Vapor Pressure Apparatus: Semi-Micro*

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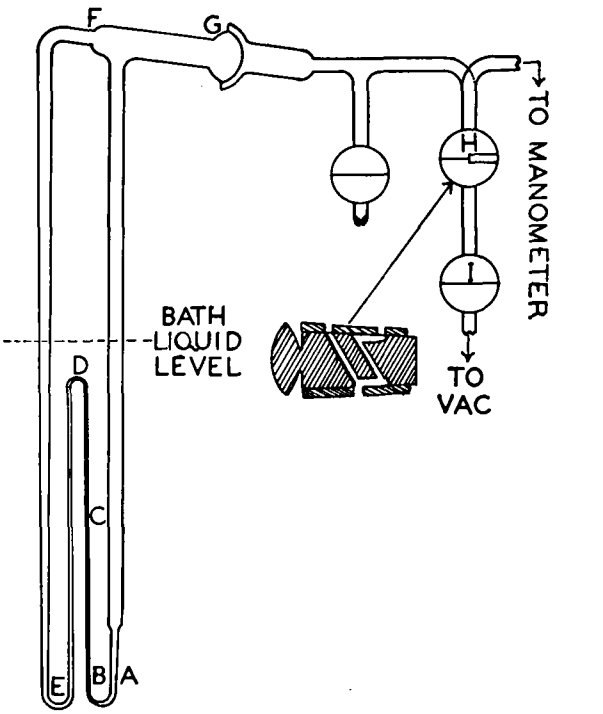
WORK in this laboratory on various fat derivatives has prompted the development of an apparatus adapted to the routine measurement of vapor pressure on small quantities of material. This apparatus is a modification of one recently described (1, 2, 3), the difference being the replacement of the air bath and open capillary by a closed system which may be immersed in a liquid bath. These changes result in a greater sensitivity and accuracy in the temperature range where liquid baths are practicable.

One form of the apparatus, which has given satisfactory results, is shown. The section ABCD is the essential part of the tube. There are a few critical dimensions in this section. While the diameter at C (upper liquid meniscus) may vary between 0.15 and 0.3 mm., it has been found that the use of the smaller diameter results in a more sensitive instrument. The inner diameter at A (lower liquid meniscus) should be 10 times that at C. The diameter should be fairly constant from B to D (factor of 2) with the smallest diameter at C or above. As the height AC varies with surface tension, the type of liquid to be studied may have to be considered although the variation is usually not great among organic liquids.

Since the difference in pressure on the two surfaces at A and C and the resultant fall of the upper meniscus are primarily dependent on the resistance to vapor flow in the section CD, the height of this section should be greater for larger diameters. However, the decrease in the height of the column of liquid supported by the capillary, as the diameter increases, as well as the lessened resistance to flow of liquid in a larger tube makes possible the maintenance of a constant height BD even though the diameter varies considerably from tube to tube.

THE rest of the tube was designed to overcome certain manipulative difficulties and may be modified without affecting the accuracy of the readings. The bend above D should be below the surface of the liquid in the bath to prevent formation of condensate, which can run back into the capillary, when the pressure is increased. A broken column of liquid in the capillary usually means that the tube must be dried and refilled before another determination can be made. The loop E serves as a heated surface where the very small amount of liquid vaporized at C may condense without forming a plug. The diameter throughout this loop should be at least 2 mm., but a larger diameter will not affect results. In constructing the tube, the seal at F should be made in such a manner that pressure or vacuum may be applied in cleaning the tube. The downward slope to the 28/12 hemispherical joint G prevents any drainage from the joint into the tube. Stopcock H was grooved to make possible the simultaneous isolation of the manometer and boiling point tube from each other and from the vacuum source.

The operation of the apparatus is the same as that of one previously described (1, 2). The stopcock I is set so that evacuation will take place at a predetermined rate. With the bath temperature either constant or changing very slowly, the stopcock H is turned so that both the manometer and the boiling point tube are connected through I to the vacuum source. When the meniscus at C begins to fall, H is closed. The temperature of the bath and the pressure as indicated by the manometer are then the coordinates of a point on the vapor pressure curve.



Although the actual operation of the apparatus is not difficult, it is important that the determinations are made under conditions nearly identical to those of the calibration. Some of these conditions are: a) the amount of liquid in the tube, b) the rate of evacuation, and c) the distance through which the liquid in the small capillary moves before the tube is isolated. Care in loading the tube, scratching and calibrating stopcock I, and use of a scale behind the capillary make it possible to reproduce readings with an accuracy of ± 0.1 mm. of Hg. As a general rule, decrease in the diameter of the small capillary reduces the need for exact reproduction of conditions.

Summary

An apparatus is described for the determination of vapor pressures on small amounts of pure substances with an accuracy of ± 0.1 mm. Use is made of the increase in resistance to gas flow which accompanies decrease in diameter of a capillary.

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*Authorized for publication on September 28, 1948, as Paper No. 1476 in the Journal Series of the Pennsylvania Agricultural Experiment Station.