

Recommended practice for calibrating vacuum gauges of the ionization type

Running title: Recommended Practice for Ionization Gauge Calibrations

Running Authors: Fedchak et al.

James A. Fedchak^{a)}, Patrick J. Abbott, Jay H. Hendricks

National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899

Paul C. Arnold

MKS Instruments, 6450 Dry Creek Parkway, Longmont, CO 80503

Neil T. Peacock

Pine Place Consulting, LLC, Broomfield, CO 80020

^{a)} Electronic mail: james.fedchak@nist.gov

This article represents a recommended practice for the calibration of ionization gauges using the comparison method. In this method, ionization gauges are compared to a working standard that has an SI traceable calibration. The ionization gauge is either of the hot-cathode ionization type or the cold-cathode ionization type. Details of the calibration apparatus, the principle of operation of the gauges, data analysis, uncertainty budget, and reporting the uncertainty are given.

I. SUBCOMMITTEE MEMBERS

This article was developed by a subcommittee of the Recommended Practice Committee of the AVS. The subcommittee is made up of users and manufacturers of vacuum gauges who have extensive experience in both the calibration and the practical use of ionization gauges and other high-vacuum gauges. The subcommittee consists of the following past and present members, in alphabetical order: Patrick J. Abbott, *National Institute of Standards and Technology (NIST)*, Paul C. Arnold *MKS Instr.* (formerly at Granville-Phillips Company), Douglass H. Baker *Teledyne*, H. M. Brady (deceased), *Electron Technology*, E. Drubetsky, *Televac*, Robert E. Ellefson, *REVac Consulting*, James A. Fedchak, *NIST*, Jay H. Hendricks (Chair), *NIST*, Neil T. Peacock, *Pine Place Consulting, LLC*, Jacob Ricker *NIST*, Julia Scherschligt, *NIST*, Loralie Smart, *Brookhaven National Laboratory*.

II. DISCLAIMER

This Recommended Practice is based on sources and information believed to be reliable, but the AVS and the authors disclaim any warranty or liability based on or relating to the contents of this Recommended Practice.

Neither the AVS, NIST, nor any of the authors nor their intuitions, endorse any of the products, processes, manufacturers or suppliers named in this article. Nothing in this publication should be interpreted as implying such endorsement. Any mention of commercial products is for information purposes only; it does not imply recommendation or endorsement by the AVS, NIST, or any other institution.

III. INTRODUCTION AND SCOPE

This article describes and recommends best practices for the calibration of ionization gauges over the pressure range of 10^{-7} Pa to 1 Pa. It contains data from many sources and represents the best current practices according to experts in the field. The only calibration method covered in this article is the comparison method, *i.e.*, by comparison of the pressure reading (or another quantity that is a function of the pressure, such as ion current) from an ionization gauge under test to that of a reference standard gauge or working standard gauge within the pressure range of 10^{-7} Pa to 1 Pa. Primary methods, such as the comparison of a gauge reading to a known pressure generated by an orifice-flow standard, are not covered in this article. Ionization gauges must be calibrated as part of an ionization gauge system, which consists of the pressure sensing element, operating electronics, and any connecting cables. In general, any part of the system can affect the calibration, and so replacing part of the system nullifies the calibration. Hereafter, an ionization gauge calibration refers to the entire system. The methods described are suitable for ionization gauges of either hot or the cold cathode type. The reference standard, or working standard, is a previously calibrated capacitance diaphragm gauge (CDG), spinning rotor gauge (SRG) or ionization gauge (IG), depending upon the pressure range over which the calibration will be performed.

It is assumed that the user is already familiar with the basic operation of ionization gauges and high-vacuum technology. Interested readers may consult general references for vacuum technology and vacuum gauge operation, which also cover gauge calibration by primary methods.^{1–6} Note that throughout the remaining article the SI unit

of pressure, Pascal, will be used to describe the vacuum level; $1 \text{ Torr} = 133.322 \text{ Pa} = 1.333 \text{ mbar}$. The terminology used in this article is reviewed in section IV.

Discussions in this article assume that the ionization gauge tube is separate from the ionization gauge controller. Two other types of ionization gauges that are not specifically discussed are gauges with a controller directly connected to the gauge tube, which we will call a “compact gauge”, and the so-called wide-range or combination gauge (or “combined gauge”). The latter is an ionization gauge combined with another gauge capable of reading high pressures, such as a Pirani gauge or quartz oscillator gauge (quartz friction gauge). The former may have a controller separable from the gauge tube (an “integrated type”) or not. Most of the methodologies covered in this article can be applied to either of these gauge styles, provided that the following caveats are considered. First, compact gauges often cannot be baked to temperatures exceeding 80°C without removing the controller head, which is not always possible. Likewise, the maximum temperature of combination gauges often prevents bake-outs. Combination gauges also typically have some type of internal calibration to force the readings of the ionization gauge and high-pressure gauge to match near their cross-over point. Often these gauges have a “calibration” procedure, such as zeroing the high-pressure gauge when the pressure is below the threshold of the high-pressure gauge or setting the value to $1.01 \times 10^5 \text{ Pa}$ when the gauge is open to the local atmosphere (of, presumably, known pressure). Such procedures must be performed before the comparison calibration is performed. This shall be noted and recorded in the report given to the customer.

This recommended practice subscribes to the philosophy that calibrations are performed “as-is”. In other words, the calibrations are performed with the instrument

settings in accordance with the customer's settings. Some settings may be changed during the initial set-up of the instrument, but the setup shall be returned to the customer's expected state before the beginning of the calibration. Regarding the controller settings, much of the discussion in this article is for the edification of the reader, and to recommend the best practice for gauge controller set-up, which may not necessarily be the same that the customer uses. Often calibrations are performed with the system set to two or more different setups (such as two different emission currents or ranges), in accordance with the agreement made with the customer before the calibration is begun.

This article is written for the calibrator of the gauge. The calibrator is providing a service to a customer, which may be internal to the calibrator's organization (indeed, perhaps the calibrator herself or himself). The scope of the calibration service must be discussed with the customer and agreed upon before the calibration begins (such as the pressure range, calibration gas, controller setup). Often the scope is part of a standard calibration service and is not negotiable; in this case the customer must be informed of the conditions of the calibration before work begins. In this work, a calibration refers to determining a calibration factor that is provided to the customer. No internal adjustment of the instrument is performed.

IV. TERMINOLOGY AND DEFINITIONS

The following terms and definitions relate to the calibration of ionization gauges and are used in this recommended practice. They are consistent with general references for terminology relating to vacuum technology and metrology.^{7,8}

- (1) *Primary Standard*. A standard that is designated or widely acknowledged as having the highest metrological qualities and produces a realization of the SI unit for pressure (pascal) without reference to other standards of the same quantity.
- (2) *Transfer Standard*. A standard calibrated with traceability to a primary standard for used in a local calibration application. Examples of transfer standards are calibrated capacitance diaphragm gauges, spinning rotor gauges and hot cathode ionization gauges.
- (3) *Reference Standard*. A measurement standard designated for the calibration of other measurement standards for quantities of a given kind within a given organization. In context of this work, this is very similar to a *transfer standard*, and we tend to use the later term.
- (4) *Working Standard*. A standard used for routine calibrations of other instruments that is traceable to a primary standard. This can be a transfer standard or a gauge calibrated against a transfer standard.
- (5) *Check Standard*. A gauge that may or may not be calibrated, but is used as a consistency check on the calibration system. This is often known as a *control standard*.
- (6) *Stability*. The ability of a measurement system to maintain metrological characteristics over a period of time; *i.e.* the change in calibration factor, between two calibrations.
- (7) *Traceability*. Property of the result of a measurement or the value of a standard whereby it can be related to the SI through documented references, usually

through an unbroken chain of comparisons to national or international standards, all with stated uncertainties.

- (8) *Static Calibration.* A method of producing a calibration pressure such that there is no gas flow while the calibration data point is obtained. Pressures are produced by introducing a quantity of gas into a closed volume to which the gauge to be calibrated and the standard are connected. The static mode is most often used at higher pressures, typically greater than 10^{-2} Pa.
- (9) *Dynamic Calibration.* Stable calibration pressures are generated by directing a constant flow of gas into a vacuum chamber that has a fixed pumping speed. Typically, the pumping speed is fixed by means of a small orifice. The dynamic method is often used to realize pressures over the range of 10^{-7} Pa to 10^{-1} Pa.
- (10) *Units of Pressure.* Pressure readings of ionization gauges are commonly displayed in pascal, Torr, or millibar. The relationships between these units are as follows: 1 Torr = 133.322 Pa and 1 mbar = 100 Pa.
- (11) *Accuracy.* Closeness of the agreement between the result of a measurement and the true value. Accuracy is a qualitative concept and should not be used in place of *uncertainty*.
- (12) *Precision.* The closeness of agreement between independent test results obtained under the same conditions. This differs from *reading precision*, which is the number of digits an instrument can display or record.
- (13) *Repeatability.* Closeness of the agreement between the results of successive measurements of the same quantities and carried out under the same conditions of measurement. This is the same as precision. This is also known as the Type A uncertainty.

