

Recommended practice for calibrating vacuum gauges of the thermal conductivity type

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This article describes and recommends various methods for calibration of thermal conductivity vacuum gauges in the pressure range of the order of 10^{-1} Pa (10^{-3} Torr) to an atmosphere and is one of a series published by the American Vacuum Society. It contains data from many sources and represents the opinions of a number of experts in the field. The text was developed by a sub-committee of the Recommended Practice Committee of the American Vacuum Society. The Thermal Conductivity Gauging Committee is made up of users and manufacturers of vacuum gauges who have a variety of experience in the practical use of thermal conductivity gauges.

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III. SCOPE

Procedures and apparatus are described for calibrating vacuum gauges of the thermal conductivity type by direct comparison with a calibrated reference instrument such as a capacitance diaphragm gauge (CDG), quartz Bourdon gauge (QBG), or spinning rotor gauge (SRG). The pressure range considered is of the order of 10^{-1} Pa (10^{-3} Torr)– 10^5 Pa (760 Torr). This recommended calibration procedure can be used for lower pressures. However, uncertainties in readings increase significantly for thermal conductivity gauges (TCGs) operated at very low pressures.

Dry nitrogen gas is commonly used as the test gas for TCG calibration although any dry inert gas may be used. In this article we do not address condensable gases, that is, gases whose saturation vapor pressure may be reached under the temperature and pressure conditions of the calibration. Potentially explosive gas mixtures are not considered and are discouraged from use since TCGs could be an ignition source.

The text is directed primarily to the calibration of TCGs with their readouts (panel meter display and analog or digital output), including thermocouple gauges, Pirani gauges, and thermistor gauges, with or without convection-mode operation. The procedure may be adapted readily to the calibration of other vacuum gauges whose operation is based on thermal conductivity or on a hybrid of pressure measurement methods. For example, there are TCGs that do not include a pressure display but rather provide a dc analog voltage output. The calibration of these gauges involves determining the analog output voltage as a function of pressure.

IV. PRINCIPLE OF OPERATION OF THERMAL CONDUCTIVITY GAUGES

Thermal conductivity gauges^{1,2} are a class of pressure measuring instruments in which the measured response is associated with energy loss from a heated element (usually a heated wire). The energy loss is due to thermal conduction through the surrounding gas and to the wire supports, and by radiation and by convection. The low-pressure limit of a TCG is reached when the pressure dependent energy loss becomes significantly less than the pressure independent losses due to radiation and conduction to the wire supports, as shown in Fig. 1. Energy loss by gas conduction has a linear pressure dependence at low pressures where the energy transfer by gas molecules is proportional to the number density of molecules. As the gas density goes up and the mean free path becomes shorter than the wire-to-wall distance, gas molecule collisions increasingly become important

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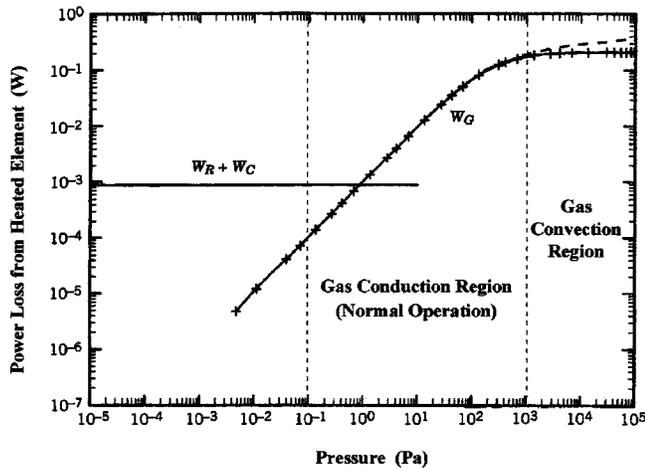


FIG. 1. Power loss from the heated element of a TCG by gas conduction (W_G) by conduction to the end supports (W_C) and by thermal radiation (W_R) as a function of pressure. As the pressure is increased above approximately 10^3 Pa the power loss due to gas conduction saturates except for convection losses, which are weakly pressure dependent (shown qualitatively as the heavy dashed curve).

in energy transport to the wall and eventually at high pressures (viscous flow) the energy transfer rate becomes pressure independent (excluding convection effects).^{1,3} Deviation from linear response in a wire filament Pirani^{4,5} and a miniature⁶ Pirani is observed to occur at pressures of 10–100 Pa (Fig. 1) where the mean free path is a few to 10 times the wire diameter. The upper pressure limit of a TCG may be extended to approximately 10^5 Pa by taking advantage of the (weak) pressure dependence of convection losses. Practical TCGs measure pressures between 10^{-1} and 10^3 Pa (10^{-3} –10 Torr). Thermal conductivity gauges that utilize convection of the gas within the gauge envelope to extend their measurement range above 10^3 Pa to 10^5 Pa (the convection mode of operation) are also commercially available. Occasionally TCGs have calibrations that extend below 10^{-1} Pa. Typical responses of a TCG to different gases are shown in Figs. 2 and 3.

