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A TECHNIQUE FOR HIGH RECOVERIES FROM VACUUM DISTILLATIONS

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The design and use of a novel apparatus for a variant of vacuum distillation is described. Relative to a conventional device, the apparatus/technique described permits superior recovery of multigram quantities of moderately volatile liquids from vacuum distillations.

Keywords: organic synthesis; vacuum distillation; apparatus.

INTRODUCTION

Vacuum distillation is commonly employed to resolve mixtures of organic liquids. A conventional apparatus consists of a distilling flask, a still head/thermometer, a jacketed (water cooled) condenser, an adapter (often with an inner tube to permit the application of vacuum without undue loss of distillate into the vacuum line) and a receiving flask.

The chief difficulty in obtaining high recoveries from vacuum distillation of a multigram quantity of an organic mixture arises from those regions of the apparatus which retain liquid. The principal regions in question are the inner tube of the condenser, the inner tube of the adapter and ground glass joint pairs located after the still head. Typically, distillation of a binary mixture results in significant volumes of the lower-boiling liquid having collected in the aforementioned locations, just prior to collection of the higher-boiling liquid. Hence, some of the lower-boiling liquid is not collected in the first distillation fraction and, as it is distilled, some of the higher-boiling liquid is contaminated by residual pools of the lower-boiling liquid. Often, this requires collection of a modest amount of impure material as a transition fraction before the higher-boiling liquid can be collected cleanly.

At the end of the distillation, residual higher-boiling liquid remains lodged in the apparatus. In the following section, an apparatus and a technique are described which largely circumvent these difficulties.

RESULTS AND DISCUSSION

In redesigning a conventional apparatus, the distilling flask was altered. The traditional round-bottomed flask was replaced with a pear-shaped bulb. A capillary bubbler was constructed, that reached very nearly to the bottom of the pear-shaped bulb to permit agitation of the liquid throughout the distillation. Next, the jacketed condenser was eliminated and a simple glass tube introduced in its place. Two ground-glass joint pairs were removed and a one-piece apparatus produced (Figure 1).

To avoid the usual loss of material trapped in the inner tube of the distillation adapter, that part of the apparatus was built into the receiving flask (Figure 2).

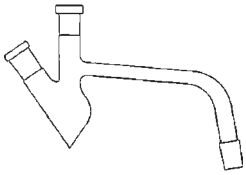

Figure 1. *A one-piece apparatus for vacuum distillation*

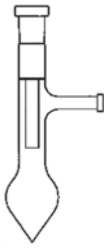

Figure 2. *A receiving flask. The top ground-glass joint is standard taper 14/20 and the side joint standard taper 5/13, over which standard one quarter inch i.d. vacuum hose fits snugly*

The apparatus, after sample loading, can be rotated so that the condenser tube is raised 30-60 degrees from the horizontal. It is then, virtually impossible to get undistilled material into the receiving flask when the vacuum is carefully introduced. Once the vacuum is established, heating with an oil bath will lead to reflux of the lower-boiling material. Tilting the apparatus, so that the condenser tube is horizontal, permits smooth collection of the lower-boiling component. In the event that the distilling bulb has been overfilled, controlled distillation may be initiated with the condenser tube remaining 30 degrees or so above the horizontal until the volume remaining in the distilling flask is compatible with horizontal orientation of the condenser tube.

When the lower-boiling liquid has been distilled, with the vacuum maintained, the oil bath is lowered. It is helpful to interpose a heat shield between the oil bath and the distilling bulb. A hot-air gun can be used to walk the condensate (from the start of the condenser tube to the receiving flask) into the cooled receiving flask. At this point, all of the high-boiling component is in the distilling bulb/still head and all of the low-boiling component is in the receiving flask.

The vacuum is broken and a new receiving flask introduced. Upon reintroducing the vacuum and the heat, the higher-boiling compound can be collected. A hot-air gun can be deployed to collect almost all of the higher-boiling component, including condensate in the upper portions of the distilling bulb, around the thermometer and throughout the condenser tube. Condensate can be driven into

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the lower region of the cooled receiving flask, if the standard taper 14/20 joint at the top of the receiving flask is heated with the hot-air gun while the system is under vacuum.

If the receiving flasks have been pre-weighed, one can conveniently determine the actual product yields without significant material loss to the apparatus. As an example, present research efforts in my lab have required the distillation of **1**[1].

$$PhCO_2CH_2SSCH_3$$

1

Typically, 12-bundle column chromatography[2] employing 1:1 chloroform/petroleum ether provides **1** that is contaminated with modest amounts of ethyl benzoate. The vacuum distillation technique, described above, permits smooth purification of **1** with very little material loss.

Direct comparison of distillation techniques

A pair of vacuum distillations was carried out. In the first distillation, propiophenone (4.07 g) was loaded into a conventional distillation apparatus which included commercially manufactured still head, thermometer, condenser and distillation adapter (14/20 standard taper joints). Propiophenone (3.25 g, 70 °C/1.7 Torr) was recovered. In the second distillation, propiophenone (4.03 g) was loaded into a distillation apparatus of the type described in the preceding section and distilled as described there. Propiophenone (3.94 g, 62 °C/0.85 Torr) was recovered. Note that the higher pressure and cold condenser of the first distillation should have diminished material loss into the vacuum line and biased results in favor of the traditional procedure. Nonetheless, with the conventional apparatus only 79.8% of the propiophenone was transferred into the receiving flask, whereas with the newly-described procedure/apparatus 97.8% of the propiophenone was transferred into the receiving flask. Clearly, very severe material loss to the conventional apparatus would be expected for vacuum distillation of a 4 g sample of a multicomponent mixture.

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REFERENCES

1. Baerlocher, F. J.; Baerlocher, M. O.; Chaulk, C. L.; Langler, R. F.; O'Brien, E.M.; *Sulfur Lett.* **2000**, *24*, 101.
2. Kabir, S. M. H.; Langler, R. F.; Smith, R. D.; Tam, N. C.; Webb, J. D.; *J. Sulfur Chem.* **2000**, *26*, 7.