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Simple Rotating Molecular Still

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A simple molecular still has been designed to purify materials which have a low vapor pressure and which are thermally unstable. The apparatus contains no ground glass joints and the distillation is accomplished in a completely closed system. The efficiency of the still was found to be better than that for either the pot-type or the falling film-type molecular stills generally used.

In recent years a number of rotating evaporators and molecular stills that employ a mobile thin film have been developed. The rotating evaporators which have been described are unsatisfactory as molecular stills because: (1) The joints which are required either leak, or if lubricated, may contaminate the sample; and (2) they are not readily adaptable to multiple stages. The mobile film molecular stills, while satisfying these objections are quite complex and even more expensive than the evaporators.

The apparatus was designed to purify materials which have a low vapor pressure and which are thermally unstable at elevated temperatures. The apparatus is simple and inexpensive. After the glass assembly is made, all other components are usually available in the laboratory. It is free of all ground glass joints, can be extended to as many stages as desired, and can be designed to remove several fractions by collecting in more than 1 ampoule. The essential features of the apparatus are shown in figure 1.

The method used is as follows: The sample is poured into bulb A while the unique axis of the apparatus is in a vertical position with bulb A on the bottom. The entrance tube C is then sealed to a vacuum line, and the sample is degassed by alternately freezing and melting under continuous pumping. When the degassing is completed, tube C is flame-sealed, and the apparatus is mounted in a nearly horizontal position on a rack as shown in figure 1. Two band clamps, D and J, beside the protruding rings, E and I, serve as supports and bearings for the apparatus. A rubber O-ring is mounted at H and connected to the drive wheel of a motor which rotates the entire apparatus at 1 to 2 rpm. A wide-mouthed Dewar flask containing liquid nitrogen or a slush of solid carbon dioxide in a suitable liquid is placed under the collecting bulb F and an infrared lamp is placed several inches above bulb A. The lamp heats the thin film of liquid on the surface of the bulb. This has the advantages that the surface film of the sample is continually renewed and that the bulk of the liquid remains cool, thereby limiting thermal decomposition. The process is repeated by placing the Dewar flask with cooling bath under bulb K and the lamp above bulb F. When the second stage of the process is completed, the entire apparatus is clamped in the inverted vertical position with ampoule M on the bottom. The frozen sample in bulb K is allowed to melt and flow into the breakoff tip, ampoule M. The residue in A and F is retained during this pouring process by the protruding ring seals at B and G. The sample is then frozen and the ampoule is flame-sealed at L.

This apparatus is 5 to 100 times as rapid as the usual pot-type molecular still. The rate can be controlled by adjusting the pressure in the system or by raising or lowering the heat source. Under normal operation the main body of the distilling sample is only a few degrees above room temperature. If necessary, the film can be made much hotter by lowering the lamp, while at the same time the bulk of the liquid is maintained at or below room temperature by supplementary cooling. The efficiency of the apparatus, as shown by the removal of colored bodies, was higher than either the pot or falling-film type of molecular stills generally used.

The method has been used to remove colored impurities and higher molecular-weight oxidation products from fluoro-, chloro-, bromo-, iodo-, methyl-, t-butyl-, and unsubstituted dimethylanilines and the analogous anisoles. Also the purification of high-boiling residues from crude ethyldichlorobenzene stocks has been successfully achieved by using this technique. With the pot-type still this same material could not be distilled.

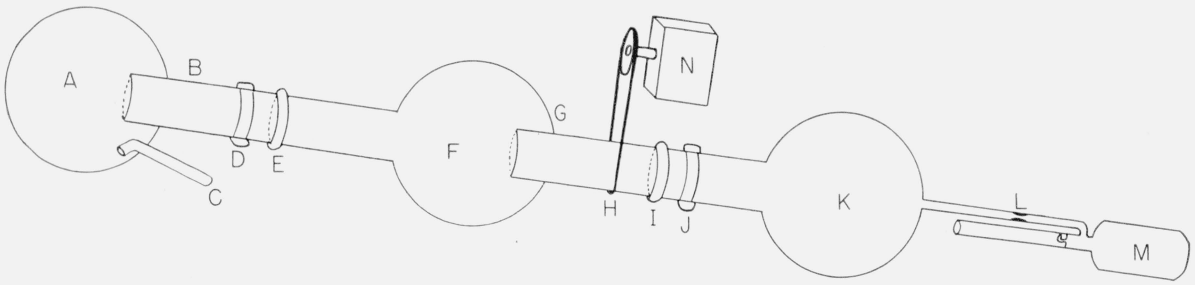

FIGURE 1. *Rotating molecular still, operating position A, F, K, 500 ml bulbs; C, entrance tube; B, G, annular rings; D, J, band clamps used as bearings; E, I, alining protrusions; H, rubber O-ring; L, sealing constriction; M, ampoule; N, motor; axis inclined at about 10° in respect to the horizon.*

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