An Automated Vacuum Gauge Calibration System

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Abstract

An automated system for calibrating vacuum gauges over the pressure range of 10^{-6} to 0.1 Pa was designed and constructed at the National Institute of Standards and Technology (NIST) for the Department of Energy (DOE) Primary Standards Laboratory at Sandia National Laboratories (SNL). Calculable pressures are generated by passing a known flow of gas through an orifice of known conductance. The orifice conductance is derived from dimensional measurements and accurate flows are generated using metal capillary leaks. The expanded uncertainty (k=2) in the generated pressure is estimated to be between 1% and 4% over the calibration range. The design, calibration results, and component uncertainties will be discussed.

Introduction

Many research and development applications rely on accurate vacuum measurements in the range of 10^{-6} to 0.1 Pa (10^{-8} to 10^{-3} torr). To ensure the accuracy of measurements, many government and private agencies have established their own calibration facilities for in-house instruments. The Department of Energy (DOE) Primary Standards Laboratory at Sandia National Laboratory (SNL) maintains a calibration facility for pressure and vacuum instrumentation that serves primarily DOE facilities, but offers services to non-DOE customers as well. The calibration facility at SNL utilizes a comparison calibration system that uses hot-cathode ionization gauges\(^1\) calibrated at NIST to serve as secondary standards for vacuum gauge calibration over the range 10^{-6} to 0.1 Pa. The total expanded uncertainty\(^2\) (k=2) for a gauge calibration utilizing these transfer standards varies between 4 and 8 %. However, several factors limit the

\(^1\) Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed-Martin company, for the United States Department of Energy under contract DE-AC04-94AL85000
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performance of this calibration system and introduce unnecessary delays in gauge calibrations including: (i.) the calibration system is not automated and therefore requires constant operator attention, (ii.) the calibration system can only accommodate two or three customer gauges at a time, and (iii.) the use of transfer standards introduces undesirably large uncertainties due to the instability of hot cathode ionization gauges over time. To overcome these limitations and to reduce the uncertainty in gauge calibration, NIST has designed and constructed an automated vacuum gauge calibration system to replace the current DOE comparison calibration system. This paper describes this new, fully automated system for calibrating vacuum gauges over the pressure range of $10^{-6}$ to 0.1 Pa., presents initial calibration data, and discusses the estimated uncertainties in the generated pressure.

I. Theory of Operation and System Design

The calibration system design is based on the theory of molecular flow of gases at low pressures. The concept is to generate a calculable pressure in a vacuum chamber by pumping a known (or measured) flow of gas through an orifice of known conductance (or resistance to flow) to generate a calculable pressure drop across the orifice. This approach, known as the orifice-flow technique, is completely analogous to Ohm's law for electricity in which the voltage drop across a known resistor can be calculated by passing a known current through the resistor. Gauge calibration systems based upon the orifice-flow system concept require both a flow-generation system and a vacuum chamber which utilizes carefully fabricated orifice plates for which the gas conductance can be accurately calculated. This approach is a well-established technique and is the basis for the primary vacuum standards developed at the NIST and the PTB.

In molecular flow the mean free path of the gas molecules is assumed to be much greater than the orifice diameter, assuring that the flow of gas through the orifice is not perturbed by intermolecular collisions in the region near the orifice. Assuming that the gas molecules impinging on the orifice have a Maxwellian velocity distribution and that the pressure above the orifice is greater than the pressure below the orifice, the kinetic theory of gases gives the relation between the net flow rate of gas molecules through an orifice (in molecules/s) in terms of the number density of molecules as:

$$n = \frac{dn}{dt} = \frac{1}{4} (n_u - n_l) \bar{v} A_{\text{eff}}$$  \hspace{1cm} (1)

