

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.

MADISON, WISCONSIN

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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several millimeters by allowing the glass enclosed soft iron, D, in arm, A, to float on the mercury in this arm or be withdrawn entirely by means of a current passing through the magnet winding, which acts as a solenoid. The quantity of mercury is adjusted so that when the iron is withdrawn the top of the meniscus at B is just a millimeter or two below the opening of the inner tube.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more convenient to constrict very slightly the arm, A, at the top so that the iron weight will stick there when it is withdrawn by means of the solenoid.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.

The size of the cut-off may be so designed that the whole apparatus can be immersed in liquid air and the mercury frozen in place, thus reducing the vapor pressure of the mercury. Otherwise a liquid air trap may be inserted between the cut-off and the system to be evacuated.

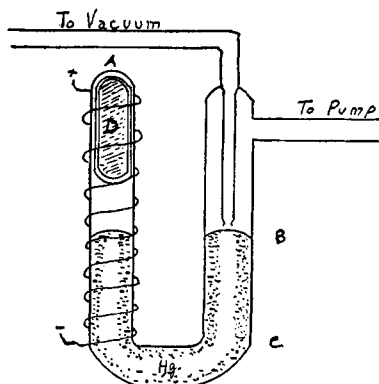


Fig. 1.—Electromagnetic vacuum cut-off.

The former procedure is practicable only when the additional volume of such a trap is detrimental. Care must be taken to adjust the volume of arm A so that the meniscus B will not descend below C when atmospheric pressure is admitted to the system. The apparatus acts as a rough vacuum gage as well as a cut-off. It has the following advantages over the usual design of "Y" cut-off. The quantity of mercury is small and may be completely de-gassed. No fresh mercury is being constantly exposed. When atmospheric pressure suddenly enters the system the mercury level will drop so rapidly that no mercury is blown into the vacuum canalization. Contrary to a stopcock there is no chance for outside leakage and no vapor pressure from stopcock grease.

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**Note in Reference to Platinum-Tungsten Welding.**—Platinum may be welded direct to tungsten by the following method. The tungsten is cleaned with sodium nitrite. Several coats of platinum are burned into the tungsten from a solution of lavender oil and platinum chloride. The platinum to be welded is then coated with borax and welded to the tungsten in an oxygen flame. This must be done quickly. Gold may very easily be welded to tungsten by first cleaning the latter with the sodium nitrite, then covering with borax and finally plunging the heated tungsten into a molten gold bead. The gold makes an excellent flux for platinum welding when baser metals are a disadvantage.

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**The Coefficient of Expansion of Bromoform.**—Our attention was called by Professor Hildebrand to the fact that the only determination of the coefficient of expansion of bromoform to be found in the literature, that of Thorpe,<sup>1</sup> was evidently made with impure material, since its melting point was recorded as 2.5° instead of the more recent value<sup>2</sup> of 7.7°. We accordingly undertook a redetermination of this coefficient.

The bromoform used was purified by washing with sodium carbonate solution and then with water, drying for seventy-two hours over anhydrous calcium chloride and distilling three times under a pressure of about 20 mm. The melting points after each distillation were, respectively, 7.65, 7.70, 7.70°, as determined by a standardized thermometer graduated to 0.1°, which was immersed in the melting solid contained in a jacketed test-tube.

The coefficient of expansion was determined from the density at a series of temperatures ranging from 9 to 50°. A Pyrex pycnometer was used, having a volume of approximately 150 cc.

Beginning at 9°, and then at a series of successively higher temperatures, the pycnometer was immersed in a water-bath until temperature equilibrium was reached. The temperature of the bath was obtained

TABLE I

## DATA AND RESULTS

Temp., °C.	Density, g./cc.	Specific volume	Molal volume
9.32	2.9187	0.34261	86.597
9.96	2.9178	.34273	86.626
13.93	2.9067	.34403	86.955
14.92	2.9048	.34426	87.014
15.00 <sup>a</sup>	2.9043	.34431	87.027
18.73	2.8938	.34556	87.343
19.90	2.8918	.34581	87.404
20.00 <sup>a</sup>	2.8912	.34588	87.423
23.21	2.8822	.34696	87.696
24.92	2.8787	.34737	87.801
25.00 <sup>a</sup>	2.8780	.34746	87.823
27.92	2.8700	.34843	88.069
29.93	2.8656	.34897	88.203
32.43	2.8581	.34989	88.436
34.93	2.8525	.35057	88.608
38.65	2.8419	.35187	88.938
40.02	2.8393	.35219	89.019
42.71	2.8317	.35315	89.259
47.04	2.8201	.35459	89.625
50.27	2.8117	.35565	89.893

<sup>a</sup> These values were calculated from Equation (1).

