

# FINAL PUBLISHABLE REPORT

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**TABLE OF CONTENTS**

1	Overview .....	3
2	Need .....	3
3	Objectives .....	3
4	Results .....	4
5	Impact .....	13
6	List of publications .....	14

### 1 Overview

The ionisation gauge is the only vacuum gauge type for high and ultrahigh vacuum. The pertinent standardisation committee for vacuum technology ISO TC 112 had indicated that important applications need better accuracy, reproducibility and the sensitivity for many gas species, properties which all current types of ionisation gauges lack. This project designed a new type of ionisation vacuum gauge that is accurate (total relative uncertainty: 1 %), robust and long-term stable, with known relative gas sensitivity factors, and provides the relevant parameters for an ISO standard gauge. The gauge can be made by any experienced manufacturer and provides the opportunity for improved traceability within industries that employ high vacuum processes.

### 2 Need

High and ultrahigh vacuum is an indispensable tool for science and industry. Fields of application for science include high-energy accelerators, plasma and fusion science, surface science, and thin film studies, which have a great impact on industry, e.g. optics, optoelectronics, and solar cells. Additional areas of application for industry include the semiconductor industry, the coating industry, and extreme ultraviolet (EUV) lithography, in which the Dutch company ASML, in cooperation with Zeiss, is the only instrument manufacturer worldwide.

The ionisation gauge is the only vacuum gauge type for high and ultrahigh vacuum but is lacking in robustness, as well as long-term and transport stability.

The pertinent technical committees ISO/TC 112 "Vacuum Technology" and the DIN NA 060-07 "Vacuum Technology" section made clear that the reliability and usefulness of ionisation gauges can be greatly improved by standardisation and encouraged research towards a standardised ionisation gauge. Their requirements in detail:

- For pumping speed measurements, ISO 21360-1 requires a standard uncertainty of 3 % of pressure measurement with an ionisation gauge. This is possible for nitrogen, but at present not for any other gas. A standardised ionisation gauge could provide this accuracy for many kinds of gases. Also, the measurement of compression ratios according to ISO 5302 and ISO 21360-1 requires an ionisation gauge with well-known relative gas sensitivity factors which are not available at present.
- Support is needed for the implementation of the two Technical Specifications ISO TS 20175 and ISO TS 20177 by means of a standardised ionisation gauge. This was one of the major needs identified in the EMRP Joint Research Project IND12 and its follow-up Support for Impact project 14SIP01, which will benefit from the standardisation developed in this project.
- Calibration laboratories for vacuum gauges in the HV and UHV ranges do not have reliable reference standards below 1 mPa. An ionisation gauge that is stable over a long-term (relative uncertainty of 1 % over 1 year) will provide this in order to apply ISO 3567 and ISO 27893.

This project has successfully produced and validated a gauge fulfilling these requirements.

### 3 Objectives

The overall objective was to determine and specify all relevant parameters to enable an ISO standard for an ionisation gauge so that this gauge is accurate, robust and long-term stable. Such a standard also strengthens the metrological and technological basis of the ISO TS 20175 and 20177 which require a reliable ionisation gauge.

The specific objectives were:

1. To provide a substantial contribution to the resolution 2015-09 of ISO TC 112. This meant in detail to determine and specify all relevant parameters that are needed to elaborate an ISO standard of an accurate ionisation gauge (total relative standard uncertainty according to GUM [2]: 1 %) in the measurement range from  $10^{-6}$  Pa to  $10^{-2}$  Pa.
2. To make a substantial contribution to the implementation of the two Technical Specifications ISO TS 20175 and 20177 by providing new data material for a stable ionisation gauge and by providing 10 relative gas sensitivity factors of this ionisation vacuum gauge. This is needed for the calibration of quadrupole mass spectrometers and outgassing rate measurement systems as outlined in the aforementioned two Technical Specifications.

3. To work closely with ionisation gauge manufacturers in order to consider their experiences and to make sure that the standard for the ionisation gauge will result in an instrument that is easy to use and economical to produce.
4. To work closely with ISO TC 112 and national standards developing organisations, as well as the future users of the standard, to ensure that the output of the project will cover their need for a reliable ionisation gauge. This includes close communication with their respective working groups in order to consider their input and to make the output of the project easily available to them. That will make the results applicable to a Standard at the earliest possible opportunity.

## 4 Results

### 1. Relevant parameters for ISO standard on ion gauge

A literature review of ionisation vacuum gauges with hot cathodes was performed by project partners PTB, IMT, LNE and CMI. 260 relevant papers dating from the 1950s to the present were identified, reviewed and conclusions drawn. A report collated the most important results. In addition, a review paper of 18 pages and 174 references was published in the journal VACUUM. This was the broadest review paper on ionisation vacuum gauges published so far. These reports identified the most relevant parameters of Bayard-Alpert type and of some other types of ionisation gauges which are important to make the gauge more stable and applicable to standardisation.

From the literature review the consortium concluded that the following effects inhibit an improvement of the metrological characteristics of the existing ionisation vacuum gauges with hot cathodes, in particular the most widespread Bayard-Alpert gauges:

1. Instability of the electron emission distribution from the cathode.
2. Unstable electron trajectories in the gauge head.
3. Secondary electrons produced on the collector by ion impingement.
4. Space charge effects around the ion collector.
5. Electron stimulated desorption of neutrals and ions from the anode.
6. X-rays produced by electrons impinging on the anode.

These effects described in the publications made it rather improbable that a BAG design as of Bayard-Alpert gauges can ever lead to a vacuum gauge with satisfying stability for metrological and scientific needs. For this reason, the consortium pursued a design different from today's commercial hot cathode ionisation vacuum gauges, although a new approach was quite risky, since experiences with existing models could not be adopted. The approaches of Bills et al. in 1984 and Klopfer in 1962 were most interesting in this respect. These designs offer both the possibility of a well-defined electron path and the possibility to separate at least some of the surface effects from volume effects. The Klopfer design also offers the possibility to use a kind of point emitter of electrons avoiding the problem of locally changing electron emission. This design with rigid mechanical parts like plates and cylinders also promises a better mechanical stability than present ionisation gauges made of thin wires and grids. Mechanical stability is important for metrological stability.