There are two principal thermal conductivity gauge types: the Pirani and the thermocouple. The typical heated-wire temperature for both gauge types is 120–150 °C. Figures 4(a) and 4(b) show basic electrical measurement circuits for the two types of gauges. In a Pirani gauge, the wire temperature (i.e., its resistance) is usually kept constant and the required voltage across the bridge provides a pressure-dependent signal. In a thermocouple gauge, the wire is usually heated with constant power and the pressure-dependent wire temperature is measured directly with a thermocouple. An alternate operating mode for a thermocouple gauge is to maintain the wire at a fixed temperature and measure the pressure-dependent input power required to maintain the constant wire temperature. In both gauge types, instrument manufacturers convert the pressure-dependent response of the heated wire into a pressure reading for a specific gas, typically, nitrogen. The manufacturer usually provides calibration curves for other gases that are obtained by

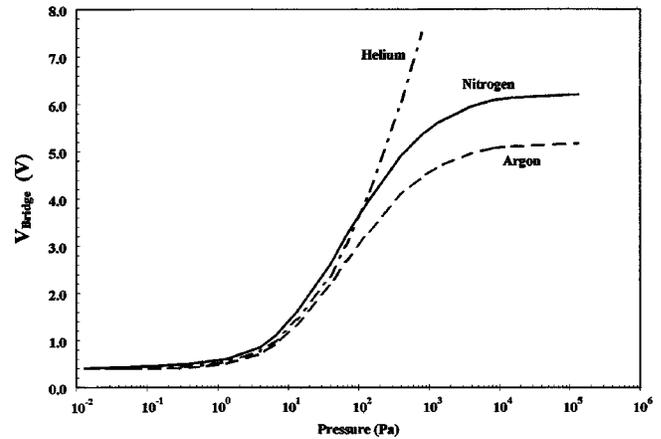


FIG. 2. Pirani gauge bridge voltage proportional to power loss for the heated element as a function of pressure for helium, nitrogen, and argon. The low pressure asymptote is due to conduction loss to the wire end supports and thermal radiation loss. Power loss in the midpressure range is dominated by the thermal conductivity of the gas present. At higher pressures the loss due to gas conduction saturates and the response of the gauge is dominated by convection losses, which are weakly pressure dependent.

multiplying the nitrogen calibration by single gas-specific correction factors (Fig. 3). More detailed descriptions of how TCGs operate are given in Refs. 1 and 2.

V. TERMINOLOGY

The following terms and definitions relate to the calibration of TCGs. Other terms and definitions may be found in the *AVS Dictionary of Vacuum Terminology*.⁷

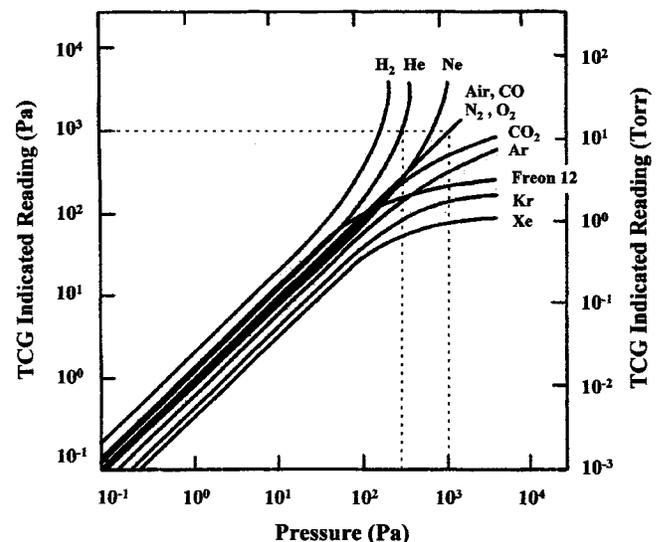


FIG. 3. Correction curves for a particular Pirani gauge relating the indicated reading to the actual pressure for common gases. For example, an indicated reading of 10^3 Pa for He corresponds to an actual He pressure of approximately 200 Pa whereas the same indicated pressure for N_2 is calibrated to be equivalent to the actual N_2 pressure. For the linear response region, a gas specific factor can be obtained from the correction curves to scale the indicated pressure to give an actual pressure for the gas present. (Used by permission from Leybold-Heraeus GmbH, Köln, Germany.)

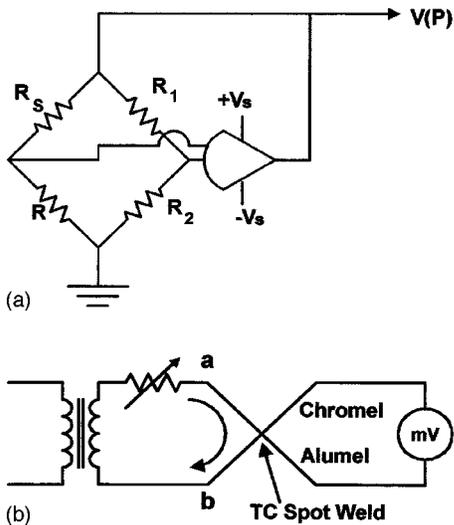


FIG. 4. (a) Pirani gauge with a bridge amplifier for constant temperature operation. The resistance R is the Pirani gauge heated element and R_s , R_1 , and R_2 are reference resistors in the bridge. The amplifier senses the error signal of the bridge and adjusts the voltage across the bridge to restore the original operating temperature of the heated element. (b) A basic thermocouple gauge circuit. Constant power is applied to the heated wire a-b and the temperature of the wire in the gas is measured at the center spot weld as a thermocouple output voltage.

