Method for Vacuum Leak Calibration

FOREWORD

This foreword is not a part of AVS 2.2–1968. This publication specifies practices tentatively approved as standard by the American Vacuum Society for the calibration of leaks in the range 10^{-5} to 10^{-3} atm cm^3/sec and is one of a series published by the American Vacuum Society. It contains data secured from many sources and represents the best thinking of a number of experts in the field. After several years of use this standard will be forwarded to the USA Standards Institute with the request that it be used as a basis for a USA Standard. Suggestions for improvement gained in the use of this Standard will be welcome. They should be sent to the American Vacuum Society, 335 East 45th Street, New York, New York 10017. This Standard was drafted by Mr. Albert Nerken. The AVS Standards Committee, which approved this Standard, had the following personnel at the time of approval:

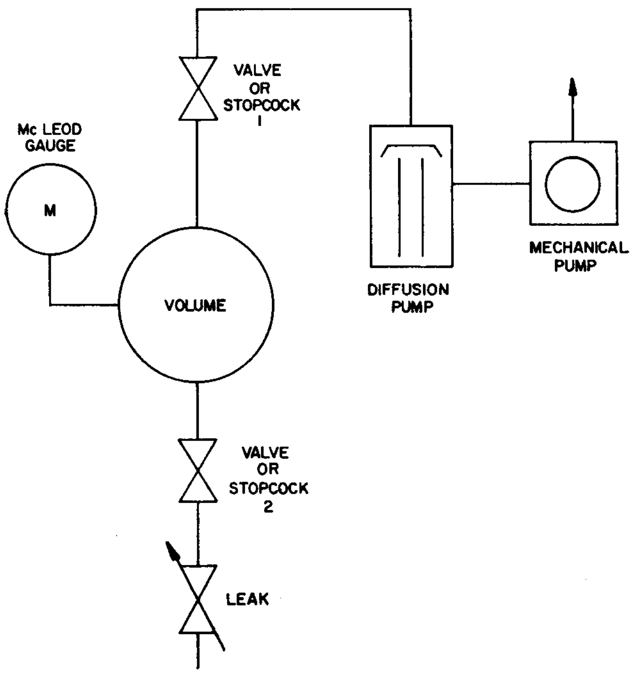

FIGURE 1. Diagram of vacuum system.

Approved by American Vacuum Society, Inc., 5 July 1968.

AVS Standards Committee

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1. SCOPE

This standard describes an apparatus for measuring the leak rate of vacuum leaks, in the range of 10^{-5} to 10^{-3} atm cm^3/sec, and a procedure for using the apparatus to determine such leak rates. The procedure involves a determination of the time rate of change of the pV product, produced in a fixed volume, by the gas entering through the leak under examination. Because a McLeod gauge is used for measuring the pressure and because the only other quantities that require determination are volume and time, the method is an absolute one.

2. APPARATUS

2.1. Vacuum System. The required vacuum system is shown schematically in Fig. 1. The component parts are described below. The material of construction may be glass and/or metal; construction methods should follow approved high-vacuum techniques.

2.1.1. Diffusion Pump. This may be of the oil type and should have a speed greater than 5 liters/sec.

2.1.2. Mechanical Pump. This should be sized to back properly the diffusion pump.

2.1.3. Cold Trap. A cold trap is not essential and is not shown in Fig. 1. However, a trap may be placed between the diffusion pump and the rest of the system.

2.1.4. Valves or Stopcocks. Either metal valves or glass stopcocks may be used in the positions shown. The construction should be such as to present a minimum of outgassing material and surface to the Volume. In the following, the term "valve" is to be understood to mean valve or stopcock, as the case may be.

2.1.5. Volume. This should have a content of not less than 1 liter nor more than 2 liters.[1] Hereafter, the term Volume, capitalized, will be used to refer to this component.

2.1.6. McLeod Gauge. This shall be useful over the range of 0.001 Torr to at least 0.250 Torr. The internal volume of the McLeod above the cutoff point should preferably not exceed one-half of that of the Volume.[2]

2.1.7. Connections. The size of connecting tubing is not critical except for the connection between the Volume and the McLeod gauge. This should be not more than 50 cm long and not less than 0.8 cm in internal diameter.