where $n_u$ and $n_l$ are the number densities of molecules above and below (upper and lower chamber) the orifice respectively, $\bar{v}$ is the average velocity of molecules (assumes a constant temperature throughout the chamber), and $A_{\text{eff}}$ is the effective area of the orifice plate. For an ideal orifice, $A_{\text{eff}}$ is equal to the geometric area of the orifice. Eq. (1) can be used, along with the ideal gas law, to develop the working relation for orifice-flow standards.
\[ P^o = \dot{n} \left[ \frac{2\pi mk_b T}{A_{eff}} \left( \frac{R_p}{R_p - 1} \right) \right]^{1/2} \]

where \( P^o \) is the calculated pressure (the superscript "o" indicates that molecular flow conditions are assumed) within the upper chamber, \( R_p \) is the ratio of the pressures in the upper and lower chambers, \( R_p = P_u/P_l \), \( k_b \) is Boltzmann's constant, \( T \) is the average temperature of the gas, and \( m \) is the molecular mass of the gas. In utilizing Eq. 2 for vacuum gauge calibration, the gas flow rate, the effective area of the orifice plate, the pressure ratio across the orifice, and the temperature are determined and the upper chamber pressure calculated. The calibration system design is driven by the desire to realize the conditions assumed in the derivation of the working relation Eq. (2), to maintain component uncertainties at acceptable levels, and to minimize the potential for systematic errors.

A. Vacuum Chamber and Orifice Design

The vacuum chamber design is schematically shown in Figure 1, and is similar in design to other systems in use at the NIST. The chamber is designed to maintain a low background pressure in the absence of a calibration gas, to provide a geometry that assures adequate pressure uniformity, and to provide room to mount a number of gauges for calibration.

The vacuum vessel is cylindrical, -30.5 cm in diameter, and is divided into two halves, each ~33.0 cm long, by a 1.8 cm thick plate which has been machined to accept and seal the orifice plate. In the upper chamber, which serves as the calibration volume, there are three rows of eight, 6.985 cm OD metal gasket knife-edge flanges for mounting the gauges that are to be calibrated. These mounting flanges are spaced equally around the circumference of the chamber. There are four mounting flanges on the lower chamber that may accommodate a residual gas analyzer for monitoring gas purity or other vacuum gauges for monitoring the lower chamber pressure. A gas inlet is provided at the top of the chamber, which is baffled to insure that gas molecules entering the top of the upper chamber will experience several collisions with the chamber walls before exiting through the orifice. Wall collisions are essential to insure that molecules thermally equilibrate with the vacuum vessel and that molecular velocities are randomized, assumptions implicit in Eq. (1). The uniform distribution of molecular flux is perturbed by the finite flow rate of the molecules passing through the calibration chamber. This perturbation can be kept within acceptable bounds by using an orifice whose area is a very small fraction of the total surface area of the chamber and sufficient baffling of the gas inlet to assure good uniformity of the flux of molecules to the walls of the chamber so that every calibration port sees the same flux. At the same time, the orifice conductance must be large enough to allow enough pumping speed to maintain a low background pressure and ensure that gauge pumping and out-gassing are minor perturbations.
The chamber is evacuated by a turbo-molecular pump attached to the bottom of the chamber, with a nominal pumping speed of 0.5 m³/s. The turbo-molecular pump is chosen for the stability of its pumping speed with time (as opposed to capture pumping, for example), its ability to produce a low base pressure, and its compatibility with most gases. Ultra-high vacuum construction techniques are employed throughout; the chamber is stainless steel, and only metal gasket seals are used. Internal quartz heater lamps are used for baking the chamber at temperatures up to 150 °C prior to commencing a calibration cycle.

