

Calibration of molecular drag vacuum gages

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In spinning-ball molecular drag gages, the logarithm of the rotational frequency varies linearly with the time integral of the applied pressure under conditions of free molecular motion. An orifice flow standard for calibrating these devices has been developed that passes a measured quantity of gas through the calibration chamber in typically 30 min. The time integral of the calibration pressure can be calculated with an estimated uncertainty less than 1%. A number of gage balls operated in conjunction with three controllers have been calibrated for N₂, Ar, and He using this system. Conditions and procedures affecting the performance characteristics of these gages have been investigated, including magnetization of the ball, baking and other conditioning of its surface, and degradation of gas purity by surface contaminants in an unbaked flow system.

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I. INTRODUCTION

In a molecular drag gage (MDG) employing active, permanent-magnet suspension of a rotating steel ball,^{1,2} the rotational frequency decreases as the result of two causes: momentum transfer to molecules colliding with the ball (molecular drag), and losses ascribed to eddy currents induced in the gage head by the rotating component of the ball's magnetic moment (residual drag). Experiments have shown that pressures can be determined from measurements of the rotational frequency and its rate of decrease, but such devices require calibration because the details of momentum transfer are not predictable from first principles. For smooth bearing balls, measurements of σ , the macroscopic coefficient of tangential momentum transfer, have yielded similar values within the range 1 ± 0.05 for He, N₂, and Ar.¹ More detailed comparisons with a primary standard over a three year period have demonstrated reproducibility within $\pm 1\%$ for this MDG type as a transfer standard in the range 10^{-3} to 1 Pa.^{2,3} The orifice flow method described in this paper has been developed in order to calibrate such gages for transfer standard application. This calibration procedure differs from the more conventional primary standards of pressure in the high vacuum range⁴ in that it is based on a standard of impulse per unit area, i.e., it provides a fundamentally calculable value for the time integral of the calibration pressure generated by the flow of gas through the system. The following considerations indicate why such a standard is particularly appropriate for MDG calibration.

Under conditions of free molecular motion the deceleration of the ball is related to the pressure P of a single-component calibration gas by the equation⁵

$$P = -(\dot{f}/f + c_1)K, \quad (1)$$

where f is the rotational frequency with time derivative \dot{f} , c_1 is the offset drag equal to the value of $-\dot{f}/f$ at base vacuum and consisting of the residual drag plus the much smaller drag exerted by background gas molecules, and K is the cali-

bration factor. Although the equations in Ref. 5 do not explicitly display the constant c_1 , its inclusion in Eq. (1) is required for all currently available spinning ball MD gages. The calibration factor is related to properties of the ball and gas by the equation⁵

$$\sigma K = \pi \rho a \bar{c} / 10, \quad (2)$$

where ρ denotes the density of the ball and a its radius, and \bar{c} is the mean molecular velocity.

Integration of Eq. (1) between time limits t_1 and t_2 can be expressed in the form

$$\int_{t_1}^{t_2} P dt = JK, \quad (3)$$

where

$$J = \ln(f_1/f_2) - c_1(t_2 - t_1), \quad (4)$$

and f_1 and f_2 are the rotational frequencies at t_1 and t_2 , respectively. All quantities on the right-hand side of Eq. (4) are readily computed from MDG output data. An impulse-per-unit area standard provides a calculable value for the integral in Eq. (3).

Sometimes by design and sometimes by mishap in the course of this work a number of procedures and conditions have been observed to alter the performance characteristics of MD gages. Although these results in no way conflict with the demonstrated reliability of the MDG as a transfer standard where carefully controlled conditions are maintained, it is important to recognize less favorable circumstances that might necessitate recalibration. Accordingly, observations of adverse effects on MDG reliability are reported in a subsequent section.

