# A primary standard high vacuum calibration station for industrial applications

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The Metrology Laboratory at Lockheed Martin Missiles & Space in Sunnyvale, CA, has developed a primary standard high vacuum calibration station. Maintaining an in-house capability for calibrating Bayard–Alpert gauge tubes and spinning rotor gauges (SRGs) at the primary level eliminates the need for external sources of calibration. This not only significantly reduces costs but also provides for continuous monitoring of the reference standards used to support research programs and manufacturing processes. A constant volume, pressure drop flowmeter is used to generate known pressures in a vacuum chamber partitioned by an orifice of calculable conductance. Data are presented which show vacuum chamber pressure to be directly proportional to flowmeter pressure drop in the three decades of vacuum spanned by the SRGs ($10^{-3}$–$10^{-6}$ Torr). Using a SRG calibrated at the National Institute of Standards and Technology, the proportionality constant is determined and shown to agree well with the constant calculated from system fixed parameters and measured pressure ratios. A new design of flowmeter is discussed. An estimate of when the system will be fully utilized as a primary reference will be made. © *1996 American Vacuum Society.*

## I. INTRODUCTION

The Lockheed Martin Missiles & Space Metrology Laboratory maintains a broad base of primary calibration standards in support of manufacturing and testing throughout the corporation. Commercial services are also offered. Vacuum gauge calibration comprises a substantial portion of the workload. As such, a program was undertaken to develop a standalone capability for the calibration of high vacuum gauges in order to improve accuracy and reliability and to lower costs.[1] The development phase of the program verified the system function and established operating parameters and procedures. The current work was focused on creating a true primary calibration capability and estimating the magnitude of uncertainty inherent to the system.

## II. SYSTEM OVERVIEW

An ultrahigh vacuum calibration chamber is constructed from two 12-in.-diam cylinders of 304 stainless steel, 15 in. long connected by a 14 in. Conflat-type flange. This flange is machined to accept a specially designed orifice plate. The section of the chamber above the flange provides measurement ports for 10 gauges and access to the orifice plate, which is demountable. A pumping bypass connects the upper to the lower section through gate valves to allow for rapid pumpdown of the entire chamber. The lower chamber has four ports for the mounting of gauges. The entire chamber is evacuated using a twin opposed rotor, turbomolecular pump of 510 ℓ/s pumping speed. Figure 1 shows details of the vacuum chamber.

The orifice is machined into a 3.75-in.-diam stainless steel plate 0.5 in. thick. The orifice geometry is based on a National Institute of Standards and Technology (NIST) design.[3] Given molecular flow conditions and constant pumping speed, the conductance of the orifice can be calculated based on its measured dimensions, by applying the work of Clausing.[2] A pressure drop will develop across the orifice for gas flowing through it from the upper to the lower chamber. The value of the drop depends only upon the flow rate and the calculated conductance:

$$P_a - P_b = Q/C, \tag{1}$$

where $P_a$=pressure above orifice (upper chamber), $P_b$=pressure below orifice (lower chamber), $Q$=flow rate, and $C$=conductance.

As such, the overall molecular distribution for a particular gas flowing through the chamber is strictly defined by the orifice conductance. Thus, if steady flow conditions are achieved, the ratio of upper to lower chamber pressures should remain constant regardless of flow rate or absolute pressure, provided the conditions for molecular flow are met and that the pumping speed remains constant. Therefore, the pressure in the upper chamber can be calculated directly from the flow rate once the pressure ratio has been determined:

$$P_a = QR/C(R-1), \tag{2}$$

where $R = P_a/P_b$.

A precision gas flowmeter supplies ultrahigh purity (99.9997%) nitrogen gas to the upper chamber through an adjustable leak valve. The pressure drop in the supply volume is monitored as a function of time. The flow rate is the product of gas supply volume and the rate of pressure drop. The flow rate can also be expressed as the product of leak valve conductance and instantaneous supply volume pressure, such that

$$Q = VdP/dt = -C_L P \quad (P \gg P_a), \tag{3}$$

where $V$=supply volume, $P$=supply volume pressure, and $C_L$=leak valve conductance.