- (14) *Reproducibility*. Closeness of the agreement between the results of measurements of same quantities using a different measurement system, location, operator, or other conditions of measurement.
- (15) *Uncertainty*. Parameter characterizing the dispersion of the quantity value being attributed to a measured quantity. In other words, the measurement result is an approximation or estimate of the quantity value. An estimated standard deviation is used to characterize the dispersion.
- (16) *Type A uncertainty*. Measurement uncertainty determined using statistical methods under defined measurement conditions. This can be used to determine the precision or repeatability of a measurement.
- (17) *Type B uncertainty*. All uncertainty components determined by other means.
- (18) *Base Pressure*. The pressure indicated by the gauge with no calibration gas admitted into the calibration chamber. Base pressure indications are typically recorded after a system has been evacuated for several days and is stable, or after the system has been baked. In this condition, the pressure in the system is often called the *background pressure*. Here, background pressure refers to a true pressure, whereas the base pressure refers to the pressure indication by the gauge, which may have an offset. This is not to be confused with *ultimate pressure*, which, in this work, refers to a property of the vacuum pump.
- (19) *UUT*. Unit under test, which is the ionization gauge to be calibrated.
- (20) *Cathode*. Electrode in an ionization gauge which is the source of electrons. This is the filament in a hot-cathode ionization gauge.
- (21) *Anode*. Electrode in an ionization gauge used to detect the electron emission current. It is usually the highest electric potential in the gauge, and must have a higher potential than the cathode. This is the grid in a hot-cathode ionization gauge.

V. THEORY OF IONIZATION GAUGES

Ionization gauges are the most commonly used gauges for measuring pressure below 1×10^{-2} Pa. To date, there are no practical alternatives to ionization gauges for measuring pressures below 10^{-5} Pa. Although numerous gauges have been designed over the last fifty years,⁹ this discussion is limited to gauges that are presently commercially available. Ionization gauges can be placed into one of two broad categories: hot-cathode ionization gauges (HCIG) and cold-cathode ionization gauges (CCIG). Both gauges sense pressure by detecting ions resulting from gas ionized by electron bombardment, but differ in the source of the ionizing electrons, electrode arrangement, and the functional dependence of the vacuum pressure with ion current. These are discussed separately below.

A. *Hot-cathode ionization gauges (HCIGs)*

In the HCIG, electrons are thermionically emitted from a heated cathode element, *i.e.* a hot filament. These electrons are accelerated into an ionization volume, typically in form of a cylindrically shaped anode (the grid). The grid or electron collector (anode) has high transmission efficiency, and, because of this, the electrons may make multiple passes through the volume. Collisions between free electrons and neutral gas molecules within the volume may result in ionizing the gas molecule by, for example, the electron detachment process which produces two free electrons and a positively charged gas molecule. Positive ions formed within the ionization volume are subsequently accelerated to an electrode (the ion collector) held at a lower electrical potential than the grid and cathode. The ion current flowing through the collector is measured using an electrometer and is a measurement of the gas density in the gauge. Under ideal operating conditions,

the ion current is directly proportional to gas density and therefore pressure. An ionization gauge of the Bayard-Alpert geometry¹⁰ is shown in Figure 1.

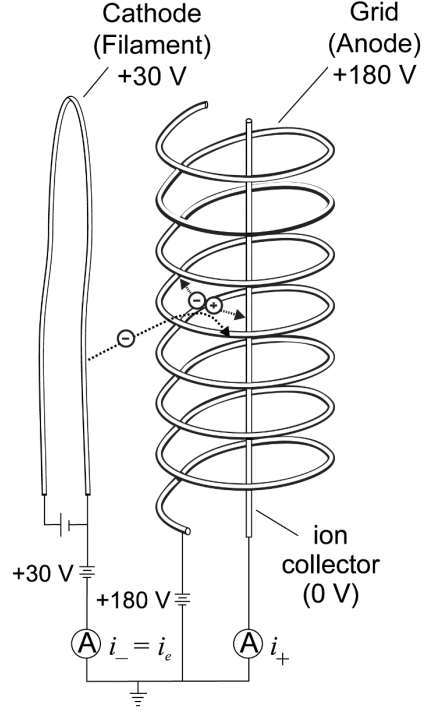


Fig. 1. Diagram of a Bayard-Alpert type HCIG.¹¹ The electron path shown is but one possible trajectory.

As shown in Figure 1, positive ions formed within the anode are accelerated toward the ion collector thus generating an ion current i_+ . In its reduced form,¹² the positive ion collector current is given by

$$i_c = K i_e p. \quad (1)$$

Here i_e is the electron emission current, p is the pressure, and K is the gauge constant, often referred to as the gauge sensitivity (sometimes denoted by S ; here we reserve S for the relative gas sensitivity factor) or gauge sensitivity coefficient, and has the units of inverse pressure. We also note that some authors have an alternative definition of gauge sensitivity in which K is defined as the ratio of the change in ion current to the

corresponding change in pressure.⁴ We discuss a similar concept in section X.A when the calibration correction factor is defined.

The ion current is not a direct measure of mechanical pressure, *i.e.* force per unit area, but is a measurement of gas number density, n_d . Pressure is related to gas density through the equation of state $p = n_d k_B T$ where k_B is the Boltzmann constant and T is the temperature of the gas. Also note the ion current is only a sampling of n_d within the gauge.

The gauge sensitivity strongly depends on the ionization probability of the calibration gas. This depends on gas species and has been determined for many gases.^{13–18} However, the user is cautioned that the relative gas sensitivity factor also depends on the gauge geometry and applies only to the particular gauge from which they were determined. Typically, K is expressed in terms of its relative sensitivity to N_2 : $K = SK_{N_2}$, where $S \equiv K/K_{N_2}$. Some controllers come with fixed values for K_{N_2} , other are more flexible and allow users to enter a value. Typically, K_{N_2} is chosen to represent a nominal value that is typical for gauges of a particular type. The “true” value of K , *i.e.* the value that yields a true measurement of pressure, not only depends on the gas composition, but on the temperature of the gas being measured, the probability of ionization (electron detachment cross section), the electron energy, the interaction length between the emitted electrons and the gas, which is determined by gauge geometry, and the collection efficiency of the ions. In addition, the measured ion current is also subject to errors and may not represent the true ion current in the collector. Inconsistent manufacturing may lead to variations among a single gauge type. Mechanical instabilities, electrical

instabilities, and changes in the surfaces can all lead to changes in gauge sensitivity over time. Hence the need for an additional correction factor.

The sensitivity factor is also weakly dependent on the ambient temperature, which, for most vacuum systems, is the same as the gas temperature. This is due to the large temperature difference between the ionization gauge tube and the vacuum chamber.^{19, 20} The temperature dependence of K has been observed to be 0.3% per degree Kelvin for several common varieties of HCIGs.¹⁹

Accurate measurement of the true ion current at the collector is critical to obtaining accurate pressure indications. The total current measured at the ion collector can include contributions from spurious sources, such as the x-ray effect and electron stimulated desorption (ESD). These effects are particularly important at pressures below 10^{-5} Pa.²¹

The most common HCIG is the Bayard-Alpert type. There are other commercially available HCIG geometries, including the older ‘triode’ geometry,²² the extractor gauge,²³ the bent beam gauge,²⁴ and the Bessel box or axial transmission gauge.^{25,26} In the extractor and Bessel box gauges, the collector is placed outside of the ionization region, so that the measured x-ray contributions are decreased. The Bessel box gauge also has an electrostatic energy analyzer that separates the ions produced from ESD from those produced in ionizing collisions.

B. Cold-cathode ionization gauges (CCIGs)

Similar to HCIGs, CCIGs indirectly determine the pressure by inferring the gas density from an ion current. In these gauges, the pressure is determined from the current of a self-sustaining gas discharge.^{27,28,29} The discharge is produced by a high voltage

between two electrodes in a magnetic field. The electrons forming the discharge arise from ionization of gas molecules in the ionization volume as well as secondary electrons created from collisions of positive ions with the cathode. The resulting current (i_{CC}) is a function of the pressure,

$$i_{CC} = K'p^n, \quad (2)$$

where n and K' are constants for a given gauge. Typical values of n found in the literature fall between 1.0 and 1.2 for a nitrogen pressure of 10^{-4} Pa.³⁰ One of the interesting properties of CCIGs is the long electron path length that can be produced, especially at ultra-high vacuum. In uniform crossed electric and magnetic fields, charged particles move in cycloidal paths. The discharge is sustained when the energy of the electron is sufficient to ionize gas molecules in the ionization volume.

Starting the gauge at low pressures may take several minutes because the discharge is initiated by some random event which generates an electron, such as a field emission or a cosmic ray. The ignition or “striking time” of a CCIG has been studied extensively,³¹ and solutions have been proposed for shortening the starting time at low pressures. Starting times are also statistical in nature, so these will vary, even for a single gauge.

There are three common geometries for CCIGs; Penning³², magnetron²⁷ and inverted magnetron³³ (See Figure 2). The anode in the Penning gauge consists of a wire loop with the cathode(s) surrounding the loop, the magnetron has a cylindrical anode surrounding the cathode, and the inverted magnetron has a single wire anode surrounded by a coaxial cylindrical cathode. In the figure, B is the magnetic field, + designates the gauge anode, and – designates the cathode. Note that the magnetron has the cathode in

the center while the inverted magnetron has the anode in the center. The electrode design of modern compact CCIGs typically use the inverted magnetron configuration.³⁴

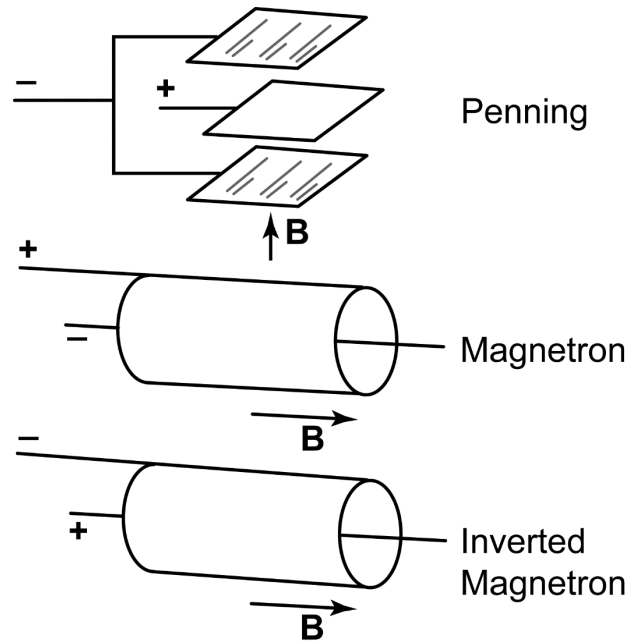


Fig. 2 Diagram of typical cold-cathode electrode geometries.