Likewise, it was also a finding from the literature review that in the past there were very few systematic investigations to use simulations to improve or design gauges. Instead, researchers in the academic or industrial field mostly used the trial and error method with hardware prototypes. The consortium concluded that the employment of simulations can lead to a significant step of an improved gauge design and also reduces the risk of a failure of a new design.

Electron and ion trajectories were simulated to design the ionisation gauge. Before this, different software options were tested by benchmarking of a commercial gauge of the extractor type. It turned out that 2 of the 4 software packages had severe problems or could not deliver the desired parameter to be compared to the real gauge. For this reason, the simulation of the proposed design was made by CERN and FCT-UNL with the remaining software packages OPERA and SIMION. Later, also the third software package COMSOL with an additional module could be used by INFICON to simulate the gauge characteristics.

For the development of the design, the consortium determined a set of requirements:

- well-defined electron paths through the ionisation volume, stable in time and space, in particular not dependent on the exact origin of the electron on the hot emission cathode
- well defined and known electron energy inside the ionisation volume
- electrons do not hit the anode to avoid reflection and electron induced desorption
- high electron transmission efficiency  $\approx 1$  through the ionisation volume, not affected by small mechanical misalignments or fluctuations of electrode potentials
- high ion collection efficiency  $\approx 1$
- ion current at  $10^{-6}$  Pa  $\geq 10$  pA
- linearity (non-linearity  $< 1\%$ ) in the pressure range from  $10^{-6}$  Pa to  $10^{-2}$  Pa
- linearity with electron emission current
- no depletion of neutral molecules inside electron beam
- ions produced outside the well-defined ionisation space must not reach the ion collector
- secondary electrons and photons produced by the impingement of electrons must not reach the ionisation volume or collector
- ion induced secondary electron yield on the ion collector must be stabilized

The resulting design is shown in Figure 1 and consists of six electrodes: an electron emitting cathode with a Wehnelt cylinder around, an anode, an ion collector, a deflector and a Faraday-cup. With this design, refined in a second step, all the requirements except the last one listed above could be met. For the last one a conditioning procedure had to be developed.

To tackle the problem of instable emission of thermionic cathodes, the project took two measures.

- a. Use of thermionic emission from a disk attached to a heating wire.
- b. Use of a Wehnelt-electrode around it.

With this assembly, all emitted electrons are guided forward and enter the ionisation volume in a rather parallel beam (Figure 1, top). No matter from which location on the emitter disk the electrons leave, they follow closely the same trajectories inside the ionisation volume. The electron path length is not affected by local changes of work-functions and temperatures on the cathode. To further define the effective electron path length, the anode as ionisation volume was carefully designed by apertures at the entry and the exit. The mechanical arrangement of these areas is designed such that the boundaries, at which ions are collected by the ion collector, coincides within the dimensions of the entry and exit plane of the cylindrical anode cage. This length of 50 mm (Figure 1, bottom left) is the nominal electron path length and is used to calculate the ion collector current and sensitivity of the gauge.

The ion collector is composed of two elements, a focussing ring and a rod. The collector ring has the function of central electrode of an electrostatic lens which focuses the electron beam through the cylindrical anode into the exit aperture. In this way, emitter misalignments of more than 0.3 mm can be tolerated in all directions. The collector rod extracts the ions from the electron beam over its full length in the anode cylinder by applying a small extraction potential gradient to overcome the space charge potential inside of the electron beam.

After exiting the ionisation chamber, a deflector directs the electrons into a Faraday cup. The ratio of electron current arriving in the Faraday cup and the emitted current leaving the emitter defines the electron transmission efficiency. Electrons hit the Faraday cup in an area where there is no direct view to the ion collector. Ions emitted from the Faraday cup due to electron stimulated desorption will be efficiently collected by the deflector. Generated X-ray photons cannot directly reach the collector. This absence of X-ray photons gave freedom in the design of the collector electrode. With the large collector surface compared to Bayard-Alpert gauges a low ion density around the collector was ensured avoiding space charge effects around the collector at high pressures. The shape of the collector also eliminates any loss of ions due to their angular momentum as is the case in present ionisation vacuum gauges of the Bayard-Alpert type.