Therefore supply volume pressure can be represented as

$$P = P_0 \exp(-C_L/V)t, \tag{4}$$

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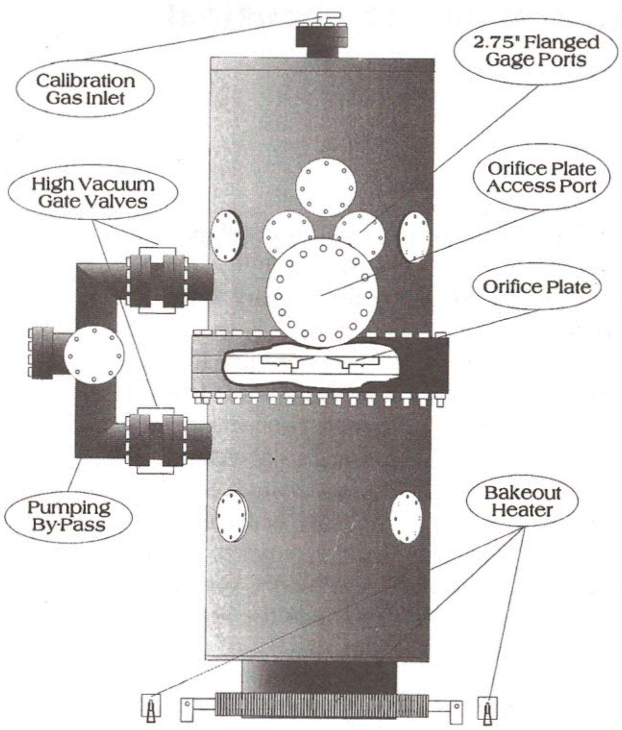


FIG. 1. Ultrahigh vacuum calibration chamber.

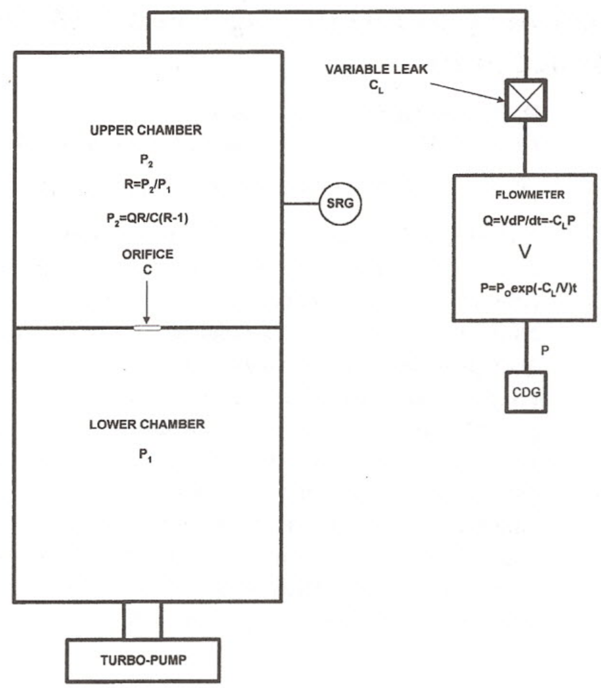


FIG. 2. System schematic.

where $P_0$=supply volume initial pressure and $t$=elapsed time.

Expanding the exponential in a Taylor series to the quadratic term:

$$P=P_0\{1-(C_L/V)t+[(C_L/V)t]^2/2!\}. \quad (5)$$

If $P/P_0 \leqslant 0.9875$ during the time $t$, which corresponds to a 1.25% drop in supply pressure, the quadratic term is 0.63% of the linear term, higher terms in the exponential can therefore be neglected. Upper chamber pressure is directly proportional to flow rate [Eq. (2)], thus the average rate of supply pressure drop is directly proportional to the mean of the upper chamber pressure during the measurement interval to the extent that the supply pressure drop is linear in time (0.63%). The proportionality constant is dependent upon system fixed parameters only and does not change as a function of flow rate. Thus, the relationship between flow rate and upper chamber pressure is established empirically. The constant is also calculated from orifice conductance, flowmeter volume, and the ratio of upper to lower chamber pressures. Figure 2 is a schematic representation of the relationship between the flowmeter and vacuum chamber.