While the use of small diameter orifice ensures good uniformity of gas flux within the upper chamber, it limits the pumping speed to the upper chamber. This places a practical limitation on the rate of evacuation of the chamber and the ultimate vacuum attainable in the upper chamber. In order to provide a high pumping speed for pump-down and bake-out, a re-usable sealing and lifting mechanism for the orifice plate was implemented. When the orifice plate is lifted, an approximately 7.5 cm diameter hole is opened between the upper and lower chamber. After bake-out, the orifice plate is replaced for gauge calibration. The orifice plate is lifted by a lever-mechanism which is driven by a UHV linear motion feed-through mounted to one of the ports in the upper chamber. The seal is made at the lip of the orifice, which is lowered into a channel located in the plate which separates the upper and lower chambers (See Fig. 1). The channel is filled with an indium-gallium eutectic that remains a liquid at room temperature and provides a hydrostatic type seal. The indium-gallium eutectic has a very low vapor pressure, much lower than the base-pressure of the system. This type of seal is utilized in the other orifice-flow systems at NIST and has been found to be a reliable and totally innocuous sealing mechanism.

The orifice itself is a sharp-edged hole approximately 1.1 cm in diameter. The outer edge of the plate includes the lip that fits into the indium-gallium-filled groove in the plate between the chamber halves. The orifice was carefully constructed so that its effective area (and hence its calculated conductance) could be calculated with high accuracy. The effective area of an orifice is the product of the geometric area of the orifice and the Clausing factor or transmission probability. The transmission probability simply represents the probability that a molecule, once it has entered the opening of the orifice, will exit. For an ideal orifice, the transmission probability is unity. For a real orifice, the transmission factor will be slightly less than unity due to the finite thickness of the orifice plate and the details of the geometry. For the orifice plate used in this system, the transmission probability was calculated to be 0.98565. This calculation is based on the approach outlined by Iczkowski et al., adapted to the specific geometry for this plate. This calculation assumes free molecular flow and the cosine law scattering at surfaces.

At 25 °C, the effective area of the orifice for nitrogen gas is 0.95945 cm² with an expanded uncertainty (k=2) of 0.071 %. The dominant component of uncertainty is the uncertainty in the calculated geometric area due to the finite accuracy of the dimensional measurements and the eccentricity of the orifice opening.
The chamber is designed so that one can valve a spinning rotor gauge (SRG) into the upper and lower chambers to measure the pressure ratio $R_p$ across the orifice. The SRG is stable and precise, and for pressures below 0.1 Pa, its response is linear with pressure.\(^{10,11}\) These attributes make the SRG ideal for measuring $R_p$, which can be done with a reproducibility of better than 1% at pressures between $10^{-4}$ and 0.1 Pa. Under molecular flow conditions, the pressure ratio $R_p$ is dependent only on the orifice conductance and pumping speed of the turbo-molecular pump. Variations in pumping speed or a leak around the orifice seal could cause significant errors in $R_p$, neither of which have been observed. We assume $R_p$ to be a system constant that does not vary with pressure.

For nitrogen gas, $R_p$ was measured to be 30.6 with an expanded uncertainty ($k=2$) of ±0.4%. This translates to an uncertainty of 0.02% in the ratio $\frac{R_p}{R_p-1}$. Errors in $R_p$ have a very small effect on the ratio $\frac{R_p}{R_p-1}$ (a one percent change in $R_p$ changes the ratio by less than 0.1%), and thus on the calculated pressure.

The average temperature of the gas molecules is determined by the average temperature of the chamber walls and is measured with a platinum resistance thermometer (PRT) having a nominal resistance of 100 Ohms. The PRT is attached to the exterior of the vacuum chamber and is used to monitor the chamber temperature. A non-uniformity in the temperature of the chamber walls and changes in the laboratory ambient temperature limit the temperature determination and introduce an additional uncertainty of 0.3 K, leading to a pressure uncertainty of 0.1% ($k=2$).