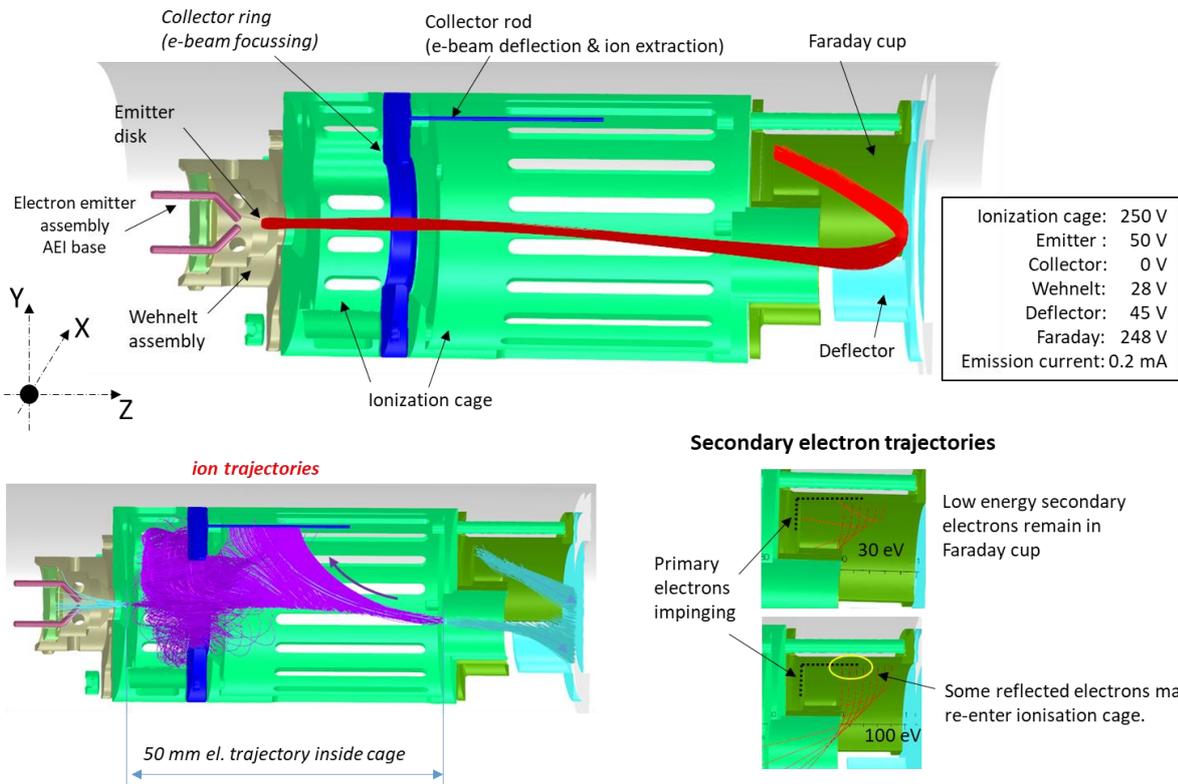


Figure 1 Design and charged particle trajectories of the new gauge. Top: the electrode arrangement and electron trajectories with the potentials and emission current applied for this simulation. Bottom left: ion trajectories. The purple trajectories correspond to those that are collected by the ion collector. The light blue ones are those that are rejected. Bottom right: Trajectories of secondary electrons leaving the surface perpendicular with the indicated kinetic energy.

Ions generated behind the exit plane of the ionisation volume are attracted to the deflector (Figure 1, bottom left). Furthermore, most of the secondary electrons that are generated inside the Faraday cup are pushed back by the deflector (Figure 1, bottom right). Only a small part of secondary electrons can reach the ionisation volume.

The robustness of the design was tested by simulations carried out in a cooperation of CERN and RISE. A statistical evaluation was carried out in order to find the tolerances of the electrode positions for manufacturing. The range of displacements in which the performance parameters (electron transmission, collection efficiency and sensitivity) remain within the target precision of 1% was determined. Varying the emitter potential, the energy distribution of the emitted electrons was considered accounting for the fact that the variation of heating currents causes variations of the emitter potential due to the change in electrical potential drop along the heating wire.

The ion induced secondary electron production on the ion collector could not be eliminated by the new design. An additional electrode would greatly complicate the design with unreliable success. The processes responsible for the electron emission in the ion energy range of 50 eV - 250 eV were also part of the literature review. They take place within the ion penetration depth, i.e. they are restricted to the first several nanometer of the material surface. Any change of this surface will directly affect the gauge sensitivity, thus introducing measurement instabilities. Since we could not eliminate the ion induced secondary electron yield, the project aimed at stabilizing it. In past publications, bombardment of the collector by Ar<sup>+</sup> ions was considered as a method to stabilise the ionisation gauge sensitivity by cleaning the collector due to sputtering. FCT-UNL, however, found that the reason for the stabilisation effect is exactly the opposite: when the partial pressure of hydrocarbons inside the gauge is sufficiently high, ion bombardment promotes hydrocarbon layer growth, so that the sensitivity stabilisation is achieved by reaching a sufficiently thick hydrocarbon overlayer on the

collector surface. Under such conditions, the ion induced secondary electron yield becomes independent of the collector material.

The surface investigations of the ion induced secondary yield were studied in an experimental ion gauge simulator built for this project by FCT-UNL. The research focused on the changes of ion induced secondary electron yield (IISEY) due to the exposure to an ionisation gauge environment. The IISEY measurements were performed on five different materials, with three ion species. The investigations revealed that there is a strong coverage of hydrocarbons on all materials. This contamination is caused by the hot cathode in the ion gauge. The contamination effect with an yttrium-oxide coated iridium cathode is somewhat lower than with a tungsten cathode, but still significant. Most interestingly, all work functions tend to reach the same value, independent of the substrate. The consortium concluded that the presently best way to stabilise the ion induced secondary electron yield is to condition the gauge by an operation in an argon environment before a measurement. This conditioning produces the hydrocarbon layer on the collector with the more stable ion induced secondary electron yield.

TRIDYN simulations of low energy ion sputtering of different materials were performed, reaching satisfactory agreement with the experiments. This software package, supported by the deterministic sampling method, could also be used to evaluate the coating lifetime of the cathode under low energy ion bombardment.

It can be concluded that the combined efforts of all partners in the literature review, the simulations and the material investigations has led to a gauge design that eliminates or significantly reduces all the problems present in ionisation vacuum gauges causing a spread and instability of the metrological gauge parameters. Describing such a gauge in a standard is now possible, therefore objective 1 has been fully achieved.

### **2. New data material for a stable ionisation gauge**

The consortium agreed on two commercial gauges to be tested as a benchmark for the laboratory and model gauge and the quantities to be investigated were fixed and measured. These results were compared to the data obtained with the new gauge developed by the project.

The NMIs adapted their existing calibration systems to the gases and measurement uncertainties needed to test the laboratory gauges being developed in the project, and to measure the 10 relative gas sensitivity factors with the new gauge.

In November 2017, the consortium agreed on the gauge design to be pursued. It was a design that cannot be found on the market at present. As already mentioned, this was somewhat risky, since experiences with existing models could not be adopted. The consortium, however, was of the mind that the technical reasons causing instabilities in ionisation gauges could not be overcome by modifying existing designs. VACOM developed the technical drawings of the laboratory gauges and produced ten laboratory gauges to be tested by the consortium. The tests of the laboratory gauges showed that the design is very successful. Issues were of technical nature only. For this reason, only technical improvements concerning robustness for transport stability and better electrical insulation between electrodes were proposed for the model gauges, maintaining the principal design.

