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Vacuum gauges — Definitions and specifications for quadrupole mass spectrometers

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National foreword

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Vacuum gauges — Definitions and specifications for quadrupole mass spectrometers

Manomètres à vide — Définitions et spécifications des spectromètres de masse quadripolaires

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Foreword

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ISO 14291 was prepared by Technical Committee ISO/TC 112, *Vacuum technology*.

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Introduction

Quadrupole mass spectrometers (QMSs) are nowadays used not only for leak detection and residual gas analysis in vacuum but also as instruments to provide quantitative analysis in processes and control processes such as physical and chemical vapor deposition, and etch processes.

Total pressure, composition of the gas mixture, QMS settings, environment conditions, etc., have a significant influence on the measured signal, its uncertainty and interpretation. For this reason, it is not possible to calibrate QMS for all its possible applications. Instead, it has either to be calibrated for the particular conditions of use or for a standardized condition.

There is also some need for standardization in order to enable QMS users to compare devices of different manufacturers and to use the QMS properly.

In continuation of efforts of TC 112 during the 1990s, this International Standard takes a first step towards establishment of a standardized calibration procedure for QMS by defining the terms and parameters.

Vacuum gauges — Definitions and specifications for quadrupole mass spectrometers

1 Scope

This International Standard defines terms relevant to quadrupole mass spectrometers (QMSs) and specifies the parameters required for specification by QMS manufacturers necessary for proper calibration and for maintaining the quality of partial pressure measurement.

This International Standard applies to QMSs with an ion source of the electron impact ionization type. Such QMSs are designed for the measurement of atomic mass-to-charge ratios m/z typically <300. QMSs with other ion sources, such as those of the chemical ionization, photoionization, and field ionization types, as well as the measurements of m/z above 300, which are mainly used to specify organic materials, lie outside the scope of this International Standard.

2 Terms and definitions

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

2.1 Definitions of components

2.1.1
quadrupole mass spectrometer
QMS
mass spectrometer in which ions are injected axially into a quadrupole lens consisting of a system of four electrodes, usually rods, to which radio frequency and d.c. electric fields in a critical ratio are applied, so that only ions with a certain mass/charge ratio emerge

[SOURCE: ISO 3529-3:1981,[2] 3.5.2.2]

Note 1 to entry Such a QMS consists of a sensor head and electronic unit.

2.1.2
sensor head
analyser tube
sensor
sensor unit
sensing head
gauge head
equipment consisting of an ion source, quadrupole mass filter, and ion detector in one enclosure

2.1.3
ion source
part of the QMS in which ions of gas molecules and atoms are produced

Note 1 to entry For the production of positive ions, the ion source generally uses an electron impact ionization process.

2.1.3.1
open ion source
ion source with a high conductance to the surrounding vacuum environment, often designed as an open grid structure

Note 1 to entry All of the operational components of this ionization hardware are exposed to the same vacuum region.

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2.1.3.2
closed ion source
enclosed ion source
differential pressure ion source
ion source that uses a nearly sealed container to ionize the gas to be analysed with openings only for passing; sample gas; energetic electrons (for impact ionization); and exciting ions

Note 1 to entry This type of ion source permits ionization at pressures that are higher than the mass filter and detector. It should be used in a sample pressure reduction system with a high vacuum pump on the mass filter.

2.1.3.3
molecular beam ion source
crossed beam ion source
ion source that accepts a focused beam of neutral gas molecules directed at the ion formation region without interference from any ion source components

Note 1 to entry The molecular beam traverses the ion formation region and is usually at right angles to the electron beam and mass filter axes.

Note 2 to entry For molecular beam epitaxy, the crossed beam ion source is also designed to accept molecular beams at various acceptance angles. Some molecular beam ion source designs include a protective shroud around the ion source with an aperture to the ion formation region. As the molecular beam exits the ion source, it may be trapped or pumped to minimize contribution to background from scattered molecules.

2.1.4
quadrupole mass filter
device consisting of four parallel conductive rods arranged in a square array with opposite rods connected electrically in parallel

Note 1 to entry A quadrupole mass filter separates the ions coming from the ion source on the basis of their mass-to-charge ratios by a critical ratio of radio frequency (r.f.) and direct current (d.c.) electrical fields applied to the rods. The rod pairs are driven with opposite r.f. phase and d.c. polarity.

