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Traceability To SI Units For Vacuum Measurement In Industrial Applications

Karl Jousten

PTB, Berlin

Abstract

In the context of international standards like the ISO 9000 series or ISO 17025 the traceability of measurement instruments of physical units in industrial processes gained more importance in the last two decades, so to say also for vacuum measurement. Traceable calibrations of vacuum gauges ensure agreement with the SI units. For this purpose vacuum primary standards are needed. The international system of metrology ensures that the vacuum primary standards registered in the system are equivalent and fulfil their specifications. Secondary and reference standards are used to disseminate the pressure scale in vacuum to calibration laboratories, to the manufacturers of gauges, and finally to industrial processes or to research facilities. Suitable vacuum gauges for this purpose including their expected measurement uncertainties will be described. Notes for the measurement uncertainties at the place of the end user will be given.

Keywords: Vacuum metrology, primary standard, traceability, vacuum gauges, accuracy, uncertainty

1. Introduction

In this review article the traceability of vacuum measurements to vacuum primary standards and hence the SI units shall be described. This includes a depiction of the uncertainties associated with vacuum measurement both for the end user in typical industrial applications as along the calibration chain.

In the first part after this introduction the methods and realization schemes of vacuum primary standards are outlined. In the following section the quality assurance of the national vacuum standards by the international system of metrology is depicted. The 4th section gives an overview on suitable reference vacuum gauges with a special emphasis on their long-term stability. In Section 5 the uncertainties caused by the calibration chain as well as by the gauges and their way of usage will be discussed. Section 6 tries to summarize the accuracies for the different vacuum gauges.

2. Primary Standards For Vacuum

The calibration measurement capabilities of the National Metrological Institutes range over 14 decades from 10-9 Pa to 105 Pa. This huge range is presently covered by 4 types of primary standards:

- Liquid manometers (mercury and oil) from 10^5 Pa to about 1 Pa
- Rotating and non-rotating piston gauges (pressure balances) from 10^5 Pa to about 1 Pa
- Static expansion systems from 10^3 Pa to 10^{-6} Pa
- Continuous expansion systems from 10^{-1} Pa to 10^{-9} Pa.

Herein only the first two can give a direct traceability to the SI units of mass (kg), time (s) and length (m) as necessary for the unit of Pa equal to 1 kg m-1 s-2. Figure 1 gives an overview of the traceability chain.

Mercury manometers give the highest accuracy around 105 Pa, oil manometers are used typically below 100 Pa. The length measurement in the column is either accomplished by optical interferometry from above to the surface [1], [2] or by phase sensitive measurement of the travel path time of ultrasound pulses within the mercury from the bottom [3], [4]. The main uncertainty contributions near 105 Pa come in this order from the uncertainty of temperature of the mercury determining its density, the uncertainty of the mercury density under standard conditions and the column height measurement.

Rotating piston gauges of high quality have been available since after the Second World War, but the accuracies of the measurement of the effective area only recently made them comparable to accuracy of mercury manometers [5]. Their lower range ends at typically 103 Pa.

Non-rotating pressure balances ([6] to [8]) became available in the 1990s and could extend the range accessible to piston gauges down to 1 Pa. Below about 30 Pa, however, the uncertainty of static expansion systems becomes superior to non-rotating pressure balances.

In static expansion systems ([9] to [12]) the ideal gas law is applied. Gas of relatively high pressure in a small volume is expanded into a large volume so that the pressure is reduced according to the volume ratio with a small correction term due to non-isothermal conditions and real gas behavior.

The static expansion method fails at lower pressures for two reasons: The gas is adsorbed on the inner vacuum surface to an amount that the pressure in the volume is significantly reduced. The outgassing of the chamber walls falsifies the pressure in the large volume, when the pump valve is closed. Dependent on the gas and the outgassing rate, the lower calibration pressure limit can be as low 10-6 Pa, but typically the static expansion method is used down to 10-2 Pa.

For the realization of lower pressures the continuous expansion method is applied [13] to [15]. In this method two largely different orifices are used to generate the low pressure. The pressure in a gas reservoir in front of the first conductance is kept constant. Since in between the two conductances there is no source or sink of gas, the conservation of flow holds and the pressure of the gas reservoir is reduced by the ratio of the conductances. If the first is very small, e.g. 10-6 L/s, and the second relatively large, 100 L/s, the pressure in the reservoir is

reduced by a factor of 10-8: A pressure of 10 Pa in the reservoir is reduced to 10-7 Pa.