II. CALIBRATION APPARATUS, PRINCIPLE, AND PROCEDURES

A schematic diagram of the apparatus is shown in Fig. 1. A calibrated metering volume V_0 initially isolates a measured quantity of gas. The subsequent flow of this gas through the

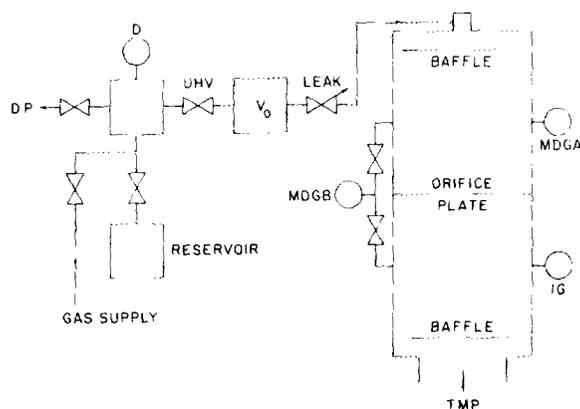


FIG. 1. Schematic diagram of the calibration apparatus. V_0 is a calibrated metering volume, which is filled to a pressure P_0 at temperature T_0 . The system is equipped with a calibrated capacitance diaphragm gage D , a Bayard-Alpert ion gage IG , and a molecular drag gage $MDGB$, in addition to $MDGA$, the molecular drag gage to be calibrated. Trapped mercury diffusion pump DP and turbomolecular pump TMP are indicated. The flow system beyond and including the ultrahigh vacuum (UHV) valve is of all-metal construction.

calibration chamber, from the baffled inlet at the top to the baffled turbomolecular pump at the bottom, is controlled with the variable leak valve shown in the figure. The chamber, which was designed as part of an orifice flow primary standard, is described more fully elsewhere.⁶ Briefly, it consists of an upper chamber separated from a lower chamber by an orifice plate, with an external bypass connecting a gage port on the upper chamber to a gage port on the lower chamber. The gage to be calibrated $MDGA$ is mounted on the upper chamber. A second MD gage $MDGB$ situated between two valves in the bypass assembly, can be valved to either chamber to provide auxiliary data required for a calibration. System pressures are monitored with the Bayard-Alpert ion gage on the lower chamber. The essential constants of the apparatus are the gas metering volume $V_0 = 128.3$ ml, and L the free molecule conductance of the orifice between upper and lower chambers. Values of L are listed in Table I for N_2 , Ar, and He. These conductances were calculated *ab initio* for the particular orifice geometry by an extension of Clausing's method.⁷ Effective speeds of exhaust from the lower chamber, by means of a baffled turbomolecular pump, are also listed in Table I. The base pressure in the upper chamber is typically 2×10^{-7} Pa.

A complete calibration consists of two procedures. In the first the ratio $R = K_B/K_A$ of the calibration factors for the two gages $MDGB$ and $MDGA$ (the gage to be calibrated on the upper chamber), is determined from the ratio of their simultaneous responses to a pressure generated by steady flow, with $MDGB$ valved to the upper chamber.

In the second procedure the metering volume is filled with calibrating gas to a pressure P_0 at temperature T_0 . This gas is then exhausted through the calibration chamber at temperature T_A , with $MDGB$ valved to the lower chamber. The time integral of the throughput at T_A is equal to $P_0 V_0 T_A / T_0$. Orifice flow theory equates this integral to the time integral of $L(P_A - P_B)$, where $P_A - P_B$ is the pressure difference

TABLE I. Calculated orifice conductance L at 25 °C; effective speed of exhaust S from the lower chamber; nominal calibration pressure P_{nom} ; and fill pressure P_0 , for N_2 , Ar, and He.

	L (l/s)	S (l/s)	P_{nom} (Pa)	P_0 (Pa)
N_2	11.68	300	4.6×10^{-3}	667
Ar	9.78	275	4.9×10^{-3}	600
He	30.89	425	1.4×10^{-2}	5000

across the orifice. The latter integral can be equated on the basis of Eq. (3) to $L(J_A K_A - J_B K_B)$, where J_A and J_B are computed according to Eq. (4) from the data obtained in this procedure from $MDGA$ and $MDGB$, respectively. Finally, using the results of the first procedure,

$$K_A L (J_A - R J_B) = P_0 V_0 T_A / T_0. \quad (5)$$

Although K_A can be obtained from Eq. (5), it is more convenient to compute results at a common reference temperature. If $T^* = 298.15$ K is taken as reference temperature, and K_A and L have reference values K^* and L^* at T^* , then from their temperature dependence

$$K^* L^* (J_A - R J_B) = P_0 V_0 T^* / T_0. \quad (6)$$

The temperature T_A does not appear in Eq. (6). It affects K^* only to the extent that spatial nonuniformity of the chamber temperature affects the pressure distribution.