## III. INSTRUMENTATION AND DATA ACQUISITION

Pressure in the flowmeter volume is measured using capacitance diaphragm gauges (CDGs). CDG heads of 100 or 1000 Torr full scale absolute are used depending upon the flow rate. Supply pressures and valve openings which maintain low leak valve conductance were determined during the development phase. Upper chamber pressures are measured using spinning rotor gauges (SRGs). Initial measurements were made using a NIST calibrated SRG in an effort to establish the proportionality constant empirically.

Data acquisition software is designed to monitor two SRGs and three CDG heads sequentially over the IEEE-488 bus of an HP 9000 series instrument controller. SRG integration time is adjusted according to the level of pressure being measured. Pressures to $5\times10^{-5}$ Torr require integration times of 30 s and use an interval of 2000 s. Pressures above $5\times10^{-5}$ and below $5\times10^{-4}$ Torr utilize integration times of 15 s with an interval of 1000 s; above $5\times10^{-4}$ integration times of 8 s are used with measurement intervals of 500 s. The parameter values above were determined to ensure that the supply pressure drop does not exceed 1.25% during the measurement interval and that a statistically significant amount of data is acquired.

## IV. CALIBRATION OF SRGs

The SRG consists of a magnetically suspended steel ball of roughly 4.5 mm diameter which is spun up and allowed to coast, the rotational frequency is maintained to within 405–415 Hz. Gas molecules impact the rotor causing it to decelerate. In addition to gas drag, the interaction of suspension and monitoring fields also decelerate the rotor; this is known as the residual drag. By monitoring the deceleration rate relative to the rotational frequency the pressure is calculated from the following formula:

$$P=(X_\rho/\sigma)\{[(d\omega/dt)/\omega]-\mathrm{RD}\}, \quad (6)$$

where $\omega$ is the rotational frequency of the rotor, $[(d\omega/dt)/\omega]$ is the relative deceleration, RD is the residual drag, $X_\rho$ is a constant determined from the density and diameter of the rotor, and $\sigma$ is the coupling constant between the gas and