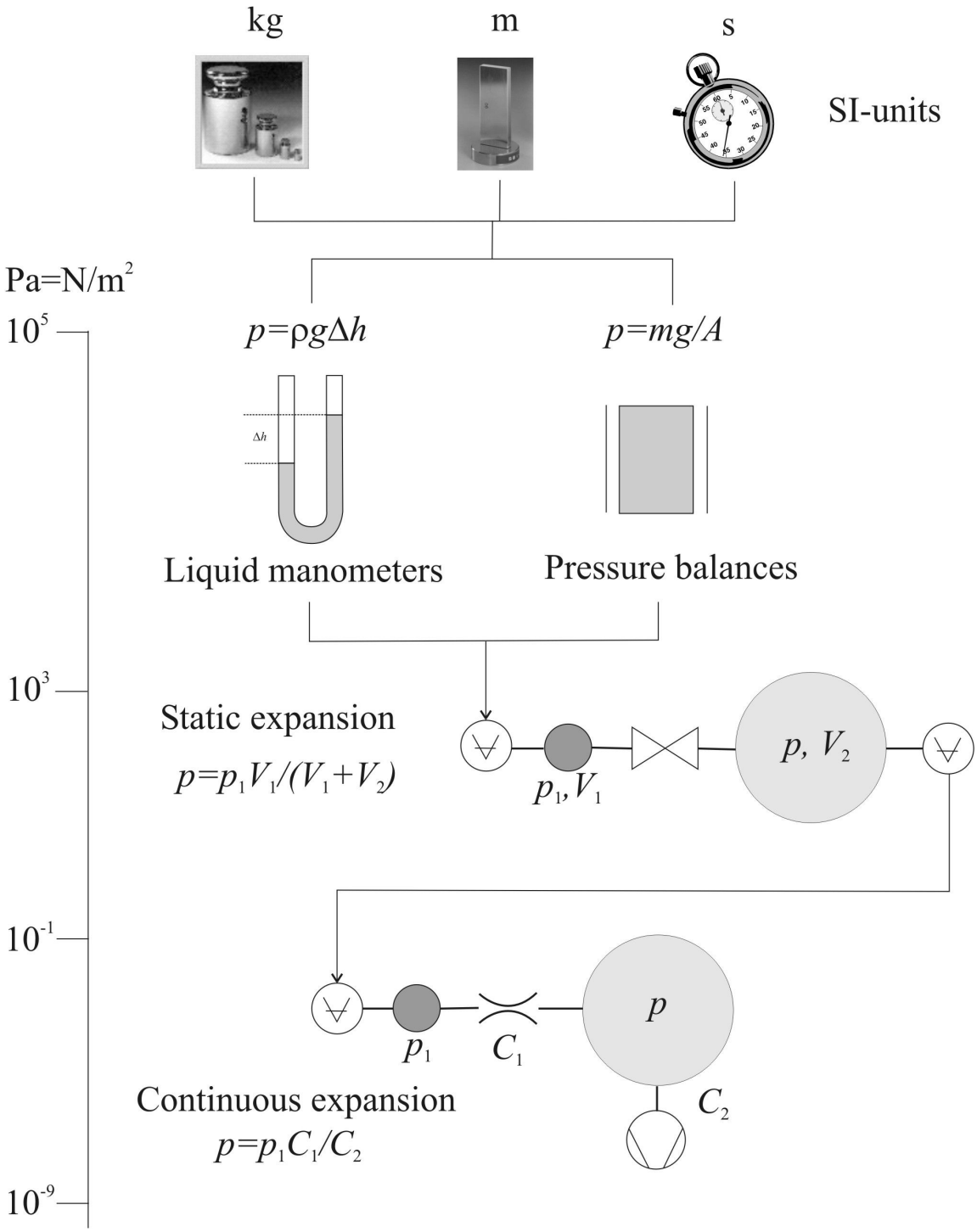

Fig 1. The traceability of the pressure scale in vacuum to the SI units. The pressure scale on the left only gives a rough guideline where the methods can be used. The ranges of the primary standards do overlap and an upper method may even replace a lower one.

