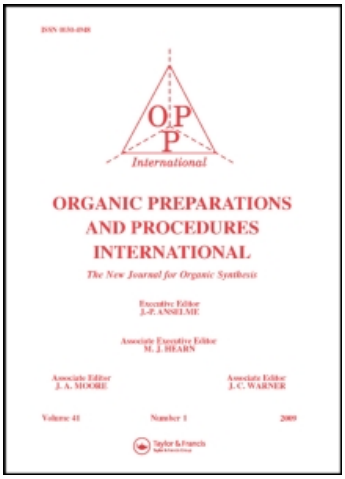

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MICRO-DISTILLATION OF ORGANIC LIQUIDS

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MICRO-DISTILLATION OF ORGANIC LIQUIDS

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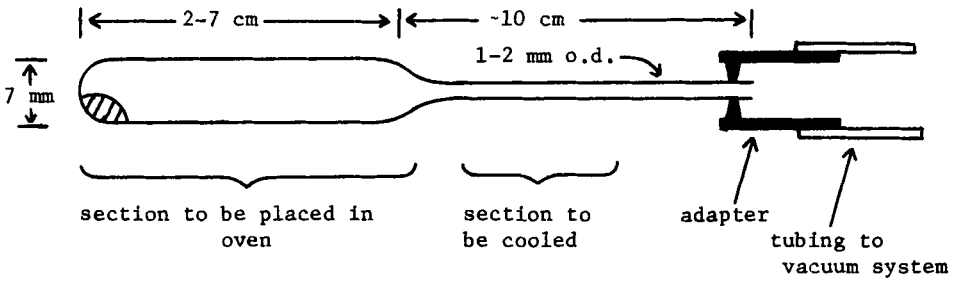
Bulb-to-bulb (Kugelrohr) distillation using an air oven is a well-known laboratory technique.[3] An obvious extension of this procedure to small samples involves use of Pyrex tubing without bulbs. We have modified this technique further for use with samples of 100μg to 10 mg by preparing distillation tubes from Pasteur-type disposable pipets.[4] This procedure is useful for purification of small samples for elemental or spectral analysis.



Cross-sectional View of Assembled Distillation Apparatus

The large end of a Pasteur pipet is sealed and the sample to be distilled is introduced as a solution in a volatile solvent through the open capillary end with a long syringe needle or a very fine capillary. The solvent is then removed by warming on a steam bath or by using a

flow of nitrogen through a needle or capillary into the sample chamber. In this manner the sample is concentrated in the sealed end of the pipet.

For distillations at atmospheric pressure the large end of the distilling tube is placed horizontally in the "Kugelrohr" oven[5] or a similar air oven with the capillary end extending out of the oven. A portion of the capillary near the oven is cooled with a wick of glass wool saturated with acetone. The oven temperature is increased until the sample slowly distills into the capillary. For samples to be submitted for microanlysis, the capillary end of the tube is separated from the large end, the end of the capillary is sealed and the capillary is centrifuged to collect the distillate in one end.

For distillations at reduced pressure the capillary end of the tube must be connected to a vacuum system. We found the adapter used to fill and empty disposable micropipets[6] to be ideal for this purpose. The rubber adapter slides easily over the capillary end of the distilling tube forming a vacuum-tight seal (a small amount of vacuum grease may be used to insure a tight seal), and the glass end of the adapter connects directly to the rubber tubing of the vacuum system (see diagram above).[7] For air-sensitive compounds the capillary end of the distilling tube may be sealed under vacuum, and the entire tube submitted to the analyst after concentration of the sample in the capillary end.[8]

REFERENCES

1. Texas A&M University

2. Hamilton College

3. Cf. inter alia M. J. Babcock, Anal. Chem., 21, 632(1949); A. W. Schrecker, Anal. Chem., 29, 1113(1957); R. Graeve and G. H. Wahl, Jr., J. Chem. Ed., 41, 279(1964).

4. Pasteur disposable pipetes may be purchased from any major chemical supply company.

5. A thermostatically-controlled Kugelrohr oven made by Buchi Instruments (Switzerland) is marketed by Rinco Instrument Company, but a self-constructed air oven is satisfactory for most distillations.

6. The adapters used with Drummond "Microcaps" made by Drummond Scientific Co. were found to work very well.

7. Fieser and Fieser have recently described a similar method for sealing an evacuated melting point capillary using an adapter prepared from a rubber vaccine stopper: L. F. Fieser and M. Fieser, "Reagents for Organic Synthesis," John Wiley, New York, 1967, p. 241.

8. The methods described herein were developed in the laboratories of Professor W. S. Johnson, Stanford University.

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