2.2. Stopwatch. A stopwatch is required in the procedure; it should have $\frac{1}{2}$-sec divisions or smaller.

2.3. Calibration of Apparatus.

2.3.1. McLeod Gauge. The McLeod gauge should be calibrated for pressure in accordance with the pertinent AVS Standard(s).

2.3.2. Total Volume of Apparatus. It will be necessary to know the volume of the apparatus between the two valves, including the volume of the McLeod gauge above the cutoff point and that of the connecting tubing between the Volume and the McLeod gauge. This combined volume will be referred to as the Total Volume. The Total Volume may be determined by filling the apparatus with a suitable liquid and then measuring the volume of the liquid by decanting into a volumetric cylinder or other measuring device. If this is not feasible, the volume of the various parts may be determined individually.

2.3.2.1. Volume. The internal content of the Volume may be determined by the method described above or by filling with a liquid of known density and weighing.

2.3.2.2. McLeod Gauge. If the general procedure of *2.3.2.* is not feasible, the volume of the McLeod gauge may be obtained from its calibration. If this latter is not available, the gauge volume should be estimated by measuring its external dimensions and calculating the volume after allowance for the thickness of the glass. The determination of volume by measuring external dimensions is permissable only when volume of the gauge is not greater than 15% of that of the Volume. If the McLeod gauge volume is larger than this, it will be necessary to find its volume from the original calibration procedure or an equivalent procedure.

2.3.2.3. Connecting Tubing. The volume of the connection between the Volume and the McLeod gauge may be determined by the liquid method or by measurement of dimensions.

3. TEST CONDITIONS

3.1. Ambient temperature shall be 23° ± 3°C.

3.2. Ambient pressure shall be 760 Torr ±5%. If the ambient pressure differs from 760 Torr by more than ±5%, air from a constant pressure source at 760 Torr pressure is to be fed to the leak. A satisfactory source is a volume, of not less than 2-liters capacity, filled with air at 760 Torr pressure at least as often as every 8 h of use.

4. BACKGROUND CORRECTION

4.1. Remove the leak and replace with a degreased metal plug. The connection shall be designed to produce a minimum of outgassing.

4.2. Open valves 1 and 2.

4.3. Evacuate the system by means of the mechanical pump.

4.4. Turn on the diffusion pump.

4.5. After 1 h, measure the pressure in the system by means of the McLeod gauge. Repeat the pressure measurement at 15-min intervals until the same reading is obtained in three successive measurements. Record this reading.

4.6. Close valve 1 and simultaneously start the stopwatch.

4.7. At the end of 15 min, read the McLeod gauge and record the reading.

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[1] The calibration procedure involves determination of the product pV. If V is 1 liter (neglecting McLeod gauge volume, see Ref. 2, below) then pV will be 0.25 Torr liter when the McLeod is at the high-pressure end of its range. Since the measurement time is 15 min (Sec. 5.6.), the largest leak that can be measured is 0.25 /(15 × 60) or roughly 2.5×10^{-4} Torr liters/sec. If larger leaks are to be measured, a larger Volume may be used and/or a McLeod which is able to measure larger pressures.

[2] Owing to the resistance of the connection between the Volume and the McLeod gauge, there will be a pressure difference between the two components. This pressure difference increases, for a given Volume, with the resistance of the connection and with the internal volume of the gauge. Assume a Volume of 1 liter and a resistance limited as in Sec. *2.1.7.* Then if the McLeod volume is one-half that of the Volume, the maximum error in the leak rate caused by the pressure difference is about 1.5%. However, the 1.5% error applies only to the first 3-min observation; the percentage error decreases directly with time from then on. Averaged over five readings (Sec. 5.6.), the error is approximately 0.5%. If the McLeod volume is equal to that of the Volume, the leak rate error is about 1%. If the Volume is 2 liters, then the error is twice as great for a given McLeod volume ratio.

4.8. If the reading obtained in **4.7** does not differ from that recorded in **4.5** above, the background correction is zero.

If the reading differs from that recorded in **4.5**, subtract the latter reading from that obtained in **4.7**. Divide the difference by 5. Call the result Δp_0; it is the pressure increment per 3-min interval in the absence of the leak, and constitutes the background correction.