2.1.5
ion detector
ion collector
device collecting the positive ions that have passed through the mass filter to measure the ion current

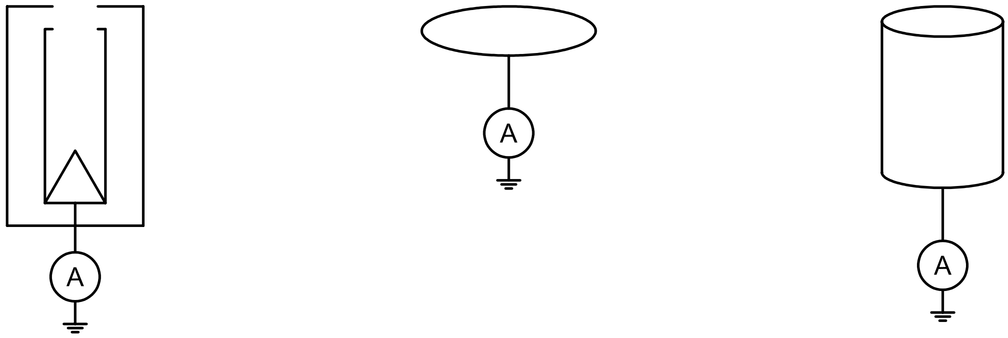
Note 1 to entry Two types of ion detectors are common: Faraday cup and secondary electron multiplier (SEM).

2.1.5.1
Faraday cup
metal plate or open cylinder or similar on which the ions from the mass filter are collected

Note 1 to entry An actual Faraday cup ion detector is illustrated in Figure 1 a). However, a metal plate, Figure 1 b), open cylinder, Figure 1 c), or similar on which the ions from the mass filter are collected is usually called a Faraday cup-type ion detector. A Faraday cup generally has a gain of unity, i.e. for each ion collected, one electron flows from the detecting electrometer.

2.1.5.2
secondary electron multiplier
SEM
detector in which the ions from the mass filter strike the entrance surface and release electrons

Note 1 to entry The released electrons are accelerated and strike another surface of the SEM resulting in multistage amplification of the electron current. SEMs can use discrete dynodes or a continuous dynode surface with a potential gradient to increase the electron current and microchannel plate electron multiplier.

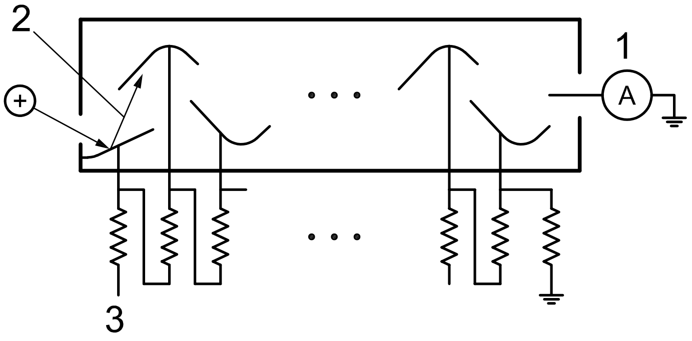


a) Faraday cup type **b) Metal plate type** **c) Open cylinder type**

Figure 1 — Faraday cup type ion detector

2.1.5.2.1
discrete dynode electron multiplier
secondary electron multiplier that uses discrete dynodes between which secondary electrons are accelerated

Note 1 to entry The voltages applied to each dynode establish the potential gradients that accelerate the secondary electrons and lead to increased numbers of electrons at each stage. Figure 2 is a schematic diagram of a discrete dynode electron multiplier.



Key

1	ammeter
2	electron
3	negative high voltage

Figure 2 — Discrete dynode electron multiplier

2.1.5.2.2
continuous dynode electron multiplier[1]
CEM
type of secondary electron multiplier with a continuous dynode, often with a horn-like channel

Note 1 to entry See Figure 3.