- (1) *Primary Standard.* An instrument where pressure readings are derived from fundamental units such as length and mass. This would include measurement of a liquid column height or pressures produced by volume expansion.
- (2) *Transfer Standard.* An instrument that has been calibrated with traceability to a primary standard for the purpose of being used for a local calibration application. Examples of such transfer standards are calibrated CDGs and SRGs.
- (3) *Working Standard.* A working standard is a calibrated instrument used for routine calibrations of other instruments. For example, this can be the transfer standard or a specific TCG.

- (4) *Check Standard.* A check standard is an instrument that may or may not be fully calibrated but which is known to have a stability comparable to or better than the device being checked. For example, the check standard can be a specific TCG.
- (5) *Static Calibration.* This refers to producing known pressures by introducing a quantity of gas into the volume to which the gauge to be calibrated and the working standard are connected.
- (6) *Dynamic (Continuous Flow) Calibration.* This refers to producing equal pressures at the gauge under test and the working standard by establishing a steady-state gas flow.
- (7) *Units of Pressure.* Pressure readings of thermal conductivity gauges are commonly displayed with units of pascal (Pa), Torr, millibar (mbar) or microns (μm). The relationships among these units are defined as follows:

$$1 \text{ standard atmosphere} = 760 \text{ Torr} = 101.325 \text{ kPa},$$

$$1 \text{ Torr} = 133.322 \text{ Pa} \approx 1000 \mu\text{m},$$

$$1 \text{ mbar} = 100 \text{ Pa}.$$

VI. CALIBRATION APPARATUS

The calibration apparatus consists of a pressure standard, the calibration chamber and vacuum system, and a test gas admission system. System construction is important. Suitable construction for the calibration apparatus uses stainless steel, metal-sealed fittings, and bellows-sealed valves.

A. Pressure standards

The pressure standards include primary, working, or check standards that are suitable for use in the range of pressures where the TCG is to be calibrated. Table I gives a list of suitable standards. The recommended choices are one or more calibrated capacitance diaphragm gauges^{8,9} of appropriate full-scale range or a combination of gauges that includes a spinning rotor gauge.¹⁰

TABLE I. Calibration standards suitable for use in the calibration of TCGs in selected pressure ranges below approximately 1 atm.

Calibration standard	Range (Pa)	Uncertainty ^a
Primary Standards:		
Liquid Column Manometers		
(i) Mercury UIM ^b	1–3.6×10 ⁵ Pa	$[(6 \times 10^{-3} \text{ Pa})^2 + (5.2 \times 10^{-6} P)^2]^{1/2}$
(ii) Oil UIM ^b	10 ⁻¹ –140 Pa	$[(3 \times 10^{-3} \text{ Pa})^2 + (3.6 \times 10^{-5} P)^2]^{1/2}$
(iii) McLeod gauge (Hg)	10 ⁻⁴ –10 ² Pa	≈2%
Transfer Standards:		
Quartz Bourdon gauges	10–10 ⁵ Pa	~0.01%
Capacitance diaphragm gauges	10 ⁻¹ –1.33×10 ⁵ Pa	0.3%–0.7%
Spinning rotor gauges	10 ⁻³ –10 Pa	1%–2%
Thermal conductivity gauges	10 ⁻¹ –10 ⁵ Pa	5%–20%

^aEstimated total uncertainty (nominally at the two-standard deviation level) arising from systematic effects; P is the pressure in Pa.

^bUltrasonic Interferometer Manometers (UIMs) developed at the National Institute of Standards and Technology.

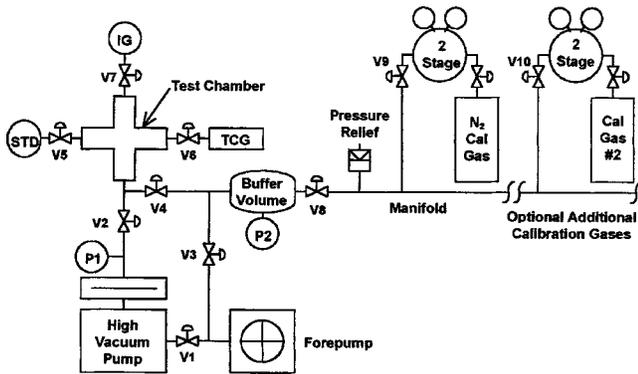


Fig. 5. Vacuum and gas admission systems for the calibration of thermal conductivity gauges with noncondensable gases by comparison with a pressure standard: IG—ionization gauge to read base pressure; P1—gauge to read inlet pressure of the high vacuum pump; P2—gauge to read buffer volume pressure (optional); valves V5, V6, and V7 are optional.

B. Vacuum system

Figure 5 shows a typical calibration apparatus. The following features are desirable for a TCG calibration apparatus.

