Simple Liquid-Liquid Partitioning Apparatus

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In pesticide residue and similar minor component determinations involving foodstuffs there is sometimes encountered a bulk of oily extractables from which it is necessary to segregate the desired component. Because of the sheer bulk of the extraneous oils, column and other chromatography are often impracticable in the initial cleanup stages and preliminary partitioning operations to reduce this bulk are essential. If restrictions on choices of solvent combinations result in very low separatory funnel-type extraction (preferential partitioning) efficiencies or in intractable emulsions, recourse is usually made to a liquid-liquid extractor. The present report is concerned with a liquid-liquid extractor of small-diameter yet long-partitioning (exchange) path and with provision to keep the solution being extracted at tap water temperatures or lower. This simple device (see Fig. 1) has been used with high efficiency for acetonitrile "extractions" of hexane solutions of avacodo, coffee bean, cottonseed, and nut meat extractables, all very oily substrates.

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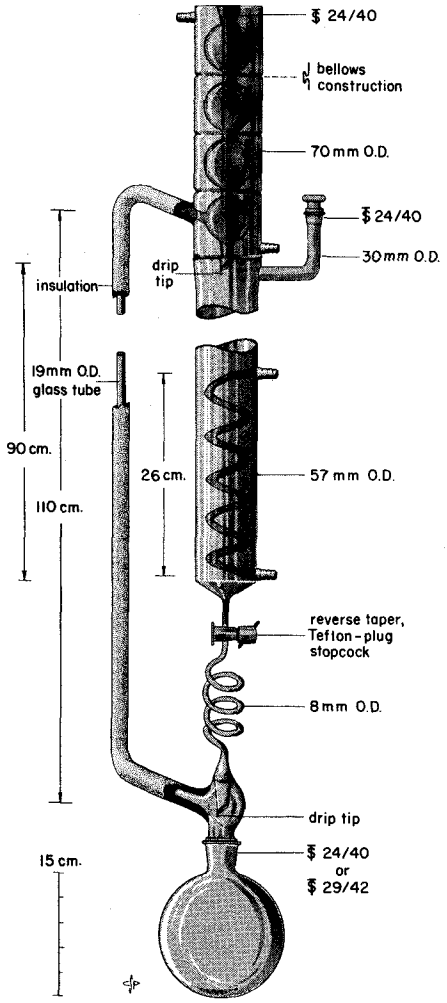

Fig. 1. Exhaustive liquid-liquid extractor. A secondary condenser may be placed on top of the bellows condenser for very low-boiling solvents. The coil below the stopcock is an expansion coil.

CONSTRUCTION AND OPERATION

All important dimensions are shown in Figure 1. The distance between the condenser drip tip and the surface of the liquid in the extraction chamber should be at least 50 cm. so that falling drops of extracting solvent break into multitudes of minute droplets upon impacting that surface. Adjustment of the Teflon stopcock permits matching of distillation rate with return of extract to the boiler flask. To minimize possible solute decomposition, cooling water is usually circulated through the lower coil then into the condenser; a second condenser can be added when highly volatile solvents are used.

DISCUSSION

With hexane-acetonitrile systems the maximum reflux rate is about 25 ml./min.; above this rate flooding may occur. The rate optimum for droplet dispersion with this system was from 10-12 ml./min. with ovex in avocado oil, chlorbenside in walnut oil, and several organochlorine and organobromine insecticides in coffee and cottonseed oils. Partitioning efficiencies at the above rates have been around 75% of the sought compound into the acetonitrile phase in 2-2½ hours. In this apparatus extraction efficiencies depend in part upon density of the oil and therefore upon concentration of oil in the hexane: for example, walnut oils are best extracted at 1 g. of ground nut meat/4 ml. of hexane, whereas coffee oils "extract" smoothly at 1 g. of ground coffee bean/2 ml. of hexane.

Hexane-extractable oil contents, in the usual stripping solutions, of the above substrates are: Fuerte avacados 20% of fresh flesh, coffee 14% of roasted bean, cottonseed 19% of whole seed, and walnuts 62% of mature nut meats.

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