Simultaneous Detection of Some Organophosphorous Pesticides in Whole Wheat Flour

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Determinations of organophosphorus pesticides and their metabolites extracted from stored wheat grains (1-3), maize (1), barley (4) and rice brans (5) were reported in conjunction with metabolism studies. A thin-layer chromatographic-enzyme inhibition (TLC-EI) procedure was proposed for screening organophosphorus pesticide residues in plant extracts without elaborate clean-up (6). Except with potato extracts, negligible interference was encountered. However, interferences from potato were circumvented by using carbon-column fractionation (7). In this report, the screening procedure (6) for detecting a mixture of 7 organophosphorus pesticides is evaluated on whole wheat flour extracts without elaborate clean-up.

Materials and Methods

Whole wheat flour samples were extracted by the procedure of McLeod et al. (8). Frothing during flash-evaporation was eliminated by adding approximately 100 ml of hexane to the extract. Each extract (equivalent to 50 g of sample) partitioned into hexane was fortified with azinphosmethyl (Guthion) at 1.00 ppm, carbophenothion (Trithion) at 0.50 ppm, diazinon at 0.01 ppm, ethion at 0.10 ppm, malathion at 8.00 ppm, mevinphos (phosdrin) at 0.25 ppm, and parathion 1.00 ppm. Two aliquots of subsamples C (control), BE (fortified before the extraction step), and F (fortified after the partition step) were analyzed by TLC-EI technique (9) on duplicate plates using 5-bromoindoxyl acetate as substrate.

Gas-liquid chromatograph (GLC) with electron capture detector (6) was used to analyse the wheat extracts partitioned in hexane.

Results and Discussion

Fig. 1 shows that carbophenothion, ethion, and parathion TLC spots were well defined even in aliquots equivalent to 100, 150, and 200 mg of sam-

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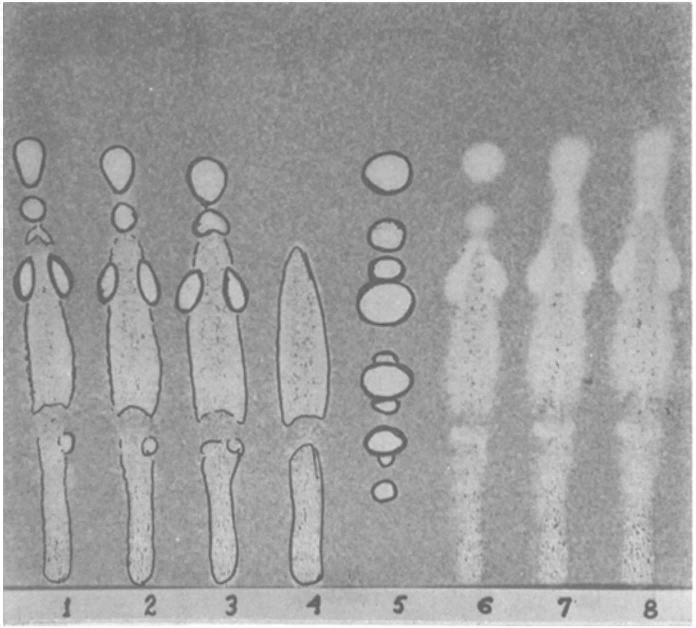

Fig. 1. Typical chromatograms of whole wheat flour fortified with pesticides. Outlines of areas of inhibition at positions 1 to 5 are traced with india ink to improve the contrast. Positions 1,6 each has 0.1 g sample; 2,7 each has 0.15 g; 3,8 each has 0.20 g; 4 has 0.20 g of control sample; 5 has a mixture of standards,*e.g.* from top to bottom,carbophenothion, ethion, diazinon, parathion, malathion impurity, malathion, carbophenothion impurity,azinophosmethyl, parathion impurity, mevinphos.

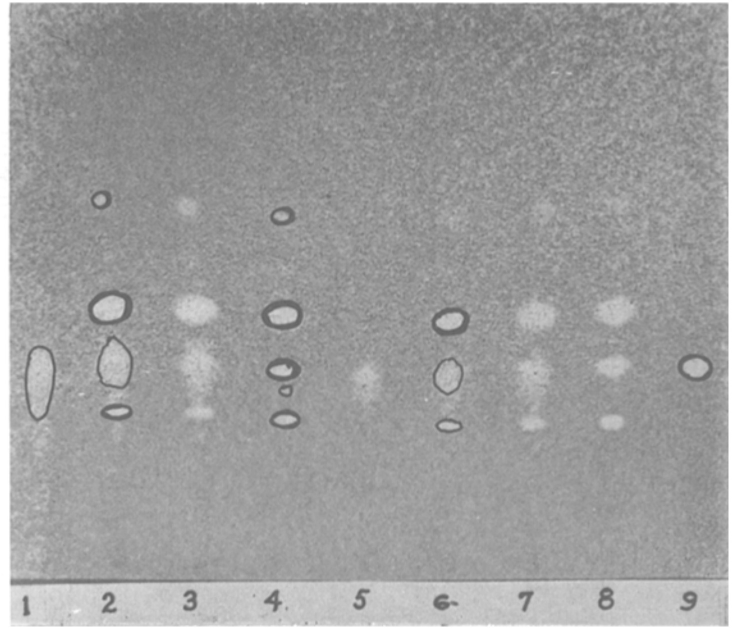

Fig.2. Typical chromatogram of whole wheat flour extracts fortified with pesticides. Outlines of representative areas of inhibition are traced with india ink to improve contrast. Positions 1,2,3 each has 5mg of sample;5,6,7 each has 2.5mg; 4,8 each have a mixture of standards from top to bottom; carbophenothion,parathion,malathion, carbophenothion impurity, azinphosmethyl; 9 has 40 ng of malathion.

ples; diazinon and azinphosmethyl spots were barely visible because of interference in the samples. Mevinphos was not detected in any of the aliquots. The results obtained with F sub-samples were comparable to those obtained with BF sub-samples.

Fig. 2 shows that carbophenothion, parathion, and azinphosmethyl TLC spots were distinct even in the presence of wheat crude extracts equivalent to 2.5 and 5.0 mg of samples, whereas diazinon gave a non-persistent spot. Malathion spots were very diffuse at the levels used and were indistinguishable from an unknown component originally present in the samples.

GLC analysis of aliquots equivalent to 50 μg of sample distinctly detected malathion, parathion and carbophenothion added to whole wheat flour extracts. The other pesticides were either barely detected or not detected. The chromatograms were similar to those obtained previously for other samples (6) and did not show any peaks that would interfere with the pesticides detected.

Thus, the results showed that the TLC-EI technique is useful for screening and determining carbophenothion, diazinon, parathion, and azinphosmethyl in whole wheat flour extracts without elaborate clean-up and that GLC can be used to rapidly screen the extracts for malathion, parathion and carbophenothion.

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