Bull. Environ. Contam. Toxicol. (1986) 36:230–233

Environmental Contamination and Toxicology

Determination of Chlorinated Pesticides in Tobacco

Stefan M. Waliszewski[1] and Grzegorz A. Szymczyński[2]

[1]Institute of Plant Protection, Poznań, Poland, and [2]Department of Andrology, Medical School of Poznań, Jackowskiego 41, PL-60-513 Poznań, Poland

One of agricultural products is tobacco which according to Bundesgesetzblatt 22 Berlin, W.Germany, June 29, 1982, may contain up to 10 ppm of total DDT, 1.0 ppm of HCH isomers and 0.3 ppm HCB. In many papers on contamination of humans with these pesticides, tobacco smoking was taken into consideration. The smokers showed increased levels of residues in fat tissue. The available analytical methods of determination of residues of chlorinated pesticides in tobacco are extremely labor-consuming and expensive as they include clean-up of raw extracts on adsorbents /Richter 1978/ or apply Sweep-Co-Distillation coupled with column chromatography /Eichner 1978; Pflugmacher and Ebing 1979; Pflugmacher and Ebing 1979/. The methods developed recently in our laboratory simplify the analysis and reduce its cost by replacing adsorbents with 1 ml of concentrated sulphuric acid. Such clean-up was successfully applied also to tobacco.

MATERIALS AND METHODS

150 ml of the acetonitrile and water mixture /65+35/ were poured over a 10 g tobacco sample in a mason jar of the OMNI Mixer - Du Pont homogenizer and the content was left for 16 h. Thereafter, it was mixed for 3 min, the supernatant was vacuum filtered through a layer of Celite 545 and transferred to a separatory funnel of 1000 ml; the tobacco sample was mixed additionally twice, each time with 100 ml of the mixture of acetonitrile and water /65+35/. 500 ml of a 5% solution of sodium sulphate were added to the combined extracts in the separatory funnel and were extracted three times, each time with 100 ml of petroleum ether. The ether extracts were dried by filtering through sodium sulphate and evaporated to approximately 5 ml in a rotary evaporator at the temperature of 40°C. The concentrated ether extract was transferred quantitatively with petroleum ether

to a calibrated tube of 10 ml, and the volume was adjusted to 10.0 ml. One ml of concentrated sulphuric acid was added and the content was vigorously shaken for about 1 min. Thereafter, it was left for 2-3 min and the upper petroleum ether phase was dried by filtering through sodium sulphate.

Qualitative and quantitative analyses were performed on Varian gas chromatogram Model 2100 with ECD Ni^{63}. The column was glass, 180 cm x 2 mm, packed with 1.5% OV-17 + 1.95% QF-1 on 80-100 mesh Gas Chrom Q. The temperatures: column - 185°C, detector - 250°C, injector - 250°C. Carrier gas was nitrogen at the flow rate of 20 ml/min.

RESULTS AND DISCUSSION

In order to determine precision of the presented analytical method recovery studies were repeated ten times for tobacco samples. A mixture of standards was added 1 h before the sample was flooded with the mixture of acetonitrile and water /65+35/. The obtained recovery for HCB, pp'-DDE and isomers of HCH and DDT, and statistical analysis of the method, fortification levels and detection limits for analyzed pesticides are presented in Table 1. The obtained recoveries for all analyzed substances were over 92%, except delta-HCH - 88.2%; standard deviation /S.D./ and coefficient of variation /V%/ were low. This indicates a very good recovery of the examined substances from the samples, and an excellent repeatability of the presented method. Besides, the detection limits presented in Table 1 point to a good sensitivity of the method, which permits for determination of chlorinated pesticides in tobacco at sub-ppm range.

Table 1. Fortification levels, recovery and detection limits of some pesticides in tobacco samples n=10

	fortification levels/ppm/	x ± SD	V%	detection limit/ppm/
HCB	0.01	97.9±1.73	1.69	0.001
alpha-HCH	0.01	98.2±2.65	2.60	0.001
beta-HCH	0.02	92.9±5.67	5.26	0.002
gamma-HCH	0.02	96.2±3.82	3.68	0.002
delta-HCH	0.02	88.2±5.68	5.01	0.002
epsilon-HCH	0.02	93.0±7.81	7.26	0.002
pp'-DDE	0.05	96.6±1.49	1.44	0.004
op'-DDT	0.07	96.5±1.57	1.51	0.005
pp'-DDT	0.12	98.5±0.50	0.49	0.007

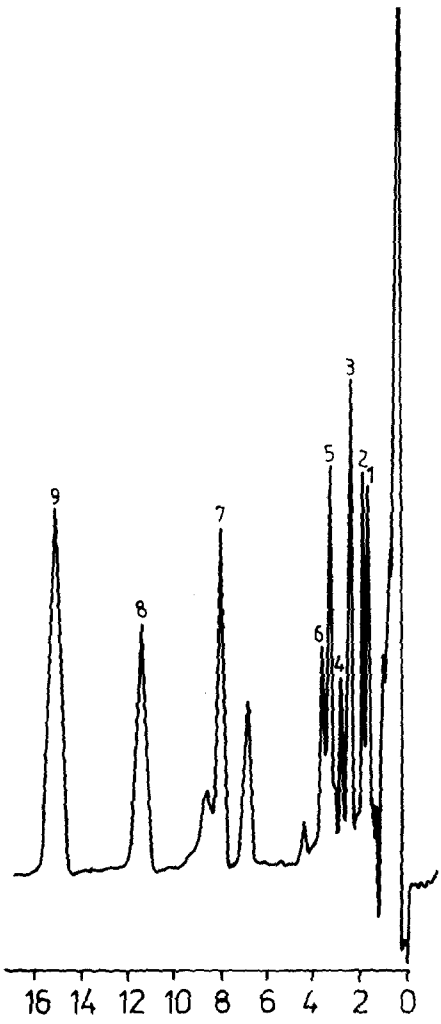

Figure 1. Gas chromatogram of a tobacco sample fortified with the amount of pesticides in Table 1.

The use of the mixture of acetonitrile and water /65 + 35/ for dry materials and the period of initial maceration of 16 h resulted in a good release of the residues of pesticides under study from analyzed samples. The application of concentrated sulphuric acid instead of conventionally used Florisil, Aluminium oxide or Silica gel not only gives substantial economical savings but the acid itself induces breakage of the complex of pesticide and endogenous substance, thus giving a truer image of contamination of tobacco samples with the residues of chlorinated compounds. Besides, the method is very little labor-consuming and uncostly, and reduces by 50% the cost of analysis in comparison to the methods using clean-up on adsorbents or Sweep-Co-Distillation. A typical gas chromatogram of a tobacco sample fortified with the amount of pesticides from Table 1 is presented on Figure 1. The chromatographic image is clear and the basic line is stable, which is very difficult to obtain in the analysis of tobacco samples.

Financial advantages of this technique and good repeatability recommend it for routine monitoring of contamination of tobacco with persistent chlorinated pesticides.

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Received March 3, 1985; accepted June 12, 1985