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PISTON MANOMETER AS AN ABSOLUTE STANDARD FOR VACUUM-GAUGE CALIBRATION

by I. Warshawsky
Lewis Research Center
Cleveland, Ohio

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Piston Manometer as an Absolute Standard for Vacuum-Gauge Calibration

I. WARSHAWSKY

NASA Lewis Research Center, Cleveland, Ohio 44135

Total pressure in a calibration volume is determined by measuring the force on a thin circular disc, of accurately-known area, that is freely suspended in a hole in the container wall, so that the disc is substantially flush with the wall. The disc almost fills the hole, so that there is a narrow annular gap. A continuous flow of calibrating gas, injected into the container in order to maintain a desired pressure, passes through the annular gap to a diffusion pump. The ratio of pressures on the two faces of the disc is on the order of 100:1, so that downstream pressure need be known only nominally in order to deduce the upstream pressure. Force on the disc is measured by a balance that is calibrated <u>in situ</u> with dead weights. In one arrangement, pressures in the range of 10-500 μTorr were measured with an estimated probable error of (1 μTorr + 1%).

INTRODUCTION

The piston manometer to be described is intended to provide a means for calibrating and intercomparing several vacuum gauges by subjecting them all to a common, accurately-known pressure. Brombacher[1] has reviewed most of the various calibration methods that were described prior to 1965. Holanda[2] lists newer volume-ratio methods. Like the volume-ratio and conductance-pressure-divider (CPD) methods, the piston manometer method depends on the ability to create a pressure that is very much lower than the lowest pressure of the gauge-calibration range. It uses a continuous flow of gas, like the CPD method, but it does not require computation or measurement of the conductance of a restriction. It yields a pressure from direct measurements of force and area rather than inferring it from the laws of gas flow, and hence does not require identification of the flow regime as continuum or free-molecule.

The principle of measuring the force on a freely-floating piston, while gas flows steadily between piston and surrounding cylinder, has been the basis of several measuring devices for the continuum regime. These range from present-day commercial precision pressure calibrators to early instruments[3], variations of which are still in commercial use today, to measure low differential pressures at 1 bar absolute. A piston manometer for the millitorr range was described in 1955[4]. Brombacher[5] summarizes related devices.

The present paper is directed toward proof of the pressure-measuring method at low absolute pressures rather than toward treatment of the gauge-calibration problem, but this latter problem is commented on.

BASIC ARRANGEMENT OF APPARATUS

Figure 1 serves to illustrate both the arrangement of apparatus and the premises of the calibration method. The gauges to be calibrated are mounted on the periphery of a multiport ring immediately above the baseplate of a conventional bell-jar type pumping system. Gauge-tubulation axes are radial and coplanar. The centrally-located opening through which the bell jar would normally be evacuated is almost fully blocked by a piston, in the form of a thin horizontal plate. The piston is suspended from a force-measuring device (dynamometer) and floats freely in the opening in the baseplate, with very small annular clearance. A second chamber, of appreciable volume and cross section, is interposed between the baseplate and the pumping system; additional, calibrated gauges are used to monitor the pressure p_0 in this chamber.

Calibration gas enters at the top of the bell jar, at a steady rate controlled by a leak valve, and is beamed upwards to facilitate uniform dispersal. All of the entering gas is removed through the annulus between piston and baseplate.

If piston area is A, and the net force on the piston, due to pressure, is ΔF, the upstream pressure p is given by

$$p = p_0 + \Delta F/A \qquad (1)$$

In the practical realizations of the apparatus, A is of the order of 100 cm^2, the annular gap b is about 0.02 cm, and $p_0 \ll p$. Thereby, the accuracy of knowing p depends principally on the measurement of $\Delta F/A$. The free-molecule volume flow rate through the annulus is on the order of 5 l/sec for N_2, but need not be known. 1 μTorr produces 1.33 μN (= 136 μgf).

It is assumed that the goal of the calibration system is to create a known boundary condition of pressure in the plane of the mouth of the gauge tubulation, while the molecular flux across this plane is negligibly small. It is hoped that the following features help to achieve this goal: (1) the plane in which the gauge axes lie is sufficiently below the point of gas injection, so that pressure in this plane is uniform; (2) the piston is very near this plane, so that its upper surface is also subjected to this pressure; (3) the annular gap constitutes a sufficiently high impedance to promote equalization of pressure in the volume above the piston; (4) the continuous mass flow rate of calibrating gas is sufficiently large to swamp the mass flow rate of gases desorbed by the bell jar; (5) the downstream plenum, for similar reasons, is also sufficiently isobaric so that the pressure indicated by the downstream gauge represents the pressure on the underside of the piston.