With these changes new or modified technical drawings of the model gauges were developed by VACOM and INFICON and 23 model gauges (Figure 5) produced by the two different gauge manufacturers of the consortium. All electrodes were made of low carbon stainless steel. As emission cathode, an indirectly heated Ta disk on an AEI base, commercially available, was used.

The potentials of Wehnelt electrode, Faraday cup and deflector were optimised by experiments. The potentials of cathode (50 V) and anode (250 V) are crucial to the sensitivity because of the energy dependence of ionization cross and to the focusing of the electron beam and were therefore fixed.

It was found that the apparent electron transmission efficiency, defined as the electron current measured on the Faraday cup divided by the total emission current, is 97 % for the nominal values of electrode potentials used for simulation by CERN and FCT-UNL. The electron transmission efficiency may be affected by secondary electrons. An increase of the Faraday potential from 248 V to 252 V increases it to approximately 98%. This is attributed to the suppression of low energy secondary electrons from the Faraday cup as found by the simulations at CERN and material investigations at FCT-UNL. A further increase of the Faraday cup potential to 350 V yields a transmission efficiency of 99%, which is attributed to the additional suppression of high energy electrons elastically scattered from the Faraday cup. Based on these findings, a Faraday potential of 280 V was selected for further measurements, for which the electron transmission efficiency is  $\approx 98\%$ .

For well aligned cathodes there is practically no dependence of the electron transmission efficiency between 15 V and 35 V of the Wehnelt potential. For misaligned cathodes, however, a significant dependence of the electron transmission efficiency on the Wehnelt potential is observed. Hence the electron transmission efficiency is a good indicator for proper alignment. Even for a misalignment, however, the sensitivity remains unchanged. The electron current reaching the Faraday increases monotonically with the Wehnelt potential. Since this current determines the ionising current, the optimal Wehnelt potential is the one, at which the emission current is maximal, and electron transmission efficiency is still at its maximum level. When the electron transmission efficiency is reduced at higher Wehnelt potentials, a defocusing of the electron beam occurs leading to the unwanted effect of electrons hitting the anode causing detrimental effects by electron induced emissions.

The main goal of the project was to achieve a design where relative gas sensitivity factors are independent of the individual gauge. The tests revealed that even absolute sensitivities are independent of the individual gauge. The values of measured nitrogen sensitivities for different manufactured model gauges are presented in Figure 2, which also contains the sensitivity values obtained by simulations.

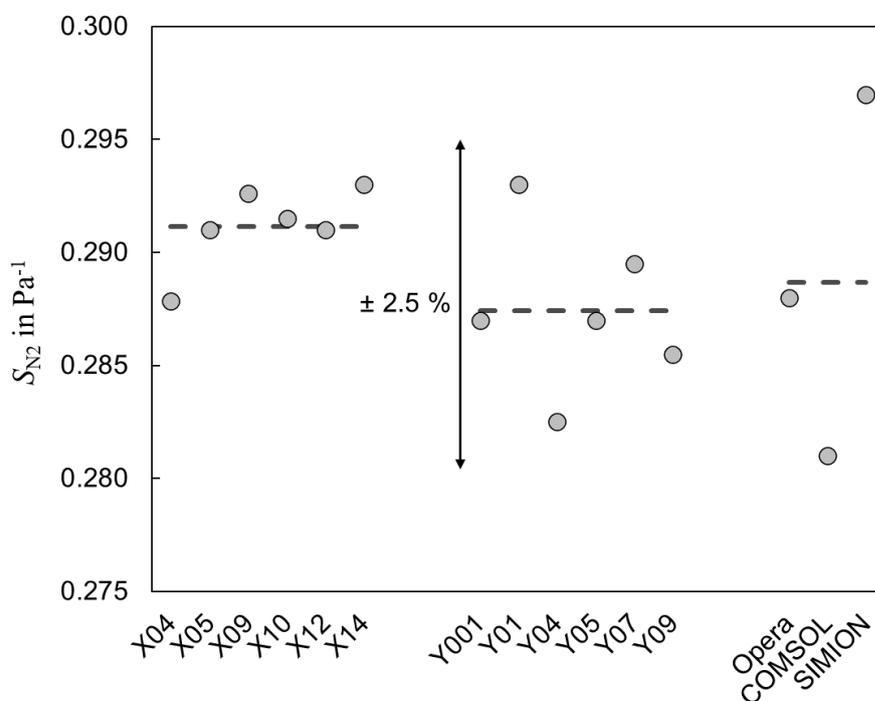


Figure 2 Measured sensitivities for  $N_2$  of six gauges from manufacturer X (INFICON) and six gauges from manufacturer Y (VACOM) as well as three calculated sensitivities from different simulation software packages

The reproducibility of manufacturing can be quantified by the standard deviation of sensitivities. These data are collected in Table 1. The relative standard deviations were 0.63% and 1.24% for manufacturer X (INFICON) and Y (VACOM), respectively, which shows the excellent reproducibility of manufacturing.

The relative difference of  $N_2$  sensitivities between the gauge types of the two manufacturers is 1.3 %. It must be noted that the number of samples is still relatively small, and the sensitivities were measured in different National Laboratories, which means that the difference, the mean values and standard deviations include possible bias between the different measurement standards.

Relative gas sensitivity factors as defined in ISO 27894 were measured for 12 gases (Table 2). The variations between the gauges were within a range from 0.1% to 1.5 % i.e. very small, which indicates the great success of this new design. Oxygen is an exception, but for this chemical active species a higher value can be expected. It needs to be noted that for some gas species the data is statistically not yet robust enough. For most gases the measured values and the values obtained from ionisation cross sections of electrons at 200 eV or obtained

from simulations which consider the actual kinetic energy of the electrons on their path through the ionisation volume agree. For some gas species ( $H_2$ , He,  $CH_4$ ), however, there are significant differences between the relative gas sensitivity factors and ratios of the ionisation cross sections. Ionisation cross sections are uncertain by about 10 %. This may be a possible reason for the discrepancy and needs to be investigated further.