- (1) The pumping system should use a liquid-nitrogen-trapped diffusion pump or a turbomolecular pump capable of handling reasonable gas loads without backstreaming when comparing gauges in the continuous flow mode. To make possible chamber pressures higher than allowed pump inlet pressures, a throttling valve, V2, is necessary between the chamber and the pumping system. This valve must be continuously adjustable from complete closure to full open. An appropriate gauge, P1, may be required between the valve and pump to insure that maximum pump inlet pressure is not exceeded.
- (2) The test chamber and associated plumbing should have a volume at least 50 times the total internal volume of the gauges. This aids in controlling pressure changes during measurements.
- (3) The test chamber ideally should have a shape that minimizes the internal surface area to volume ratio. However, for small systems, a six-way cross conveniently approximates this goal. Other shapes with additional test ports that provide symmetry in mounting the TCGs and the standard (STD) to minimize pressure gradients are acceptable.
- (4) The system should be clean and leak tight and should be capable of a base pressure less than 1% of the lowest pressure at which the gauge is to be calibrated. Alternatively, the isolated calibration system should have a rise in pressure from leaks and outgassing that is less than 10% of the lowest pressure for calibration during the time it takes to establish and measure the lowest calibration point.
- (5) During the calibration, the system should be maintained at the desired ambient temperature, which is then recorded several times during calibration. *Note: If the gauge is used at a different ambient temperature than*

that of the calibration, the user should determine the effect of ambient temperature on the gauge from vendor literature or by measurements on the test gauge over the temperature range of interest.

- (6) All gauges involved in the calibration should be operated long enough to reach a stable temperature before beginning the calibration. The time required for a TCG to temperature stabilize depends on pressure. The temperature-stabilization times should be given in the TCG Operations Manual as part of the TCG specifications.
- (7) The gauges being calibrated and the standard should be mounted symmetrically with respect to the flow stream to assure equal pressures even with flowing calibration gas.
- (8) The system should include a gauge capable of reading the base pressure. A hot or cold cathode ionization gauge (IG) or a spinning rotor gauge is suitable for this and can be isolated from the test chamber with a valve (V7, optional). A residual gas analyzer (RGA) could also be used to provide base pressure, test gas purity, and an easy way to leak check the system at base pressure.

C. Test gas admission system

The purpose of the gas admission system is to provide either conveniently adjustable flows or intermittent admission of the gas to obtain desired pressures. Figure 5 shows a typical gas admission apparatus.

Since TCGs have differing sensitivity for various gases, the system must be capable of calibrating the gauges for different gases. For example, a pressure of less than 10^3 Pa (10 Torr) of a high thermal conductivity gas such as hydrogen, helium, or water vapor will produce the same indication as one atmosphere of nitrogen. Thus the gas admission system must provide pure test gas with the partial pressure of impurities low enough to avoid errors. This means that

- (1) the chamber must be evacuated to at least 10^{-3} Pa (10^{-5} Torr) before starting a calibration;
- (2) the gas admission system must be vacuum purged back to the high-pressure gas cylinder valves as necessary;
- (3) the cylinder gas should be at least 99.95% pure. *Note: A small mass spectrometer with an appropriate gas sampling system can be helpful in ensuring that purity requirements are satisfied.*
- (4) Valve V4, which is used for gas admission, must be capable of fine control. Flow adjustment from 10^{-6} to 10^3 Pa m³/s (10^{-5} – 10^4 atm cc/s) is desirable. This range of flow can be accomplished with a wide-range variable leak valve. The goal is to produce a stable flow rate to establish a constant pressure in the test chamber in the dynamic calibration operating mode.

The following features are desirable for a gas admission system.

- (1) The gas system should be compatible with the two modes of calibration:¹ dynamic and static. In the dy-

dynamic mode, which is typically used from 10^{-1} Pa (10^{-3} Torr) to about 100 Pa (1 Torr), gas flows continuously through the system. This mode is used to prevent small leakage and outgassing rates from affecting the measurements, and makes it easier to set a stable low pressure. The valve (V4) and the throttling valve (V2) are used to obtain the desired equilibrium pressure while maintaining an allowed pump inlet pressure within the manufacturer's specifications. In the static mode, which is used at higher pressures, the throttling valve (V2) is fully closed, and the valve (V4) is opened to increase the pressure incrementally, and then closed.

- (2) The system should include a pressure relief valve or burst disk to prevent the manifold pressure from rising to dangerous values in the event of gas cylinder regulator failure. If calibrating with toxic gases, an appropriate venting system is required.
- (3) The system should include porous metal filter/seals in the metal-sealed fittings between the cylinder pressure regulator and the regulator-to-manifold valves (V9, V10) to prevent particulates from damaging the expensive flow adjustment valves.
- (4) An optional buffer volume with pressure gauge, P2, is shown, which is helpful in maintaining stable flow in those instances where it is desired to work with the cylinder valve closed.

VII. CONDITIONS FOR TESTS

A. Pressure

The gauge to measure base pressure should be calibrated to read nitrogen pressure. The transfer standard(s) used to measure calibration pressures should be calibrated for all gases used if their response is gas species dependent. Spinning rotor gauges are species dependent over their entire operating range. Capacitance diaphragm gauges are species dependent at pressures below 100 Pa (1 Torr) if operated at a temperature different from that of the calibration system.