Note 2 to entry The applied voltage from entrance to exit of the channel establishes the potential gradient along the channel which accelerates secondary electrons and leads to increased numbers of electrons from entrance to exit.

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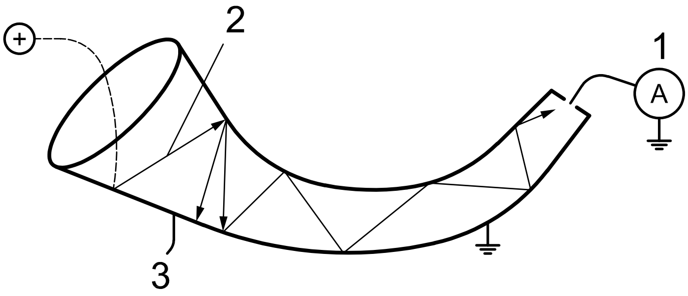
[1] One of the original CEMs is known as Channeltron®. Channeltron® is the trademark of a product supplied by Burle. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named.



Key

1	ammeter
2	electron
3	negative high voltage

Figure 3 — Continuous dynode electron multiplier (CEM)

2.1.5.2.3
microchannel plate electron multiplier
MCP electron multiplier

type of secondary electron multiplier which consists of a large number of small, parallel continuous-dynode channels (typically 5 µm to 25 µm in diameter), in a planar array, or “plate”

2.1.6
electronic unit

unit consisting of a radio frequency source and several regulated power supplies and amplifiers which operate the ion source and mass filter as well as measure detected ion current

Note 1 to entry The electronic unit usually incorporates a microprocessor and firmware to control the electronics and usually pass data to an external computer. Electronic units may be integrated or separate.

2.1.6.1
integrated type

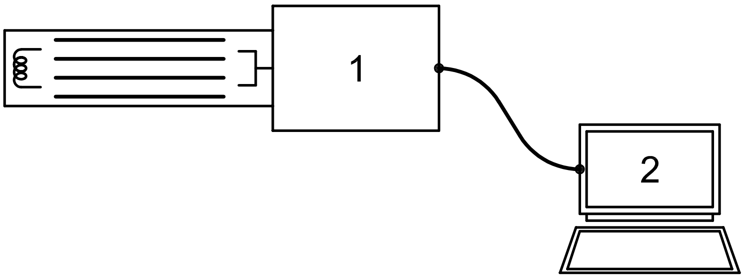
electronic unit mounted directly on sensor head

Note 1 to entry See Figure 4.

2.1.6.2
separated type

electronic unit separated from the sensor head but connected to it via one or more cables

Note 1 to entry See Figure 5.



Key

1	electronic unit
2	computer

Figure 4 — Integrated electronic unit

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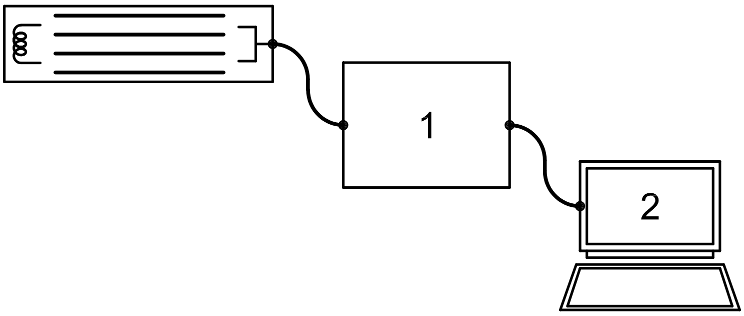

Key

1 electronic unit

2 computer

Figure 5 — Separated electronic unit

2.2 Definitions of physical parameters

2.2.1
sensitivity

S

ratio of the change in spectrum peak height (ion current), $I - I_0$, to the corresponding change in partial pressure, $p - p_0$

$$S(p) = \frac{I - I_0}{(p - p_0)} \tag{1}$$

where

I is the ion current measured at partial pressure p;

I_0 is the ion current measured at residual pressure p_0

Note 1 to entry Sensitivity is expressed in amperes per pascal.