Table 1 Mean values and standard deviations of measured sensitivities for nitrogen of manufactured gauges by two manufacturers. The difference of the mean values is also given.

	Mean value [Pa <sup>-1</sup> ]	Standard deviation [Pa <sup>-1</sup> ]	Relative standard deviation
Manufacturer X (INFICON) (6 gauges measured)	0.2912	0.0018	0.63%
Manufacturer Y (VACOM) (5 gauges measured)	0.2874	0.0036	1.24%
	Difference of two manufacturers		relative difference
	0.0038		1.29%

Table 2 Mean relative gas sensitivity factors determined for the new ionisation vacuum gauge design. The variation between different gauges is expressed in the 3<sup>rd</sup> column as standard deviation.

Gas species	$S_x/S_{N_2}$		St.dev.
$H_2$	0.374		0.0151
He	0.176		0.0049
$CH_4$	1.385		0.0150
Ne	0.337		0.0151
$H_2O$	0.821		0.0156
$N_2$	1.000		
CO	1.021		0.0030
$O_2$	0.964		0.0565
$CO_2$	1.433		0.0289
Ar	1.134		0.0125
Kr	1.523		0.0062
Xe	2.215		0.078

Another important characteristic of any measuring instrument is linearity. This was tested with nitrogen gas in the pressure range from  $1 \times 10^{-6}$  Pa to  $1 \times 10^{-2}$  Pa. All gauges tested showed a linearity well within  $\pm 0.5$  %. It was also checked if there is a depletion of molecules within the electron beam. This would be revealed by a non-linearity of ion current versus electron current. In  $N_2$  at  $1 \times 10^{-2}$  Pa and up to 300  $\mu A$  electron current no non-linearity was found. For the gauge tests by the consortium, 30  $\mu A$  electron current was used.

The gauge also showed an excellent repeatability. IMT took 10 sensitivity measurements at the same pressure within one hour. Between the measurements the system was evacuated to a base pressure below  $1.5 \times 10^{-7}$  Pa. Relative standard deviations were 0.019% at a pressure of  $1.0 \times 10^{-5}$  Pa and 0.044 % at  $1.0 \times 10^{-4}$  Pa.

The resolution limit is mainly determined by the resolution of the current meter measuring the ion current and the stability of the residual current. So far, a resolution down to  $2 \cdot 10^{-14}$  A was demonstrated by PTB and IMT at measured residual offset current of 1 pA. With an emission current of 30  $\mu$ A and the nominal sensitivity for nitrogen of  $0.29 \text{ Pa}^{-1}$ , the pressure resolution limit is  $2 \cdot 10^{-9}$  Pa.

Another important goal of the design was stability over time and stability after regular operations with the gauge like venting, bake-out, idling at residual pressure, exposure to other gases, cathode exchange, and transport. To quantify the reproducibility of sensitivity over time of individual gauges the quantity

$$\Delta = \frac{S_i - \bar{S}}{\bar{S}} \tag{1}$$

was defined as relative difference of sensitivity  $S$  at measurement  $i$  to the mean value of sensitivity  $\bar{S}$  obtained for this gauge and gas.

Figure 3 shows  $\Delta$  over a period of 5 days or more, for gauges Y04, X04 and X10, measured at three different laboratories. Sensitivities were measured for argon and nitrogen gas. Before the first measurements the gauges were baked at  $150 \text{ }^\circ\text{C}$  for a period of 48 h to 110 h, followed by an operation at an Ar pressure of 5 mPa ("conditioning"). At PTB and LNE the duration of this conditioning was 1 h, and at IMT 2.5 h for gauge Y04 and 1.6 h for X04.

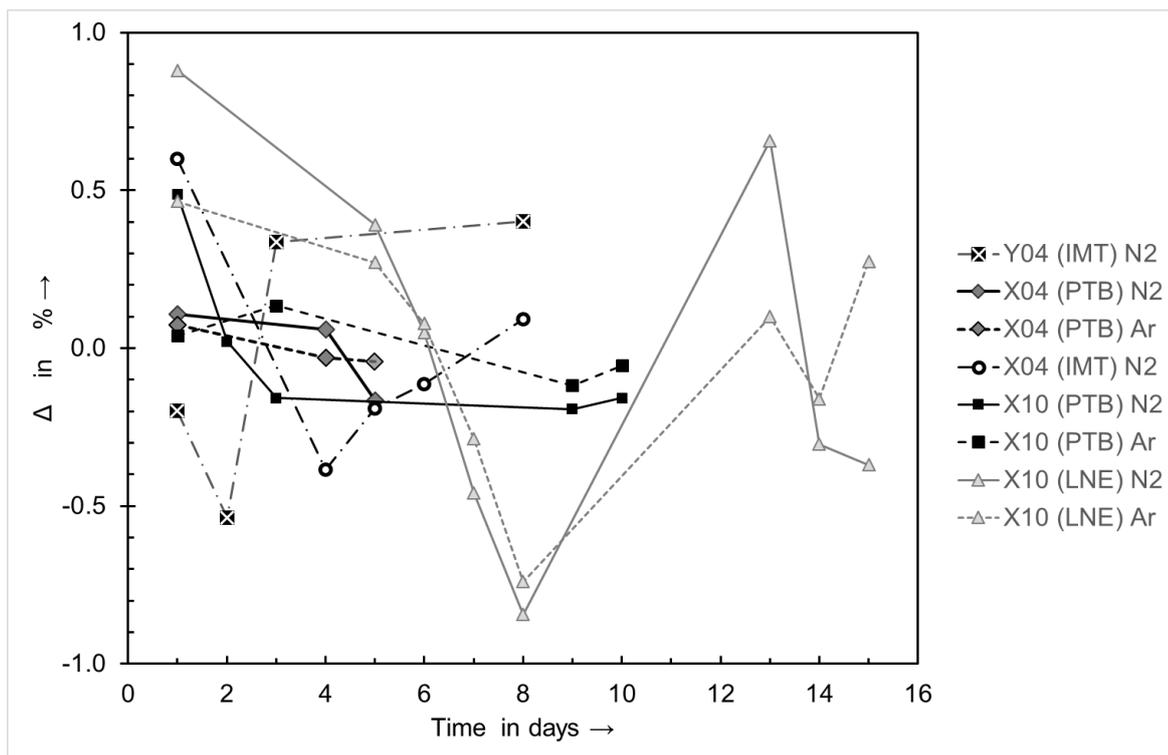


Figure 3 Reproducibility of the sensitivity over 5 or more days for three selected gauges Y04, X04 and X10 (see text). Gauge X10 at LNE was vented to atmosphere and baked between measurements on Day 8 and Day 13.