B. Temperature

The calibration of a TCG should be at an ambient temperature of (23 ± 3) °C or at a specialized temperature for the desired application. It is important that the temperature conditions during the calibration resemble, as closely as possible, those under which the gauge is to be used (see Sec. XC).

If there are significant ambient temperature differences between calibration and use of either the transfer standard(s) or the TCGs, thermal transpiration effects can become important at pressures below 100 Pa (1 Torr). For example, the effects of thermal transpiration in capacitance diaphragm gauges⁹ can be as large as $\sim 1\%$ for each 5 °C difference as discussed by Poulter *et al.*¹¹ and by Jitschen and Röhl¹². However, in the case of TCGs, the magnitude of the thermal transpiration effect cannot be readily estimated due to the large temperature distribution of gas molecules inside the gauge.

VIII. PROCEDURES

A. Assembly

- (1) Assemble the calibration apparatus and connect the standard (as well as any check standards) to the calibration chamber. Check with manufacturer's recommendations for mounting orientation of the standard.
- (2) Connect the thermal conductivity gauge(s) to be calibrated to the vacuum system. Orientation of a TCG during calibration and use should be the same as that recommended by the manufacturer; for example, most convection-enhanced Pirani gauges need to be mounted with the filament in a horizontal position to operate correctly above ~ 10 Torr. *Note: Components from several TCG measuring systems of the same model may be interchangeable and be within the manufacturer's specifications. However, it is recommended that elements comprising a given measurement system, including all ancillary equipment, be calibrated as a unit for the lowest uncertainty.*
- (3) Complete the electrical connections between the TCGs and their controllers (according to manufacturer's instructions) using the cable and input connections that will be used in a subsequent application. Verify operation according to the manufacturer's recommendation.

B. Preparatory operations

- (1) TCGs that have an atmospheric pressure reference adjustment (ATM) require exposure to a specific gas or the application gas for end use. In preparation for calibration, TCGs should be exposed to the test gas at 1 atm and the ATM gauge adjustment made.
- (2) Evacuate the test chamber, gauges, and associated plumbing. If necessary, bake out the system, applying heat to the components within their temperature limits to reduce outgassing to an acceptable amount to reach the desired base pressure.
- (3) After achieving base pressure, close the valve to the pump (V2) and the gas supply (V4) to determine the pressure rate of rise. If a RGA has been installed on the system, most have software packages to determine the pressure rate of rise. It is recommended that the rise in pressure be less than 10% of the lowest calibration pressure in the time it takes to establish and measure the lowest calibration point. If the rate of rise is too high, take corrective measures as necessary to eliminate leaks and reduce outgassing before proceeding.
- (4) Just prior to calibration, with the system at base pressure, adjust or readjust all gauges on the system to zero according to the manufacturer's recommendations.
- (5) Close valve V7 to the base-pressure gauge (or turn off the gauge if there is no isolation valve).

C. Gas delivery operation

- (1) Isolate the gas supply cylinders from test chamber and vacuum system by closing appropriate valves. Connect pressurized gas cylinder(s) to the manifold as needed.

Purge the air trapped when connecting a cylinder to the system by using a general laboratory procedure that involves a sequence of evacuations of the manifold and purging using the cylinder gas. Three cycles of evacuation and purge are a common practice. For evacuation of the manifold, establish a path from the cylinder to the forepump by closing V1 and V4 and opening V3, V8, and V9 or V10 as appropriate (Fig. 5) with the regulator adjusted to deliver gas. To purge the manifold with cylinder gas, close V9 (or V10) and open the gas cylinder valve. Adjust the regulator to a positive pressure in the range of 15–30 kPa (2–5 psig). *Note: Suitable two stage regulators are needed for low-pressure delivery. Manufacturers also will supply regulators specially cleaned for pure gas handling.* With the manifold under vacuum and valve V4 fully closed, the manifold is filled with gas to the regulator pressure by opening V9 (or V10). A Bourdon or similar pressure gauge, P2, is helpful to indicate the manifold pressure. Thereafter, gas admission to the test chamber is controlled by the leak valve, V4.

- (2) Calibration is possible using increasing or decreasing pressure. However, calibration is normally performed from low to high pressure for convenience and to minimize effects of outgassing, wall temperature variations, gauge zero instabilities, or leaks on low pressure calibration points. Calibrations using increasing pressure may differ from a calibration obtained with decreasing pressure if the gauge wall temperature changes significantly due to self-heating.
- (3) After a calibration run the chamber pressure is usually higher than the maximum recommended inlet pressure of the high vacuum pump. Chamber pressure can be reduced by closing V1, V2, and V8 and opening V4 and bypass valve V3 to a mechanical roughing pump. If an oil forepump is used, avoid backstreaming of oil by closing bypass valve V3 before molecular flow is reached in the foreline¹³ at about 30 Pa (0.3 Torr). Use of a suitable molecular sieve trap in the foreline of an oil forepump is recommended.