4.9. Close valve 2.

4.10. Remove the plug in valve 2 and replace the leak.

5. PROCEDURE

5.1. If the volume between valve 2 and the leak is not less than 1% of the total volume between the stopcocks, its volume should be determined, either by a displacement method or by measurement of the external dimensions. This volume shall be added to the previously determined Total Volume (*2.3.2* above).

5.2. Open valves 1 and 2.

5.3. Measure the pressure in the system by means of the McLeod gauge. Repeat the pressure measurement at 15-min intervals until the same reading is obtained in two successive measurements. Record this reading.

5.4. Close valve 1 and simultaneously start the stopwatch.

5.5. At the end of 3 min raise the mercury in the McLeod gauge. The level of the mercury in the gauge should be as close as feasible to the cutoff point so that the gauge may be cut off as close to the end of the 3-min period as possible. Read and record the reading.

5.6. Repeat the McLeod gauge reading at 3-min inter vals up to a total elapsed time of 15 min, recording each reading.[3]

5.7. Close valve 2 and open valve 1 so that the system may be pumped down for another determination.

6. CALCULATION

6.1. Determine the increase in pressure in each 3-min interval by subtracting the initial pressure reading for the interval from the final pressure reading for the interval.

[3] If the volume of the McLeod gauge is substantially in excess of 150 to 200 ml, the 3-min period between readings may not be sufficient for a careful reading to be taken. In such case, the time interval may be increased, with obvious changes in the determination and calculation of background correction (Sec. 4.) and of leak rate (Sec. 5 and 6.).

6.2. Determine the average of the five pressure increments. Call this average Δp_{av}.

6.3. The leak rate is then determined from the formula:

L. R. $= (\Delta p_{av} - \Delta p_0)V/(3 \times 60)]$ Torr liters/sec at 23°C.

where

L. R. = Leak Rate

Δp_{av} = Average pressure increment, in Torr, as determined in **6.2** above.

Δp_0 = Background correction (**4.8**).

V = Total Volume of apparatus in liters.

6.4. To convert the above leak rate to atm cm³/sec at 23°C, multiply by 1000/760.

APPENDIX I: ERROR DERIVATION

In Ref. 2 it is pointed out that a difference in pressure will exist between the Volume and the McLeod gauge. This pressure difference results in an error in the leak rate determined by the procedure of this Tentative Standard. Estimates of this error are also given in the Ref. 2. These estimates are based on a theoretical calculation. In the calculation, the content of the Volume is denoted by V_1, the volume of the McLeod gauge by V_2, and these volumes are assumed to have no resistance. The two volumes are connected by a tube of resistance R, and the tube is assumed to have negligible (zero) volume. (In this procedure, R is assumed to be zero.) The gas from the leak enters V_1, part of the gas then flowing to V_2 through R; the flow is assumed molecular in type.

Then, if L denotes the leakage rate of the leak being calibrated, P_1, the pressure at any time in V_1, and P_2, the pressures at any time in V_2,

$$L dt = V_1\, dP_1 + V_2\, dP_2. \tag{1}$$

Let Q be the rate at which gas flows from V_1 into V_2. Then,

$$V_2\, dP_2 = Q\, dt.$$

But, for molecular flow

$$Q = (P_1 - P_2)/R$$

so that

$$V_2\, dP_2 = [(P_1 - P_2)/R]\, dt. \tag{2}$$

Equations (1) and (2) can be solved to give

$$P_2 = \frac{L}{V_1 + V_2} \cdot \left\{ t + \frac{RV_1}{\frac{V_1}{V_2} + 1} \left[\exp\left(-\frac{\frac{V_1}{V_2} + 1}{RV_1} t \right) - 1 \right] \right\}. \quad (3)$$

For values of R, V_1, and V_2 likely to pertain to apparatus such as that of Fig. 1, the exponential becomes insignificant after 20 sec or much less. Equation (3) then reduces to

$$P_2 = \frac{L}{V_1 + V_2} \left[t - \frac{RV_1}{\frac{V_1}{V_2} + 1} \right] \quad (4)$$

Now, $[L/(V_1 + V_2)]t$ is exactly the pressure that would be measured in the McLeod if no resistance existed between it and the Volume, as is assumed in the procedure. The error in the determination of leak rate is, then, given by the second expression in the brackets. Evaluation of this expression leads to the error figures cited in Ref. 2.