7th International CCM and IMEKO TC-16 Conference on Pressure and Vacuum Metrology
May 17-19, 2023
Rockville, Maryland, USA

ISO standards for vacuum metrology

Karl Jousten

Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin, Germany
karl.jousten@ptb.de

***Abstract* – During the last decade, several important standards and technical specifications (TS) for vacuum metrology have been published by the International Organisation for Standardisation (ISO) or are under development. The pressure ranges for high and ultra-high vacuum have been redefined, and extreme high vacuum has been defined internationally for the first time. Basic standards describing vacuum gauge calibration equipment and methods, as well as the uncertainties associated with a calibration, are supplemented by specific standards for characterising or calibrating specific vacuum gauges. Procedures have also been developed for the characterisation of quadrupole mass spectrometers for partial pressure measurement and for the measurement of outgassing rates in a traceable and comparable manner. A TS for the design of a high-precision ionisation vacuum gauge has recently been published. Reliable vacuum measurement is also important for characterising the performance of vacuum pumps.**

I. INTRODUCTION

Written standards are not only useful to make things “fitting together”, but also to transfer knowledge from science to industry and all-day work. To this end, in the past decade the Technical Committee (TC) 112 of the International Standardization Organisation (ISO) has developed standards for the correct calibration of vacuum gauges, to perform outgassing rate measurements in a comparable way and to improve the measurement of high vacuum.

TC 112 is supported by three working groups (WGs), WG 1 for vacuum pumps, WG 2 for vacuum instrumentation, WG 3 for vacuum hardware. This report will mainly deal with standards developed by the WG 2 (Table 1).

Table 1. Standards and Technical Specifications published by ISO TC 112 since 2011 related to vacuum metrology.

ISO number	Title
3529-1	Vocabulary- Part 1: General terms
3567	Vacuum gauges – Calibration by direct comparison with a reference gauge
TS 6737	Characteristics for a stable ionisation vacuum gauge
14291	Definitions and specifications of quadrupole mass spectrometers
19685	Specifications, calibration and measurement uncertainties for Pirani gauges
20146	Specifications, calibration and measurement uncertainties for capacitance diaphragm gauges
TS 20175	Characterization of quadrupole mass spectrometers for partial pressure measurement
TS 20177	Procedures to measure and report outgassing rates
24477	Specifications, calibration and measurement uncertainties for spinning rotor gauges
27893	Evaluation of the uncertainties of results of calibrations by direct comparison with a reference gauge

II. VACUUM RANGES

Technological progress has made it necessary to redefine some vacuum ranges. The first definition of vacuum ranges by ISO were published in 1981 and revised in ISO 3529-1:2019 [1].

Vacuum is defined as the state of a gas with a pressure or a molecular density below the prevailing atmospheric level. The atmospheric pressure on ground depends on weather conditions and altitude and ranges from 31 kPa (altitude of the Mount Everest, weather condition "low") up to 110 kPa (altitude Dead Sea, weather condition "high").

The vacuum ranges (Table 2) are defined according to the technology needed to achieve the corresponding pressure. While relatively simple materials and positive displacement pumps are sufficient for low or medium vacuum, high vacuum requires elaborate materials such as stainless steel and a pump system with different pump principles in series. Still, elastomer sealings are sufficient for high vacuum and systems need no or only a mild and short bake-out to remove water from the surfaces. Due to

improved pumping technology since 1981 the lower limit of the high vacuum range was reduced by a factor of 10 to 10^{-6} Pa in the new standard. This pressure can still be achieved with elastomer sealings on flanges. Ultra-high vacuum needs metal sealings and high-temperature bake-outs ≥ 150 °C, special materials like low-carbon stainless steel and special surface preparations and cleaning. Extreme-high vacuum needs sophisticated materials like vacuum-fired low carbon steel, extensive surface preparations and cleaning and getter pumping technology. This range has been newly defined by ISO.

Table 2. Vacuum ranges as defined in ISO 3529-1:2019 [1]. Brackets indicate alternative terms.

Pressure range	Definition
100 Pa to prevailing atmospheric pressure	Low (rough) vacuum
0.1 Pa to < 100 Pa	Medium (fine) vacuum
10^{-6} Pa to < 0.1 Pa	High vacuum
10^{-9} Pa to < 10^{-6} Pa	Ultra-high vacuum
< 10^{-9} Pa	Extreme-high vacuum

3529-1:2019 also defined ultra clean vacuum, which is a medium or high vacuum that requires special conditions for some gas species equivalent to UHV conditions.

III. VACUUM GAUGE CALIBRATION

The basics of a calibration apparatus and also the procedures for the calibration of vacuum gauges have been laid down in the standard ISO 3567 [2], the treatment of the uncertainties for the calibration of vacuum gauges in ISO 27893 [3]. These two standards are supplemented by ISO 19685 [4] for the calibration of Pirani gauges, ISO 20146 [5] for capacitance diaphragm gauges (CDGs), ISO 24477 [6] for spinning rotor gauges (SRGs). The latter two types of gauges are also suitable to serve as reference gauges in a calibration system according to ISO 3567.