VI. CALIBRATION APPARATUS

A typical calibration apparatus is shown in Figure 3. There are three main components: a working standard (WS), a calibration vacuum system (3(a)) and the gas admission system (3(b)). The calibration vacuum system is comprised of calibration chamber to which the working standard(s) and units-under-test (UUTs) are connected, and a pumping system. The pumping system is typically comprised of a high-vacuum pump and fore-pump or rough pump, and pressure gauges to monitor pressure at various points in the system. The working standard may be a transfer standard or a gauge calibrated against a transfer standard. More than one working standard may be used, either to cover a range of pressures or to provide redundancy. The gas admission system

provides a calibration gas to set the gauge pressure. These three components will now be covered in detail in the following subsections.

A. Working standard

The working standard must be suitable for use in the range of pressures over which the ionization gauge is to be calibrated. Calibrated capacitance diaphragm gauges are recommended as a working standard for pressures greater than 10^{-2} Pa, spinning rotor gauges are recommended in the pressure range of 10^{-1} Pa to 10^{-4} Pa, and ionization gauges for pressures below 10^{-4} Pa. The working standard must have a calibration traceable to the SI (*i.e.*, documented calibration traceability to an a primary standard operated by an accredited institute, such as a national metrology institute, *e.g.* NIST) over the pressure range of interest, including a clearly stated estimate of the uncertainty of the calibration results. The working standard should be operated under the same conditions as it was calibrated, often including the same controller and cabling, with which it was calibrated. Table 1 gives a list of suitable working standards and their associated uncertainties for transfer standards calibrated at NIST. U_{Total} is the total relative expanded uncertainty (*i.e.* a coverage factor of $k = 2$, or a confidence level of 95 %) of the transfer standard gauge reading. It is given by $U_{\text{Total}} = \sqrt{U_{\text{Cal}}^2 + U_{\text{LTS}}^2}$, where U_{Cal} is the uncertainty determined at the calibration facility at the time of the calibration, and U_{LTS} is the uncertainty due to long-term instability of the transfer standard. Gauges calibrated at other facilities or made by other manufacturers will have a different value for U_{Total} . The values of U_{LTS} given in Table 1 are typical for the gauge type listed in the table; in general, the best practice is to use stability data for the particular gauge used as the working standard.

The gauge models and manufacturers listed in Table 1 are not the only gauges suitable as working standards or transfer standards. Those in Table 1 are gauges that NIST has had enough repeat calibration to estimate U_{LTS} and, in some cases, have had these results published (references given in the table).

It is worth taking a moment here to consider what makes a HCIG of metrological quality. First and foremost, it must have excellent long-term stability in regards to the sensitivity coefficient or calibration factor. The gauge sensitivity (gauge constant) depends on the electron trajectory which is highly affected by the location of the surrounding envelope (if it exists), cathodes, anode, grid posts, and ion collector.^{35,36} These must be mechanically stable. The controller must also be electrically stable such that a consistent voltage is applied to these electrodes. It is best if the filaments are mounted in tension to minimize twisting or sagging as the filaments age. Coated filaments, such as those coated by thorium or yttrium, are generally preferred because these are less prone to burn-out and operate at a lower temperature than tungsten filaments. Another consideration is the desired accuracy. Controllers which give readings to one decimal place, for example, are limited to an accuracy of 10% at the lowest pressure reading of each decade scale. Glass BAGs (Bayard-Alpert Ionization Gauges) were the dominant variety of HCIGs more than two decades ago, but are less common now. These may be used as a transfer standard, but selecting a suitable glass BAG is difficult because a wide variety of models and options are available. We generally recommend gauges enclosed in a metal envelope because the electric potential surrounding the gauges is well-defined, and the metal envelope offers some protection to the electrodes during transfer. There is an abundance of stability data available in the literature, too much to

review here, and one must be careful because many older reports concern gauges no longer commercially available. We suggest the relatively recent studies by various National Metrology Institutes as excellent resources for presently available gauges of high metrological quality.^{37–40}

TABLE I. Typical uncertainties for metrological quality gauges calibrated at NIST. All of the uncertainty data comes from NIST’s calibration data base and from the referenced published data.

	Calibration Pressure (Pa)	$U_{\text{Cal}}(k = 2)$	$U_{\text{LTS}}(k = 2)$	$U_{\text{Total}}(k = 2)$	References
		Percent			
0.13	MKS Baratron CDG Full Scale 133 Pa (0.13 Pa to 133 Pa)	0.7	> 0.7	> 0.7	41
	MKS Baratron CDG Full Scale 13 Pa (0.013 Pa to 13.3 Pa)	6.0	> 0.7	> 6.0	41
	0.013	0.6	0.7	0.9	41
0.13		0.3	0.7	0.8	41
1.3					
	SRG				
10 ⁻⁴ to 10 ⁻¹		0.3	2.0	2.0	42,43
	Stabil-Ion ⁴⁴ Ionization Gauge				
10 ⁻⁷ to 10 ⁻²		1.3 to 0.4	3.8	4.0 to 3.8	37

B. Calibration vacuum system

Two basic techniques may be used to set the calibration pressure in the calibration chamber: A static method, or a dynamic method. The schematic chamber of Figure 3 allows both techniques to be used. For the static technique valves V2 and V3 are fully closed and gas is admitted into the chamber until the desired pressure is obtained, as indicated by the working standard, then V4 is closed. The optional orifice is not used. In the dynamic expansion technique, gas is continually flowed into the chamber, and either

valve V2 must be a throttling valve or the optional orifice must be used to restrict the gas flow to the high-vacuum pump. The chamber pressure p is determined by

$$p = \frac{q_{pV}}{S_{\text{eff}}} , \quad (2)$$

where S_{eff} is the effective pumping speed of the orifice or throttle valve in combination with the vacuum pump, and q_{pV} is the flow throughput into the chamber. The dynamic expansion has disadvantages in that pressure gradients are a possible source of uncertainty and it is a more complicated to operate than the static system, but the static system has the disadvantage that both gauge chamber outgassing will cause a pressure rise in the system that must be taken into account. This is a serious concern at low pressures and for chambers with small volumes.

For high-vacuum calibrations, calibration chambers are typically constructed of stainless steel with flanges and fittings sealed with metal gaskets. Chambers made of aluminum, titanium, or other low-outgassing materials may also be suitable for high-vacuum calibrations. When using aluminum or titanium chambers, follow the manufacturer's instructions regarding seals. There are several different technologies available for aluminum all-metal seals, choose the gasket material appropriate for the seal technology. Most ionization gauges are fitted with stainless steel flanges. Models with ranges extending below 10^{-6} Pa are typically available with DN40 fittings (*i.e.* metal-gasket sealed 2.75" CF (knife-edge fitting). KF type flanges (quick release , typically elastomer-sealed) are often available for ionization gauges intended for operation above 10^{-6} Pa only. High-quality elastomer seals, *i.e.* those with low outgassing rates and low

permeability of atmospheric gases such as those made of Viton or Kalrez, may be used in circumstances where higher base pressures ($>10^{-6}$ Pa) are acceptable.

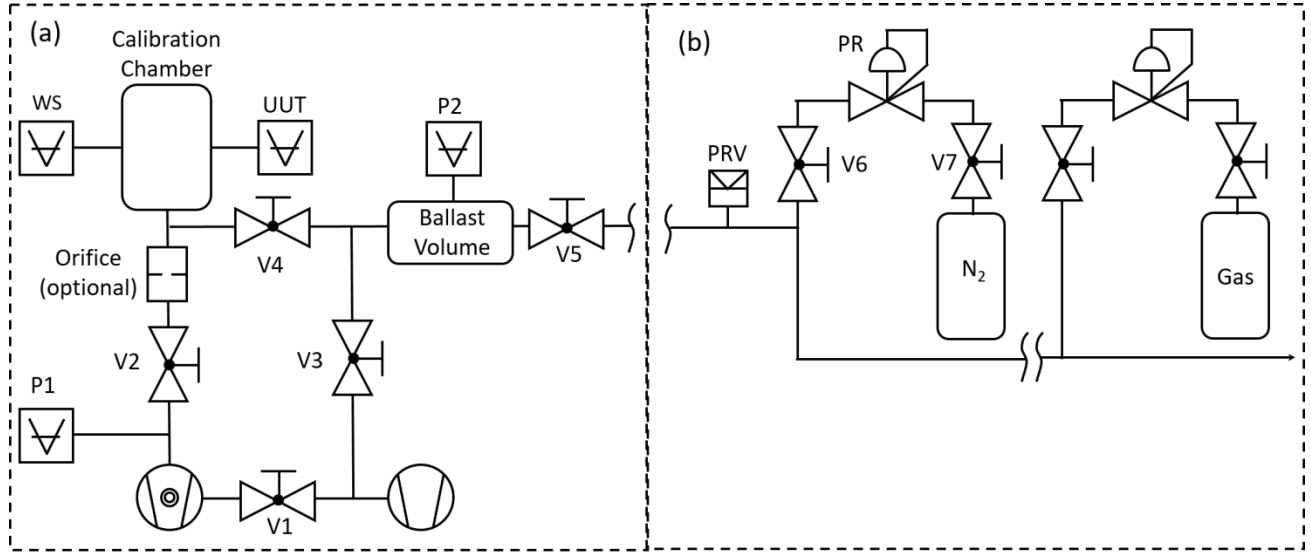


Fig. 3. Calibration vacuum system (a) and gas admission system (b) for the calibration of ionization gauges using the comparison method. The unit under test (UUT) is compared to the working standard (WS); P1- gauge to read inlet pressure of the high vacuum pump; P2 - gauge to read buffer volume pressure (optional); V1—V7 are valves; V4 is a leak valve; PRV is a pressure relief valve; PR is a pressure regulator.