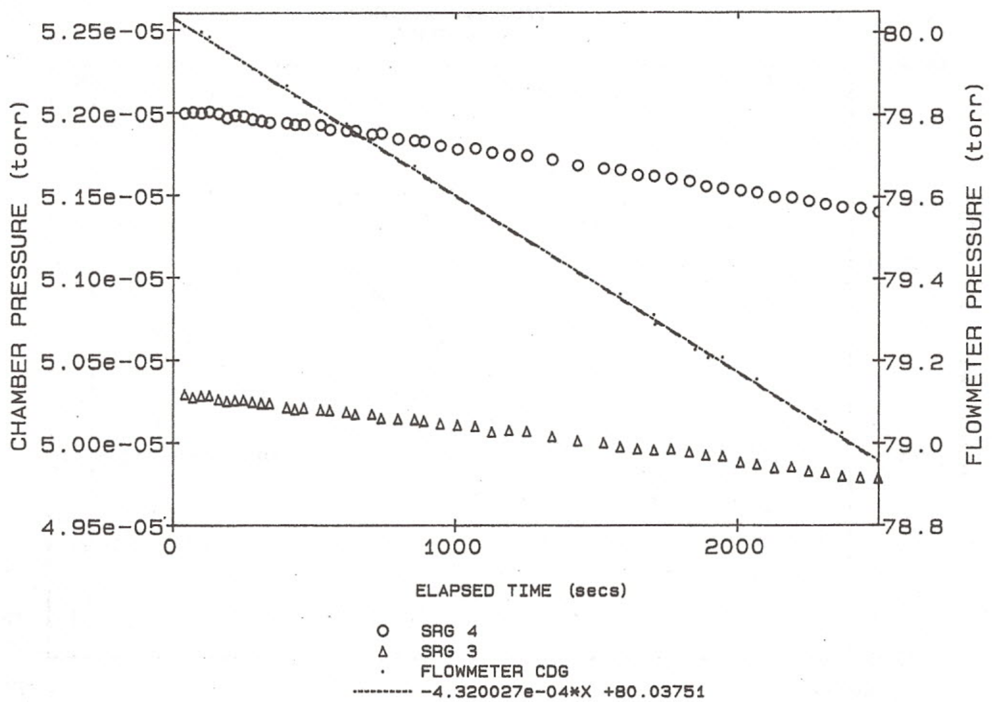


FIG. 3. Typical graph of data.

rotor.[3] Once RD is determined and the mass and diameter of the rotor are measured SRG calibration is simply the determination of $\sigma$.

The residual drag constitutes an offset in the pressure measurement. It is dependent upon spin frequency. In order to correct for the offset, each gauge is monitored at the chamber base pressure for several days and a fit of residual drag versus frequency is made. During the course of actual pressure measurements the spin frequency is recorded and when measurements are completed the offset is automatically subtracted based on the curve fit of residual drag versus frequency.

Four SRGs were utilized in this work, one of which was calibrated at NIST. The NIST gauge was used to establish the relationship between flowmeter pressure drop and upper chamber pressure. Measurements were made at upper chamber pressures from $1\times10^{-6}$ to $1\times10^{-3}$ Torr. Flowmeter and indicated upper chamber pressure were graphed versus time (see Fig. 3). A straight line fit was made to the flowmeter CDG data to yield a value of $\Delta P/\Delta t$. Mean upper chamber pressure during the measurement interval was determined from the SRG data. At least five points per decade were obtained in this way. Mean upper chamber pressure within the measurement interval ($\Delta t$) was graphed versus $\Delta P/\Delta t$ (Fig. 4) for each point in the three decades of pressure measured to obtain the overall system proportionality constant $K_{cal}$, where

$$P_{cal}=K_{cal}\Delta P/\Delta t. \tag{7}$$

Once a value of $K_{cal}$ was established, a measurement sequence was initiated wherein all four gauges were measured two at a time. The diameter and mass of each of the three unknown rotors were determined and $\sigma$ set to one. A previously measured gauge was used to set upper chamber pressures by incorporating an average offset value. The pressures indicated by the SRGs were graphed versus $\Delta P/\Delta t$ in each of the calibration sequences and a value of $K_{unk}$ obtained as for the calibrated gauge. The only variable to be determined in SRG calibration is $\sigma$. Therefore, the ratio $K_{unk}/K_{cal}$ can be taken as $\sigma$ of the unknown provided that $\Delta P/\Delta t$ truly defines upper chamber pressure.

## V. DATA AND ANALYSIS

The four SRGs were denoted as $SRG_1$, $SRG_2$, $SRG_3$, $SRG_4$, with the last one holding a recent NIST calibration. Data were first obtained for $SRG_4$ and $SRG_3$ vs $\Delta P/\Delta t$. The values of $K_{unk1}$ and $K_{unk2}$ as determined for these two gauges are shown in Fig. 4. Using the value of $K_{unk1}$ for $SRG_4$ as $K_{cal}$ (0.1203), a value of $K_{unk}$ was obtained for $SRG_1$ and $SRG_2$ by monitoring them simultaneously vs $\Delta P/\Delta t$. In this way a $\sigma$ value was assigned to each of the three unknown gauges. This constituted a "calibration" of the unknown gauges based on the system constant $K_{cal}$ as determined by the NIST calibrated rotor.

