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Loss of Mirex on Evaporation of Aqueous Solutions

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Mirex is a polycyclic chlorinated insecticide in the Southeastern United States for control of the imported fire ant (*Solenopsis richteri and Solenopsis invicta*) (COON and FLEET, 1970). Toxicological studies on Mirex are being carried out in our Institute which involve analysis of plasma, tissue, fat and feces by gas, thin layer and high pressure liquid chromatography (WIENER, 1976). Often these methods require clean-up procedures and concentration of solutions to small volumes. This is usually accomplished by drying over a stream of nitrogen, evaporating under a hood or using a rotary evaporator. It has been demonstrated that considerable losses of pesticides can result from evaporation of solutions to small volumes (BURKE, 1966). The work described here attempts to show the large losses of Mirex that result when concentrating aqueous solutions containing Mirex.

METHOD

Solutions of ^{14}C Mirex (obtained from Mallinckrodt) in various organic solvents were prepared by adding 10 microliters of stock solution to counting vials containing various organic solvents and aqueous-organic mixtures. All organic solvents used in this experiment were pesticide grade (Anachemia, Inc.). Experiments were performed in triplicate using low (0.1 to 0.2 µg/ml) levels of Mirex and determinations made by liquid scintillation spectrometry using a Packard Tri-Carb Model 3380.

Evaporation was carried out by drying over a stream of nitrogen (22°C), evaporating under a hood (22°C), and using a rotary evaporator. Solutions were evaporated to dryness and to 0.1 ml and samples of various percentages of water in organic solvents were evaporated, usually to dryness, for a constant period of time.

Controls treated in the same manner as the experimentals were used for each set of solutions and were prepared by adding ten microliters of a ^{14}C Mirex stock solution to counting vials containing pure organic solvents. The percent recovery was calculated from the ratio of counts of controls to counts of solution after evaporation.

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RESULTS AND DISCUSSION

Solutions of Mirex in various organic solvents were evaporated to dryness using the methods mentioned above. Table I is a summary of the data and shows nearly total recovery. It was determined that, of the three methods, evaporation using a rotary evaporator was the most convenient. Therefore, all solutions were evaporated using a rotary evaporator.

TABLE I

Evaporation of Mirex from Hexane and Ethanol Using Various Methods[a]

Sample	Over N_2	Under a Hood	Rotary Evaporator
Hexane	99%	99%	99%
Ethanol	99%	96%	98%

[a]Samples evaporated at 22°C.

Various organic solvents (methanol, acetone, ethanol, ethyl acetate, petroleum ether, acetonitrile, and hexane) containing Mirex were evaporated to dryness and the percent recovery determined to be near 100%.

Solutions containing different percentages of water in organic solvents were prepared and evaporated to dryness at constant time. The percent recovery decreases as the percent of water increased as shown in Table II. Samples containing greater than 50% water were not used since the solubility limit of Mirex was approached in solutions containing greater than 50% water. Solutions of 50% organic - H_2O (v/v) were also evaporated to dryness and to 0.1 ml using a rotary evaporator. There appeared to be little difference in the recovery obtained between samples evaporated to dryness and those evaporated to 0.1 ml. The results are by no means intended to represent absolute values because the rate of loss of Mirex may vary according to conditions (mean of evaporation, vacuum obtained from rotary evaporator, etc.).

These data, however, indicate that losses of Mirex can be expected if aqueous solutions containing Mirex are allowed to evaporate. Therefore, necessary precautions should be taken when concentrating Mirex in aqueous solutions containing organic solvents.

TABLE II

Loss of Mirex from Water Using A Rotary Evaporator[a]

Percent Water	% Recovery			
	Acetonitrile	Ethanol	Methanol	Acetone
10	90	94	86	87
20	82	81	64	76
40	55	48	28	65
50	52	41	18	61

[a]Samples evaporated at 35°C for a constant time.

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