A few more stability tests of the different gauges are described in the following (see Figure 3): For gauge Y04, after  $\text{N}_2$  sensitivity measurements on Day 2, sensitivities for active gases  $\text{O}_2$ ,  $\text{CO}$  and  $\text{CO}_2$  at pressure  $3 \times 10^{-4}$  Pa were measured, followed by operation at residual pressure below  $3 \times 10^{-7}$  Pa for 16 h until the next  $\text{N}_2$  sensitivity measurements on Day 3. Then, the gauge was left in operation at residual pressure for 5 days before the final measurement on Day 8.

Gauge X04, measured at IMT, received a second and third conditioning with a duration of 3 h at Ar pressure of 5 mPa before measurements on Day 4 and 5, respectively. On Day 7 sensitivities for the "active" gases O<sub>2</sub>, CO and CO<sub>2</sub> at pressures of 3×10<sup>-5</sup> Pa (10 times lower pressures than with gauge Y04) were measured. The gauge was then left in operation at base pressure below 2×10<sup>-7</sup> Pa for 14 h until the final measurement on Day 8.

Gauge X10 at LNE was included in a study of repeated conditioning at an Ar pressure of 5 mPa. The duration of conditioning was 1h before measurements on Day 5 and 6, and 2h before measurements on Days 7 and 8. Then the gauge was exposed to atmosphere and baked at 150 °C for three days. After bakeout, the sensitivity was first measured without Ar conditioning on Day 13. After that, two more sensitivity measurements were performed on Days 14 and 15 after Ar conditioning for 2 h on each day.

It can be seen that  $\Delta$  after all these procedures was within  $\pm 1\%$  for the 3 gauges.

To test the robustness of the design after repeated venting and baking, RISE made an experiment with 5 repeated cycles (Figure 4). Each cycle started with venting to atmosphere for 1 h, followed by a vacuum bakeout at 150 °C for 24 h and a cool-down phase to 30 °C. The gauge was put in operation at residual pressure for at least 12 h, after which it was conditioned in Ar for 1 h at 30  $\mu$ A emission current. The pressure was 1×10<sup>-2</sup> Pa for runs No. 1, 4 and 5, and 1×10<sup>-3</sup> Pa for runs No. 2 and 3. Measurements of the sensitivity for Ar and N<sub>2</sub> followed at a pressure of 1×10<sup>-3</sup> Pa. Values of the sensitivity after these repeated venting and baking periods were within  $\pm 1\%$  (Figure 4).

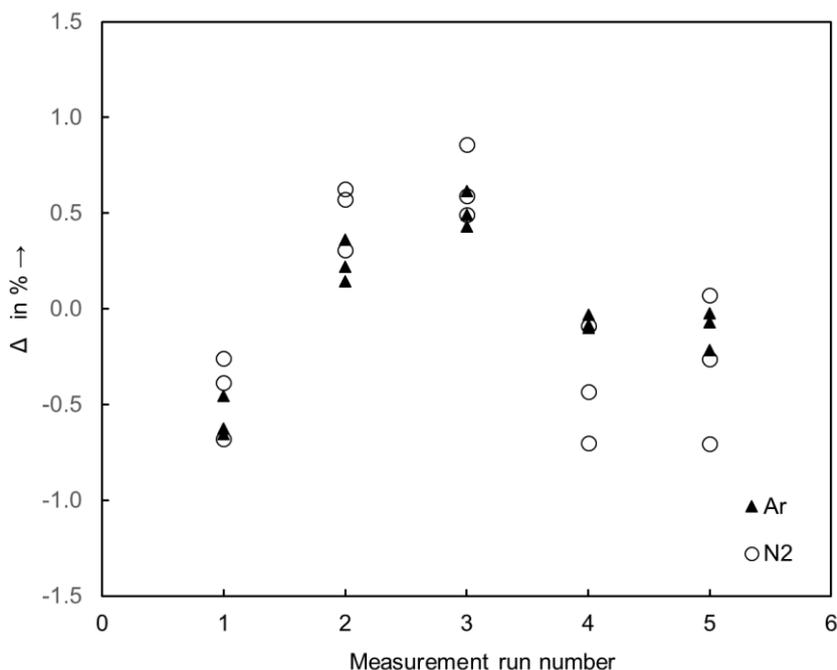


Figure 4 Reproducibility of the sensitivity for Ar and N<sub>2</sub> after repeated venting and baking (gauge X014). For runs No. 1, 4 and 5, conditioning was done at a Ar pressure of 1×10<sup>-2</sup> Pa, and for runs No. 2 and 3 at 1×10<sup>-3</sup> Pa.

For one of the model gauges (X012) PTB checked the sensitivity after a replacement of the cathode. The sensitivity for N<sub>2</sub> was 0.291 Pa<sup>-1</sup> for the original cathode and changed to 0.290 Pa<sup>-1</sup> after the cathode exchange which is within the measurement uncertainties. This indicates that a cathode exchange does not require recalibration. The electron transmission changed by 0.4% only.

To check the stability after a transport, CMI packed a gauge copy in a cardboard box and exposed it to a road transport in a car trailer for a total of 740 km, followed by an impact test. The box with the gauge was dropped 10 times from 1 m height (1 × corner, 3 × edge, 6 × face). Measurements after the transport and drop-down tests showed a reduction of the electron transmission efficiency to 90 % from 95 % before the test. The shape of the transmission function over Wehnelt potential was typical for a displaced cathode. This displacement of the cathode, however, had little effect on the N<sub>2</sub> sensitivity. Its value was 0.2933 Pa<sup>-1</sup> before the test, and 0.2923 Pa<sup>-1</sup> after, so the relative change was only -0.34%.

