Concentration of Pesticide Residue Solutions with a Modified Commercial Multi-tube Solvent Evaporator

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Almost every pesticide residue analytical procedure requires at least one concentration step involving partial or complete removal of some normally present organic solvent. Various techniques have been used for this purpose. A common technique for relatively small volumes is to direct a gentle jet of clean, dry air or nitrogen over the surface of the solution, maintained at a slightly elevated temperature to avoid condensation of airborne water vapor; alternatively, the solution may be maintained at room temperature while providing warm dry air. Another common technique is to apply a partial vacuum over the surface of the warmed solution while it is rotated or otherwise stirred.

Several devices based on one or the other technique are commercially available. The Rotary Evapo-Mix[2], originally designed for use with the partial vacuum technique, had special merit after the simple modification described herein. This modification permits it to be used as a multi-air-jet evaporator or it can be quickly changed back to work in its original vacuum mode if desired. The entire unmodified device is shown in Fig. 1. Modification involves the following simple steps: a) substitution of ten glass air-jet tubes (Fig. 2) onto the standard manifold, at the flexible tubing connections above the stopcocks (see Fig. 1), to replace the ten standard glass vacuum-trap heads and b) connection of a clean dry-air source to the manifold instead of a source of vacuum as in the original mode. The entire unit is placed in a fume hood when in use in the modified mode.

The standard provisions of heating bath and of controlled circular vibrating motion for the sample tubes are unchanged. The individual stopcocks permit adjustment of air flow rate into each sample container to cause only a gentle disturbance at the liquid surface. The break vacuum stopcock (see Fig. 1, slightly left of center) is kept partially open to serve now as an air bleeder in reverse manner. The condenser jacket for the manifold is unused except when using short sample tubes or vials;

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[1] Present address: 3735 Mt. Alma Gosa Pl., San Diego, CA 92111.

[2] Buchler Instruments Div., Searle Analytic, Inc., Fort Lee, N.J. 07024.

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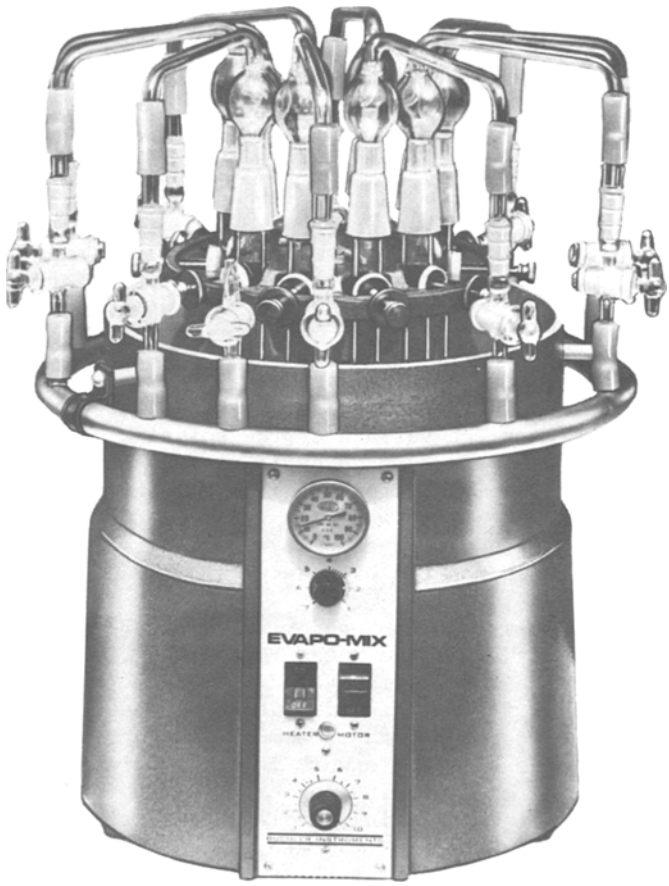

Fig. 1. Unmodified multi-tube solvent evaporator. An earlier model but with ground-glass joints for mating sample tubes to the vacuum-trap heads was used. This later model uses polyethylene or optional Teflon caps on the vacuum heads to press fit with the sample tubes, permitting use of a greater selection of less specialized tubes; however, screw cap vials are usable only with special adapters (photo generously provided by Buchler Instruments Div.).

if they are less than 70 mm long they cannot be warmed directly by the water bath so warm water from the bath should be pumped through the manifold jacket, by an added external pump, to warm the air before it blows over the samples.