The heart of the calibration system is the flowmeter. Figure 2 is a schematic representation of the system showing the relationship of flowmeter pressure drop to upper chamber pressure. Pressures become completely defined provided rates of pressure drop can be accurately determined. Thus, evaluating the reproducibility of the system constant using the gauges as calibrated above is a measure of flowmeter accuracy as all other system parameters are fixed. As such, all four gauges were compared to each other in combinations

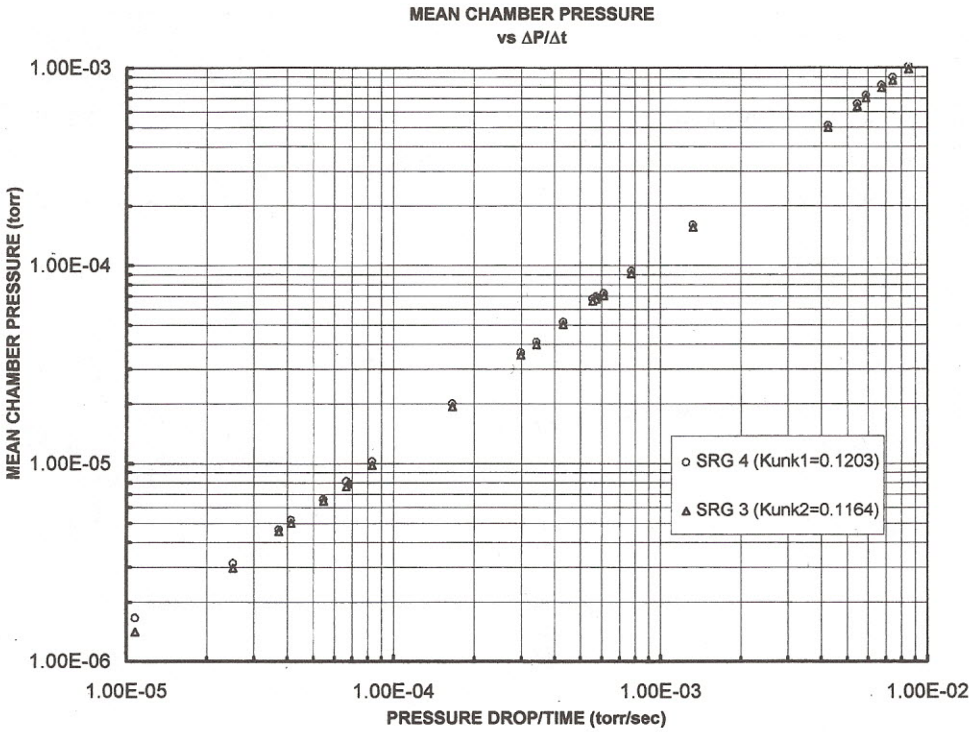


FIG. 4. Determination of system constant.

other than those used in establishing the initial calibration. Table I summarizes the data obtained for all gauges.

Rows 1 and 2 show the $\sigma$ values established for $SRG_1$, $SRG_2$, and $SRG_3$ based on the initial value of $K_{cal}$ ($K_{unk1}$ for $SRG_4$) determined in run 1. Rows 3–6 show values of the system constant $K_{cal}$ based on SRG vs $\Delta P/\Delta t$ measurements and the $\sigma$ values displayed in rows 1 and 2.

The mean of all values for the system constant obtained using calibrated rotors is 0.120 25 with a standard deviation of 0.73%. The overall variation as listed in rows 3–6, is about 2.25% compared to this value. The largest variation seen in an individual gauge is 1.5% for $SRG_4$. The smallest variation is 0.5% for $SRG_2$. There are two manufacturers of SRGs, two from each were used in this work. One specifies $\pm 1\%$ for the accuracy and the other $\pm 1.5\%$. The reproducibility is excellent and well within what can be expected of the gauges. In essence, flowmeter measurements were used to transfer the calibration from the NIST rotor to the other three. Thus it appears that $\Delta P/\Delta t$ measurements are sufficient for determining calibration chamber pressures in the current system configuration.

All that remains is to establish the system constant absolutely by virtue of orifice conductance calculations and flowmeter volume measurements.