It should be noted that $K_B = R K_A$, hence both gages are calibrated by carrying out these two procedures. With slight added uncertainty the first procedure can be omitted if $MDGB$ is already calibrated. To the extent that turbomolecular pumping speeds remain constant, the ratio $R J_B / J_A$ is a species-dependent constant of the system (the lower chamber: upper chamber pressure ratio). If this ratio has been measured, further use of $MDGB$ can be omitted, with the additional uncertainty in K^* not exceeding a few tenths of 1%.

The fill pressure P_0 is measured with a calibrated capacitance diaphragm gage shown in Fig. 1 as D . The effect of the slight (~ 50 ppm) compression of the gas in the metering volume upon closing the UHV valve is included in the calibration of V_0 . The adjustable all-metal leak valve is controlled manually. A typical time of 30 min suffices to exhaust calibrating gas from V_0 until the residual pressure is negligible compared with the uncertainty in P_0 . The flow rate is adjusted to hold the calibration pressure constant within $\pm 10\%$, apart from rise and fall times, as monitored indirectly with the ion gage on the lower chamber. Nominal calibration pressures, calculated on the basis of theory⁸ to limit those uncertainties ascribed to deviations from free molecular motion to less than 0.1%, are listed in Table I. Also in Table I are fill pressures for total exhaust in 30 min. The higher calibration pressures allowed for lighter gases largely compensate for the lower MDG sensitivities to these gases. Corrections for nonlinear MDG response are less than 0.1% to pressures approximately one decade above these nominal calibration pressures.⁹ The reason lies in the fact that fractional nonlinearity caused by departure from free molecule

conditions is 8–10 times greater for the orifice flow than for the gage response.

A. Uncertainties

Component random uncertainties of 0.1% are assigned to each of P_0 , T_0 , and the effect of residual drag variations on the quantity $J_A - RJ_B$. Limits of systematic errors are estimated as 0.1% for the calibration of V_0 , 0.2% for the orifice conductance, 0.2% for the deviations from pressures calculated for infinite chambers, and 0.2% for the effect of spatial nonuniformity of the chamber temperature. From these components an overall uncertainty of less than 1% is estimated for the calibration procedure. This calibration method avoids uncertainties associated with measuring and maintaining constant flow as is required in an orifice flow pressure standard. Observations indicate that uncertainties from adsorption and desorption in the clean, well-baked chamber and from gas uptake by ion gages are negligible. This result is reasonable, considering that this procedure employs several hundred times as much gas as does a final chamber of the same size in a series expansion apparatus at the same calibration pressure.⁴ Results of calibrations performed in close sequence are free from trends, suggesting that any residual contaminants flushed from the baked system by calibrating gas do not contribute a significant uncertainty component to the gage response.

III. RESULTS

A number of gage balls operating in conjunction with three controllers were calibrated in the present work. To investigate the reproducibility of the results, two gage balls were calibrated simultaneously in N_2 , and the procedure was repeated a total of seven times in 2 weeks. The σ values showed standard deviations of 0.1% for each ball. The ratio of the two σ values obtained in each calibration showed a standard deviation of 0.05%, suggesting that gage performance was even more reproducible than the calibration standard. It would be misleading not to point out that these gages were subject only to minimal disturbance between calibrations. Such reproducibility is not likely to be realized in transfer standard applications.²

As an illustration of MDG reproducibility in a typical application at NBS, two such gages were calibrated together in nitrogen. Using these calibration results four days later, with the gages disturbed in the interim only to the extent required to maintain rotational speeds, they were operated simultaneously as reference gages to calibrate an ion gage. At 3×10^{-4} Pa the two reference gage indications differed by 0.3%; from 10^{-3} Pa to 10^{-1} Pa they agreed within 0.1%, indicating continued mutual consistency of the MDG calibration factors. The discrepancy at 3×10^{-4} Pa is ascribed to the fact that one of the gages exhibited such a large residual drag that its use as a transfer standard would not be recommended under normal circumstances.