Aside from this minor complication for short tubes, the device in its modified form conveniently and quickly evaporates most of the commonly used solvents in pesticide residue methodologies from almost any tube or vial (including screw-cap vials) from about 15 mm o.d. to 28 mm without the need for a ground-glass joint at the top. For example, 30 ml of n-hexane in each of ten tubes can be completely and simultaneously evaporated in about 15 min with the water bath at 55--60°C.

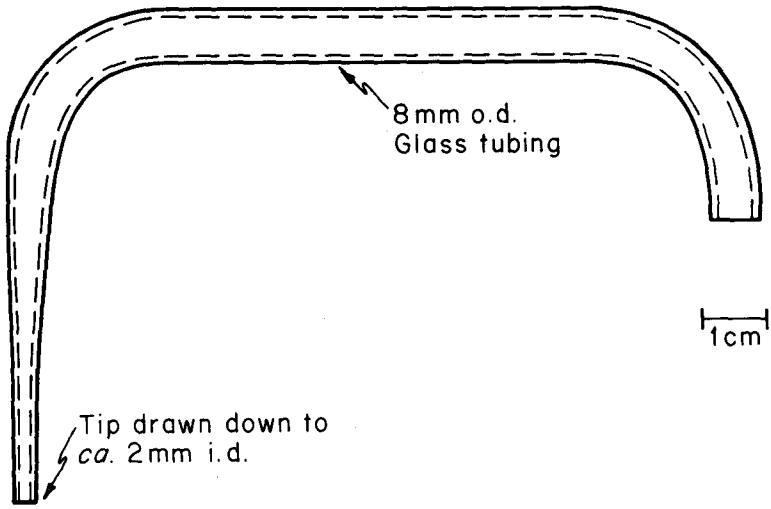

Fig. 2. One of ten glass air-jet tubes required for modification of the commercial evaporator.

The usual precautions of avoiding excessive heat and of incorporating suitable "keepers," e.g., mineral oil (GUNTHER and BLINN 1955, BEROZA and BOWMAN 1967), must be employed when concentrating some pesticide solutions to small volumes by this type of technique. To illustrate, BURKE et al. (1966) obtained recoveries above 94% for petroleum ether solutions (1--4 μg/ml) of aldrin, DDT, heptachlor, heptachlor epoxide, and lindane evaporated to solvent dryness under an air stream when 2 mg of oil extracted from butterfat was added before evaporating any of the solvent; samples were removed from the air stream just as the last trace of solvent disappeared.

The modified solvent evaporator has at least two advantages over the standard version: a) freedom of choice of type and of size of vial or tube and b) long tubes can be raised during the concentration step so that small volumes of solution in the bottoms of the tubes can be more readily seen, whereas tubes in the standard version must remain in a fixed position well below the water level of the bath, causing poor visibility of samples. To preclude the possibility of needless pesticide losses, it is important to be able to see the samples so that they may be isolated individually from the air stream (or from the vacuum source, standard version) just as the last trace of solvent disappears. Often, however, it is not necessary to watch so closely because a frequently used concentration technique is to concentrate only to a small volume, rather than to solvent dryness; typically, calibrated tubes are used, in which case concentration proceeds to any point below some required volume and then adjustment is made with added solvent to the exact volume requirement.

From presently available commercial equipment, it is difficult to find a solvent evaporator system offering more versatility, convenience, and efficiency than this modified version offers.

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