TABLE I. Data for determining system constant.

| Run No. | Gauge 1 | Gauge 2 | $K_{unk1}$ | $K_{unk2}$ | $\sigma_1$ | $\sigma_2$ |
|---|---|---|---|---|---|---|
| 1 | $SRG_4$ | $SRG_3$ | 0.1203[a] | 0.1164 | 0.980[a] | 0.967 |
| 2 | $SRG_2$ | $SRG_1$ | 0.1154 | 0.1165 | 0.960 | 0.969 |
| 3 | $SRG_1$ | $SRG_3$ | 0.1201 | 0.1202 | 0.969 | 0.967 |
| 4 | $SRG_4$ | $SRG_2$ | 0.1210 | 0.120 | 0.980[a] | 0.960 |
| 5 | $SRG_2$ | $SRG_3$ | 0.1206 | 0.1197 | 0.960 | 0.967 |
| 6 | $SRG_4$ | $SRG_1$ | 0.1185 | 0.1209 | 0.980[a] | 0.969 |

[a]Value based on NIST calibration.

## VI. CALCULATION OF SYSTEM CONSTANT

The volume of the flowmeter was determined using a stainless cylinder with a volume of 0.969 ℓ. The cylinder was calibrated to 0.5% traceable to NIST. It was connected through a shut-off valve to the flowmeter output. The entire system was evacuated and the calibrated volume valved off. The flowmeter volume was then backfilled with ultrapure nitrogen to a pressure of approximately 1 Torr and the change in pressure recorded. The shut-off valve was opened and the pressure allowed to equalize. The final pressure was noted such that the change in pressure due to the added volume could be determined. The ratio of the two pressure differences and the value of the calibrated volume were than used to calculate flowmeter volume. This process was repeated in steps to 1200 Torr. The process was then reversed by pumping the flowmeter volume incrementally and then allowing equalization to take place. These measurements yielded a flowmeter volume of 1.2516 ℓ with a standard deviation of 0.26%.

Using Eqs. (2) and (3), it can be seen that

$$K_{cal} = VR/(R-1)C, \tag{8}$$

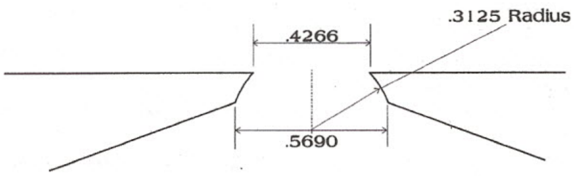


FIG. 5. Detail of calibration orifice.

where $Q=V\Delta P/\Delta t$, $C$=conductance, and $V$=flowmeter volume.

The orifice conductance for nitrogen is calculated through the use of Clausing factors and the aperture radius and can be written as

$$L=\Pi r_1^2 W K_1 K_2 c/4, \quad (9)^3$$

where $W$=Clausing factor for the orifice used (0.9971), $K_1=1$, $K_2$=correction factor for 70° conical surface below the spherical surface (0.9992), $c$=mean molecular velocity for nitrogen at 294.3 K, and $r_1$=radius of orifice entrance aperture (0.2133 in.).

This yields an orifice conductance ($C$) of 10.86 ℓ/s. The pressure ratio ($R$) across the orifice has previously been determined to be 20.2 (see Ref. 1). Given these values $K_{cal}$ is calculated to be 0.12125. The value obtained empirically is 0.1203. The difference between the calculated and measured values of the system constant is 0.8% with respect to the calculated value. Thus, it appears that the flowmeter measurements are accurate. Given the proper conditions, it is possible to generate known pressures in a vacuum chamber which are directly proportional to the rate of pressure drop in a fixed volume. Figure 5 shows the details of the LMMS orifice.

## VII. ESTIMATING SYSTEM UNCERTAINTY

There are several sources of uncertainty in the Primary Vacuum Calibration Station. Uncertainties are evaluated according to the guidelines adopted by NIST[4] and are classified into two categories: type "A" and type "B." Type A uncertainties are those which are evaluated by statistical methods and type B are those evaluated by other means. The rate of pressure drop in the flowmeter is basically exponential as described above. Parameters are chosen in such a way as to ensure that the linear term in the exponential dominates. The only type A uncertainty arises from the extent to which the rate of pressure drop is not linear—from Sec. II this can be seen to be 0.63%. The other components of uncertainty derive from the parameters used to calculate the pressure: the orifice conductance calculation (dimensional), flowmeter volume and pressure drop, the measured pressure ratio, and gas temperature. These components are all considered type B as details of their distributions are not known.