The following recommendations are given for the calibration system design and operation:

1. *Temperature and environmental conditions*

The calibration system for gauge calibration should be in a temperature controlled environment. As pointed out in Section V.a, ionization gauge sensitivity depends on temperature. Because the calibration uncertainty of ionization gauges will be several percent in most cases, a temperature stability of about 1 °C, i.e. ± 0.5 °C over the period of calibration will be sufficient in most applications.

The calibration vessel should be placed in areas away from significant vibration sources. Vibrations can potentially affect gauge performance. SRGs are particularly susceptible to vibration noise. Vibration checks can be made by turning off potential sources of vibration and monitoring the stability of gauge readings.

2. *Pumping system*

Throughput-type pumps, such as a turbomolecular pumps, are recommended for the high vacuum pump. These pumps have high pumping speeds and can pump all gases without long-term changes in pumping speed. Caution is advised before using capture pumps, such as ion pumps and some cryopumps, as these may not be suitable for pumping high gas throughput calibration chambers. Cryopumps offer high pumping speeds for most gases, but can only pump a finite amount of gas before regeneration is required. Properly sized cryopumps can be used. Ion pumps do not, in general, have a constant pumping speed, which can complicate the operation of a dynamic expansion system. Their pumping speed is highly gas dependent and most do not pump noble gases well, but they can produce a low ultimate pressure, and can be used in combination with a throughput pump to produce a low ultimate pressure system with high throughput. The use of liquid-nitrogen-trapped diffusion pumps is discouraged because of the potential for contamination.

Dry pumps, such as scroll pumps or diaphragm pumps, are ideal for using as forepumps or rough pumps. These will minimize hydrocarbon contamination as compared to, for example, oil-sealed pumps. If oil-sealed forepumps and rough pumps are used, an oil trap in the foreline is recommended.

3. *Construction techniques and materials*

The system should be designed and constructed using materials, seals, and cleaning techniques appropriate for high and ultra-high vacuum systems.⁴⁵ It must be clean and leak-tight, and capable of attaining a base pressure at least one decade (preferable two) lower than the pressure at which the gauge calibration begins. For example, to calibrate at a pressure of 5×10^{-5} Pa, the base pressure should be below 5×10^{-6} Pa. Most vacuum systems cannot obtain pressures below the 10^{-6} Pa range within a few days of operation. Baking the test chamber between 100 °C and 150 °C is necessary to achieve base pressures lower than 10^{-6} Pa. Care must be taken not to exceed the maximum temperature of the vacuum pumps and/or elastomer seals, consult the manufacturer's specifications. For calibrations requiring these low base pressures, the entire vacuum system must be constructed using metal seals. Elastomer seals are acceptable for systems requiring base pressures above 1×10^{-6} Pa, but their use should be minimized to reduce outgassing and permeation, both of which will limit the ultimate pressure of the system.

4. *Gauge ports*

Gauges measure the pressure inside themselves. Ionization gauges tend to outgas, which can cause a pressure rise within the gauge if the conductance is restricted. The port diameter should have a diameter that meets or exceeds that of the gauge. In most cases, a DN 40 flange is sufficient. The use of valves or other components between the chamber and any ionization gauge is discouraged. Extra components can affect conductance and can outgas, causing locally high pressures. If valves are used, they should be all metal and have a large conductance. Any ionization gauge should be mounted to avoid line-of-sight charged particle sources, such as another ionization gauge or a mass spectrometer.

5. *Static systems*

For static calibration systems, the calibration chamber ideally should have a shape that minimizes the internal surface area to volume ratio. Spherical chambers have the smallest area to volume ratio, but cylindrical chambers with a length that is equal to the diameter are a very close approximation. Both chamber and gauge outgassing can be a problem in static systems because it causes a steady pressure rise. Ionization gauge outgassing rates have been measured to be in the range of $1 \times 10^{-6} \text{ Pa L s}^{-1}$ to $1 \times 10^{-8} \text{ Pa L s}^{-1}$.^{39,46,47} Chamber volumes must be large enough to minimize the pressure rise due to outgassing. For example, if the lowest calibration pressure is 10^{-4} Pa , then the vacuum chamber must be greater than 30 L to insure the ionization gauge reading is stable to better than 10% over 5 minutes. In most cases the static method is not practical for calibrations below 10^{-4} Pa .

6. *Dynamic flow method*

Pressure gradients can be a concern in the dynamic flow method. Ideally, the gauges under calibration and the working standard gauge should be mounted in the same plane, transverse to the gas flow, to minimize possible pressure gradients between the standard and UUT. The gas inlet location should be such that the calibration gas undergoes many wall collisions before exiting the chamber. This is to achieve thermal equilibrium and prevent pressure gradients. To achieve this, it may be necessary to place a baffle in the chamber where the flow of gas enters.

An excellent way to account for the uncertainty of pressure gradients is to perform a Monte Carlo analysis of the calibration chamber. Alternatively, the pressure at the various ports can be measured using the same calibrated pressure gauge, making sure that the first and last location of the gauge is at the same port to account for any drifts in

the gauge sensitivity. Such a measurement of the pressure gradient only needs to be done once to characterize the pressure gradient in the system and the associated uncertainty, and only needs to be repeated if a relevant change is made to the system (such as, for example, replacing an orifice or changing a baffle). Pressure gradients may also be minimized by design. In addition to the recommendations given above, one way to insure minimal pressure gradients is to use a sufficiently small conductance limiter located between the chamber and the vacuum pump, as shown in Figure 3. If an orifice is used, for example, the ratio of the area of the orifice to the surface area of the largest sphere that may be enclosed by the vacuum chamber is an indication of pressure consistency. A ratio of 1000 or greater will effectively eliminate relevant pressure gradients. Dynamic flow chambers with ratios smaller than 1000 may also be used with success, but the uncertainty due to potential pressure gradients must be carefully characterized.

An high-vacuum pressure gauge, P1, may be required between the valve and pump to insure that maximum pump inlet pressure is not exceeded.

7. *Partial pressure analyzers*

Residual gas analyzers (RGAs), also known as quadrupole mass spectrometers, are useful for monitoring the test gas purity, and performing leak detection. Leak detection is often performed by spraying He gas near seals, welds, and likely leaking spots, and looking for a He signal to appear on an RGA. Most units have a “leak check” mode where He, relative atomic mass 4, is continuously monitored. The mass spectrum can also be an indication of leaks. Mass spectrometer signals in unbaked systems are typically dominated by relative molecular mass 18 (H₂O); a dominant relative molecular mass 28 peak (N₂ or CO) together with a relative molecular mass 32 peak (O₂) is a strong indication of an air leak.

Baked systems should have a dominant H₂ peak (relative molecular mass of 2), with very small or negligible signals at relative molecular masses 18 and 28. RGAs usually require high-vacuum for operation; they cannot be used to detect “gross” leaks. However, small leaks can adversely affect the calibration of ionization gauges, making the use of RGAs as leak detectors worth consideration.

RGAs should never be used as a working standard. Their calibration highly depends on the various settings of the device, they have poor long-term stability, and can be highly non-linear.

C. Calibration gas admission system

The gas admission system provides either a continuous gas flow (dynamic flow method) or periodic gas flow (static method) to obtain desired set pressures. It is also needed for venting the system to atmospheric pressure when installing or removing gauges. Venting the system with N₂ or Ar is recommended. This reduces the time that the vacuum chamber is exposed to ambient laboratory air and consequently water, thus minimizing water adsorption on the chamber walls and providing a faster “turn-around” time for the vacuum system. Furthermore, venting with clean gas reduces the amount of dust and other contaminants entering the vacuum system. This is further accomplished by establishing a positive flow of calibration gas such as dry nitrogen when the system is open. A separate gas admission system designed for venting is necessary in cases where the gas admission system is not capable of producing a high enough flow to vent the system in a reasonable time, or if the admission system is not designed for inert gas operation. Figure 3(b) depicts a typical gas admission apparatus. A pressure regulator (PR) must be used to limit the maximum pressure entering gas lines. Choose a regulator specified for vacuum on the low-pressure side. In

most cases, the pressure regulator must limit the pressure to less than 1 atmosphere gauge pressure. It is recommended that a burst disc be used as a fail-safe for over-pressurization.

Because the sensitivity and calibration factor of an ionization gauge depends upon the gas being measured, the apparatus must maintain the purity of the calibration gases.

Therefore, the gas admission system must provide pure test gas with the partial pressure of impurities low enough to avoid errors. The following are recommended to maintain purity:

- (1) The chamber must be evacuated to a base pressure of at least ten times below the lowest desired calibration pressure before admitting the calibration gas and beginning a calibration;
- (2) The gas admission system must be capable of evacuating the gas lines that run from the chamber to the regulator on the high-pressure gas cylinder to at least a rough vacuum. This should be done before a calibration is performed;
- (3) The calibration gas should be at least 99.95% pure;
- (4) Metal gas lines (preferably stainless steel or copper), metal seals and metal sealed valves should be used.