A major influence of the uncertainty of vacuum gauges, unfortunately often forgotten to include in the uncertainty budget, is the long-term instability of the calibration constant. This long-term instability includes transport instability when the gauge is sent to another laboratory for recalibration. The long-term instability δ_t was both defined in ISO 19685 and 20146. At least three calibrations must be performed in order to quantify δ_t by two possible equations:

$$\delta_t = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}\left(\frac{\Delta p_i}{p_i} - \overline{\left(\frac{\Delta p}{p}\right)}\right)^2} \qquad (1)$$

or

$$\delta_t = \frac{\sum_{i=1}^{n-1}\left|\frac{\Delta p_{i+1}}{p_{i+1}} - \frac{\Delta p_i}{p_i}\right|}{n-1} \quad , \qquad (2)$$

where n is the number of calibrations i, Δp_i is the measurement error at the indicated pressure p_i of the gauge determined by calibration, and

$$\overline{\left(\frac{\Delta p}{p}\right)} = \frac{1}{n}\sum_{i=1}^{n}\frac{\Delta p_i}{p_i} \qquad (3)$$

is the mean measurement error of the n calibrations. For a CDG, the measurement error should be evaluated near full scale, for a Pirani gauge in the medium range where accuracy is highest. Eq. (1) is recommended, when the measurement error does not show a significant drift but random variations, and Eq. (2), when the measurement error shows a systematic and monotonic drift. When not yet three calibrations were performed, commonly accepted values should be used [7].

Since some Pirani gauges use convection in the viscous flow regime, an arrow marker is recommended to show the upside direction in which the gauge was calibrated. The calibration report should also include a warning that significantly higher measurement uncertainties can occur when using the Pirani gauge. Sources of measurement errors and additional uncertainties are: using a different gas species as calibrated, even when corrected for known gas species, exposure to corrosive or non-inert gas species, use of interpolated values between calibration values, use at different ambient temperature than at calibration, contamination, and thermal relaxation at higher pressures. In particular in load locks, the response time of vacuum gauges due to sudden pressure changes may be important. For this reason, response times were defined both in ISO 19685 and 20146. Since Pirani gauges use thermal effects for measurements, they react usually slower than CDGs [8].

IV. QUADRUPOLE MASS SPECTROMETERS

Some applications of quadrupole mass spectrometers (QMS) require quantitative results. Leak rate measurements have been identified as such, to comply with safety or environmental regulations or to enable the proper operation of particle accelerators or fusion reactors. Outgassing rate measurements, most critical nowadays for components in EUV lithography systems, require also traceable and quantitative data. The process industry also requires quantitative data to control processes such as physical and chemical vapour deposition, and etch processes. There is also a need for standardization in order to enable the users of QMS to compare the devices of different manufactures and to use the QMS properly.

Unfortunately, the indicated partial pressures of QMS are highly unreliable: The total pressure, the composition of the gas mixture, the settings and the operational history of QMS (long-term instability) [9], to name a few, 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 special conditions at use or for a standardized condition. The ISO TS 20175 [10] specifies such conditions and attempts to provide standardised calibration procedures for QMSs for some important applications.

Together with ISO 14291 [11] it also specifies important terms for QMS, e.g. the interference effect ratio, which quantifies the change of sensitivity of a gas species when another gas species or gas mixture is present.

V. OUTGASSING RATE MEASUREMENTS

In the past, the results of measurements of the outgassing rate were not sufficiently comparable and not traceable to the SI (International system of units). ISO TS 20177 [12] was developed to standardize the measurement of outgassing rates in such a way that values obtained at different laboratories and by different methods are comparable.

To this end, for any of the described methods, some traceability was provided to the System International (SI) for the most important parameters of each method and according to the metrological level. For this purpose, the nitrogen equivalent outgassing rate was defined since many vacuum gauges are calibrated for nitrogen only. Nitrogen equivalent outgassing rate is the outgassing rate, when all gases released from the sample are assumed to be nitrogen molecules. Unfortunately, in some cases this still does not lead to comparable values, but the routes to the nitrogen equivalent value are known and, with additional information, can be converted to absolute and comparable values.

ISO TS 20177 standardised the measurement systems (Fig. 1) and procedures and gives estimates for the uncertainties of outgassing rates obtained from the different systems and procedures. An informative annex gives recommendations of which system and method should be used for which application.

The measurement procedures and systems can be distinguished in those applying the gas accumulation method measuring a pressure rise and those applying different throughput methods to measure the outgassing rate emerging from a sample or a chamber as a whole. These throughput methods range from a simple system with a known effective pumping speed to flow rate measurements across an orifice or to comparator systems where known gas flows are injected into a continuous expansion system with known pump conductance.

Accumulation systems can be built with and without gas analysis.

