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Organochlorine Pesticide Residues in Animal Feed by Cyclic Steam Distillation

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It is well established that organochlorine pesticides (OCP's) are among the most persistent and toxic pollutants both in the terrestrial and aquatic ecosystems. The use of these pesticides has been banned in many countries since several years, but they are still used in Chile especially to protect the large beet crops in the south of the country. Only DDT and its derivatives were banned in 1986, after its findings in meat and milk. The other organochlorine insecticides have a widespread application and only a few data about their levels in the Chilean environment have yet been reported.

As part of our environmental contamination studies by pesticides, we have investigated the presence of organochlorine insecticide residues in cossettes, a by-product from the beet sugar industry, extensively utilized as animal feed.

The beet sugar industry in Chile reaches 300.000 tons per year and about 15.000 tons are represented by the cossettes which are mainly used as animal feed, specially for cows and swines. In the sugar production process, beets are washed with water and sliced into spaghetti-like shapes that are water extracted, leaving residues which after being dried and pressed into different shapes are called cossettes. The cossettes contain about 10% water and are directly used as animal feed.

Currently, there are many multiresidue methods available for the extraction and cleanup of chlorinated pesticides, but these methodologies are usually time consuming and/or expensive (Pesticide Analytical Manual 1982). In view of these limitations, the extraction and cleanup of the pesticides from the cossette samples was performed with a modified Nielsen-Krieger steam distillation apparatus (Veith & Kiwus 1977) constructed in our laboratory (Albornoz & Ober 1980).

Cyclic steam distillation of OCP residues has been succesfully applied by other authors to soils, sediments and birds of Prey (Cooke et al. 1979; Cooke et al. 1980; Chang-Yen & Sampath 1984). We utilized this technique for fruits and vegetables (Santa María et al. 1986).

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Table 1. Recovery of organochlorine insecticides from cossettes by cyclic steam distillation.

	Fortification level (ppm)	Recovery* (ppm)	% Recovery
Lindane	0.035	0.028	80.0
Heptachlor	0.023	0.012	53.8
Aldrin	0.012	0.010	87.5
Heptachlor epoxide	0.021	0.018	85.6
Dieldrin	0.080	0.077	96.5

* Mean value of a sample run in triplicate.

Here we report the recovery studies of the organochlorine pesticide residues from cossette samples using cyclic steam distillation and their contents in samples as used for animal feed.

MATERIALS AND METHODS

Analytical standards of the organochlorine pesticides were supplied by the Environmental Protection Agency (U.S.). Standard stock solutions (200 ug/uL) were prepared in iso-octane and kept refrigerated. All solvents used were pesticide grade.

The cossette samples were obtained directly from the sugar-derived feed factories. The samples, about 5 kg, were taken randomly for analysis in order to get an average for the feed.

The distillation-extraction of the chlorinated pesticides was performed as described previuosly (Santa María et al. 1986). The sample (25 g) was blended with water (1 L) and boiled for 1 hour. Hexane (10 mL) was used as the extraction solvent. The hexane extracts were suitable for direct gas chromatographic analysis without any time-consuming concentration or cleanup procedure.

Gas chromatographic analysis were performed using a Perkin-Elmer Sigma 3 chromatograph equipped with a Ni-63 electron capture detector and fitted with a 3.5 m x 2.0 mm coiled pyrex glass column packed with 1.5% OV-17/1.95% QF-1 on Chromosorb W HP (100-120 mesh). The injector, column and detector temperatures were 220, 200, and 300 C respectively. 95% argon/5% methane (Matheson, U.S.) was used as carrier gas and its flow rate was adjusted to 35 mL/min. Quantification was performed with a Perkin-Elmer Sigma 3 Data Station.

The recovery studies were carried out by fortification of the less contaminated cossette sample with lindane, heptachlor, aldrin,

Table 2. Organochlorine insecticide contents in the cossette samples as used for animal feed (ppm). Average values of a sample run in triplicate.

	Sample No.				
	1	2	3	4	beet
Lindane	0.012	0.009	0.011	n.d.	<0.01
Heptachlor	0.008	0.022	0.004	n.d.	n.d.
Aldrin	0.028	0.029	0.026	0.01	<0.01
Heptachlor epoxide	0.040	0.180	0.003	n.d.	n.d.
Dieldrin	0.041	0.056	0.008	0.003	<0.001

n.d.= not detected

heptachlor epoxide, and dieldrin. The mixture was tumbled for several hours and two assay portions were taken. After the distillation-extraction process, the feed samples were analyzed directly by gas chromatography as described above.

