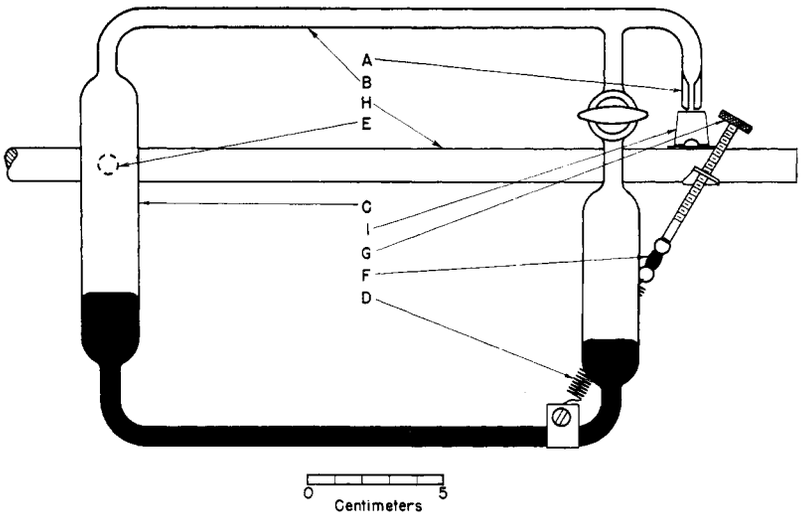


FIGURE 2. PRESSURE REGULATOR WITH LOOP TYPE OF MANOMETER

a stiffer spring. The diameter of the tubing in the closed limb of the manometer controlled the weight of mercury shifted for a given pressure change, hence larger tubing in the closed limb of the manometer greatly increased sensitivity; because of location the size of the open limb was unimportant. A bubble of air was always trapped in the closed end of the manometer in the Figure 2 type of regulator; a larger bubble resulted in increased sensitivity. A bubble trapped above the mercury in the Figure 1 type of regulator increased the sensitivity and increased the pressure range over which the device was operative. While the pivot bearing on these regulators was the glass tube, better practice would be to sheath this tube with a machined, split metal bushing, since glass tubing is rarely round and is apt to stick in the bearing. It would likewise be better practice to connect to the vacuum connection, *E*, with a loop of thin-walled rubber tubing, prevented from collapsing by Raschig rings inserted at intervals, and with the other end of the loop fastened permanently to the support bar, *H*, so that torque on the regulator by changing the position of the suction tubing would not change the adjustment of the regulator.

Both regulators were independent of variations in atmospheric pressure. Adjustment to a predetermined pressure by means of the thumbscrew, *G*, having 32 threads per inch, was easily effected. Both regulators controlled pressure with uniform facility over the entire pressure range. Both regulators were susceptible to "bouncing" under certain conditions—i. e., an oscillation of the mercury column and concomitant movement of the manometer-beam, causing intermittent admission of air. It was found to be due to parallelism of valve seat and face, and was eliminated by an imperceptible bending of the valve seat support, so that valve seat and face were not quite parallel and hence the valve could not close completely. The bouncing apparently was due to complete closure of the valve. In order to open the valve, pushing against atmospheric pressure required overcompensation of the mercury-balance, and then rapid overcompensation in the opposite direction resulted owing to the sensitivity of the device, again causing sealing of the valve. This bouncing phenomenon has been noted in other regulating devices (*1*) using a mercury column and is due to similar causes.

Generally speaking, the Figure 2 type of regulator was more flexible and sensitive, but no stopcock under vacuum can be considered completely dependable under long periods of operation. Either regulator may be made capable of increased sensitivity by increasing the diameter of the tubing in the closed manometer limb, lengthening the beam, using a more elastic spring, or trapping a larger volume of air in the closed end of the manometer. Where great sensitivity is not required, a rubber cushion can be used to replace the spring suspension, but all attempts to use rubber have necessitated continual adjustment, owing to the fatigue of the rubber cushion.

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# Electrolytic Preparation of Quinhydrone

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AN ELECTROLYTE used extensively in pH measurements is quinhydrone, which consists of bright platinum immersed in a saturated solution of quinhydrone. In view of the fact that quinhydrone is an equimolecular compound of hydroquinone and quinone, it was thought possible that hydroquinone, a relatively cheap product used in photography, might be oxidized to quinone by the use of an electric current. The resulting product would be free of foreign materials.

The apparatus consists of an outer cup of nonporous material 20 cm. (8 inches) high and 20 cm. (8 inches) in diameter and an inner cup of porous material 17.5 cm. (7 inches) high and 7.5 cm. (3 inches) in diameter. A carbon electrode, obtained from a dry battery such as those used in residential doorbell circuits, is placed in the outer cup (anode) and another is placed in the inner cup (cathode).

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TABLE I. PREPARATION OF QUINHYDRONE

| Expt. No. | Electrolysis Time *Hours* | Amperage *Amperes* | Hydroquinone *Grams* | Yield *%* |
|---|---|---|---|---|
| 1 | 4.82 | 1 | 10 | 72.6 |
| 2 | 2.41 | 2 | 10 | 74.2 |
| 3 | 1.61 | 3 | 10 | 74.8 |
| 4 | 1.21 | 4 | 10 | 75.3 |
| 5 | 0.96 | 5 | 10 | 74.1 |

The hydroquinone has an equivalence of 2; therefore one mole of hydroquinone would take 2 faradays for complete oxidation. As only half the quantity of hydroquinone used is to be oxidized, half of the required faradays for complete oxidation should be used, or one faraday per mole of hydroquinone.