In addition, we consider the following features to be essential to the gas admission system:

- (5) Valve V4 (Figure 3), used for gas admission, must be capable of fine control. Flow adjustment from 10^{-9} Pa-m³/s to 10^3 Pa-m³/s (10^{-8} atm-cm³/s to 10^{-4} atm-cm³/s) is desirable. This can be accomplished with a wide-range variable leak valve, or multiple valves and a volume in series.
- (6) A pressure relief valve or burst disc is recommended to prevent the manifold pressure from rising to dangerous values in event of gas cylinder regulator failure.
- (7) If calibrating with toxic gases, an appropriate venting system is required.

- (8) A porous metal filter should be used after the cylinder pressure regulator to prevent particulates from damaging the flow adjustment valves,
- (9) The fill indicator gauge on the buffer volume must be gas species independent, such as a Bourdon tube gauge or CDG.
- (10) If different calibration gases are to be used, an interlocking system should be utilized to prevent cross-contamination of gases.

A quadrupole mass spectrometer attached to the calibrating vacuum system, discussed in Section VI.B.7, may be used to monitor gas purity.

VII. PREPARATION OF IONIZATION GAUGES FOR CALIBRATION

A visual inspection of the gauge system to be calibrated should be performed prior to installation. This includes the gauge tube, gauge controller, and any interconnecting cables). Basic checks of the system are performed prior to calibration to assure the system is working properly.

A. Gauge tube or sensor preparation and checks

The inside of the gauge tube and its sealing flange should be clean according to ultrahigh vacuum practices. Gauge tubes with visible contamination or excessively dirty internal electrodes are best replaced; the long-term stability of the sensitivity may be compromised. It is not recommended that the internal gauge structures be exposed to cleaning solvents due to the possibility of damaging the electrodes. The sealing region (knife-edge sealing flanges are assumed) should be free from scratches, or other damage that may prevent a proper seal. A leak test prior to operation of the gauge is good practice.

The gauge electrodes should be inspected for damage, deformity, and any sign of mechanical instability. Filaments in HCIGs should be checked for electrical continuity. Filament coatings should be intact and uniform in appearance. A check that the individual electrodes are electrically isolated (i.e., at least $10^{10} \Omega$) from one another and from ground (chamber potential) should be made, as any short will result in faulty operation.

It is good practice to clean external contacts or pins with a fine abrasive cloth to eliminate high resistance contacts. Any bent connections or pins should be carefully straightened with a pair of long-nose pliers. To prevent stress on the vacuum feedthrough, use a second pair of pliers to hold the base of the pin. Leak check gauge pin feedthroughs if it is suspected that they have been subject to stress from bending.

Clean UHV installation techniques must be used. The gauge tube should be mounted to the vacuum chamber according to the recommendations of the manufacturer. In general, all sealing flanges should be vacuum clean and free of lint and other contaminants. For all-metal seals, new UHV cleaned metal gaskets must be used.

Consideration should be given to mechanical and electrical safety, e.g., mounting a glass enveloped tube to minimize the chance of glass breakage and implosion. Glass tubes fitted with metal seals are recommended over those requiring a compression fitting. If the latter is used, it is particularly important that compression mounts be tight on glass gauges. Cables that connect the gauge tube to the gauge controller should be strain-relieved. Since potentials of up to 180 V (500 V during electron bombardment degas) are present, it is recommended that appropriate warnings be posted to protect laboratory personnel.

Nude gauge sensitivity is profoundly affected by the mounting configuration. It is strongly recommended that nude gauges (i.e., a gauge without an envelope or enclosure around the electrodes) be mounted inside of a stainless-steel nipple that is long enough to cover the entire electrode structure. The gauge should then always be used with this nipple to maintain a consistent electrical boundary.^{48,49}

The orientation of the gauge during calibration should be the same as the orientation during use. The preferred mounting orientation for a Bayard-Alpert HCIG is with the collector axis vertical, to avoid shorting the filament to the grid if the filament sags during use. HCIGs are best mounted so that there is free air circulation around the tube, and with no excessive heat sources or stray magnetic fields nearby. When mounting multiple gauges, distribute them uniformly about the chamber to minimize thermal gradients. When attaching cables to the gauge and controller, insure that no undue strain is placed on the electrode feedthroughs. Cables should also be routed so that they do not pick up electrical noise or interference.

CCIGs must be located so that stray magnetic fields will not interfere with other equipment. Likewise, CCIGs must not be mounted in large external magnetic fields, such as near a large ion pump, since these fields can cause calibration shifts. Most tools are ferromagnetic and are strongly attracted to magnets of CCIGs, therefore the use of non-magnetic tools is recommended when working near the CCIG magnet.

B. Controller preparation and checks

Inspect the controller for any damage, including to the chassis, controls, display, and connectors. Damage to the chassis may indicate rough treatment and potential problems. Check that the power cable is free from cracks along its insulation, and rated to

meet the power requirements of the controller. Inspect all cables connecting the gauge tube and controllers for damage, including corrosion to any connecting pins or sockets. A good cable insulation is free from cracks. Note the condition of the controller and cables, report damage to the customer.

After the system is under vacuum and the gauge is operating, check the zero of the controller. This can be done by disconnecting the ion collector current lead at the controller (if possible). If the controller has multiple electrometer ranges, check the zero on each range.

Carefully note the setup of the controller after it is powered on. Some controllers have a plethora of settings and information that may be read from the front panel, others do not. Most HCIG controllers will allow the user to choose between two or more emission currents (often referred to as “ranges”). As discussed below in section VIII.a, it is best to select the lowest emission current before gauge ignition. However, the gauge calibration will depend on the emission current, and so this will need to be set to the desired range before the calibration is begun. Other possible settings to note are the electrode potentials, gauge sensitivity, relative gas sensitivity scale factor, and factory adjusted calibration factors. Many controllers do not have the latter, and for some controllers the latter is combined with the gauge sensitivity factor and is not accessible to the user, for others gauge-specific factory calibrations are contained in memory modules attached to the controller. If these have been loaded into the controller, it is important to note the model and serial number of the module. Relevant gauge parameters are discussed in Section VIII.d.

VIII. OPERATION OF IONIZATION GAUGES

A. *Leak test*

A leak test of gauge(s) and vacuum connections with a mass spectrometer leak detector or an RGA is advisable. If using an RGA, this may not be possible. Most RGAs require pressures to be below 10^{-3} Pa before operation. To check that the proper pressure has been reached, at least one ionization gauge must be ignited. Further details of using an RGA as a leak checker are discussed in Section VI.B.4. Portable leak checkers may be attached to the calibration chamber before the ionization gauges are operated, which is the most ideal time to leak check.

B. *Gauge operation*

Ionization gauges may be operated after the calibration system has been pumped down to a pressure below the maximum pressure of the gauge as given by the manufacturer, typically 10^{-2} Pa or lower. This pressure could be determined by another type of gauge, such as a CDG or thermocouple gauge. Otherwise, if the system pumps are fully operational (turbomolecular pumps at full speed or cryopumps at operational temperature, for example), most systems will realize this pressure within the first hour of operation. For most HCIG controllers, the ionization gauge filament is not ignited upon power-up, although the electrode potentials are often automatically applied. This allows the user to check the setup before operating the filament. It is best to set the emission current to the lowest value possible before filament ignition. Afterwards, the emission can be set to the value desired for the calibration, provided the system has reached a pressure below that recommended for the specific current. Usually the manufacturer provides this information. Otherwise, we generally recommend that emission currents

much less than 1 mA be used above 10^{-4} Pa and an emission current in the range of 4 mA to 10 mA be used below 10^{-5} Pa.

C. *System bake*

A bake of the calibration system to at least 100 °C is required to obtain the system base pressures required for calibrations below 1×10^{-6} Pa. Such bakes remove water and other condensable material from the surfaces within the vacuum system. Consult the manufacturer's information for bake preparations and temperature limitations on gauges and other vacuum components. Typically, more than 24 hours of baking is required; higher temperatures allow shorter bake times. In general, we recommend a bake in the range of 125 °C to 150 °C for a minimum of three days. Bake temperatures exceeding 200 °C are not recommended.

Many HCIGs have more than one filament. The calibration will depend upon the filament that is used. All filaments to be calibrated should be operated prior to the bake. This aids in degassing the filament and provides a check that the filaments and gauge function properly. We recommend operating the filaments for at least an hour before baking, but a full day of operation is preferred.

Whether it is necessary to operate the gauges during a bake is a matter of some debate. The main concern is that an operating filament is much hotter than 150 °C and may produce significant off-gassing when ignited, even if it has been previously baked. This may increase the vacuum pressure, perhaps, in some cases, more in the gauge than in the entire system; in addition, it is possible that ESD ions result from the off-gassed products, also causing a high pressure reading in the gauge. The latter effect is typically only relevant at ultra-high vacuum pressures. We recommend operating ionization gauges

during bakes if the calibration pressures are to be $\leq 1 \times 10^{-6}$ Pa. Otherwise, it is optional, depending on the experience of the user; *i.e.*, if the user finds that their system does or does not obtain the desired base pressure. Many gauge manufacturers offer gauges and cables that can be baked while operational. Alternatively, bake-out cables can be fabricated from wire rated for high-temperature and crimp-on socket and pin connectors. The wire must also be rated for currents of at least 3 A.

D. Operating parameters

The gauge operating parameters are essential to the calibration report (see section XI). Parameters relevant to the calibration of the gauge are discussed in this section. These parameters should be measured or read from the controller before calibration begins. Some parameters are settable, depending on the controller. The gauge parameters shall be set to those used by the customer. Where applicable, we make recommendations below for the operating parameters; however, the customer's practice takes precedence over these recommendations.