Additional practical refinements, not shown in Fig. 1, are

(1) Grounded sheet-metal baffles are used to prevent line-of-sight particle transfer between gauges.

(2) The piston-and-dynamometer assembly may be raised 10 cm or more, by means of mechanical manipulators operated through high-vacuum seals. This operation serves two separate purposes: it permits obtaining a "zero" reading for the condition $\Delta F = 0$, and it permits initial evacuation of the bell jar at full speed of the pumping system.

DESIGN CONSIDERATIONS

Piston and Orifice

Two of the piston-orifice combinations that have been used are shown in Fig. 2. They are intended to keep the ratio of piston weight to full-scale live force ΔF_{max} adequately small.

The design of Fig. 2a has been used for pressures below about 1 mTorr. A circular disc of soft Al foil, 20 μm thick, is formed into the shape of a shallow dish with vertical sides. Final diameter is about 11.3 cm, producing a nominal horizontal area of 100 cm^2. The forming, which is performed with dies, produces wrinkled vertical surfaces, since the foil is folded over on itself. Thus, the 0.6 cm-high vertical surfaces are 20 to 60 μm thick. Mass of the dish is about 0.8 g.

Alternative methods of supporting the dish are shown on the right- and left-hand sides of Fig. 2a. In one method, a 0.3 mm diam hole is punched in the vertical side, about 0.2 cm from the upper edge, and the 0.25 mm diam supporting wire is hooked through the hole. An advantage of this method is that the protruding wires protect the dish against falling through the orifice. In the other method, a wire hook is cemented near the inside corner of the dish, with high-vacuum cement. This method of support is the more rugged of the two.

The baseplate used with this design of piston has a sharp-edged orifice with 120° included angle. To further ensure roundness, the edge is blunted to form a vertical cylinder about 0.1 mm high. Baseplates are usually at least 3 cm thick.

The piston-and-orifice design of Fig. 2b has been used for the range 10-500 mTorr. This piston can be used in the pumping port in the baseplate of a conventional pumping system of nominal 10 cm size, if the sides of the port are adequately round, smooth, and square. The piston is a disc of hard-drawn stainless steel, 25 μm thick, machined to a diameter 0.1-0.2 mm less than the hole diameter. Its mass is about 2g. Each supporting wire passes through a hole punched in the disc; the end of the wire is twisted into a loop that will provide firm support under the heavier forces exerted by the higher pressures.

All support designs use three supporting wires, hooked to the bent corners of a small triangular plate, shown at the top of Fig. 2. This plate is free to rotate about a central wire that is hooked to the dynamometer. The supporting wires are made very long, 20-40 cm, so that horizontal misalignment between the geometric center of the orifice and the upper central wire will produce very little transverse force between piston and orifice at their point of contact.

When horizontal misalignment is adequately small, the piston swings constantly inside the orifice, so that static friction at the orifice is negligible.

Dynamometer

The dynamometers that have been used will be sketched only briefly because of space limitations. Details will be presented in a NASA Technical Note now in preparation.

For pressures below 1 mTorr, the dynamometer is a special modification of a high-quality d'Arsonval microammeter with conical pivots. Two stiffened pointers, 180° apart, create the appearance of an equal-arm balance. The piston is suspended from one pointer, and a counterbalancing Al weight from the other. Natural period of the assembly is 8 sec. Current through the moving coil is adjusted to keep the piston (Fig. 2a) vertically centered in the orifice; a 60 Hz dither current is also injected to overcome pivot friction. Spring torque is less than 0.2% of electrodynamic torque.

Two methods of galvanometer-current control have been used: (1) Manual adjustment of a multi-turn rheostat while the vertical position of the piston is observed visually. The self-protective protruding-wire method of support (Fig. 2a) is then particularly useful. (2) Optical servosystem. A 20 μm Al flag is attached to the piston to partially intercept the light from a projection lamp, when the piston is in proper vertical position. The lamp image falls on a photocell, connected in a feedback circuit with the galvanometer coil, to maintain the piston in proper vertical location. Optical elements, except for the flag, are outside the vacuum system; two windows, 180° apart in the calibration ring, are used. Oscillations of this servosystem produce more violent motion of the piston, at about 9 Hz, than is obtained in the manual method, but convenience, stability, and safety of operation are higher. Vertical position can be maintained to 0.1 mm.