A further reduction of pressure can be realized by applying a flow divider between the two conductances as realized in a system by PTB [16] establishing known pressures down to 10-10 Pa.

Figure 2 gives a survey of the uncertainties associated with the realized pressures in the primary standards. Also shown are the lowest measurement uncertainties of some types of vacuum gauges as discussed in the Sections 4 to 6.

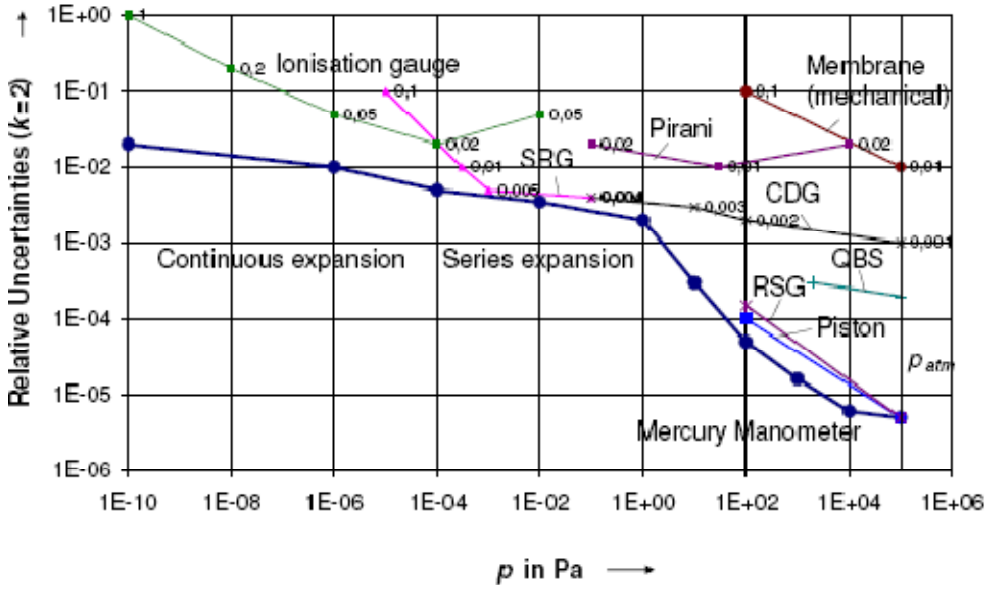

Fig 2. Uncertainties associated with generated (in the case of primary standards) or measured (vacuum gauges) pressures. For clarity of the graph the curves are much simplified and values can be taken only as rough guideline. The range below 10^{-9} Pa is presently not internationally recognized.

3. International Quality Assurance Of National Vacuum Standards

Since on the one hand vacuum primary standards are expensive to built and to maintain and since on the other hand the demand for highest accuracy of calibration of vacuum gauges is not so high, for only a few countries in the world it makes sense to keep vacuum primary standards while other countries are better off with secondary standards as their national standards. Fig. 3 gives an overview of places where national standards from atmosphere into the high vacuum are available.

Independent of the fact which kind of national standard is used, the measurement procedures including the equipment have to be tested by comparisons among the national standards. All institutes that operate vacuum primary standards have signed the Mutual Recognition Arrangement (MRA), which commits each institute to operate a quality management system according to ISO 17025, to publish the calibration measurement capabilities (CMC) including their uncertainty budget, and to participate in comparisons suitable to test the CMC before they are entered in the data base of the International Bureau of Weights and Measures BIPM [17].

Table 1 The published comparisons in vacuum metrology performed or accepted within the Mutual Recognition Arrangement (MRA) of 1999. The Laboratories represented the following countries: BNM-INM – France; CENAM - Mexiko; CEM - Spain; CMU – former Czechoslovokia; CMI – Czech republic; ETL (now NMIJ) – Japan; IMGC (now INRIM) – Italy; IMT – Slovenia; KRISS – Korea; LIP – China; LNE – France; MIKES – Finland; NIST – USA; NMi – Netherlands; NPL – United Kingdom; NPL/I – India; NRC – Canada; NRLM – Japan; OMH – Hungary; PTB – Germany; SMU – Slovakia; SP – Sveden; UME – Turkey.