RESULTS AND DISCUSSION

Gas chromatograms of the organochlorine residues in the cossette extracts are shown in Figure 1. The identity of the peaks was further confirmed by running the samples in a 2 m x 2 mm pyrex glass column packed with 3% OV-225 on Chromosorb W HP (100-120 mesh).

Table 1 shows the fortification levels and recoveries of the individually tested insecticides. The organochlorine contents of the analyzed cossette samples are shown in Table 2.

As it can be seen from Table 1, recoveries of the OCP from the spiked cossette sample, except for heptachlor, ranged from 80 to 96%. Recovery precision was good, generally within less than 4% variation between successive samples. The extracts were clean enough as to be analyzed directly without any further purification process (Figure 1). The benefits of the extraction-distillation method utilized here are easily recognized by observing the chromatograms presented in Figure 1.

All the analyzed feed samples appeared to contain organochlorine pesticide residues (Table 2). Though the level of the residues is not extremely high, the reported values are very close to the recommended maximum limits for similar products, especially in what heptachlor and heptachlor epoxide is concerned. Table 2 also shows the OCP contents of a fresh beet sample collected from one of the

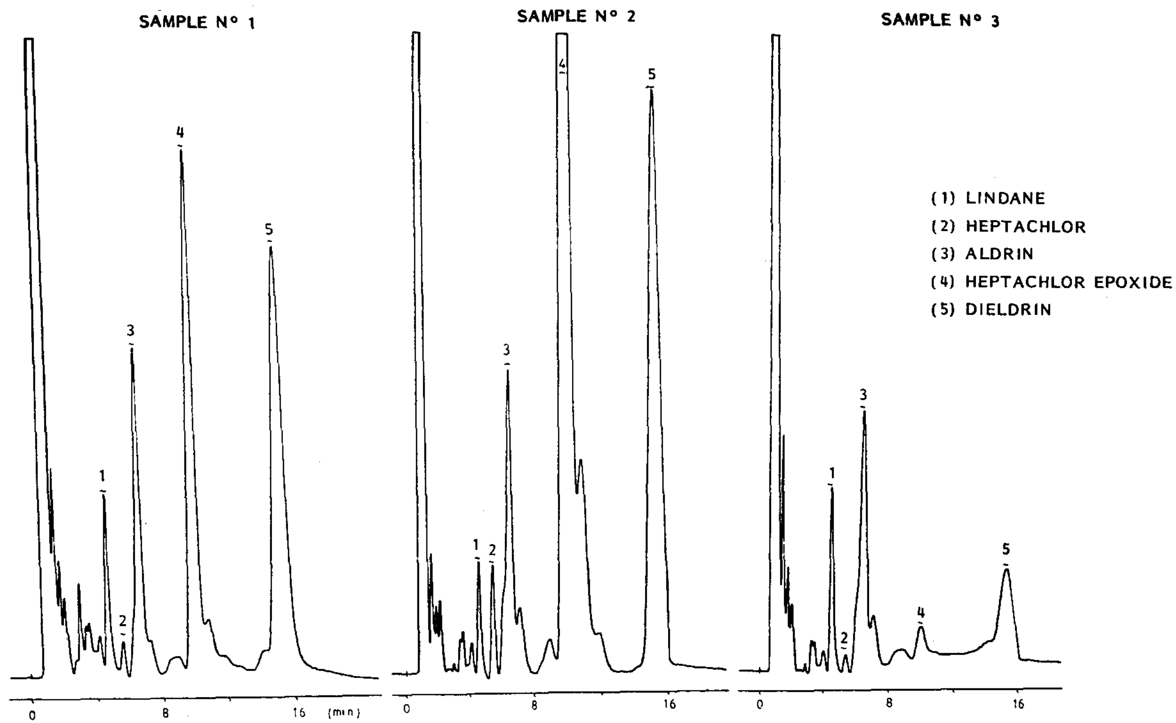

Figure 1. Gas chromatograms of the cossette samples extracts. Glass column (3.5 m x 2.0 mm) packed with 1.5% OV-17/1.95% QF-1 on Chromosorb W HP (100-120). Temperatures: column oven, 300 °C; Ni-63 ECD, 200 °C.

crop fields nearby one of the factories. The presence of organochlorine pesticide residues in these animal feeds indicates a potential source of OCP contamination for the Chilean population. These results seem to indicate a way to explain the presence of OCP's in milk and beef which forced the authorities to ban the use if DDT and its derivatives since 1986.

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