In another set of measurements, in each of which two gage balls were calibrated simultaneously, one ball showed σ values of 0.973, 0.984, and 0.964 for N_2 , Ar, and He, respectively. For the other, σ values of 1.023, 1.031, and 1.050 were

obtained for these gases in the same order, suggesting that the first was the smoother of the two balls. The different trends in σ from Ar to He for the two balls are consistent with other indications of incomplete accommodation of He on the rotor surface.¹ These results also illustrate errors that might be introduced by assuming that σ is independent of gas species, or by taking $\sigma = 1$ for an uncalibrated ball.

Conditions and procedures affecting the performance characteristics of such gages have been encountered in this work. MD gages are likely to find applications on unbaked flow systems. Even if the entire system cannot be baked, the gage ball in its tubular housing and any small diameter gas inlet tube should be baked separately. This recommendation is based on the following experiences. Two new balls were calibrated before and after vacuum bakeout. Baking only the balls and rotor housings increased their σ values in N_2 by 1.9% and 2.1%. In the second experience, because of an interlock malfunction, a brief power interruption during a thunderstorm stopped the turbomolecular pump but left the forepump in operation upon restoration of power. As a consequence, oil contamination was carried back into the gas inlet tube between the leak valve and the chamber. A subsequent helium calibration made before this tube was baked gave σ values spuriously 15% too large, because the helium flow swept heavy contaminant species into the calibration chamber.

Baking is not without its hazards, however. An ion gage developed a massive leak during system bakeout, exposing MDG balls on the system to an oxidizing atmosphere at 200 °C. Surface changes from this mistreatment increased σ values for nitrogen of two previously calibrated balls by 1.1% and 2.3%.

It has been reported that "crashing" of a spinning ball against the housing, either during a power interruption or as a result of turning off the suspension circuit without first applying braking action, can alter its σ values.⁹ Although changes from this cause have not exceeded experimental uncertainty limits in this investigation, such occurrences should be avoided.

Unsatisfactory magnetization has been encountered on several occasions, in new as well as in previously satisfactory gage balls. Symptoms include low signal amplitude from the inductive rotation pickup, poor precision in timing rotations, and excessive residual drag. Attempts to demagnetize or remagnetize balls exhibiting such symptoms have frequently been unsuccessful. An attempt to use an ac solenoid for *in situ* demagnetization of one ball exhibiting excessive residual drag caused the ball to chatter so violently in its tubular housing that its surface was visibly roughened. The treatment left it with a σ value for N_2 of 1.05, but with no significant improvement in residual drag. Reliable, benign procedures for demagnetizing and remagnetizing balls in place under vacuum would constitute valuable contributions.

A history of σ values for one gage ball, obtained in seven calibrations with N_2 gas, spanning a 20 month period of use at NBS, is reported in Table II, together with suspected causes of change. The first two calibrations were performed at the Physikalisch-Technische Bundesanstalt Berlin (PTB).

TABLE II. Successive determinations of σ for one ball in N_2 . Suspected causes of changes during 20 month use at NBS are: (a) successive exposures to H_2 and air, (b) crashing, (c) demagnetizing with an ac solenoid, (d) oil contamination with subsequent bakeout, and (e) baking in an oxidizing atmosphere.

Suspected causes	σ	Laboratory
(Initial value)	0.998	PTB
a, b, c	1.014	PTB
a(?)	1.022	NBS
c	1.035	NBS
d	1.023	NBS
e	1.034	NBS
b(10 times)	1.037	NBS

Since the last two entries differ by no more than twice the estimated random uncertainty of a single calibration, these results provide no unambiguous evidence regarding the effect of crashing on the σ values of MDG balls.

IV. CONCLUSION

The calibration method described in this paper has exhibited reproducibility represented by a standard deviation of 0.1% in a series of measurements. The estimated total uncertainty is less than 1%. Some of the reported experiences suggest types of circumstances that might necessitate recalibration of transfer standard gages.

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