<sup>1</sup> Thorpe, *Proc. Roy. Soc. (London)*, **24**, 283 (1876).

<sup>2</sup> International Critical Tables, Vol. I.

by using a thermometer graduated to  $0.1^\circ$ , which was compared with one recently calibrated by the Bureau of Standards. The filled pycnometer was weighed at each temperature and the density, given in Table I, was calculated from these weights. The coefficient of cubical expansion of Pyrex glass used was that recently obtained by Buffington and Latimer.<sup>3</sup> All weights were reduced to vacuum.

No recent determination of the density of bromoform has been made and those in the literature are not in close agreement. However, that of Perkin,<sup>4</sup> 2.9045 g./cc. at  $15^\circ$ , and the value selected for the International Critical Tables, 2.890 at  $20^\circ$ , agree well with our values.

The following empirical equation for the variation of specific volume with temperature was obtained by the method of least squares from the data in Table I.

$$V_t = 0.24204 [1 + 0.00090411 (t - 7.7) + 0.0000006766 (t - 7.7)^2] \quad (1)$$

The values calculated by the aid of this equation differ by not more than three units in the last decimal place from the experimental points.

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**The Freezing Point and Density of Pure Hydrogen Peroxide.**—The above constants are of importance as criteria of the purity of peroxide. The freezing point as previously given<sup>1</sup> was taken when complete solidification took place, and the freezing bath was kept at a temperature several degrees below the freezing point of the peroxide. This gave too low a value, since the rate of crystallization of hydrogen peroxide is slow and the liquid has a great tendency to supercool. By a sufficient number of crystallizations a sample of peroxide was obtained which remained at a constant temperature during the whole process of solidification. The precaution necessary is to maintain the cooling bath at a temperature less than  $0.1^\circ$  below the freezing point, the liquid at the same time being continuously stirred. Under these conditions the melting point is  $-0.89^\circ$ . The melting point  $-0.89^\circ$  differs from that previously found by  $0.90^\circ$ , and this large difference must be ascribed to the slow rate of solidification which was not taken into account in the former work.

Special precautions were taken with regard to the dilatometer in the measurement of the density at  $0^\circ$  so that a minimum of decomposition occurred. Pyrex glass was found to be most suitable but only one out of a

<sup>3</sup> Buffington and Latimer, *THIS JOURNAL*, **48**, 2305 (1926).

<sup>4</sup> Perkin, *J. Chem. Soc.*, **45**, 533 (1884).

<sup>1</sup> *THIS JOURNAL*, **42**, 2548 (1920).

large number of dilatometers could be used successfully, as too much decomposition was evident in the others. The value 1.4649 found for the density at 0° agrees within 1 part in 1500 with the previous<sup>1</sup> determinations 1.4633.

In a dilatometer slight decomposition often occurs as is evidenced by the formation of minute bubbles. The following method is then recommended. The dilatometer is connected as indicated in the diagram by means of a rubber tube "B" to one end of an open manometer having an adjustable mercury reservoir A. The reading of the meniscus of the dilatometer is taken at atmospheric pressure,  $p_1$ , and then the mercury is rapidly raised so that the enclosed gas in the dilatometer and connecting tubing is compressed to a pressure  $p_2$  given by the manometer. The meniscus then gives a reading  $R_2$  at the pressure  $p_2$  as compared to the reading  $R_1$  at the pressure  $p_1$ . The volume  $v$  of the oxygen produced in the decomposition of the peroxide is given by  $v = K(R_1 - R_2) \div (1 - p_1/p_2)$ , where  $K$  is the volume of the unit length of the capillary of the dilatometer. As an example, in an inferior dilatometer (5cc. capacity), three sets of meniscus readings were made at twenty-minute intervals and the volumes of the bubbles in the liquid estimated by the above method as 0.0086, 0.0156 and 0.0214 cc. With these corrections the densities calculated for each of the three sets of observations agreed within 1 part in 7000.

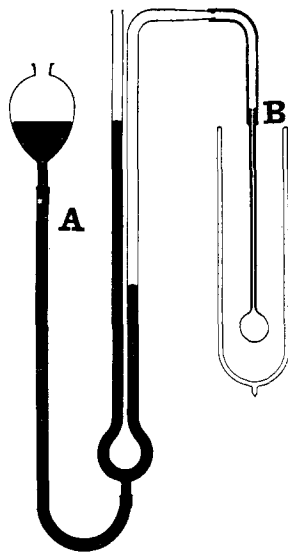


Fig. 1.

It was found that for concentrated aqueous solutions of peroxide containing up to 5% of water, the density could be accurately expressed by the equation  $D = 0.9486 + .005163 A$ , where  $A$  is the per cent. by weight of hydrogen peroxide.

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