MICRO VACUUM DISTILLATION.

By

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One of the most important operations in organic chemistry is that of fractional vacuum distillation. This operation is of equal importance to the organic chemist who wishes to deal with very small quantities of liquids. However, the micro technician is faced with the difficulty that, to date, no satisfactory description of a micro vacuum fractional distillation with efficient temperature control has been reported. PREGL\(^1\) has reported a simple and entirely satisfactory apparatus for simple vacuum distillation. A disadvantage of the PREGL apparatus is that the temperatures of the gases leaving the boiling liquid cannot be taken accurately; the nearest approach to this is to estimate the boiling point from the temperature of the heating bath employed. Obviously this is not satisfactory. A second objection is that a fractional distillation cannot be carried out with the apparatus. It has been found possible to modify the PREGL apparatus so that all its advantages are retained, and so that the two difficulties referred to are overcome.

The revised apparatus is illustrated in Figure 1. The vertical chamber (A) contains the liquid to be vacuum distilled. A suitable vacuum pump is connected at point (B). The horizontal chambers (C), (C'), etc. serve as receiving chambers; the number of these chambers is governed by the number of fractions which are to be taken. A small thermocouple is placed in the distilling chamber at (a) and serves as a means of recording the tempera-

\(^1\) F. PREGL, Quantit. Org. Mikroanalyse, Julius Springer, 1930.
tures of the distilling gases. A heating bath is placed around (A); suitable heating and cooling baths are placed around the collecting receivers (C), (C'), etc. The use of these heating and cooling baths will be explained in detail later. From the foregoing descrip-

![Figure 1. Apparatus for Fractional Micro-Vacuum Distillation.](image)

(½ Actual Size)

tion of the apparatus, it can be seen how the difficulties of the Pregl apparatus have been overcome. A thermocouple acts as a micro-thermometer for recording the temperatures of the gases given off by the boiling liquids; and suitable collecting receivers are introduced into the apparatus to allow of the collection of different distillation fractions.

**Preparation of the apparatus.**

The apparatus is most conveniently made from an ordinary soft glass test tube. Glass tubing of the proper bore, or Pyrex glass, may be used. If a test tube is used, the sealed end is drawn out to a capillary of from 1 to 2 mm. inner diameter and about 25 to 30 cm. long. Two or more other capillaries are drawn out at points of the remaining part of the test tube. This leaves three or more original sections of the test tube connected by capillary tubing, and with a long capillary at one end. The apparatus is then bent with the aid of a small luminous gas flame to conform to the shape indicated in the diagram. The vertical section near the long capillary is to perform in the capacity of a distilling chamber;