at one end of the leak, and a pressure at the other end so low as to have a negligible effect on the leak rate.

2.4.8 virtual leak : The semblance of a leak due to evolution of gas or vapour within a system.

2.5 Leak rates

2.5.1 leak rate : The throughput¹⁾, in $\text{Pa}\cdot\text{m}^3\cdot\text{s}^{-1}$, of a specific gas which passes through a leak under specific conditions.

2.5.2 standard air leak rate : The throughput, through a leak, of atmospheric air of dewpoint less than -25°C under standard conditions specified as follows : the inlet pressure shall be $100\text{ kPa} \pm 5\%$, the outlet pressure shall be less than 1 kPa , and the temperature shall be $23 \pm 7^\circ\text{C}$.

1) Differentiated from volume rate of flow, which is also called "pumping speed". Throughput is equivalent to mass rate of flow, when the temperature and molecular mass of the gas are specified.

2.5.3 equivalent standard air leak rate : Short-path leaks having standard air leak rates less than 10^{-7} to 10^{-8} Pa·m³·s⁻¹ are of the molecular type (see 2.4.4). Consequently, helium (relative molecular mass 4) passes through such leaks more rapidly than air (relative molecular mass 29,0), and a given flow rate of helium corresponds to a smaller flow rate of air. In this International Standard, helium flow is measured and the "equivalent standard air leak rate" is taken as $\sqrt{4/29,0} = 0,37$ times the helium leak rate under standard conditions (see 2.5.2).

2.6 Operation of the leak detector

2.6.1 peak (n) : The trace showing a maximum on the chart recorder when the leak detector is mass scanned (see below) with gas present, usually the search gas, to which the detector is sensitive.

2.6.2 peak (v) : To so set the scanning control (see 2.6.3) of a leak detector that the output due to a given search gas input is maximized. A form of tuning.

2.6.3 scan (v) : To vary the accelerating voltage (or other equivalent operating parameter) of a leak detector, particularly across that range of voltage which includes the voltage necessary to produce a search gas peak.

2.6.4 tune (v) : In leak-detection technology, to adjust one or more of the controls of a leak detector so that its response to a search gas is maximized. Tuning by means of the scanning control only is called "peaking".

2.6.5 zero (v) : To adjust the backing-off or zero control so that the output indication of the leak detector is at the zero of the indicating scale or at some other reference point.

2.7 Relative gas concentration

2.7.1 concentration ratio : Same as mole fraction (2.7.2).

2.7.2 mole fraction : The ratio of the number of atoms (or molecules) of a given constituent of a mixture to the total number of atoms (or molecules) in the mixture. For ideal gases, the mole fraction has the same value as the fraction based on volume; in general, leak detectors are operated in the pressure range where gases behave ideally. Same as concentration ratio.

2.7.3 partial pressure : In a mixture of gases, the partial pressure of a constituent is the product of the total pressure of the mixture and the mole fraction or concentration ratio of the given constituent.

2.8 Sensitivity terms

2.8.1 sensitivity : The sensitivity of a device is the change in output of the device divided by the change in input which caused the response.

2.8.2 minimum detectable signal : An output signal due to

incoming search gas which is equal to the sum of the noise and the drift.

2.8.3 minimum detectable leak, minimum detectable leak rate : The smallest leak, as specified by its standard air leak rate, that can be detected unambiguously by a given leak detector (see clause 1). The minimum detectable leak rate depends on a number of factors. Physically, it depends on the volume rate of flow, q_{vi} , of the search gas, measured at the ion source, and minimum partial pressure p_g , of the search gas in the ion source that can be detected, according to the formula

$$\text{minimum detectable leak rate} = p_g \times q_{vi}$$

The minimum detectable leak rate will be calculated as the ratio of the minimum detectable signal and the sensitivity.

NOTE — One of the purposes of this International Standard is to describe practical procedures for determining minimum detectable leak rate, taking into account background, volume rate of flow (pumping speed), and time factor.

2.8.4 minimum detectable concentration ratio : The smallest concentration ratio of a given search gas in an air mixture that can be detected unambiguously by a given leak detector when the mixture is fed to the detector at such a rate as to raise the pressure in the instrument to some optimum high value. In this International Standard, the minimum detectable leak rate is calculated — by a somewhat arbitrary procedure — from observations of the response of the leak detector to a helium-air mixture of known helium concentration ratio (see 3.5).

2.9 Time factors

2.9.1 time constant τ : The time interval required for the output of an instrument or system to change by $1 - 1/e$ or 63 % of the ultimate (steady-state) output change produced by an abrupt change in input.

2.9.2 response time : The time constant corresponding to a change from a zero or small leak-rate indication, to a positive or larger leak-rate indication.

2.9.3 cleanup time, clearing time : The time constant corresponding to a change from a positive leak-rate indication to a small or zero leak-rate indication.

NOTE — In this International Standard response time and cleanup time are assumed to be equal.

3 Test conditions

3.1 Ambient temperature

Ambient temperature should be 23 ± 7 °C.

3.2 Ambient pressure

Ambient pressure should be 100 kPa $\pm$ 5 %. When the deviation from 100 kPa exceeds 5 %, an appropriate correction shall be made, with 5 % tolerable inaccuracy.