D. Calibration

- (1) Admit nitrogen or other test gas to the calibration chamber as required to establish the pressure desired for a calibration point. The gas may be admitted and the pressure adjusted by either of the following two methods.¹
 - (a) *Dynamic method* (for pressures below about 100 Pa). Close V3 then open V8. Adjust both the inlet valve V4 and exhaust valve V2 to produce the desired pressure in the calibration chamber with a constant flow of test gas into and out of the calibration chamber. *Note: The dynamic method makes adjustment of pressure easier than controlling the introduction of small quantities of gas. However, the gas load may get too large if the pressure produced for dynamic flow calibration measurements exceeds the maximum inlet pressure of the high vacuum pump. The static method is preferred for establishing higher pres-*

ures.

- (b) *Static method* (for pressures greater than the maximum inlet pressure of the high vacuum pump). Admit only as much gas as required through valve V4 (V2, V3 closed) to raise the pressure to the desired value with the pumps valved off.
- (2) Observe the reading on the pressure standard until a stable pressure is achieved. Observe the reading of the TCG until the reading stabilizes to a steady average value with a precision that is applicable for the purpose of the calibration. *Note: The precision of all TCGs is pressure dependent, which is a direct consequence of their nonlinear response to pressure.* Successive readings should be taken on the TCG and standard until no trend or the same trend in pressure readings is observed in both gauge and standard. Record the readings and compute the average values for the TCG and standard.
- (3) Repeat for other test points with a minimum of two different points per decade of pressure over the complete pressure range desired. For two points per decade, use [approximate values (1, 3, 10, ...)] and for three points per decade use (1, 3, 7, 10, ...) over the pressure range. This procedure generates a calibration table of pressures measured by the pressure standard and by the gauge(s) under test. These are used to generate a calibration report as described in Appendix C.

IX. ACCURACY AND PRECISION

The precision of readings of the TCG under test is limited to the least significant stable digit on a digital display or readability/interpolation of an analog display or analog output for the decade of pressure being measured. The measurement uncertainty of the TCG can be about 5%–20% of indicated pressure. The use of a single gas-specific factor (Fig. 3) to scale the calibration with nitrogen to another gas will generally produce uncertainties that are larger than direct calibration for that gas.

X. GOOD PRACTICES AND USAGE OF TCGS

A. Safety issues

1. Overpressurization protection

Figure 3 indicates that gases with molecular weights greater than that of nitrogen result in a TCG pressure indication that is lower than the true pressure in the convection-mode pressure region ($>10^3$ Pa). For example, the TCG response to argon saturates at about a 10^4 Pa (100 Torr) reading on a nitrogen calibrated scale as pressure is increased.^{14,15} As the argon pressure is increased to an atmosphere and above, there is no increase in the TCG reading. If this gauge were used for monitoring when bringing a system to atmospheric pressure using argon, the potential for overpressurization exists. Thus a pressure relief device is recommended if a TCG with a convection-mode calibration is planned to be used to control process pressure near atmospheric pressure.

2. Explosive gas mixtures

Heated TCG filament materials include W, Pt, Ni, Nichrome, and Chromel-Alumel. Platinum and nickel can be sources of ignition if explosive gas mixtures are present even if the gauge is turned off because the surface can be auto-catalytic for some reactions.¹⁵ Hot spots or broken wires that could generate a spark can also become a source of ignition for any filament material. It is not recommended that TCGs be used when potentially explosive gas mixtures are present; a single-sided CDG with no electrodes present would be a better choice for pressure measurements of potentially explosive mixtures.

B. Cleaning of TC gauges

The calibration of TCGs may be affected by changes in the surface of the heated elements, which will affect their heat transfer characteristics, especially at low pressures. These changes may result from oxidation of the heated surfaces or the accumulation of foreign matter. Oil deposits are especially troublesome. If the heated elements operate at temperatures above, say, 300 °C, the oil may decompose and form coatings that are difficult to remove. If the gauge is suspected of being contaminated, consider the consequences of attaching the unit to the calibration apparatus. Consult the operating manual of the TCG for cleaning instructions, if any. The safest plan to assure reliable pressure readings is to replace the gauge head with a new calibrated unit.

C. Ambient temperature effects

The response and calibration of the TCG depend on the gauge wall temperature, which is affected by the ambient temperature of the gauge. Some TCG manufacturers have temperature compensation for the wall temperature but unusually high or low ambient temperatures can cause inaccuracies in readings. Gauges should be used at an ambient temperature within the range specified by the manufacturer. For applications where the gauge environment is unusually warm or cold, and accurate readings are needed, the calibration of the gauge should be done with the gauge tube placed in a temperature-controlled environment and adjusted to the temperature of the application environment (see also Appendix A 3 a).

APPENDIX A: GENERAL

1. Laboratory safety

Caution: General laboratory safety and the use of hazardous materials are beyond the scope of this article.

2. Calibration standard

For a device or instrument to qualify as a standard, its measurement performance should be predictable and thoroughly understood, and its random and systematic uncertainties should be well characterized and documented. Only if the above conditions are met can the comparison of the TCG with the standard be called a calibration.

3. Environment

a. Temperature considerations. All thermal conductivity gauges (TCGs) respond to external temperature changes, as do the standards against which they are being calibrated. Although many TCGs have temperature compensation, they should be protected from extreme temperatures during both calibration and subsequent use and also from changes in temperature from local air currents, such as those from air conditioning vents and cooling fans. If extreme temperature variations are encountered, it may be necessary to actively control the temperature of the immediate surroundings, even for TCGs with built-in temperature control or temperature compensation. The degree to which lack of temperature stability is a problem must be inferred from behavior in a particular environment.