Note 2 to entry Sensitivity is defined differently in ISO 27894:2009[4] for an ionization gauge with an emissive cathode. The sensitivity of a hot-cathode ionization gauge is defined as

$$S = \frac{I_c - I_0}{I_e(p - p_0)} \tag{2}$$

where

I_c is the ion current measured at pressure p;

I_0 is the ion current measured at residual pressure p_0;

I_e is the emission current.

2.2.2
relative sensitivity factor

r_x

sensitivity S_x for a specified gas species, x, divided by sensitivity S_{N_2} for nitrogen for the same device at the same pressure and the same operation conditions:

$$r_x = \frac{S_x}{S_{N_2}} \tag{3}$$

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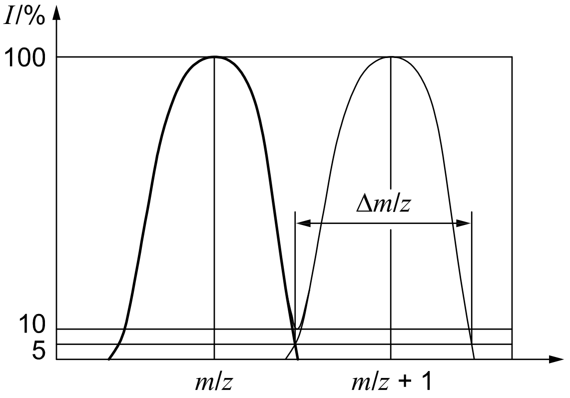
Note 1 to entry Relative sensitivity factors depend on the operational parameters of QMS as shown in References [13][16][17]. If necessary for reasons of accuracy, it is recommended that relative sensitivity factors be measured before each measurement.

2.2.3
mass resolution

smallest mass difference Δm between two equal magnitude peaks so that the valley between them is a specified fraction of the peak height

Note 1 to entry See Figure 6.



Key

I	ionic current
m/z	mass-to-charge ratio
$m/z + 1$	mass-to-charge ratio plus one
$\Delta m/z$	mass difference to charge ratio

Figure 6 — Mass resolution defined at 10 % valley of neighbouring mass/charge ratios

Note 2 to entry Mass resolution defined at 10 % valley is technically close to the peak width at 5 % of peak height.

Note 3 to entry Peak shape (i.e. asymmetry) has influence on mass resolution.

2.2.4
mass resolving power

ratio of the mass, m, to the mass resolution Δm:

$$\frac{m}{\Delta m}$$

Note 1 to entry See Reference [13].

2.2.5
mass number stability

extent to which a position of a peak of a single charged ion is stable within a specified time on the mass scale

2.2.6
minimum detectable partial pressure
MDPP

$p_{\text{p, min det}}$

partial pressure of a specific gas which produces a peak height equal to three times its current noise limit σ_I in a given period of time divided by its sensitivity S

$$p_{\text{p, min det}} = \frac{3\sigma_I}{S} \tag{4}$$

Note 1 to entry The standard deviation of the current can be used for current noise.

Note 2 to entry The noise limit depends on the integration time. It is recommended that an integration time of 1 s be used because this time is typical for current measurement in a mass spectrum peak.

Note 3 to entry Minimum detectable partial pressure is usually determined at the base pressure of the vacuum system.

Note 4 to entry Background noise or peak overlap from other gases of a gas mixture can influence MDPP substantially.

2.2.7
amount of substance fraction
ratio of the number of molecules of a given constituent of a mixture to the total number of molecules in the mixture

Note 1 to entry For ideal gases and for the conditions applicable for QMS, the mole fraction has the same value as the fraction based on volume.

2.2.8
minimum detectable concentration
minimum detectable partial pressure in a specified carrier gas

Note 1 to entry Generally, minimum detectable concentration is expressed as a volume fraction, e.g. as a percentage volume fraction or in microlitres per litre.

Note 2 to entry The MDPP to be used here is different to the one at base pressure. It shall be determined at the operating pressure.

Note 3 to entry Background noise or peak overlap from other gases of a gas mixture can influence minimum detectable concentration substantially.