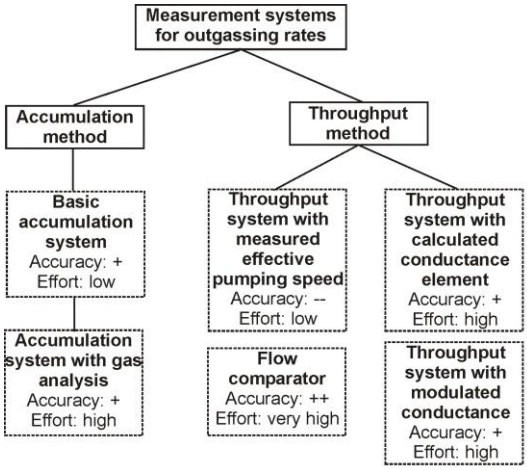

Fig. 1. Measurement systems for outgassing rate according to ISO TS 20177.

VI. STANDARDISED ION GAUGE

Vacuum gauges are usually calibrated for nitrogen. When pressures in the high vacuum range or lower need to be measured, all vacuum gauges rely on measurement principles which are gas sensitive. To calibrate QMS for other gas species than nitrogen, relative gas sensitive factors of ionisation gauges are used. This is also the case when measuring pumping speed of high vacuum pumps for different gas species. The relative gas sensitive factors, however, depend on the type of ionisation gauge and the individual gauge and are therefore unknown or, when using tables, known with large uncertainty only.

To overcome this situation, the European project 16NRM05 developed an ionisation vacuum gauge [13-15], where the relative gas sensitive factors are well known. This gauge had to be developed to be transport stable in order not to lose any calibration information. It turned out that the design of this gauge was so reproducible that even the nitrogen sensitivity is predictable without any calibration, provided that a measurement uncertainty of 5 % is enough. For even lower uncertainties, a calibration will reduce the uncertainty for the sensitivity of nitrogen to 1%.

The success of this development let ISO TC 112 standardise this ionisation vacuum gauge in ISO TS 6737, which was published in November 2023. This gauge can be produced by any experienced manufacturer and will show the same characteristics independent of the producer [14].

ACKNOWLEDGEMENTS

Part of this work has received funding from the EMPIR programme (projects 14IND06, 16NRM05 and 20SIP01) co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation

programme.

REFERENCES

[1] ISO 3529-1:2019, Vacuum technology – Vocabulary-Part 1: General terms, 2nd edition 2019-07.

[2] ISO 3567:2011, Vacuum gauges - Calibration by direct comparison with a reference gauge, 1st edition 2011-12-15.

[3] ISO 27893:2011, Vacuum technology - Vacuum gauges — Evaluation of the uncertainties of results of calibrations by direct comparison with a reference gauge, 1st edition 2011-08-15.

[4] ISO 19685:2017, Vacuum technology - Vacuum gauges - Specifications, calibration and measurement uncertainties for Pirani gauges, 1st edition 2017-09.

[5] ISO 20146:2019, Vacuum technology - Vacuum gauges - Specifications, calibration and measurement uncertainties for capacitance diaphragm gauges, 1st edition 2019-01.

[6] ISO 24477:2022, Vacuum technology – Vacuum gauges - Specifications, calibration and measurement uncertainties for spinning rotor gauges, 1st edition 2022-08.

[7] Chapter 15 in Handbook of Vacuum Technology, 2nd edition, ed. K. Jousten, Wiley-VCH, Weinheim, 2016.

[8] K. Jousten, “Vacuum metrology and its impact on research and industry”, Vakuum in Forschung und Praxis, Vol. 31, Nr. 4, 2019, DOI:10.1002/vipr.201900715

[9] A. Elkatmis et al., “Time stability characterization of quadrupole mass spectrometers”, Measurement 165, 108143, 2020, doi.org/10.1016/j.measurement.2020.108143.

[10] ISO/TS 20175:2018, Vacuum technology - Vacuum gauges -Characterization of quadrupole mass spectrometers for partial pressure measurement, 1st edition 2018-04.

[11] ISO 14291:2012, Vacuum gauges – Definitions and specifications of quadrupole mass spectrometers, 1st edition 2012-07-15.

[12] ISO/TS 20177: Vacuum technology - Vacuum gauges - Procedures to measure and report outgassing rates, 1st edition 2018-06.

[13] B. Jenninger et al., “Development of a design for an ionisation vacuum gauge suitable as a reference standard”, Vacuum 2020,109884, https://doi: 10.1016/j.vacuum.2020.109884

[14] K. Jousten et al., “Electrons on a straight path: A novel ionisation vacuum gauge suitable as reference standard”, Vacuum 189 (2021) 110239, https://doi.org/10.1016/j.vacuum.2021.110239

[15] K. Jousten et al., “Evaluation and metrological performance of a novel ionisation vacuum gauge suitable as reference standard”, Measurement 210, 112552, 2023, https://doi.org/10.1016/j.measurement.2023.112552