For pressures between 10 and 500 mTorr, the piston (Fig. 2b) has been supported by an unbonded resistance-strain-gage force transducer, energized with 1/10 of its normal supply voltage in order to reduce excessive temperature rise of the strain-gauge wires in the vacuum environment. Bridge output voltage is a measure of force. Vertical deflection of the transducer at maximum pressure is about 0.2 mm.

Calibration

In situ determination of dynamometer sensitivity (calibration-curve slope) is made by adding an accurately-known weight to the piston and noting the change in dynamometer output indication. The weight is of the order of ΔF_{max}. The addition is performed with mechanical linkages operated, through high-vacuum seals, from the outside of the bell jar.

Dynamometer zero, corresponding to $\Delta F = 0$, is ordinarily determined with the piston well above the orifice, and with the leak valve closed, so that both sides of the piston are at the same pressure. However, at pressures above 1 mTorr, where sorption effects are negligible, the zero can also be determined with piston in place in the orifice, by closing a high-vacuum valve between the pumping system and the downstream plenum.

TESTS AND ANALYSIS OF PERFORMANCE

Performance possible in practical cases was determined by building two types of systems for gauge calibration--a low-pressure system and a high-pressure one. To conserve space, the high-pressure system (10-500 mTorr) will not be considered any further than to state that its estimated probable error (e_p) is (0.3 mTorr + 0.1%). The following discussion deals with the low-pressure system (10-500 μTorr), which is the less accurate and more interesting one.

Test System

A vacuum system used to prove the manometer consisted of a 46x76 cm (18x30 in) glass bell jar and an aluminum-alloy calibration ring, baseplate, and downstream plenum. A Viton L-ring sealed the bell jar; Viton O-rings sealed all other components. The multiport calibration ring held a magnetic-sector mass spectrometer and five ion gauges (a nude Bayard-Alpert (B-A) type, two of a tubulated B-A type, and two of a tubulated conventional-triode type). Other ports were used for windows, manipulators, electrical connections, and gas injection. A 25 cm, water-baffled diffusion pump constituted the pumping system.

Factors That Affect Accuracy

Downstream Pressure

This pressure was measured with two tubulated B-A type gauges, of the same type as those used in the upstream plenum, but not previously calibrated. The indications of the two gauges differed by 28% of their mean. The measured sensitivities of the two upstream gauges differed by 20%. The downstream pressure was computed by assuming the same mean sensitivity for both pairs of gauges. Probable error, e_p, for one pair is about $\frac{1}{4}$ the difference.

The ratio p/p_0 at various values of p, for Ar, is shown in Fig. 3. If we conservatively assume an e_p of 20% in knowledge of p_0, the corresponding e_p in p will be less than 0.2%. If calibrated gauges had been used downstream, the e_p in p would have been negligible, since the present work and that of Holanda[6] have shown that a single gauge may have an e_p of about 3%.

Gas Composition

The relation between pressure, as measured by the piston manometer, and molecular density, as measured by an ion gauge, depends on the identity of the gases present. This relation affects deduction of downstream pressure from the ion gauge reading. It also affects the upstream-gauge calibration computation. When contaminating gases are present together with the calibration gas (subscript G), we have the simultaneous relations

$$p_t = {}_T\Sigma\, p_j = p_G + {}_C\Sigma\, p_j \qquad (2)$$

$$i^+ = {}_T\Sigma\, i_j^+ = {}_T\Sigma\, s_j p_j = s_G p_G + {}_C\Sigma\, s_j p_j \qquad (3)$$

where j is a running index, s is sensitivity (collector current/pressure), p_j is partial pressure, p_t is total pressure, i^+ is total ion collector current, and subscripts T,C respectively indicate that the summation is to be taken over the totality of gases or over only the contaminating gases.

For an ion gauge used in the presence of contaminating gases, we define p_{EIG}, the equivalent indicated pressure of gas G, by

$$i^+ = s_G p_{EIG} \qquad (4)$$

The ratio p_{EIG}/p approaches unity as $({}_C\Sigma\, p_j)/p_G \rightarrow 0$.

Determination of p_0 requires a knowledge of s_j for the mass spectrometer; determination of p_{EIG} for the ion gauges being calibrated requires a knowledge of s_j/s_G for the principal contaminants. However, the knowledge need not be accurate if ${}_C\Sigma\, p_j \ll p_G$. For the mass spectrometer,

s_j must be determined empirically; for an ion gauge, s_j/s_G may be taken as the average value listed by Summers[7] in his comprehensive review of the published literature on ion gauge sensitivity.