Year of publication	Pressure range in Pa	Participating Laboratories	Published in
1989 (accepted for MRA)	10^{-4} to 1	CMU, ETL, IMGC, LNE, NIM, NIST, NPL, NPL/I, PTB	[18]
1998 (accepted for MRA)	10^{4} to $1.4 \cdot 10^{5}$	BNM-INM, CSIRO, NIST, NPL, NRC, NRLM, PTB, IMGC, NRC, NPL/I, SMU	[19]
2002	1 to 10^{3}	CSIRO-NML, IMGC, KRISS, NIST, NPL, NPL/I, PTB	[20]
2005	$3 \cdot 10^{-4}$ to $9 \cdot 10^{-2}$	CEM, IMGC, IMT, LNE, NPL, PTB, UME	[21]
2005	$3 \cdot 10^{-4}$ to $9 \cdot 10^{-2}$	CENAM, PTB	[22]
2005	1 to 10^{3}	CEM, IMGC, LNE, MIKES, NMi, NPL, OMH, PTB, SP, UME	[23]
2007	$3 \cdot 10^{1}$ to $7 \cdot 10^{3}$	CMI, PTB	[24]

Both the key comparisons and the regional comparisons showed that most, but not all of the national standards were equivalent. Details of this the reader should get from the references listed in Table 1. As a consequence the non-equivalent ones had to change their CMCs accordingly.

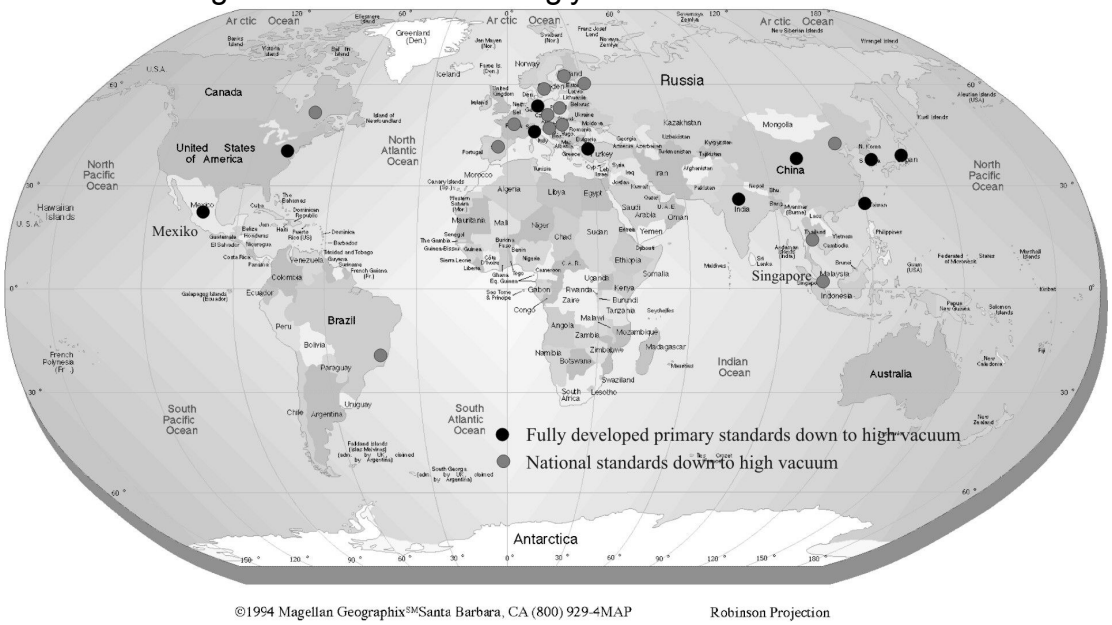

Fig 3. Places in the world with national vacuum primary standards. Fully developed means primary standards from atmosphere to at least high vacuum are available, national standards down to high vacuum means that the standards rely fully or partly on commercial vacuum gauges as secondary standards.

4. Vacuum Gauges Suitable As Secondary And Reference Standards

Secondary standards are calibrated by comparison with primary standards. Reference standards, which may be secondary standards, do have the highest metrological quality at the considered calibration site. Secondary standards or reference standards must have a high accuracy, i.e. mainly a high resolution, good linearity, good repeatibility, a fair robustness against changes in environmental conditions, and show a good transport and long-term stability in order to conserve the calibrated value for a different site.