A second transport test was carried out as a laboratory comparison between PTB, CMI, IMT and LNE. Two gauges (one from each manufacturer) were selected as transfer standards and circulated in in separate loops. The N<sub>2</sub> sensitivity of the first gauge measured at PTB at the beginning of the comparison was 0.2809 Pa<sup>-1</sup>. When the gauge had returned to PTB after 4 months the sensitivity was remeasured to 0.2793 Pa<sup>-1</sup>, corresponding to a relative change of -0.57%. For the second gauge the initial value was 0.2868 Pa<sup>-1</sup> and the final value (measured after 7 months) was 0.2861 Pa<sup>-1</sup>, corresponding to a relative change of -0.25%. These are excellent stabilities compared to transport instabilities of past comparisons, where the changes after transport were in the range from 2% to 7%.

In conclusion, the tests of the model gauges in the different NMIs produced outstanding results and justified the risk that the consortium took with its decision to try a completely new design. This new design exceeds the performance of the existing designs of ionisation vacuum gauges by far in terms of predictability of sensitivity, linearity, repeatability, reproducibility, robustness, and transport stability. The relative uncertainty from these effects is below 1 % of measured vacuum pressure values. On top, the measured sensitivity for nitrogen agrees with the one expected from the simulation with the three software packages. The electron transmission through the ionization region is close to 100% so that the electron path length is well-defined. Relative gas sensitivity factors were measured for 12 gas species. This objective was successfully completed.

### 3. Cooperation with gauge manufacturers

The consortium incorporated the two-gauge manufacturers INFICON and VAVOM as partners who by contributing their experience helped ensure that the standard for the ionisation gauge resulted in an instrument that is easy to use and economical to produce. VAVOM delivered the design of the new type of ionisation gauge according to the results gained from the literature review and simulations and produced ten laboratory gauges to be tested by the consortium. After the tests of the laboratory gauges, some improvements were decided for the design of the model gauges. These were implemented by both manufacturers and each produced at least ten model gauges of the same electrode design, but with different technical layouts (see Figure 5) using their respective production lines. The tests described in the previous Section 4.2 (see e.g. Fig. 2) showed agreement between the different layouts of the manufacturers. This ensures that, when the gauge type is standardised, it will be independent of the characteristics of the gauge manufacturer.

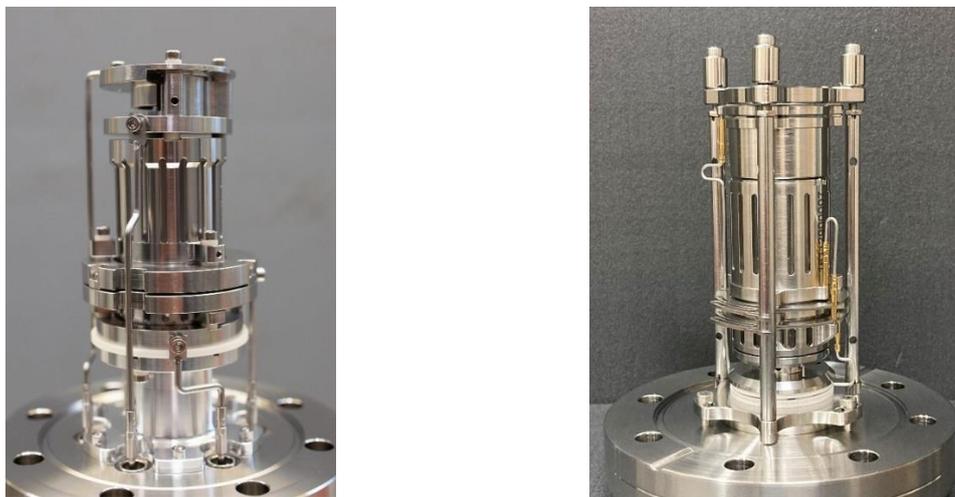


Figure 5 Photographs of two model gauge types realising our design, each from a different manufacturer (INFICON and VAVOM)

The inclusion of two industrial gauge manufacturers in this project has been a complete success, their experience and advice ensured that the instrument can be realised in an economical manner and is practical to use. By having two different technical drawings and two different production lines realise the same new ionisation vacuum gauge design it could be shown that the metrological parameters are independent of the manufacturer. Therefore, demonstrating that nothing stands in the way of ionisation gauge standardisation.

## 5 Impact

### *Key Highlights of Dissemination Activities:*

- Consortium partners presented the project and its research results to the ISO TC 112 Working Group 2 on Vacuum Instrumentation (the advisory group of the project) in November 2017, May 2019, and November 2020
- The consortium gave two presentations and presented one poster at the 15<sup>th</sup> European Vacuum Conference in June 2018
- The consortium gave four presentations and presented a poster at the 21<sup>st</sup> International Vacuum Congress in July 2019
- The consortium published four articles in open access peer reviewed journals, three of them with authorship from at least two partners, a fifth article has been submitted.

### *Impact on industrial and other user communities*

As a result of this project, vacuum gauge manufacturers now have the option of producing in-house a reference ionisation gauge that due to its inherent sensitivity enables significant reductions in calibration costs and the time penalties that this introduces. In addition, the ability to change the gauge's cathode, or to replace it in a production process without the need of recalibration or readjustment offers substantial benefits to users in the semiconductor, coating and aerospace industries.

For pumping speed measurements, ISO 21360-1 requires an accuracy of 3 % of pressure measurement with an ionisation gauge. By introducing the project designed ionisation vacuum gauges, high-vacuum pump manufacturers will be able to fulfil this requirement not only for nitrogen, but also for other gas species – an important attribute for many of their industrial and academic customers.