In the special case where the amount of contaminants does not change after gas G is injected, the s_j's need not be known. Sensitivity is given simply by

$$s_G = (i^+ - i_C^+)/(p-p_C) \qquad (5)$$

where i_C^+ and p_C are, respectively, the gauge current and the measured total pressure prior to opening of the leak valve.

The principal contaminants in the test system were H_2O and CO. Their partial pressures were substantially independent of p when the calibration gas was He, N_2, or Ar; ${}_C\Sigma\, p_j$ was about 4 μTorr. The effect on p_0 is negligible when $p \geq 10$ μTorr.

Effective Piston Area

It is convenient to define an effective piston area A which allows for the presence of drag forces as well as for purely geometric dimensions.

Measurable geometric dimensions. The dish shown in Fig. 2a is too flimsy to allow direct measurement of diameter. A lower limit to its diameter is the measurable diameter of the steel die with which it was formed, plus 40 μm. An upper limit is the measurable diameter of the orifice. However, a closer estimate is obtainable from an inferential measurement of annulus width.

Annulus width. An electrical analogue of the vacuum system of Fig. 1 is shown in Fig. 4, where only the principal impedances are indicated. Pressure p_1 is usually slightly higher than atmospheric. The time constant of response of bell-jar pressure p to a change in p_1 (or to a small change in R_1) is

$$\tau = CR_1R_2/(R_1 + R_2). \tag{6}$$

If $R_1 \gg R_2$,

$$\tau \approx CR_2 \tag{7}$$

In the free-molecule regime, the value of CR_2 for an infinitely-thin orifice of area A_a gives

$$\tau \approx (V/A_a)(2\pi M/R_0T)^{1/2} \tag{8}$$

where $A_a = \pi D_o b$, D_o = orifice diameter, b = annulus width, V = upstream volume, $4(R_0T/2\pi M)^{1/2}$ = average molecular velocity.

Neglecting the possible increase in τ due to nonzero orifice-edge thickness[8,9], the width b may be determined by measuring the time constant of response when the leak-valve setting is changed slightly or when p_1 is reduced abruptly. We have done this and find, typically, that $\tau \approx 22$ sec, leading to b = 0.16 mm. However, the piston area thus computed should be augmented by a correction representing the effect of drag.

Drag forces. The flow of gas through the annulus may exert a drag on the piston of Fig. 2a. It is not necessary to treat this phenomenon in detail because simple limits may be set on its magnitude. The lower limit is zero. The upper limit is the frictional force that would be exerted at low Knudsen numbers if piston and orifice were long, concentric cylinders; this force is merely $\frac{1}{2}$ the product of annulus area πDb and pressure-difference $p - p_0$. Taking one half of this force as a median between upper and lower limits is tantamount to assuming that orifice diameter D_o is diminished by annulus width b. This conclusion is independent of the nature of the flow regime.

Thus, the effective piston diameter is taken as $D_o - b$, with a maximum uncertainty of $100\ b/D_o$ percent. In the present case, this latter quantity was 0.14%.

Force Measurement

Dead weight calibrations with class M weights have established that nonlinearity of the dynamometer is negligible. Peak-to-peak variation in sensitivity (slope) has been 0.07% over a 100-day period. A more significant error is the variation in zero during one day; this has an average value equivalent to 0.1 μTorr.

Correlation Between ΔF and Pressure Difference

The principal source of error appears to lie in the random uncertainty with which dynamometer current represents pressure difference. An overall evaluation of this effect was obtained in the following manner.

The leak valve opening was changed in small steps to obtain many values of p_t. At each pressure, grid current i^- and collector current i^+ of each of the five ion gauges were measured to 0.1%, and the quantity

$$s = (i^+/p)/(i_0^-/i^-) \tag{9}$$

was computed. The quantity i_0^-/i^- is a normalizing factor (≈ 1) to correct for the deviation of i^- from its nominal value i_0^-. A smooth curve was drawn through the points. Random deviations of individual points from the curve were attributed to defects of the piston manometer technique. For $10 \leq p \leq 100$ μTorr, these deviations did not exceed 0.7 μTorr; for $100 \leq p \leq 500$ μTorr, 90% did not exceed 2%.