Table 2 Long-term instabilities over a one-year period (standard uncertainty) of vacuum gauges suitable as reference standard and references where available. "PTB" means our data from our experience (unpublished data). Variations show the range of the majority of the individual gauges tested, higher values may occur for some gauges. It is understood that all gauges are metrologically well treated. FS: Full scale.

Type of gauge	Long-term instability (relative)	at pressure	Reference
Quartz Bourdon spiral (QBS)	$5 \cdot 10^{-5}$ … $2 \cdot 10^{-4}$	30 kPa … 100 kPa	PTB
Resonance silicon gauge (RSG) FS 130 kPa	$2 \cdot 10^{-6}$	100 kPa	[32]
Resonance silicon gauge (RSG) FS 1 kPa and 10 kPa	$1 \cdot 10^{-5} \ldots 5 \cdot 10^{-5}$	1 kPa	[20], [32]
Resonance silicon gauge (RSG) FS 1 kPa and 10 kPa	$1 \cdot 10^{-4}$	100 Pa	[20],[32]
Capacitance diaphragm gauge (CDG) FS 13 and 130 kPa	$1 \cdot 10^{-4} \ldots 1 \cdot 10^{-3}$	FS	[33], PTB
Capacitance diaphragm gauge (CDG) FS 1.3 kPa	$5 \cdot 10^{-4} \ldots 2 \cdot 10^{-3}$	1.3 kPa (FS)	[33], PTB
Capacitance diaphragm gauge (CDG) FS 130 Pa	$7 \cdot 10^{-4} \ldots 3 \cdot 10^{-3}$	100 Pa	[20], [23], [33], PTB
Spinning rotor gauge (SRG)	$1 \cdot 10^{-3} \ldots 5 \cdot 10^{-3}$	$5 \cdot 10^{-3}$ Pa… $5 \cdot 10^{-2}$ Pa	[18], [21], [22], [35], PTB
Ionization gauge (IG), hot emissive cathode	$1 \cdot 10^{-2} \ldots 7 \cdot 10^{-2}$	10^{-7} Pa… 10^{-3} Pa	[31], [36] to [43], PTB

Suitable vacuum gauges with these qualities are high level, zero compensated, Quartz Bourdon spirals (QBS) in the range from 1 kPa to 100 kPa [25], [26], resonance silicon gauges (RSG, 100 Pa to 100 kPa) [27], capacitance diaphragm gauges (CDG, 0.1 Pa to 100 kPa) [28], spinning rotor gauges (SRG, 10-4 Pa to 0.1 Pa) [29], and high-quality emissive (hot) cathode ionization gauges (IG, 10-7 Pa to 10-2 Pa), where the latter have some limits in their transport and long-term stability.

The SRG and the IG are sensitive to the gas species and therefore need pure gases to determine their calibration values.

Both high-quality Pirani gauges as well as crossed fields (cold cathode) ionization gauges exhibit good repeatibility and long-term stability under clean conditions [30], [31] but lack of linearity, which makes them only suitable as reference standards when calibrated very extensively at many pressure points to

determine the degree of non-linearity. In general, their use as secondary standard cannot be recommended.

Quadrupole mass spectrometers (QMS) should only be used as reference standard for very low pressures (< 10-7 Pa), when calibrated for the gas mixture or the pure gas species under consideration and when routinely cross checked with a total pressure gauge as a SRG or IG.

Data for long-term stabilities of the different gauges are not too numerous. In Table 2 the typical values for the different gauges are given including the references. "PTB" refers to data so far unpublished and based on our calibration data for customers.

5. Measurement Uncertainties With Vacuum Gauges

The measurement uncertainties with vacuum gauges have the following major sources:

- Uncertainties due to the calibration chain
- Uncertainties due to the vacuum gauge itself including its long-term instability
- Uncertainties due to the installation of the gauge

In the second point we include the uncertainties due to the physical principle of measurement and the operation of the gauge.