The hydroquinone (20 grams) was dissolved in a suitable amount of water and poured into the outer cup of the electrolytic apparatus. Two to 3 grams of sodium sulfate were added and the level of the liquid was raised to within 2.5 or 5 cm. (1 or 2 inches) of the top of the cup. The inner cup was half filled with distilled water, 5 ml. of glacial acetic acid were added, and the cup was filled with distilled water to within 1.9 cm. (0.75 inch) of the top. The current was then turned on at various amperages and for various times (Table I). Sodium sulfate was added to the outer cup liquor in small amounts from time to time to maintain a constant amperage. At the completion of the required oxidation period, the solution in the inner cup was concentrated and cooled, and the quinhydrone was filtered off. The yield was about 75 per cent of the theoretical.

The purity of the quinhydrone in each experiment was 98 per cent or better in each case. In the opinion of the author a higher yield may be obtained by using the mother liquor repeatedly.

# Accurate Low-Pressure Gage

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LOW-PRESSURE gages commonly found in physics and chemistry laboratories are either of the Bennert closed U-type, or one of the many modifications of the McLeod type. The Bennert type is satisfactory for pressures above 1 cm. of mercury, but is not very accurate for lower pressures. For the low-pressure range the McLeod gage is usually used, but its application is limited by the fact that it depends on increasing the pressure in one part of the system. This increase in pressure will, in the case of vapors from liquids having high boiling points, lead to a condensation of the vapors, which will make the measurements useless. Thus in the case of measuring the vapor pressure of water of crystalline hydrates, the pressure cannot be increased above that over pure water at the same temperature without bringing about dew formation.

A number of methods have been used for increasing the accuracy of such measurements, one of which involves the replacement of mercury by a liquid of low density and negligible vapor pressure, always being certain that the vapors being measured are not soluble in the confining liquid.

The floating tube barometer described by Caswell (*1*) in 1704 and later adapted to use in what has been called the steelyard barometer (*3*) embodies the basic principles of a recent instrument known as the Dubrovin (*2*) gage, which may be purchased from scientific apparatus houses. Since there appears to be no published description of the theoretical background of the floating barometer, it seemed worth while to develop the equation for the magnification factor, and to give directions for making a gage which would be useful in vacuum distillation work, as well as around physical chemistry and physics laboratories.

Figure 1 shows an inverted closed cylinder floating freely over mercury. Its lateral motion is controlled by three point guides at top and bottom lightly touching the inside walls of the containing vessel. The device is placed on its side and a high vacuum is created by means of a good oil or diffusion pump. This removes all the air and adsorbed gases from the inside and outside of the floating tube. It is then set up on end and atmospheric pressure is slowly restored. If the density of the tube material is less than that of mercury, the tube will float and the inside of the floating tube will be entirely filled with mercury. Now if the external pressure is gradually reduced, the tube remains stationary until the external pressure becomes equal to the height of the mercury column inside the tube above the outside level. (Capillary forces are for the moment neglected.)

Reducing the pressure still further results in the condition shown in the figure. The height, $N$, measures the pressure in the large cylinder. As the pressure is progressively reduced, the mercury column drops while the tube itself rises. Thus a drop of 1-cm. pressure results in a drop of 1 cm. in height $N$, while $H$ may increase 10 or 20 cm., corresponding to multiplying factors of 10 or 20.

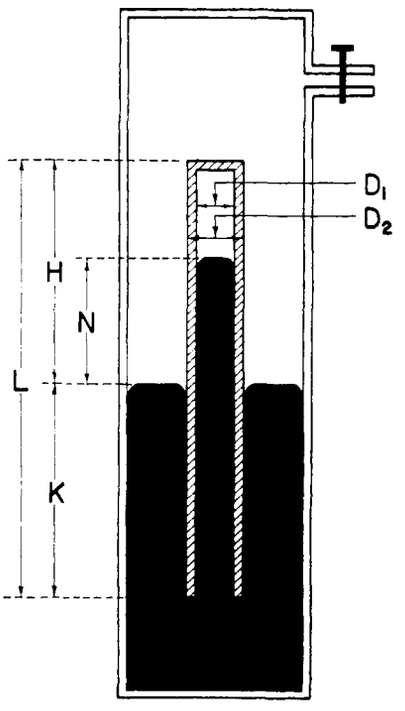


FIGURE 1

Regarding the tube as a freely floating body at equilibrium, constrained by guides to move only in a vertical direction, and assuming the gas pressure inside the floating tube to be zero, we may equate the sum of the downward forces to the sum of the upward forces. Height $N$ of the liquid inside the tube depends only on the external pressure, $P$.

The pressure in the liquid at the base of the floating tube is made up of the sum of the gas pressure, $P = Ng\rho$, and the pressure of the liquid column, $K$, which is $Kg\rho = (L - H)g\rho$, in which $g$ and $\rho$ represent the force of gravity and the density of the liquid. This total pressure $g\rho(N + L - H)$ over the annular ring area of