Summary of Estimated Errors

A simplified formulation for the summation of errors treated above is that e_p in the range $10 \leq p_t \leq 500$ μTorr is (1 μTorr + 1%). There is a 90% probability that errors will not exceed twice this value.

DISCUSSION

The effectiveness of the piston manometer depends on using a piston whose area is large compared to the annulus area, maintaining sufficient downstream pumping speed to create a high pressure ratio and an isobaric downstream plenum, and having a dynamometer of adequate sensitivity and capacity. We point out some compromises involved in achieving these goals.

Increasing annulus width facilitates mechanical construction and operation and also reduces the time required to establish a new pressure. But it reduces the pressure ratio and increases the uncertainty in effective area.

If downstream-plenum volume is not isothermal, the gauges may not represent the pressure on the underside of the piston. If the pumping system uses a cryogenic trap, radiation shielding may be considered. However, such shielding may significantly raise p_0.

Since the time of construction of the dynamometer described here, there have been numerous developments in vacuum microbalances[10]. They may provide a means for downward extension of the useful range. However, such extension will require deeper examination of how isobaric a condition can be created in the upstream plenum, where there are present several gauges that operate at a variety of tubulation, envelope, and element temperatures.

The substitution of a bakeable upstream plenum to achieve lower pressures is straightforward, albeit expensive and more complex.

Final proof of the manometer will require (1) a group of ion gauges of sufficiently constant sensitivity to allow their use as transfer standards and (2) intercomparison of the calibration of these gauges with calibrations in other calibration systems.

CONCLUSION

The piston manometer in its present realization offers the advantages of independence of the identity of the gas and of the Knudsen number of the flow, facility of creating an isobaric condition for gauge calibration, and principal dependence on measurements of length and mass. In the range above 10 μTorr, it appears to offer inaccuracies acceptably small for most purposes.

ACKNOWLEDGEMENTS

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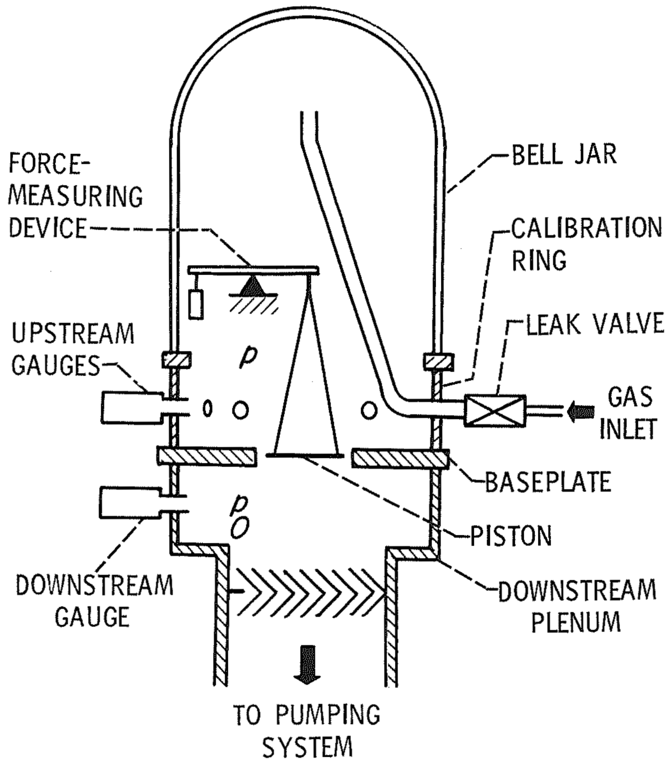

Fig. 1 Basic arrangement of piston manometer for vacuum-gauge calibration.