1. Hot-cathode ionization gauges (HCIGs)

A. Electrode potentials

The Bayard-Alpert type HCIG has three electrodes: The filament or cathode, the grid or anode, and the ion collector. Other types of hot cathode gauges, such as the extractor gauge or Bessel box gauge, have other electrodes in addition to these. The various electrodes operate at different potentials which are automatically set by most modern controllers. Typical values (referenced to ground potential) are shown in Table

2. Modern controllers typically regulate these potentials to better than 1%. A 1.0 %

uncertainty in each electrode potential, for example, contributes about 0.25 % uncertainty in the ion current.⁵⁰ Some glass Bayard-Alpert type gauges have a metal coating that can function as a fourth electrode that should be maintained at a defined potential which is consistent between calibration and use. In addition, most modern controllers use DC for the cathode potential, but many older units still in operation use AC.

TABLE II. Typical values for electrode potentials in Bayard-Alpert ionization gauges.

Electrode	Potential (volts)
Cathode (Filament)	30
Anode (Grid)	180
Ion Collector	0

Consult the manufacturer’s literature for correct values and methods to measure and verify the potentials. Electrode potentials which are out of tolerance may indicate a problem with the controller or gauge, and it is best practice to repair or replace malfunctioning controllers. Gauge tubes can be checked for continuity as discussed in section VII.a. However, we recognize that electrode potentials and tolerances are often not available. It is ultimately the stability of these values over the course of the calibration and the stability compared to the previous calibrations (if any) that is the most important. We recommend an electrode stability of 1% or less for calibrated gauges. Checking the values of the electrode potentials at the beginning and end of the calibration is recommended. We also recognize that many controllers do not indicate the values of the electrode potentials, and the manufacturer may not give a procedure to measure these. In this case, we do not recommend measuring the electrode potentials. On some gauges, for example, it is possible to measure the electrode potentials at the gauge connection

using a voltmeter. However, shorting the electrode during such a check can damage the controller. Custom built “break-out” boxes may also be used to measure the electrode potentials, and these are discussed in the section below. As indicated in that discussion, we do not generally recommend the use of these for comparison calibrations.

B. Emission current

Ionization gauge controllers regulate the emission current, which is the thermionically emitted electrons from the hot cathode that ionizes the gas in the gauge. Many controllers allow the emission current to be set by the user to values ranging from less than a tenth of a milliamperere to several milliamperes.

In general, the best emission current value depends on the pressure being measured, which is typically given by the manufacturer of the gauge. Some guidelines are given in Table III.

TABLE III. Recommended emission current, i_e , setting for Bayard-Alpert ionization gauges

Pressure (Pa)	Emission Current Range (mA)
$< 1 \times 10^{-6}$	$1 \leq i_e \leq 10$
$\geq 1 \times 10^{-6}$	$0.1 \leq i_e \leq 1.0$

Emission currents are usually increased at low pressure to increase the ion current at the collector and to reduce the effects of ion created by electron stimulated desorption (ESD).¹⁵ Higher emission currents serve to reduce the gas desorbed on the filament and grid, thereby reducing the ESD effect. On the other hand, the sensitivity of many gauges can be more consistent at reduced emission currents.⁵¹ Therefore, low emission currents can have an advantage at higher pressures, where there is ample ion current. In many

controllers, the emission current is set by the user and is constant, however, some controllers set this automatically and will vary the emission current according to the pressure range.

Both emission current and electrode potential can be measured by use of a break-out box. This is an electrical device that allows the electrode potentials to be probed by a voltmeter, and is connected to the controller where the ionization gauge cable would normally be attached; the ionization gauge cable is attached to the break-out box. The emission current is determined by placing a 1 k Ω (0.25 watt) resistor in series with the anode (grid) connection. An emission current of 1 mA will produce a 1 V potential drop across the resistor. It can be helpful to verify that there is no emission current (current in the grid supply) when the filament is turned off, but the grid remains powered. A current observed in this situation indicates a problem with leakage currents. Note that any error in the emission current will cause the same error in the ion current, which is why it is best that the emission current is regulated better than 1%. Break-out boxes are often used in calibrations by National Metrology Institutes (NMIs), and have been used in international intercomparisons among NMIs.^{52,53} During intercomparisons, calibration factors for the same gauge determined by various NMIs are compared, and so the electrode potentials and emission current help to assess whether the gauge was in the same condition at each NMI. However, we do not generally recommend the use of break-out boxes because these must be custom constructed, the user may be exposed to high voltages, and the information obtained may not be that useful to many users. These are mentioned here because they are useful in intercomparisons, and some NMIs use these devices in their calibrations.

C. Sensitivity

Most controllers allow the user to program a relative gas sensitivity factor S , such as N_2 or air, for the gauge tube in use. For example, the relative gas sensitivity factor is defined to be 1 for N_2 . Some controllers allow the user to set a gauge constant K , with typical values for N_2 ranging from 0.1 Pa^{-1} to 0.5 Pa^{-1} . Consult the tube manufacturer's literature for this parameter, and verify the controller's setting. Note that it is critical to record the relative gas sensitivity factor (and K , if settable) used during the calibration. Once calibrated at a specific gas sensitivity factor and gauge constant, the obtained calibration factor cannot be used for any other value of S or K .

D. Pressure units

Many controllers have user selectable units (e.g., Pa, mbar, or Torr). The pressure unit setting shall be recorded and used consistently throughout the calibration.

E. Degassing

HCIGs should be degassed prior to a calibration run. Degassing is an operation by which the grid is heated to remove adsorbed gas. Gas desorbed from the gauge structure can cause large errors in pressure readings.⁹ Controllers employ one of two methods to degas the gauge: The grid is either resistively heated by passing a current through the grid, or by electron bombardment, where the grid is heated by impacting electrons originating from the filament which is passing high current.

Baking a gauge is not the same as degassing, which is a procedure initiated by the controller. During degassing, the temperature of the grid can reach 700°C , which is much higher than typical chamber bake temperatures. Even if the gauge has been baked, degassing is still necessary. The typical degassing time recommended by gauge

manufacturers is 10 minutes at 40 watts. Degassing should always be done in accordance with manufacturer's recommendations.

HCIGs can be damaged by being degassed at too high a pressure. Most controllers will shut off the degas if the pressure exceeds a safety threshold. If the controller does not self-monitor the system pressure during degassing, another gauge system should be used to monitor the system pressure. This gauge can then initiate shut down of the gauge(s) during degas, should the system pressure exceed a safe threshold.

Following degassing, the gauge should be operated with the emission current to be used during the calibration. Calibration should not proceed until the requirements for stable base pressure are met; this can take 24 hours or more.

2. *Cold-cathode ionization gauges (CCIGs)*

CCIG controllers typically offer little user control. CCIGs do not have any adjustment that corresponds to the emission current on a hot cathode gauge. Although it can be beneficial to measure the operating potential of a cold cathode gauge, we do not recommend measuring this potential. Controllers can generate voltages on the order of several thousand volts. Extreme caution and appropriately rated equipment must be used for measuring these high voltages. The operating potential varies from manufacturer to manufacturer, so it is necessary to consult them or their literature to find the nominal value.

IX. CALIBRATION PROCEDURE

A. *Base pressure*

The base pressure is the pressure indication with no calibration gas flowing into the chamber, and the chamber open to the high-vacuum pumps. It corresponds to the

equilibrium between gas flow in, such as due to outgassing, leaks, or permeation, and gas flow out of the calibration chamber prior to adding calibration gas. Gas flow out is determined by the throughput of the pump and conductance of the vacuum plumbing between the pump and chamber. In practice, a stable base pressure may be achieved after a period of pumping, *e.g.*, several days. The base pressure reading of each gauge shall be recorded prior to taking calibration data. For most unbaked calibration systems, the largest gas constituent at base pressure is water vapor, but the gauge relative sensitivity shall be set to that of the calibration gas.

The base pressure reading is subtracted from all subsequent calibration pressure readings to correct for non-calibration gas contributions to the gauge's total pressure indication. The base pressure should be lower than the first calibration pressure by at least a factor of 10, but a factor of 100 is more ideal.

The base pressure is considered stable when the change in the working standard pressure indication is negligible compared to the lowest generated calibration pressure, over the time of data acquisition (approximately 1 hour in most cases). Likewise, a UUT gauge's change should also be negligible over the same time interval.

The calibration chamber temperature should also be stable to within 1 °C for at least an hour. If the gauge is to be used at a different ambient temperature than that of the calibration, the user should determine the effect of ambient temperature on the gauge, as discussed in section X.c.

B. Establishing calibration pressure

It is recommended that the calibration proceed from the lowest to the highest pressure. Lower calibration pressures are obtained using the dynamic method.

Procedures for both the dynamic and static methods are given below. The following procedures are general in nature and can be easily adapted to specific systems. Both dynamic and static methods can be used in tandem to cover the calibration range.

1. *Dynamic method*

First, the gas admission system should be purged by pumping back to the gas regulator or gas bottle (with valves V1, V2, V3, V5 open and V4 closed -- see Figure 3). The gas handling system leak valve (V4) should be adjusted so that the chamber pressure, as indicated by the reference gauge, approaches the lowest desired calibration pressure. For finer control, the valve V5 is used to fill the ballast volume to a lower pressure than that of the pressure regulator. The chamber pressure should be allowed to stabilize until any change or fluctuation in indicated pressure within a 5-minute period is less than the measurement precision of the working standard.