At the highest calibration pressures, the gas density increases to the point where the mean free path of the gas molecules becomes of the order of the orifice diameter. Collisions in the vicinity of the orifice opening in turn lead to small deviations from ideal molecular flow.\(^{12,13}\) The deviation from ideal molecular flow introduces a small correction to the working relation, Eq. (2). Specifically, Eq. (2) now becomes

$$P_v = P_u^\circ \left(1 - \kappa P_u^\circ\right)$$

(3)

The value of $\kappa$ depends upon the calibration gas and orifice geometry. For nitrogen gas, and the orifice plate used in this system, $\kappa$ has a value of 0.1137 Pa\(^{-1}\) with an uncertainty of ±0.1%. The deviation from molecular flow requires that an approximately 1% correction be made at the highest calibration pressure, 0.1 Pa.

**B. Flow Generation System Design**

The requirements for the flow generation system are that it be compatible with the calibration gas and generate stable and reproducible flow rates with sufficient accuracy.
and dynamic range to generate the pressures needed for the calibration system. The flow rates required for this application vary from about \(1 \times 10^{-11}\) to \(5 \times 10^{-7}\) mol/s (\(2 \times 10^{-7}\) to \(1 \times 10^{-2}\) std. cc/s assuming STP conditions of \(T = 273.15\) K and \(P = 101.325\) kPa). To generate nitrogen flow rates in this range, a flow generation system based upon the use of metal capillary leak elements was constructed. A crimped metal capillary leak can be used to generate stable flows of gas assuming that the conductance is measured as a function of fill pressure. NIST has used this method for several years to generate reliable gas flows in a number of applications\(^{14}\), and the fundamental techniques and accuracy have been established.

The flow generator is shown schematically in Figure 2. The present system employs three independently selectable crimped metal capillary leaks to cover the range of desired gas flows. The leak rates of the three crimped metal capillary tubes were calibrated as a function of upstream pressure (the downstream pressure is many orders of magnitude smaller and thus is effectively zero). The leaks were chosen so that their leak rates would overlap with one another, providing an internal consistency check. The upstream pressure is set and maintained to 0.1 % or better by a commercial pressure controller. Calibrated pressure transducers (capacitance diaphragm gauges and a quartz bourdon tube gauge) with full scale ranges of \(1.33 \times 10^3\) to \(1.33 \times 10^5\) Pa are used to measure the upstream fill pressure. An additional pressure transducer \(P_4\) having a full scale range of \(1.58 \times 10^5\) Pa is used as part of a feedback loop to the pressure controller.

The crimped capillary leak elements were calibrated using two different methods. For leak rates below \(5 \times 10^{-10}\) mol/s, measurements were made with a direct flow technique using the NIST Primary Leak Standard\(^{15}\). To calibrate leak rates greater than \(5 \times 10^{-10}\) mol/s, an orifice flow technique was used in which flow from the leak element was directed through an orifice of known conductance. The leak rate was determined by measuring the resulting pressure drop across the orifice. The expanded uncertainties (\(k = 2\)) in the leak rate calibrations range from approximately 4 % at \(1 \times 10^{-11}\) mol/s to about 0.6 % at \(5 \times 10^{-7}\) mol/s. A calibration curve for one of the crimped capillary leak elements is shown in Figure 3. The leak elements were calibrated in situ at a nominal temperature of 26 °C. Based on experience with other crimped capillary leaks of this type, the uncertainty of the leak rate as a function of temperature is estimated at ± 0.1 % per degree C away from 26 °C, though the temperature dependence was not measured directly.

II. Uncertainty in the Calculated Pressure.

The following uncertainty analysis assumes the standard propagation of errors concepts as detailed in reference 2. Combining Eqs. 2 and 3, the fractional uncertainty in the calculated pressure, \(P_u\), is found to be given by:
\[
\frac{\delta P_u}{P_u} = \left[ \left( \frac{\delta n}{n} \right)^2 + \left( \frac{\delta R_p}{R_p} \right)^2 \left( \frac{1}{R_p - 1} \right)^2 + \left( \frac{\delta A_{\text{eff}}}{A_{\text{eff}}} \right)^2 \right]^{1/2} + \frac{1}{4} \left( \frac{\delta T}{T} \right)^2 + \kappa^2 P_0 (\frac{\delta \kappa}{\kappa})^2
\]  

Eq. (4)

Table 1 summarizes the component uncertainties and the total expanded uncertainty (k=2) of the generated pressure for this calibration system calculated using Eq. (4).