The exchangeability of a standardised gauge, one of its most important features, will have a significant impact in the vacuum market by reducing maintenance costs. The company ASML, the only producer of EUV lithography equipment, has set-up a working group with its suppliers and PTB to establish guidelines for traceable outgassing rate measurements based on ISO TC 20175 and 20177. The role of a standardised ionisation gauge has been emphasised in this group.

### *Impact on the metrological and scientific communities*

Achieving ultrahigh vacuum is important to European researchers at for example the high energy and fusion physics facilities of CERN, DESY, ESRF, and ITER. They rely on the ability to make reliable measurements of many different gas species. This is another area expected to benefit from the use of the project developed reference/standardised vacuum gauge. Examples of uses are determinations of nuclear/atomic collision cross sections, ionisation probabilities, nuclear/atomic absorption cross sections and gas exposure measurements in surface adsorption experiments.

The impact in the metrology area is twofold: (i) The function of ionisation gauges as reference and transfer standards for calibration services has been improved and widened to relevant gases other than nitrogen. National Metrology Institutes now have a reliable transfer gauge for high and ultrahigh vacuum to compare their primary standards. (ii) The use of ionisation gauges to calibrate quadrupole mass spectrometers in situ is much more accurate with a standardised ionisation gauge as relative gas sensitivity factors are reliably known. Such an in-situ-calibration is frequently needed for outgassing rate measurements.

The consortium held a web meeting with the CCM WG PV. The group appreciated the success of this development and qualified the new ionisation vacuum gauge as good candidate for a transfer standard in future CCM KCs in the high vacuum range. NMIJ as pilot laboratory for the next comparison in this range indicated interest to test it as transfer standard and asked for a copy.

### *Impact on relevant standards*

As a result of this project it will be possible to develop an ISO standard (Technical Specification) for an ionisation vacuum gauge as outlined in the business plan of ISO TC 112. The consortium held a web meeting with the ISO TC 112 WG 2. The group appreciated the success of this project in specifying the requirements for a standard ionisation gauge and recommended starting the ballot process within ISO TC 112 for a new project to cast our draft 'working' technical specification into a standard Technical Specification. The ballot of ISO/NTS 6737 was started in January 2021. With such a standardised ionisation gauge, ISO TS 20175 and 20177 will be able to be effectively implemented. ISO 3567, 5302 and 21360-4 and other newly developed standards for high vacuum pumps in the 21360 series will also greatly benefit from a standardised gauge.

#### *Longer-term economic, social and environmental impacts*

As a wider impact, more accurate pumping speed values for a greater number of gases will provide designers of vacuum plants with reliable data for sizing pumps and gas flows. With more accurate and more reliable data, safety margins can be reduced, and gas consumption minimised. This will save resources, improve work security for explosive and poisonous gases in the semiconductor and coating industry and reduce environmental pollution.

The vacuum gauge manufacturers will benefit from a standardised ionisation gauge design as less effort will be required for the calibration of the manufactured gauge with no need to calibrate sensitivity for gases other than nitrogen.

When the project developed standardised ionisation gauges are used in plants, an exchange of the gauge will be possible without readjusting process parameters in the semiconductor and coating industry, because the sensitivities for all process gas species will be known with high accuracy. The exchangeability of the standardised gauge may have a great impact on the vacuum market where time consuming vacuum process readjustments lead to unscheduled production stoppages and lost productivity.

The ionisation gauge developed by the project will improve the control of vacuum processes in the semiconductor and coating industry by the more accurate pressure measurements at the different places in a plant. At present, due to the sensitivity scatter of ionisation gauges gas flow measurements under high vacuum conditions are unreliable. This better control will lead to higher cost efficiency, greater safety, and improved environmental protection due to a better waste management of vacuum processes. It will improve quality assurance procedures of European vacuum equipment manufacturers helping increase customer confidence in European products.

The European vacuum industry is traditionally at the forefront worldwide, especially in the excellence of its mechanical engineering. There are several companies in Europe that manufacture ionisation gauges. The knowledge gained by the project will give them an improved competitive edge compared with Asian and American companies.

The improvement of the measurement possibilities brought about by this project will enable European manufacturers of process tools, vacuum pumps, and vacuum and partial pressure gauges to improve their products.

## 6 List of publications

1. R. Silva, N. Bundaleski, A.L. Fonseca, O.M.N.D. Teodoro, *3D-Simulation of a Bayard Alpert ionisation gauge using SIMION program*, Vacuum **164** (2019) 300-307  
<https://doi.org/10.1016/j.vacuum.2019.03.039>
2. I.G.C. Figueiredo, *Investigation and characterization of materials towards building ionization vacuum gauges* (Master's thesis), Nova University of Lisbon (2018) <http://hdl.handle.net/10362/52578>
3. R.A.S. Silva, *Desenvolvimento de um manómetro de ionização de elevada estabilidade* (Master's thesis), Nova University of Lisbon (2018) <http://hdl.handle.net/10362/59610>
4. Jousten K, Boineau F, Bundaleski N, Illgen C, Setina J, Teodoro OMND, Vicar M, Wüest M, *A review on hot cathode ionisation gauges with focus on a suitable design for measurement accuracy and stability*, Vacuum 179 (2020) 109545, <https://doi.org/10.1016/j.vacuum.2020.109545>.
5. Jenninger B, Anderson J, Bernien M, Bundaleski N, Dimitrova H, Granovskij M, Illgen C, Setina J, Jousten K, Kucharski P, Reinhardt C, Scuderi F, Silva RAS, Stöltzel A, Teodoro OMND, Trzpił-Jurgielewicz B, Wüest M, *Development of a design for an ionisation vacuum gauge suitable as a reference standard*, Vacuum 183 (2021) 109884, <https://doi.org/10.1016/j.vacuum.2020.109884>.
6. I. Figueiredo, N. Bundaleski, O.M.N.D. Teodoro, K. Jousten, C. Illgen, *Influence of ion induced secondary electron emission on the stability of ionisation vacuum gauges*, Vacuum 184 (2021), 109907, <https://doi.org/10.1016/j.vacuum.2020.109907>.

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