Table 3 List of uncertainties that can be attributed to the vacuum gauge itself and its operation

Sources of uncertainties for vacuum gauges
Offset measurement
Offset instability (drift)
Resolution
Influences of environment (mainly temperature)
Non-Linearity
Integration time (scatter of data), repeatibility
Reproducibility (stability of calibration constant)
Hysteresis
Gas species correction
Prior usage, cleanliness

The uncertainties due to the calibration chain usually can be taken from a calibration certificate. It is clear that a vacuum gauge can never be as accurate as the primary standard at the top of the calibration chain (Fig 4). Instead with each lower level the measurement uncertainty of a reference gauge is increased, since the uncertainties at the time of the usage of the vacuum gauge as reference standard have to be added. Except at the lower end of the measurement range, normally the long term instability is the dominating term that increases the uncertainty given in the certificate. Generally it is understood that the long-term instability also includes influences of transport from the calibration place to the place where it is used as reference standard or ordinary gauge.

Typical uncertainties due to the gauge itself are listed in Table 3.

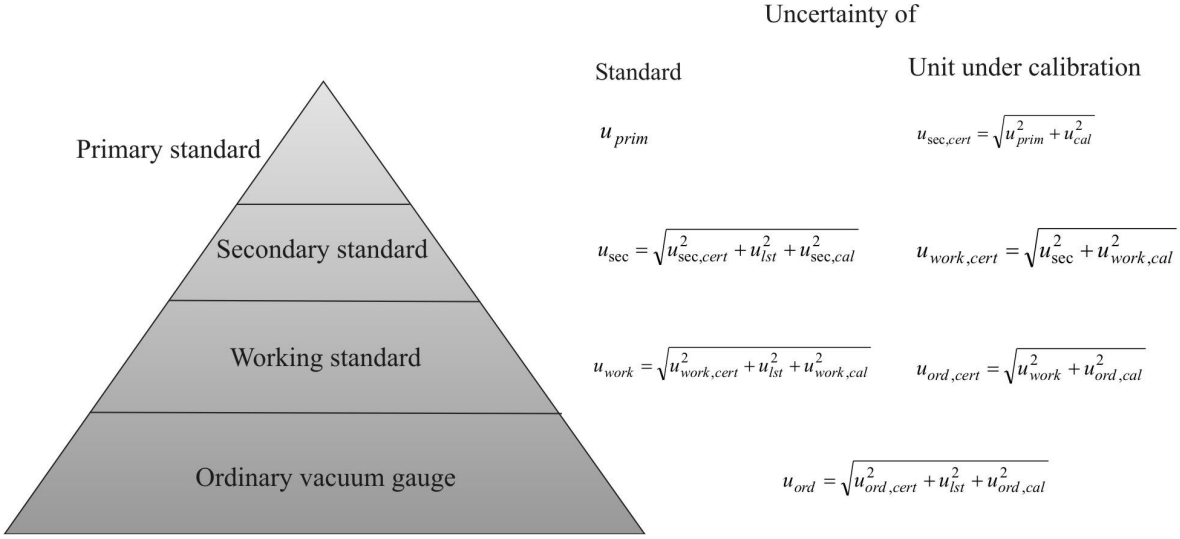

Fig 4. The traceability chain and the uncertainties associated with each level.

The reading of some total pressure gauges is sensitive to the gas species, i.e. the heated CDG in the thermal transpiration regime from about 0.3 Pa to 100 Pa, the SRG, and the IG, and the types of vacuum gauges not recommended as reference gauge as the thermal conductivity gauges and the cold cathode gauges.

In industrial applications quite often the gauge has to be used for a different gas than the gas species used for the calibration which is mostly nitrogen. In this case, a gas correction factor must be applied which, if given, exhibits generally a quite high uncertainty. The sensitivities of vacuum gauges to different gas species is not only due the parameters of the gas species (e.g. its ionisation probability, thermal speed or thermal conductivity) but also due to parameters of the gauge itself, e.g. dimensions of the tubing in a CDG, the accommodation factor of the rotor of a SRG, or the electron energy distribution in an IG. For this reason, the gas correction factor is normally individual to each gauge and, if a generalized factor from the manufacturer for this gauge type is applied, its uncertainty must consider the production scatter as well as the prior treatment of the gauge. The situation becomes even more complicated when gas mixtures are used and weighted correction factors have to be used.