If condensable gases are present in the system, care must be taken to avoid temperature and pressure conditions under which liquid can form.

b. Cleanliness. Strict attention must be paid to maintaining cleanliness in order to insure leak-tight joints and low outgassing rates and to prevent contamination of the instruments under test and/or the calibration system. It is important to know the history of the TCG to be calibrated before connecting it to the calibration chamber to prevent a possible transfer of contaminants.

c. Stability of equipment. It is suggested that the transfer standard and check gauges be kept turned on and evacuated when not in use. This keeps them in a relatively stable temperature environment, clean, and outgassed before the next use. All of these precautions will help prevent shifts in their calibration and minimize the next start-up time.

4. Personnel

Personnel performing calibrations should be familiar with the principle of operation and general characteristics of both TCGs and whatever device is being used as a standard, plus all ancillary equipment.

5. Certification of equipment

If applicable, all ancillary equipment (voltmeters, etc.), should be within their certification period and have reliable estimates of their uncertainties that can be factored into the final uncertainty of the instrument being calibrated. Proper operation of computers and other data gathering devices should be verified. Be aware that the addition of data-logging equipment can introduce errors such as noise, nonlinearity, and voltage offsets.

APPENDIX B: DOCUMENTATION

Maintenance of documentation relating to calibrations, including procedures and results, is a matter of user policy. The following guidelines are suggested.

1. Calibration intervals

In principle the required calibration intervals could be estimated from the TCG manufacturer's specifications pertaining to accuracy and calibration stability, if given. However these data may not represent the user's TGG and until a history is obtained which indicates otherwise, it is prudent to keep the initial calibration intervals for a TCG relatively short. Past calibration history and interim use (or misuse) should also be accounted for rather than adhering to an inflexible schedule. Comparisons at several pressures against a check standard (see definition in Sec. V) can be used between regularly scheduled calibrations to determine if gross changes have occurred, and to determine if a scheduled full calibration is even necessary. Drift of the ZERO or ATM readings may also indicate a calibration problem.

2. Records

Records should be kept for each TCG that is calibrated as well as for any transfer standard used for comparison to allow assessment of their long-term stability with time (which may not be initially available) and to provide objective evidence that calibration schedules are complied with.

a. Retention. Calibration data and ancillary records should be retained in sufficient detail to permit repetition of the calibration, if necessary, and for a sufficient period of time to satisfy all regulatory and contractual requirements.

b. Record content. The records should include TCG identification, calibration history including any unusual observations or circumstances, necessary information required to locate each TCG in the calibration system (if applicable), and a clear indication of when the next calibration should be performed on any gauge. Similar information concerning the primary or transfer standard used in the calibrations should also be kept.

c. Traceability documentation. If required, documentation must state the traceability to national, international, or intrinsic standards of measurement. If traceability to the National Institute of Standards and Technology (NIST) is being claimed for a transfer standard, be sure that its calibration certification is current and contains references to the date of calibration and the NIST calibration test number upon which the claim is based. If a primary standard is used for TCG calibration, a suitable program of interlaboratory comparisons or proficiency testing may satisfy traceability requirements.

d. Labeling. Calibration labels should be used when the accuracy of a TCG is critical to the integrity of a measurement process in order to alert the user to its calibration status. The label, which may be applied to the TCG itself or on the TCG readout, should include information such as an identification or control number of last calibration, name or initials of the calibrator, and the scheduled recalibration date.

APPENDIX C: THE CALIBRATION REPORT

The calibration report, at a minimum, should properly identify the TCG and the standard used, the date of calibration, the calibration gas, the calibration method, the calibrator, the critical temperatures in the system such as temperatures of the standard, the calibration chamber, and the TCG, and any special circumstances.

The report should include some method for recovering "true" pressure from the TCG indicated readings (e.g., see Fig. 3). A table containing the indicated readings of the TCG and those of the standard (true pressure) along with the associated differences (or corrections) is a common method of presenting data in a report. *Note: At the time of use for pressure determinations, these data may be entered into a computer as a look-up table or a least-squares-fitted equation giving "true" pressure as a function of TCG indication.*

The use of control charts in the calibration report is highly recommended. Examples of control charts are graphs that give the history of past calibrations, which may be used to spot trends or changes in the behavior of either the TCG or the standard. Plots of differences between indicated readings of the TCG and those of the standard as a function of the TCG reading are also useful because they provide an easy method for comparison with previous calibrations. They also provide a quick method for predicting corrections to the indicated readings at different pressures.

The report should also contain a statement of the estimated uncertainty in the calibration test results (see Sec. C 2) as well as the estimated uncertainty in subsequent use of these results to measure pressure with the calibrated TCG (see Sec. C 3).

