[Contribution from the Laboratory of Physical Chemistry, University of Wisconsin]

AN IMPROVED GLASS MANOMETER

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There is a considerable demand for devices which permit pressure measurements of a system enclosed entirely by glass. They are valuable for use with corrosive materials or for experiments at high temperatures. Several typical ones are shown in Figs. 1 to 8.[1] In every case a measured air pressure is balanced through a sensitive diaphragm. The first and second use the principle of the optical lever, the third and fourth depend on the movement of a pointer, the fifth displaces a liquid in a capillary tube, the sixth makes a clicking sound when the wrinkled diaphragm moves, and the rest operate by closing an electrical circuit.

A new glass diaphragm developed in this Laboratory has proved to be so satisfactory that a brief description is offered. Its construction is shown in Figs. 9 to 12. A short platinum wire is sealed into a small glass rod about 1 cm. long and let down by a fine platinum wire to rest on the bottom of a thin glass bulb. The bulb is then flattened in a flame and the protruding mass of glass under the rod is melted flat with a small, hot flame. It is well to have one side of the diaphragm less flexible than the other, so that the rod is given a lateral motion. A second platinum wire is next sealed through the tube just above the bulb. It is broken off on the outside and covered over with melted glass. The fine platinum wire connected to the diaphragm contact is pushed down while the tube is being sealed into a flask or other container. It is then pulled out and attached to a heavier platinum wire which is sealed through the tube near the top. Another fine platinum wire provided with a loop is caught over the platinum contact at the side of the tube, pulled taut and sealed through the tube. The side contact is then adjusted with a stout wire to give a suitable zero reading, that is, to give electrical contact through a galvanometer and a high resistance when the pressure on the manometer side is slightly greater than the pressure in the flask. A radio grid leak or a beaker of distilled water serves as a convenient resistance. Asbestos wool is stuffed gently into the tube to keep the lead wires separated and to prevent cooling effects resulting from air circulation. The operation and calculations are similar to those described before.[1i]

With ordinary glass the small rod attached to the diaphragm must be made of lead glass to avoid cracking of the diaphragm. With Pyrex glass the platinum wires are held firmly but the seals are not gas tight. This difficulty is removed by melting Pyrex glass over the end of the side contact and by covering the seals through which the lead wires pass with DeKhotinsky cement or lacquer.

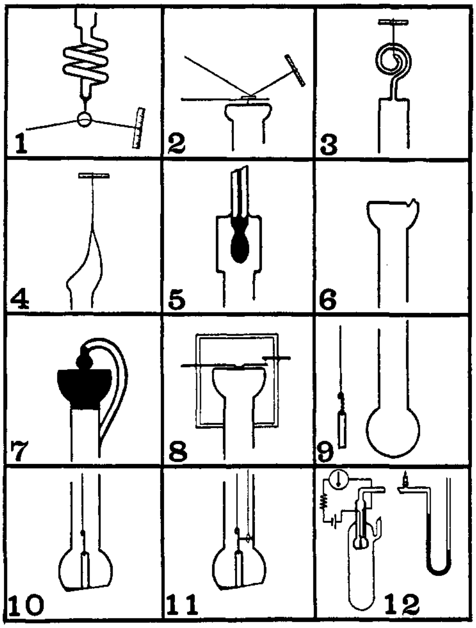
Sections of tungsten wires may be used to advantage for sealing the lead wires through the Pyrex.

[1] (a) Ladenberg and Lehman, *Ber.*, **8,** 20 (1906); (b) Johnson, *Z. physik. Chem.*, **61,** 457 (1907); (c) Bodenstein and Katayama, *ibid.*, **69,** 26 (1909); (d) Warburg and Leithauser, *Ann. Physik,* **24,** 25 (1907); (e) Gibson, *Proc. Roy. Soc. Edinburgh,* **33,** 1 (1912); (f) Jackson, *J. Chem. Soc.*, **99,** 1056 (1911); (g) Baume and Robert, *Compt. rend.*, **168,** 1199 (1919); (h) Smith and Taylor, This Journal, **46,** 1393 (1924); (i) Daniels and Bright, *ibid.*, **42,** 1131 (1920); (j) Karrer, Johnston and Wulf, *J. Ind. Eng. Chem.*, **14,** 1015 (1922).