<table>
<thead>
<tr>
<th>Generated Pressure (Pa)</th>
<th>Generated Pressure (Torr)</th>
<th>Fractional Component Uncertainties (k=2) (%)</th>
<th>Total Expanded Uncertainty (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>(\frac{\delta n}{n})</td>
<td>(\frac{\delta R_p}{R_p})</td>
</tr>
<tr>
<td>2.0 \times 10^{-6}</td>
<td>1.5 \times 10^{-8}</td>
<td>4.0</td>
<td>0.40</td>
</tr>
<tr>
<td>2.7 \times 10^{-6}</td>
<td>2.0 \times 10^{-8}</td>
<td>2.5</td>
<td>0.40</td>
</tr>
<tr>
<td>6.7 \times 10^{-6}</td>
<td>5.0 \times 10^{-8}</td>
<td>0.9</td>
<td>0.40</td>
</tr>
<tr>
<td>1.3 \times 10^{-5}</td>
<td>1.0 \times 10^{-7}</td>
<td>0.6</td>
<td>0.40</td>
</tr>
<tr>
<td>6.7 \times 10^{-5}</td>
<td>5.0 \times 10^{-7}</td>
<td>0.7</td>
<td>0.40</td>
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<tr>
<td>1.3 \times 10^{-5}</td>
<td>1.0 \times 10^{-6}</td>
<td>0.6</td>
<td>0.40</td>
</tr>
<tr>
<td>6.7 \times 10^{-4}</td>
<td>5.0 \times 10^{-6}</td>
<td>0.5</td>
<td>0.40</td>
</tr>
<tr>
<td>1.3 \times 10^{-4}</td>
<td>1.0 \times 10^{-5}</td>
<td>0.7</td>
<td>0.40</td>
</tr>
<tr>
<td>6.7 \times 10^{-3}</td>
<td>5.0 \times 10^{-4}</td>
<td>0.7</td>
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</tr>
<tr>
<td>1.3 \times 10^{-1}</td>
<td>1.0 \times 10^{-3}</td>
<td>0.7</td>
<td>0.40</td>
</tr>
</tbody>
</table>

The total system uncertainties represent a factor of two improvement over the uncertainties of the comparison calibration system currently in use.

**III. System Automation.**

Calibration costs are a function of the amount of time in which an operator is directly involved with a calibration, so it is highly desirable to minimize the time an operator spends in the data taking process. All calibration system controls and components are accessible via computer (IEEE-488 or RS-232) interfaces, including measurement of the temperatures and pressures necessary for calculating flow rates and upper chamber pressures, and all valve controls. The chamber baking is achieved using quartz heater lamps and a programmable temperature controller. These provisions enable the entire
calibration process to be automated through a computer program. After baking the operator needs only to provide a list of desired calibration pressures to the control software, and the program calculates the necessary flow rates and the nitrogen fill pressures needed to generate them. The pressure controller sets and maintains the necessary fill pressures; an automatic routine monitors the fill pressure for stability, and a suitable waiting period is allowed before calibration data taking can begin. The only operator involvement required is to manually re-zero the fill pressure gauges and to ensure that a suitable base pressure has been attained. About four hours are required to generate pressures for an entire calibration sequence covering four pressure decades at five calibration points per decade (total of 20 calibration points).

IV. Results.

Experiments were undertaken to check the accuracy of this standard by using a spinning rotor gauge previously calibrated on the NIST primary high vacuum standard. The accommodation coefficient of the rotor measured on the two systems agreed within the combined uncertainties of the two standards at the k=1 level. As a further check for systematic errors, the accommodation coefficient obtained for the rotor using the flow generation system was compared to that measured using one of the NIST primary flow meters. Again, good agreement at the k=1 level was found. Together, these tests give assurance that the system does not have any significant systematic errors.