Guidelines¹⁶ for evaluating and expressing uncertainty in measurement results adopted at NIST are recommended as a general approach. The components of uncertainty are commonly identified either as those arising from random effects or those arising from systematic effects. These components of uncertainty may be classified alternatively according to the method used to estimate their numerical values: (a) those which can be evaluated by statistical analyses as standard deviations, termed **standard uncertainties** (type A evaluation), and (b) those which are evaluated based on scientific judgment using all relevant information available (type B evaluation). In a type B evaluation, the estimated uncertainty is converted to an equivalent **standard uncertainty** by dividing the estimated uncertainty by a multiplier¹⁶ associated with the model probability distribution (rectangular, triangular, or normal) that best describes the statistical behavior of the quantity being measured. The **combined standard uncertainty** of a measurement result can then be obtained by combining the individual standard uncertainties, whether arising from a type A evaluation or a type B evaluation, using the "root-sum-of-squares" method. The current international practice (at NIST as well) is to report the measure of uncertainty as an **expanded uncertainty** at the two-standard deviation level, which is obtained by multiplying the standard uncertainty by a **coverage factor** equal to 2. Thus, when normal statistical distributions apply, the expanded un-

certainty defines an interval having a level of confidence of approximately 95%.

1. Uncertainties associated with the standard

Primary and transfer standards that are suitable for use in the calibration of TCGs are listed in Table I along with a typical value or range of values for their uncertainty due to systematic effects. The systematic effects in the liquid-column manometer primary standards include uncertainties in the density of the liquid, the measurement of liquid column heights, and the local acceleration of gravity. The largest systematic effect in a transfer standard is often the long-term instability in its calibration. The uncertainty due to calibration shifts can only be evaluated by repeat calibrations of the particular transfer standard against a reliable standard over a period of time.

Systematic effects in different types of calibration standards such as ultrasonic interferometer manometers,^{17,18} capacitance diaphragm gauges,⁹ and spinning rotor gauges¹⁰ (also called molecular drag gauges) are described in detail in the cited references. The references give uncertainties that were representative of a particular instrument or a group of instruments at the time of study. These uncertainties and those in Table I should be used only as a guide in evaluating uncertainty in any selected calibration standard since some instruments perform better than this, and some worse.

All systematic effects that can contribute to the uncertainty of the standard used for calibration should be identified and the magnitudes of the associated uncertainties should be estimated at appropriate points within its measurement range. The uncertainty of the standard, U_{STD} , can then be evaluated by taking the root-sum of squares of the component uncertainties:

$$U_{STD} = \sqrt{\sum_i (U_{SYS})_i^2}, \quad (C1)$$

where $(U_{SYS})_i$ is the uncertainty due to the i th systematic effect (e.g., zero instabilities of the standard, calibration instability of the standard, etc.). The uncertainty due to the randomness (noise) of the standard is best evaluated at the time of the calibration test and included as part of the uncertainty of the calibration results.

2. Uncertainty in the calibration test results

The uncertainty of the calibration results, U_{CAL} , that is, the uncertainty in the "true" pressures associated with given TCG readings, will include uncertainties arising from the calibration standard, from the calibration method used, as well as from the TCG itself. It may be estimated by combining the component uncertainties by the root-sum-of-squares method,

$$U_{CAL} = \sqrt{U_{STD}^2 + \sum_j (U_{SYS})_j^2 + U_{RDM}^2}, \quad (C2)$$

where U_{STD} is the uncertainty due to systematic effects in the standard as evaluated in Eq. (1), $(U_{SYS})_j$ is the uncertainty due to the j th systematic effect associated with the calibra-

tion method, and U_{RDM} is the combined contribution due to random errors of the standard, the TCG under test, plus other ancillary instruments (if used). Examples of uncertainties that are associated with the calibration method may include operator bias, inaccuracy of ancillary instruments such as a digital voltmeter used to read outputs from the standard and/or the TCG, etc.

The random uncertainty of the standard and that of the TCG can be evaluated individually from the standard deviation of repeated measurements at a series of stable pressures spanning the range of measurements. The total random uncertainty at each pressure is then obtained by taking the root-sum square of the component contributions.

When calibration results are represented by a least-squares fitted equation, the combined random uncertainty arising from the standard and the TCG, as well as from ancillary instruments (if used), is evaluated from the standard deviation of the predicted values of the least-squares fit.

3. Uncertainty at the time of TCG use

It is important to note that the test results are valid *only at the time of calibration* and that, *at the time of use*, additional random and systematic effects must be taken into account. The uncertainty in the measured pressure, U_P , at the time of TCG use will include not only the calibration uncertainty, U_{CAL} , but also additional uncertainties that must be combined in quadrature:

$$U_P = \sqrt{U_{CAL}^2 + \sum_k (U_{SYS})_k^2 + U_{RDM}^2 + U_{LTS}^2}, \quad (C3)$$

where $(U_{SYS})_k$ is the uncertainty due to the k th systematic effect in the measurement method (e.g., operator bias, single-factor scaling, etc.) and U_{RDM} is the uncertainty due to random errors in the TCG at the time of use, which can be evaluated from the standard deviation of repeated measurements at a stable pressure. The uncertainty due to long-term shifts in the TCG response between calibrations, U_{LTS} , is often the largest uncertainty and yet the most difficult to evaluate. It can be estimated from historical calibration data for the particular TCG, if available, and/or from the calibration histories of other similar TCGs. With or without historical data, confidence in the stability of the TCG's calibration can be improved if it is periodically compared with one or more check standards.

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