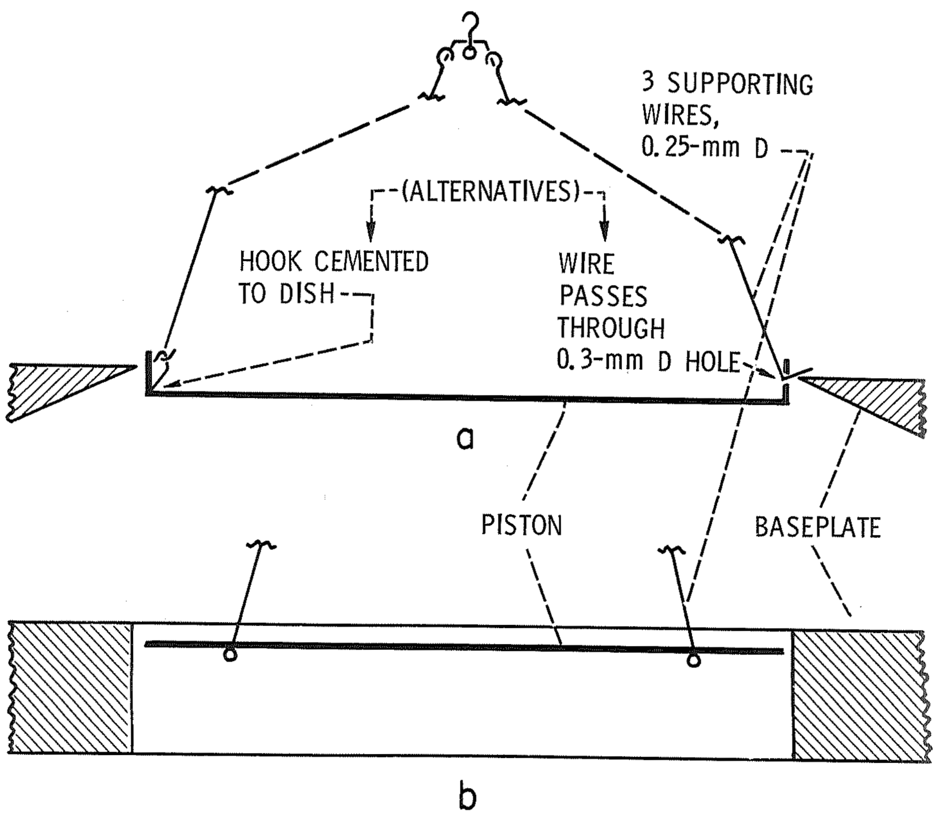

Fig. 2 Piston and orifice arrangements.

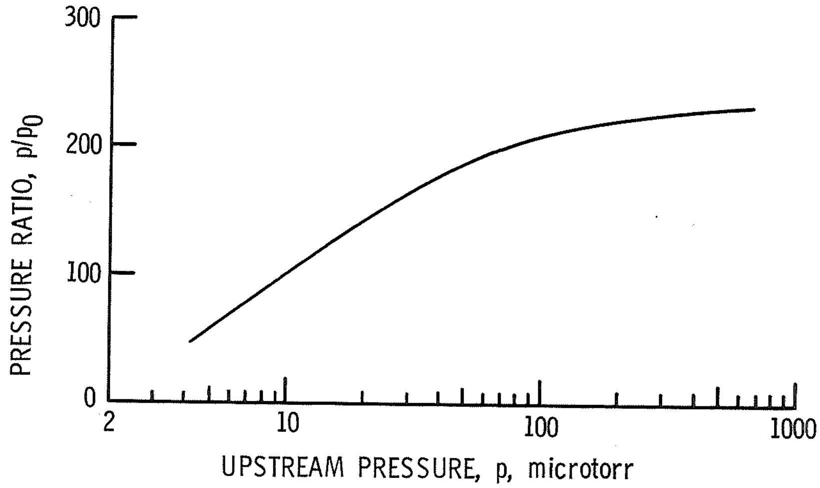

Fig. 3 Pressure ratio for a calibration with argon.

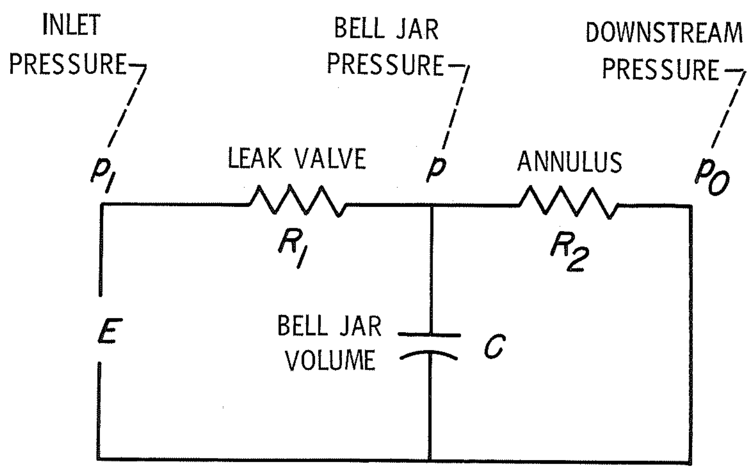

Fig. 4 Analog representation of the gas flow system.