2. *Static method*

Here, valve V2 to the pump is closed and gas admission valve (V4) is opened long enough to achieve the desired pressure and then is closed. The pressure must be allowed to equilibrate after making a change. This method is most useful at higher calibration pressures where outgassing does not significantly contribute to the pressure reading.

C. *Data collection*

Once a stable calibration pressure has been achieved, readings may be taken. Record the UUT's indicated pressure, the working standard gauge(s) indicated pressure (or other raw output unit), and the calibration chamber temperature. Several readings

shall be taken (we recommend a minimum of 5) and averaged. The standard deviation is a measurement of stability and repeatability.

Increase the pressure in the system to successively higher pressures and repeat the above data taking procedure. Ideally, a minimum of three approximately equal steps per pressure decade should be taken over the calibration range of interest. All data must be gathered under the same gauge operating parameters, *e.g.*, emission current. If the controller switches emission current automatically, make a note of this and allow time for the pressure indication to stabilize.

If the test gauge is a HCIG having more than one filament, and it is desired to calibrate both, it is recommended that only one filament be calibrated per day. At the end of data taking for one filament, its emission may be turned off and the other filament emission turned on. A degas of the gauge is recommended after switching filaments. The newly activated filament should be set to the desired emission current and be allowed to equilibrate for several hours (preferably 12 hours) at the system base pressure prior to taking calibration data.

X. DATA ANALYSIS

A. Definition and calculation of the correction factor

The raw calibration data must be transformed into a correction factor so that the indicated pressure from the UUT can be converted to accurate pressure reading with a known uncertainty. A pressure dependent correction factor, $f_c = f_c(P_{\text{UUT}})$ is defined as

$$f_c \equiv \frac{\Delta P_{\text{std}}}{\Delta P_{\text{UUT}}} . \quad (3)$$

We use capitalized P to indicate pressure readings from gauges, and we write, f_c instead of $f_c(P_{\text{UUT}})$ for convenience. Here, ΔP_{std} is the pressure change above base pressure caused by the calibration gas, as indicated by the working standard, *i.e.*,

$$\Delta P_{\text{std}} = P_{\text{std}} - P_{\text{std},0} \quad (4)$$

Here P_{std} is the pressure indicated by the working standard due to the admission of calibration gas, and $P_{\text{std},0}$ is the pressure indicated by the working standard when the system is at base pressure. Similarly, for the UUT we have

$$\Delta P_{\text{UUT}} = P_{\text{UUT}} - P_{\text{UUT},0} \quad (5)$$

where P_{UUT} is the pressure indicated by the UUT due to the admission of calibration gas, and $P_{\text{UUT},0}$ is the pressure indicated by the UUT when the system is at base pressure.

The astute reader may notice that f_c has been defined as a function of P_{UUT} and not ΔP_{UUT} , as would seem natural from eq. (3). Users of the calibration factors want $f_c(P_{\text{UUT}})$ and not $f_c(\Delta P_{\text{UUT}})$ because the later would require the user to have the same base pressure in their system as was obtained in the calibration lab, which is not practical or reasonable. The difference between $f_c(P_{\text{UUT}})$ and $f_c(\Delta P_{\text{UUT}})$ is negligible for $P_{\text{UUT}} \gg \Delta P_{\text{UUT}}$, which is why it is important that the lowest calibration pressure be at least 10 times the base pressure. Otherwise, large errors in f_c can result if f_c is not constant.

The correction factor is pressure dependent factor applied to the controller reading. This differs from K or S , discussed in section V.A., which are values programmed into the controller that produce the reading. Here we also assume that once the values of S and K are set in the controller, they are not changed.

B. Adjustment to a common pressure

It may be desirable to adjust the correction factors f_c to pressures different than those set during the calibration. For example, the f_c may have been determined at calibration pressures 1.1×10^{-4} Pa and 5.9×10^{-4} Pa, but a calibration factor, f'_c , is desired at pressures of 1.0×10^{-4} Pa and 5.0×10^{-4} Pa. This may be in accordance to the calibration lab's own procedures, or in agreement with the customer. In addition, the end user of the f_c will need to adjust these to use at an arbitrary pressure. A simple linear interpolation (or extrapolation) may be used if there are two or more determined f_c on the same scale (decade) as f'_c :

$$f'_c(P_{\text{UUT},i}) = f_c(P_{\text{UUT},1}) + \frac{f_c(P_{\text{UUT},2}) - f_c(P_{\text{UUT},1})}{P_{\text{UUT},2} - P_{\text{UUT},1}} \Delta P_{\text{UUT},i} . \quad (6)$$

If three or more f_c were determined per decade, then a linear least-square fit may be applied to obtain a slope, m and an intercept b for each decade. The f'_c may then be determined from

$$f'_c = m \Delta P_{\text{UUT},i} + b , \quad (7)$$

We do not recommend using a linear interpolation or extrapolation between decades. This is because the difference between two successive decades often corresponds to a real change in scale, such as a different amplifier gain, and the correction factors may be discontinuous across scale changes. Linear fits on each scale can be arduous task if calibrations are performed over many decades of pressure. In this case, a polynomial of the form

$$f'_c = \sum_{n=0}^j a_n (\log(P_{\text{UUT},i}))^n \quad (8)$$

can be used to perform a general interpolation of the correction factor. The best fit and number of coefficients is best determined from statistical analysis; however, as a rule of thumb, the number of coefficients should be at least one greater than the number of decades covered in the calibration. Polynomial fits shall not be used for extrapolation of data.

C. Adjustment to a common temperature

Often is desired to adjust the f_c to a temperature that is different from that what used during the calibration. Ideally, the temperature dependence for a particular gauge would be measured, but, realistically, this is difficult to do in most calibration labs. If this is not known, we recommend using the coefficients given in Abbott et al.¹⁴ The typical adjustment for a hot cathode ionization gauge is $0.26\% \pm 0.02\%$ per degree Celsius.

XI. UNCERTAINTY

The uncertainty of f_c shall be reported in the form

$$u_c = \sqrt{u_B^2 + u_A^2}, \quad (9)$$

where u_A^2 is the Type A standard uncertainty estimated from statistical methods, and u_B^2 is the Type B standard uncertainty estimated by methods which may or may not include statistical methods. Typically, u_A^2 is taken from the standard deviation of the mean from repeat measurements. If a model is used to interpolate data, as in eqs. (7) and (8), then u_A^2 may be taken from the statistics of the fit.

The pressure-dependent Type B uncertainty must contain at least the following components:

$$u_B(p) = f_c(p) \sqrt{\frac{u_{\text{std,CAL}}^2}{p^2} + \frac{u_{\text{std,ind}}^2}{p^2} + \frac{u_{\text{std,LTS}}^2}{p^2} + \frac{u_{\text{UUT,ind}}^2}{p^2} + \frac{u_{\Delta p}^2}{p^2} + \frac{u_{p_0}^2}{p^2} + \frac{u_{\text{GAS}}^2}{n_{\text{GAS}}^2}}. \quad (10)$$

Here p is the nominal pressure and may be taken the pressure read by the UUT, P_{UUT} . We use the notation that u_x is that standard uncertainty component for a given quantity x , and

$\frac{u_x}{|x|}$ is the relative uncertainty component. The molar quantity of calibration gas is given

by n . The individual components are discussed below.

$u_{\text{std,CAL}}$ is the uncertainty in the calibration of the working standard. This is taken from a calibration report.

$u_{\text{std,ind}}$ is the uncertainty due to the resolution of the pressure indicated by the working standard. For example, if the working standard has three digits of resolution, such as a reading of 5.01×10^{-4} Pa, then $u_{\text{std,ind}} = 0.005 \times 10^{-4}$ Pa.

$u_{\text{std,LTS}}$ is the uncertainty due to the long-term drift of the working standard. This can be estimated from repeat calibrations of the device, if available. Otherwise, it can be estimated from the performance of similar working standards.^{37--43, 54, 55}

$u_{\text{UUT,ind}}$ is the uncertainty due to the resolution of the pressure indicated by the UUT, as in $u_{\text{std,ind}}$ above.

$u_{\Delta p}$ is the uncertainty due to a pressure gradient between the working standard and the UUT. Often this is given by a relative uncertainty such as, for example,

$$\frac{u_{\Delta p}}{|p|} = 0.1\%$$

u_{p_0} is the uncertainty due to the base pressure measurement. It is estimated from the stability of the base pressure over the course of the calibration.

u_{GAS} is the uncertainty due to the gas purity. For example, if N₂ with 99% purity is used, we might presume, for HClGs, that the remaining 1% of the gas could be gases such as H₂O, O₂, CO₂, or Ar, and the relative gas sensitivity of these gases is $S=1.0 \pm 0.2$. Thus, in this example, we can estimate the relative uncertainty due

to gas purity to be $\frac{u_{\text{GAS}}}{|n|} = 0.02\%$. Note that this is only considering the UUT,

and is the correct uncertainty estimate of if the working standard is not sensitive to gas composition. If an ionization gauge is used as a working standard, then, in

this example, $\frac{u_{\text{GAS}}}{|n|} = \sqrt{2} \times 0.02\% = 0.03\%$. As demonstrated by this example, gas

purity has a negligible contribution the Type B uncertainty if high purity gas is used.