The orifice conductance is thought to have no greater than a 0.2% uncertainty related to dimensional measurements; the cylinder used to calibrate the flowmeter volume has an uncertainty of 0.5%. The pressure ratio uncertainty is 0.15%, as measured. Ambient temperature within the laboratory was observed to vary by no more than ±0.75 °C or 0.25% throughout the course of the measurement program. The effect is manifested directly as a variation in flowmeter supply volume gas pressure. This is the worst case as ambient temperature drift monitored during the longest measurement intervals (2000 s) was typically less than ±50 mK. Table II lists the source, type, and magnitude of the uncertainty components and combines the type A and total B in quadrature. The type B uncertainties are normalized to a rectangular distribution as only the limits of the distribution are known.

TABLE II. System uncertainties.

| Source | Type | Combined as | Magnitude |
|---|---|---|---|
| Flowmeter $\Delta P/\Delta t$ | A | A | 0.63% |
| Orifice conductance | $B_1$ | $B_1/\sqrt{3}$ | 0.12% |
| Flowmeter volume | $B_2$ | $B_2/\sqrt{3}$ | 0.29% |
| Pressure ratio | $B_3$ | $B_3/\sqrt{3}$ | 0.09% |
| Temperature | $B_4$ | $B_4/\sqrt{3}$ | 0.14% |
| Total | U | $\sqrt{A^2+\Sigma(B_i/\sqrt{3})^2}$ | 0.72% |
| | $U_T=kU$ $(k=2)$ | | 1.44% |

A coverage factor of $k=2^4$ is used to define the overall uncertainty in the generated pressure as 1.44%. The largest contributor is the departure from linearity of the flow rate as a function of time. The standard deviation of the system constant as measured and the difference between the calculated and measured system constant fall well within the estimated overall uncertainty.

## VIII. CONCLUSIONS AND FUTURE PLANS

A facility for the primary calibration of vacuum gauges in the range from $10^{-3}$ to $10^{-6}$ Torr has been created and verified. Known pressures are generated in a vacuum chamber which are directly proportional to the measured pressure drop in a fixed volume flowmeter. The proportionality constant determined empirically using a gauge calibrated by NIST is in excellent agreement with that determined by virtue of dimensional, volumetric, differential pressure, and pressure ratio measurements only. The overall estimated uncertainty is ±1.44% due mainly to departures from linearity in the determination of flow rate. This work has established the calibration of three SRGs both absolutely and by comparison to a NIST calibrated gauge. As such, these gauges can now be used as reference standards from $10^{-3}$ to $10^{-6}$ Torr.

A new flowmeter has been designed and is currently under construction. It will experience smaller pressure drops for the same flowrates and provide higher resolution of $\Delta P/\Delta t$. This should reduce the contribution from nonlinear terms in the flow rate, thus reducing the uncertainty in the generated pressure. The next generation flowmeter should also allow for the extension of calibration to the $10^{-7}$ and $10^{-8}$ Torr range using Bayard–Alpert type ion gauge tubes. Integration and verification should be completed within the next year making the Lockheed Martin Missiles & Space Primary Vacuum Calibration Station fully operational from $10^{-3}$ to $10^{-8}$ Torr.

[1]P. Clausing, Ann. Phys. **12**, 961 (1932); English translation appears in J. Vac. Sci. Technol. **8**, 636 (1971).
[2]S. Dittmann, "High Vacuum Standard and its Use," NIST Special Publication 250-34, March 1989
[3]R. P. Iczkowski, J. L. Margrave, and S. M. Robinson, J. Phys. Chem. **67**, 229 (1963).
[4]B. N. Taylor and C. E. Kuyatt, NIST Technical Note 1297, January 1993.