2.2.9
mass range
range from the lightest to the heaviest single charged ion which can be detected

2.2.10
scan
mass scan
act or process of varying the scanning parameters over all or part of the mass range of QMS

Note 1 to entry The mass scan is also identified with the output of the QMS that shows the kinds and relative amounts of the ions produced from the gas. The scan is usually displayed as a graph on a PC monitor; the abscissa is ion mass per charge, m/z, or time and the ordinate represents the ion current on a linear or logarithmic scale. Typical scan parameters are mass range, scan speed, time per separated signal, resolution.

2.2.11
peak number
mass number
position of the maximum ion current of a certain mass/charge ratio

Note 1 to entry It is referred to, for example, as the “peak number 28”. For single charged ions it is called mass number.

2.2.12
peak height
peak intensity
distance from baseline to the apex of a given peak

Note 1 to entry The peak height is expressed in amperes or pascals.

Note 2 to entry The output in pascals is a calculated value and not the originally measured signal (raw data).

2.2.13
peak tail
$\Delta_{\pm}$
position on the mass scale where mass peak has dropped to v % of the peak height

Note 1 to entry Since peaks are not, in general, perfectly symmetrical, the $+$ and – subscripts are to distinguish values of peak tails obtained on the positive side and negative side of a mass peak.

2.2.14
baseline
reference level from which peak height is measured

Note 1 to entry The baseline is determined from the signal in mass ranges where no ions arrive at the detector.

2.2.15
fragmentation pattern
cracking pattern
pattern (i.e. kinds and relative amounts) of ions produced by a given pure gas in a given mass spectrometer under given conditions

Note 1 to entry The term derives from the fact that molecules are decomposed, cracked or multi-charged in the ion source and produce a characteristic set of atomic or molecular fragments.

Note 2 to entry The cracking pattern in a pure gas may be different from the one of the same gas species in a gas mixture.

2.2.16
fragmentation factor
cracking factor
“relative abundance”
f_i
ratio of an ion signal I_i at a mass number m_i originating from the fragmentation of an molecule to the ion signal I_0 at the main mass number peak m_0

$$f_i = \frac{I_i}{I_0} \tag{5}$$

Note 1 to entry Cracking factors depend on the operational parameters of a QMS as shown in References [12][14][15]. It is recommended that relative sensitivity factors be measured before each measurement.

2.2.17
non-linearity
extent to which the change in ion current is not proportional to the corresponding change in partial pressure

Note 1 to entry The non-linearity is equal to the change of sensitivity in a given range.

2.2.18
linear response range
partial pressure range over which the non-linearity is within a specified limit

2.2.19
stability of peak height
stability of peak intensity
change of the peak height at constant partial pressure in a specified time period

2.2.20
maximum operational pressure
maximum pressure specified to avoid damages of filament, detector or others, and to ensure a reasonable lifetime of the QMS

2.2.21
warm-up time
time after start of operation of QMS when measurement signal with stability as specified by the manufacture is achieved

3 Symbols and abbreviated terms

See Table 1.

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Table 1 — Symbols and abbreviated terms

Symbol	Designation	Unit
f	fragmentation factor	1
p	pressure or partial pressure	Pa
p_0	residual pressure or residual partial pressure	Pa
r_x	relative sensitivity for a specified gas species x divided by sensitivity S_{N_2} for nitrogen	1
I	ion current at partial pressure p	A
I_0	ion current at residual pressure p_0	A
$p_{\mathrm{p,\ min\ det}}$	minimum detectable partial pressure	Pa
S	sensitivity (coefficient)	A/Pa
S_{N_2}	sensitivity for nitrogen	A/Pa
CEM	continuous dynode electron multiplier	
MCP	micro-channel plate	
MDPP	minimum detectable partial pressure	Pa
QMS	quadrupole mass spectrometer	
SEM	secondary electron multiplier	

4 Principle of QMS

Positive ions are produced in the ion source. These ions are accelerated towards the mass filter consisting of four symmetrically arranged hyperbolic, cylindrical rods or rods with a square cross-section. The combination of a direct current (d.c.) potential and radio-frequency (r.f.) potential applied to the rods separates the ions according to their mass-to-charge ratios which are detected by QMS detector. For more details see, for example, Reference [12].

Some types of QMS also allow the generation of negative ions and the measurement of their masses by changing the potentials accordingly.