XII. CALIBRATION REPORT/ DATA PRESENTATION

The following minimum information must be included in the calibration report:

1. Test gauge system controller make, model, and serial number or identification number
2. Test gauge tube make, model, and serial number
3. Working standard(s) used and evidence of their SI traceability (such as a calibration report reference number)
4. Data acquisition method, i.e. front display, digital output, (RS232, IEE-488) and or DC volts
5. UUT operating parameters. For HCIGs, this includes:
 - a. Filament(s) used during the calibration
 - b. Emission current
 - c. Grid voltage (if known)
 - d. Filament voltage (AC and/or DC, if known)
6. UUT relative sensitivity setting
7. Calibration gas
8. Date(s) of calibration
9. Name of calibration technician
10. Identification of calibration system
11. Chamber temperature
12. Base pressure prior to start of calibration
13. The determined calibration factor(s) f_c and their uncertainty; if calibration factors are determined at multiple pressures, a table should be presented with

f_c as a function of either the calibration pressure, or to an adjusted pressure (see section X.B)

14. The mathematical model used to determine f_c , such as eq. (3), eq. (6), or eq. (8)

Additionally, we recommend noting any pre-calibration conditioning such as soaking at a pressure in the calibration gas, baking, degassing, and total time that the system has been under vacuum prior to calibration starts. The gauge orientation should also be noted. In addition, any relevant problems with the operation of equipment, malfunctions, disruptions in data taking, and environmental conditions must be included in the report.

XIII. SUMMARY AND CONCLUSIONS

We have recommended practices for the calibration of ionization gauges using the comparison method. The recommendations are based upon the experience of experts in the field. Moreover, we have given detailed theoretical explanations of the behavior of ionization gauges and practical discussion regarding the calibrations of gauges. This information is intended to be informative and helpful.

The methods described here are specific to HCIGs and CCIGs, but many of the considerations will be generally true even as new working standards and vacuum sensors are developed.^{56,57} The design and construction of the comparison apparatus, the consideration in the uncertainty budget, and attention to detail, for example, can be of general applicability to comparison calibrations.

- ¹J. F. O'Hanlon, *A User's Guide to Vacuum Technology*, 3rd ed. (John Wiley & Sons, Hoboken New Jersey, 2003).
- ²*Foundations of Vacuum Science and Technology*, edited by James M. Lafferty, (John Wiley and Sons, Inc. New York, NY, 1998).
- ³A. Roth, *Vacuum Sealing Techniques* (Springer-Verlag, New York, 1993).
- ⁴*Handbook of Vacuum Technology*, edited by K. Jousten (WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim, 2008).
- ⁵*Handbook of Vacuum Science and Technology*, edited by D. M. Hoffman, B. Singh, and J. H. Thomas III (Academic Press, 1997).
- ⁶A. Berman, *Total Pressure Measurements in Vacuum Technology*, Academic Press, Orlando, FL, (1985).
- ⁷*The International Vocabulary of Metrology-Basic and general concepts and associated terms (VIM)*, 3rd Edition, JCGM 200:2012 (2012).
- ⁸B. N. Taylor and C. E. Kuyatt, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, (1994);
<http://physics.nist.gov/TN1297>.
- ⁹P. A. Redhead, J. P. Hobson, E. V. Kornelsen, *The Physical Basis of Ultrahigh Vacuum*, (American Institute of Physics, New York, NY, 1993), pp 304 - 339.
- ¹⁰R. T. Bayard and D. Alpert, *Rev. Sci. Instr.* **21**, 571 (1950).
- ¹¹Figure is courtesy of and generated by Paul Arnold at Granville-Phillips Company circa 1988.
- ¹²*Foundations of Vacuum Science and Technology*, edited by J. M. Lafferty (John Wiley and Sons, Inc. New York, NY, 1998), p 414.
- ¹³R. Holanda, *J. Vac. Sci. Technol.* **10**, 1133 (1973).

- ¹⁴R. L. Summers, *Empirical Observations on the Sensitivity of Hot Cathode Ionization Type Vacuum Gages*, NASA Technical Note TN-D-5285 (Washington D.C., 1969).
- ¹⁵F. Nakao, *Vacuum* **25**, 431 (1975).
- ¹⁶K. Nakayama and H. Hojo, *Jpn. J. Appl. Phys.* **13**, 113 (1974).
- ¹⁷J. E. Bartmess and R. M. Georgiadis, *Vacuum* **33**, 149 (1983).
- ¹⁸K. Tämm, C. Mayeux, L. Sikk, J. F. Gal, and P. Burk, *Int. J. Mass Spectrom.* **341–342**, 52 (2013).
- ¹⁹P. J. Abbott, J. P. Looney, P. Mohan, *Vacuum* **77**, 217 (2005).
- ²⁰K. Jousten, *Vacuum* **49**, 81 (1998).
- ²¹B. R. F. Kendall, *J. Vac. Sci. Technol. A* **17**, 2049 (1999).
- ²²S. Dushman, *Scientific foundations of Vacuum Techniques* (John Wiley and Sons, Inc. NewYork, 1949), pp 332—366.
- ²³Redhead, P. A., *J. Vac. Sci. Technol.* **3**, 173 (1966); doi: 10.1116/1.1492470.
- ²⁴J. C. Helmer and W. H. Hayward, *Rev. Sci. Instrum.* **37**, 1652 (1966); doi: 10.1063/1.1720076.
- ²⁵H. Akimichi, T. Tanaka, K. Takeuchi, Y. Tuzi, and I. Arakawa, *Vacuum* **46**, 749 (1995).
- ²⁶H. Akimichi, N. Takahashi, T. Tanaka, K. Takeuchi, Y. Tuzi, and I. Arakawa, *Vacuum* **47**, 561 (1996).
- ²⁷P. A. Redhead, *Can. J. Phys.* **37**, 1260 (1959).
- ²⁸P. A. Redhead, *Can. J. Phys.* **36**, 255 (1958).
- ²⁹F. M. Penning and K. Nienhuis, *Philips Tech. Rev.* **11**, 116 (1949).
- ³⁰N.T. Peacock and R. N. Peacock, *J. Vac. Sci. Technol. A* **8**, 2806 (1990).

- ³¹B. R. F. Kendall and E. Drubetsky, J. Vac. Sci. Technol. A **14**, 1292 (1996).
- ³²F. M. Penning, Physica **4**, 71 (1937).
- ³³P. A. Redhead, Can. J. Phys. **36**, 271 (1958).
- ³⁴B. R. F. Kendall and E. Drubetsky, J. Vac. Sci. Technol. A **18**, 1724 (2000).
- ³⁵P. A. Redhead, J. Vac. Sci. Technol. **6**, 848 (1969).
- ³⁶L. G. Pittaway, J. Phys. D **3**, 1113 (1970).
- ³⁷J. A. Fedchak and D. R. Defibaugh, J. Vac. Sci. Technol. A **30**, 061601-1 (2012).
- ³⁸H. Yoshida, K. Arai, H. Akimichi, and M. Hirata, Vacuum **84**, 705 (2009).
- ³⁹D. Li and K. Jousten, J. Vac. Sci. Technol. A **21**, 937 (2003).
- ⁴⁰A. R. Filippelli and P. J. Abbott, J. Vac. Sci. Technol. A **13**, 2582 (1995).
- ⁴¹A. P. Miiller, Metrologia, **36**, 617 (1999).
- ⁴²R. F. Berg and J. A. Fedchak, *NIST Calibration Services for Spinning Rotor Gauge Calibrations*, NIST Special Publication 250-93 (2015), p 26;
<http://dx.doi.org/10.6028/NIST.SP.250-93>.
- ⁴³R. F. Chang and P. J. Abbott, J. Vac. Sci. Technol. A **25**, 1567 (2007).
- ⁴⁴P. C. Arnold, D. G. Bills, M. D. Borenstein, and S. C. Borichevsky, J. Vac. Sci. Technol. A **12**, 580 (1994).
- ⁴⁵Y. Tito Sasaki, J. Vac. Sci. Technol. A **9**, 2025 (1991).
- ⁴⁶R. N. Peacock, N. T. Peacock,,D. S. Hauschulz, J. Vac. Sci. Technol. A **9**, 1977 (1991).
- ⁴⁷M. Saitoh, K. Shimura, T. Iwata, J. Vac. Sci. Technol. A **11**, 2816 (1993).
- ⁴⁸A. R. Filippelli, J. Vac. Sci. Technol. A **14** (1996).

- ⁴⁹H. C. Hseuh and C. Lanni J. Vac. Sci. Technol. A **5** (1987).
- ⁵⁰P. C. Arnold, *Results of Performance Dependencies of Bayard-Alpert type Hot Cathode Ionization Gauges Upon Operational Parameters of Their Controllers*, AVS 52nd International Symposium, Nov. 1 (2005).
- ⁵¹R. N. Peacock and N. T. Peacock, J. Vac. Sci. Technol. A **8**, 3341 (1990).
- ⁵²D.A. Olson, P.J. Abbott, K. Jousten, F.J. Redgrave, P. Mohan, S.S. Hong, Metrologia Tech. Suppl. **47**, 07004 (2010).
- ⁵³J. A. Fedchak, Th. Bock, and K. Jousten, Metrologia **51**, 07005 (2014).
- ⁵⁴J. A. Fedchak, K. Arai, K. Jousten, J. Setina, and H. Yoshida, Measurement **66**, 176 (2015).
- ⁵⁵K. Jousten and S. Naef, J. Vac. Sci. Technol. A **29**, 11011 (2011).
- ⁵⁶J. Scherschligt, J. A. Fedchak, D. S Barker, S. Eckel, N. Klimov, C. Makrides, and E. Tiesinga, Metrologia **54**, S125–S132 (2017).
- ⁵⁷K. Jousten, J. Hendricks, D. Barker, K. Douglas, S. Eckel, P. Egan, J. Fedchak, J. Flügge, C. Gaiser, D. Olson, J. Ricker, T. Rubin, W. Sabuga, J. Scherschligt, R. Schödel, U. Sterr, J. Stone and G. Strouse, Metrologia **54**, S146 (2017).