Uncertainties due to the installation of gauges are typical for industrial applications. It is caused by the geometrical distance of the point of interest where the pressure is to be known (e.g. in front of the target in an ion plater) and the flange where the gauge is mounted onto. The pressures at these two points may be different due the gas flow inside the vacuum chamber or due to different temperatures.

In calibration chambers both flow effects and temperature gradients are minimized, but in industrial applications they often cannot be avoided. In these cases the process is often optimized by data from the reading of the gauge, but when the optimum process parameters have to be transferred to a newly installed gauge or to a different apparatus, this may fail. For this reason it is advisable to use calibrated gauges and to estimate or measure the pressure difference or pressure ratio between the point of interest and the position of the gauge. A

conservatively estimated uncertainty should be attributed to this deviation term or correction factor.

6. Uncertainty Ranges Of Vacuum Gauges

In Table 4 we have summarized the uncertainty ranges of the main types of vacuum gauges, not only the ones suitable as reference standards. It is also noted, if the sensitivity of the gauge depends on the gas species. Fig 2 gives a rough overview of values listed in as best measurement capabilities. As reference those vacuum gauges are chosen which uncertainties are closest to the ones of primary standards.

Table 4 Typical uncertainties associated with measurements by the different types of vacuum gauges. The uncertainty values are given as expanded uncertainty (k =2 or two times the standard uncertainty). The values are rough guidelines. Individual gauges may exhibit higher uncertainties, but rarely lower ones.

Gauge type	**Measurement range in Pa**	**Normal uncertainty**	**Optimum range in Pa**	**Lowest uncertainty**	**Gas sensitive**
Pressure balance (Piston gauge)	$10...10^5$		$10^2...10^5$	$1{\cdot}10^{-4}$ ... $5{\cdot}10^{-6}$	no
Quartz-Bourdon-manometer	$10^3...10^5$		$10^3...10^5$	$3{\cdot}10^{-4}$ ... $2{\cdot}10^{-4}$	no
Resonance silicon gauges	10 ... 10^5	$3{\cdot}10^{-3}$ ... $5{\cdot}10^{-4}$	100 ... 10^5	$2{\cdot}10^{-4}$ ... $5{\cdot}10^{-5}$	no
Mechanical vacuum gauge	10^2 ... 10^5	0.1 ...0.01			no
Membrane vacuum gauge	10^2 ... 10^5	0.1 ...0.01			no
Piezo	10^2 ... 10^5	1 ...0.002			no
Thermocouple gauge	10^{-1} ... 10^2	1... 0.3			yes
Pirani gauges	10^{-1}... 10^5	1 ... 0.15	1 ... 100	0.05 ... 0.01	yes
Capacitance diaphragm gauges	10^{-4} ...10^5	10^{-1}... $3{\cdot}10^{-3}$	10^{-1} ... 10^5	$4{\cdot}10^{-3}$ ... $1{\cdot}10^{-3}$	partly
Spinning rotor gauges	10^{-5} ...10	0.1 ... $7{\cdot}10^{-3}$	10^{-3} ...10^{-1}	$5{\cdot}10^{-3}$ ... $4{\cdot}10^{-3}$	yes
Penning gauges	10^{-7} ... 1	$5{\cdot}10^{-1}$...$2{\cdot}10^{-1}$	10^{-5} ... 1	$3{\cdot}10^{-1}$...$1{\cdot}10^{-1}$	yes
Magnetron gauges	10^{-8} ... 1	1 ...$1{\cdot}10^{-1}$	10^{-6} ... 1	$1{\cdot}10^{-1}$...$2{\cdot}10^{-2}$	yes
Ionisation gauges (Emission cathodes)	10^{-10} ... 1	$1.5{\cdot}10^{-1}$...$5{\cdot}10^{-2}$	10^{-8} ... 10^{-2}	$5{\cdot}10^{-2}$...$2{\cdot}10^{-2}$	yes

7. Conclusion

In industrial applications it is very difficult to meet the conditions for accurate pressure measurement in vacuum chambers due to "dirty" processes that change the parameters of the gauge, due to strong gas flows in chambers, and due to temperature differences. For this reason great care has to be taken to validate and optimize processes with a correct vacuum measurement in order to make the information obtained valuable for the future and other systems.

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