When the pressure of the gas to be analysed is higher than the maximum operating pressure of the QMS, a separate pumping system plus a number of conductances are used to maintain a low enough pressure inside of the QMS. This method is usually done by using a differential pumping system.

5 Specifications for a QMS to be provided by manufacturers

The following features and specifications shall be provided by the manufacturer in order to enable users of their QMS to minimize measurement error and to give guidance on safe operation. A QMS supplier can be mandated by the manufacturer to provide these specifications or part of them.

5.1 Mass range

The mass range of the supplied QMS shall be specified in m/z.

5.2 Type of ion source

The type of ion source shall be specified: open ion source; closed ion source; molecular-beam ion source; etc.

5.3 Type of ion detector

The type of detector, such as Faraday cup or SEM, shall be specified.

5.4 Mass resolution

The mass resolution should be specified in m/z. In order to have comparable specifications, it is strongly recommended that the value at 5 % of the peak height be given over a specified mass range or at a m/z. In addition, the mass resolution may be given at any other suitable percentage of the peak height (e.g. 50 %). Since mass resolution is normally adjustable, the best (lowest value) under the specific conditions shall be given.

5.5 Mass number stability

Mass number stability is the extent to which a position of a mass peak is stable within a specified time on mass scale. It should be given for the main mass peak of Ar or for He.

5.6 Sensitivity

The sensitivity shall be provided at least for N_2 and for the available detector or detectors in a pressure range to be specified and at a given mass resolution. The sensitivity shall be expressed in amperes per pascal. Other units are also allowed. The supplier shall describe the conditions such as pressure, emission current and temperature, and the settings to evaluate the sensitivity.

5.7 Linear response range

Minimum and maximum limit of the partial pressure over which the non-linearity is within a specified limit defined by manufacturer. The pressure range shall be expressed in pascals. Other units are also allowed.

The linear response range should be given at least for N_2.

As the linear response range depends on many adjustable parameters, e.g. emission current, the parameters should be specified.

5.8 Minimum detectable partial pressure

MDPP shall be provided for a specified gas and a detector. It shall be expressed in pascals. Other units are also allowed.

This quantity should be given for at least He, optionally also for Ar.

5.9 Minimum detectable concentration

MDC shall be provided for a specified gas mixture and a detector. The minimum detectable concentration can be determined from the measurement of minor constituents of the atmosphere, i.e. He, Ar, and Kr.

5.10 Maximum operational pressure

The maximum operational pressure shall be specified. Maximum operational pressure should be expressed in pascals. Other units are also allowed.

Automatic shut down point of overpressure should be provided.

NOTE Highest allowable pressure under operation depends on the gas (composition), filament materials, and detector type or setting. It is generally provided for N_2.

5.11 Scanning parameter

Scanning parameters shall be specified which are scan speed, scan time, point per m/z, resolution, number of scan, emission current, and SEM voltage, etc. If they are changeable, the ranges shall be specified.

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5.12 Signal output

Signal output shall be specified, e.g. ion current, in amperes, partial pressure, in pascals, and/or total pressure, in pascals.

5.13 Potentials

The potentials of electrodes such as cathode, anode, extraction, field axis or ion energy, detector shall be specified.

If the potentials can be adjusted, this fact should be stated. Restrictions or recommendations for values and ratios (e.g. ratio between repeller voltage and electron energy) shall also be mentioned.

5.14 Detector specifications

For SEM, the maximum ion current (up to saturation) for a mass number shall be expressed in amperes. The typical amplification factor for a given potential shall be given and also the typical loss of amplification with lifetime and bake-outs.

5.15 Set point

The manufacturer shall specify whether trigger signal outputs (ion current or pressure if available) can be set to control other units or processes.

5.16 Maximum bake-out temperature

The maximum temperatures shall be specified respectively for QMS sensing unit, electronic unit, and cables. If the electronic unit of an integrated type can be removed, this fact shall be stated and the maximum bake-out temperature for either the sensing unit or electronic unit shall be given.

5.17 Nominal operating conditions

Temperature and humidity range at which the QMS can be operated for reliable measurements shall be specified.