With quartz it is necessary to fuse the stout platinum wire contacts onto heavy tungsten wires in the oxygen flame and to anchor the tungsten wires in the quartz. A small capillary tube is fitted snugly over the tungsten wire and melted down with the oxygen flame. The quartz is then sealed in place. The lead wires are passed through holes and rendered gas tight with cement. For high temperatures the exit holes must be at a considerable distance from the furnace.

Several advantages of this diaphragm may be enumerated.

1. The construction and operation are relatively simple.



Devices for measuring gas pressures in all-glass vessels. Fig. 1.—Ladenberg and Lehman; Johnson. 2.—Warburg and Leithauser. 3.—Bodenstein and Katayama. 4.—Gibson; Jackson. 5.—Baume and Robert. 6.—Smith and Taylor. 7.—Daniels and Bright. 8.—Karrer, Johnston and Wulf. 9–11.—Construction of improved diaphragm. 12.—Arrangement of finished apparatus.

2. The lever construction magnifies the motion and gives an excellent sensitivity.

3. The working parts are on the inside, instead of on the outside of the diaphragm, and a clumsy housing vessel for the diaphragm is not required.

4. The diaphragms always withstand a greater pressure outward than inward and the new construction makes it unnecessary to evacuate the manometer side before carrying out a measurement.

5. The whole of the diaphragm is well immersed in the chamber, giving isothermal conditions. Some of the other types are unsatisfactory for high temperatures because part of the diaphragm may be cooled by circulating air.

6. The contacts of platinum wire do not wear out and the zero point may be easily adjusted at any time. At temperatures below room temperature it is necessary to use a drying tube to prevent condensation of moisture on the contacts.

7. The zero point is independent of temperature over a wide range and it is independent of pressure until the total pressure becomes as low as 10 mm.

8. The apparatus may be made of ordinary glass, Pyrex or quartz. Pyrex glass is satisfactory up to 375°.

9. The chamber may be set up at a distance in a thermostat or other apparatus where direct observation is awkward.

One distinct disadvantage in comparison with the optical types lies in the fact that no warning is given when the zero is being approached. This difficulty is minimized by adjusting the manometer slowly through a capillary leak, or by placing a large bottle in the system as a pressure "buffer."

A full atmosphere's pressure on the manometer side does not break the diaphragm, even though the chamber is completely evacuated. The readings are usually accurate to 0.2 mm. of mercury. The sensitivity may be increased still further if the diaphragm is not required to withstand an atmosphere's difference in pressure. Occasionally the contacts become fouled, presumably by a speck of grease from the stopcock driven along by an inrush of air, but they may be readily regenerated by momentary sparking with a weak induction coil.

This type of manometer has been in constant use for three years in several different researches in this Laboratory and it has proved very satisfactory through many thousand pressure readings.

Summary

A device for measuring the pressure of a gas enclosed entirely in glass is described. A measured air pressure is balanced against the pressure of the enclosed gas through a glass diaphragm and the point of balance is obtained by closing an electrical circuit through a platinum contact fused to the diaphragm. Although similar to earlier devices the apparatus described here is more reliable and easier to construct.

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NOTES

Note on Electromagnetic Vacuum Cut-Off.—The accompanying diagram explains a type of mercury cut-off suitable for high vacuum work. The meniscus at B may be raised and lowered several millimeters by allowing the glass enclosed soft iron, D, in arm, A, to float on the mercury in this arm or be withdrawn entirely by means of a current passing through the magnet winding, which acts as a solenoid. The quantity of mercury is adjusted so that when the iron is withdrawn the top of the meniscus at B is just a millimeter or two below the opening of the inner tube. This permits an unimpeded flow of gas from the system to be evacuated to the pump. When the system is evacuated the iron is permitted to drop and the meniscus B then rises until the opening of the inner tube is effectively sealed off. It is more convenient to constrict very slightly the arm, A, at the top so that the iron weight will stick there when it is withdrawn by means of the solenoid. This permits the use of the cut-off either open or closed without a continuous flow of current. The weight will fall when the arm is lightly tapped with a pencil.