A T.L.C. Procedure For Identification of DDT and Its Metabolites in Presence of PCB

L. Laitem and P. Gaspar
Laboratory of Food Analysis
Veterinarian Medicine Faculty
University of Liège
Bruxelles, Belgium

G.L.C. is used in most procedure for the detection of organochlorinated pesticides in food. For some years, T.L.C. has been used as a purification and confirmation method for these pesticides in presence of PCB* (ABBOTT 1969, FEHRINGER 1971, VISWESWARIAH 1971, SANDRONI 1971, COLLINS 1972).

In this paper we describe a screening procedure allowing the detection of DDT and its metabolites and their identification in presence of PCB in fatty tissues.

MATERIAL AND METHODS.

25 g. of fatty tissues were extracted with 200 ml of light petroleum (60-80° C) for 4 hours in a Soxhlet apparatus. In order to precipitate the dissolved fat, the extract was kept at 4° C during 4 hours. After filtration, the extract was transferred in a separatory funnel and washed with 25 ml of concentrated sulfuric acid. In order to complete separation, the lower layer was centrifugated. Combined organic layers were dried over anhydrous sodium sulfate and concentrated to a volume of 1 ml. This solution, transferred on the top of a micro-column (25 cm height, 0.5 cm inner diameter) filled with alumina (5 % deactivated, 3 cm height), was eluted with 2 ml of hexane. The eluate was then evaporated under a nitrogen stream to a volume of 0.1 ml. 40 ul of this concentrated were spotted on an alumina plate and development was performed with hexane in a lined tank. Spots were visualized classicaly by spraying plates with tolidine (1 % in acetone) and irradiated with U.V. light (360 nm) for 20 min.

As confirmation test, the remaining 60 ul solution was evaporated to dryness. Add 0.5 ml sodium hydroxyde in alcohol (10 %) to the residues. After 2 minutes, and addition of 5 ml water followed by 1 ml saturated sodium sulfate water solution, the mixture was extracted with 1 ml light petroleum. The separated organic layer was reduced to a volume of 50 ul and 40 ul of this solution were chromatographied as described above.

* polychlorinated biphenyls.

Bulletin of Environmental Contamination & Toxicology,
0007-4861/78/0019-0264 $1.00

T A B L E

	Rf	
	Detection	Confirmation after dehydrochlorination
DDT	47	69
DDE	69	69
DDD	21	59
PCB	72	73

RESULTS AND DISCUSSION.

The purification procedure described above is an improvement of the methods of MURPHY (1972) and LAW (1970). The use of a micro-column as purification step presents many advantages with respect to classical adsorption columns (speed, smaller volumes of solvent and adsorbent). The sensitivity obtained in our procedure is about 20 ppb for DDT and its metabolites and 50 ppb for PCB. To confirm the detection of DDT and related compounds at these levels it was necessary to dehydrochlorinate the samples and perform a second chromatographic step.

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