Another critical test of this calibration system involved calibrating a hot cathode ionization gauge that had been previously calibrated against NIST Primary Standards. The results of the test calibration are shown in Figure 4. This figure plots the gauge correction factor as a function of pressure displayed on the ionization gauge controller. The correction factor $CF$ is defined to be the ratio of the calculated pressure, $P_c$, (using Eqs. 2 and 2) to the displayed gauge reading, $P_{\text{display}}$, corrected for the displayed reading at base vacuum, $P_{\text{base}}$:

$$
CF = \frac{P_c}{P_{\text{display}} - P_{\text{base}}} 
$$

Also shown in Fig. 4 are the expanded uncertainties in the measured correction factor, at the k=1 level. The data taken with the automated calibration system shows excellent agreement with the NIST calibration data (no uncertainty due to drift in the gauge’s calibration was assumed). This level of agreement over the entire range of calibration pressures give us confidence that no serious systematic errors are present in the automated calibration system.

Only one calibration point, taken at a pressure of $8.5 \times 10^{-4}$ Pa with the automated system, fails to overlap the NIST data at the k = 2 level. This data point may encompass a small
error in the calibration of the leak element used to produce this calibration pressure. Further testing of the system is currently underway to characterize and correct this error, which is unaccounted for in the uncertainty budget of Table 1.

V. Conclusions.

The automated vacuum gauge calibration system developed at NIST for DOE transfers technology implemented in NIST standards to meet the measurement needs of the SNL vacuum gauge calibration facility. The system uses the orifice flow technique to generate stable nitrogen calibration pressures in the range of $10^6$ to 0.1 Pa. Calculation of calibration pressures using the orifice flow technique relies on knowing the flow rate of gas that passes through an orifice whose effective area is known. The orifice has been carefully constructed and accurately measured so that its effective area can be calculated using established methods. The flow generator relies on the stability of flow rates produced by metal capillary leaks. The calibrations of the flow generator leak elements will need to be checked periodically as the uncertainty in the generated flow of gas drives the uncertainty of the calibration pressure calculation. This can be done in-situ as the elements were chosen so that part of the range of leak rates generated by each element would overlap with the range of at least one other leak element. When necessary, the pressure transducers that are used to measure the backing pressure of the leaks will need to be recalibrated.

The new system reduces customer turn-around time, lowers the calibration uncertainty by a factor of two over the existing system, and lowers costs by reducing operator time in the calibration process. Initial testing of a calibrated spinning rotor gauge and a calibrated hot-cathode gauge has shown that no serious systematic errors exist in the new calibration system. Further testing is currently in progress at SNL.
References


**Figure 1.** Vacuum chamber for the orifice-flow calibration system. For clarity, only two of the gauge mounting ports are shown in the upper chamber and one shown in the lower chamber. The orifice plate can be lifted by a lever mechanism (not shown). Stainless steel tubing connects the vacuum chamber to the flow generator.
Figure 2. Flow generator for orifice-flow calibration system. P1-P3 are calibrated pressure transducers for measuring leak fill pressure. P4 provides feedback to the pressure controller. The reference vacuum for the pressure controller is provided by a separate rotary vane pump (not shown). All connecting lines are stainless-steel. The flow generator is completely automated.
Figure 3a. Calibration curve for one of the metal capillary leak elements contained in the flow generator. A third order polynomial was used to fit the data.

Figure 3b. Residuals (100% x [data-fit]/data) for analytical fit to capillary leak calibration data shown in Figure 3a.
Figure 4. Comparison of the results of ionization gauge calibrations performed at NIST and with the automated DOE system. The error bars indicate the uncertainty of each calibration data point at the $k=1$ level. Error bars for the NIST calibration data are approximately the width of the symbols.