5.18 Warm-up time

Warm-up time shall be specified.

5.19 Filament material

The number and material of filament(s) shall be specified.

5.20 Electron emission current

The manufacturer shall specify the electron emission current or the range of the electron emission current when adjustable. The emission current is generally expressed in milliamperes.

5.21 Filament exchange

The manufacturer shall specify whether a filament can be replaced by the user. If replacement is possible by the user, the procedure for resetting the QMS should be provided.

5.22 Detector exchange

If the detector (e.g. SEM) is replaceable, the method shall be stated.

5.23 Fitting to chamber

The flange type and its dimension for the QMS sensor head should be specified.

5.24 Mounting orientation

The manufacturer shall specify the mounting orientation of the sensor head to the chamber.

5.25 Dimensions

The dimensions of the QMS sensor head and electronic unit shall be specified in outline drawings in SI units. Dimensions may be expressed as width, depth and height. Other units (e.g. inch) can additionally be used.

5.26 Internal volume

The volume added by the sensor head to the vacuum chamber to which it is attached should be given in SI units. An uncertainty of 20 % or 50 cm^3 (whichever is higher) is sufficient. The internal volume is the additional volume when a blind flange is replaced by the sensor head. The vacuum walls of the sensor head may increase the volume attached to the chamber (positive internal volume), built-in components reduce the internal volume.

The internal volume may be a negative value.

5.27 Mass of sensor head and electronic unit

Manufacturers shall specify the mass of the sensor head and its electronic unit in SI units. Other units can additionally be used.

5.28 Input power of electronic unit

Voltage, current, frequency, and phase shall be specified.

5.29 Cable

The cable type shall be specified. Their lengths shall be specified in SI units.

5.30 Software

Manufacturers shall specify the necessary information on the software that controls the QMS hardware. The information may include how to acquire and display data and how to optimize the hardware and software setup for reliable measurements. Generally, a software manual is provided by the manufacturer. It shall also contain the minimum specifications of the computer required.

5.31 Interface

The interface for communication (e.g. RS-232c, LAN) shall be specified.

5.32 Storage and transportation condition

The manufacturer is recommended to specify gas environment, cleanliness, temperature, relative humidity, vibration, shock, etc., for storage and transportation in order to avoid damage.

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6 Optional specifications for QMS to be provided by manufacturers

6.1 Mass resolution

The full width at half maximum (FWHM) of the height of a given peak on the mass/charge scale may be specified in addition to that specified in 5.4.

6.2 Fragmentation or cracking pattern

It is recommended that the fragmentation factors of the cracking patterns of several important gas species, e.g. water vapour, methane, nitrogen, carbon monoxide, be given for defined settings of the QMS.

6.3 Temperature coefficient of sensitivity

The temperature coefficient of the sensitivity that is specified in 5.6 may be specified.

6.4 QMS sensor cleaning

The manufacturer may specify the cleaning procedure of the QMS sensor head. The materials of the ion source shall be provided for the cleaning. It usually contains the procedures such as demounting and disassembling of the QMS sensor head, chemical cleaning, re-assembling, and testing.

6.5 Degassing

The manufacturer shall specify the degassing method and time. A maximum pressure for degassing shall be specified to avoid filament damage.

For accurate measurements, most QMSs are often degassed to remove contaminants from electrodes and enclosure of sensing head. Degassing time is often adjustable using the QMS software. Some QMSs automatically return to measurement mode after degassing. It is recommended that the manufacturer specify the time interval and the automatic returning function if it is available.

6.6 Degassing power

The manufacturer should specify the degas power (e.g. filament to grid and/or electrode voltage and electron current).

Degassing power is often adjustable using the QMS software. If adjustable, this fact should be stated.

6.7 Photographs

For clear outlook and details, the provision of photographs of the upright gauge tube and front and back panels of the gauge controller are recommended.

6.8 Inspection record

The manufacturer should provide some specific information on the QMS supplied, such as a typical mass spectrum.

6.9 Outgassing rate

The outgassing rate at specified clean conditions and settings may be provided by the manufacturer.

NOTE The outgassing rate depends on the conditioning and history of the QMS.

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