Environ Monit Assess (2011) 174:327–335
DOI 10.1007/s10661-010-1460-1

Persistent organochlorine pesticide residues in animal feed

Subir Kumar Nag · Mukesh K. Raikwar

Received: 4 September 2009 / Accepted: 7 April 2010 / Published online: 5 May 2010

Abstract Animal products like milk and meat are often found to be contaminated with residues of persistent pesticides and other toxic substances. The major source of entry of these compounds to animal body is the contaminated feed and fodder. So, unless the residues are managed at this stage, it is very difficult to prevent contamination in milk and meat. Therefore, the status of residue level of most persistent organochlorinated pesticides (OCP) in feed and fodder should be monitored regularly. The frequency of occurrence and contamination levels of OCP residues in different kinds of animal concentrate feed and straw samples collected from Bundelkhand region of India were determined. Out of 533 total samples, 301 i.e. 56.47% samples were positive containing residues of different OCPs like hexachlorocyclohexane (HCH) isomers, dichlorodiphenyltrichloroethane (DDT) complex, endosulfan and dicofol. Among different HCH isomers, the mean concentration of β-HCH was highest, and total HCH varied from 0.01 to 0.306 mg kg^{-1}. In case of DDT complex, i.e. DDD, DDE and DDT, the concentration ranged between 0.016 and 0.118 mg kg^{-1} and the pp$^{|}$ isomers were more frequently encountered than their op$^{|}$ counterparts. Endosulfan was also found in some samples in concentration ranging from 0.009 to 0.237 mg/kg, but dicofol could be recorded in very few samples. Although feed samples were found to contain OC residues, after comparing their levels in positive samples with the limiting values of respective pesticides, only very few were found to exceed the threshold level. Otherwise, they were mostly within safe limits.

Keywords Organochlorine pesticide residues · Feed

S. K. Nag (✉) · M. K. Raikwar
Plant Animal Relationship Division,
Indian Grassland and Fodder Research Institute,
Jhansi, 284 003 (UP), India
e-mail: subirknag@yahoo.com,
nagsk_67@rediffmail.com

Introduction

Organochlorine (OC) pesticides, an important group within the POPs, have potential of polluting the environment during the last few decades almost everywhere. They have high bioaccumulation potential due to chemical inertness, persistence, lipophilic nature and very less biodegradability. Although banned and/or restricted in most of the developed countries, some developing countries are still using some of the OC pesticides, mainly because of their low cost,

to get rid of the pests damaging agricultural and horticultural crops and also to eradicate vectors. The residues of these compounds are still found to some extent in various substances in some countries. So, there is a need for further monitoring of samples for residues of this group of pesticides.

Feed and fodder, when contaminated, act as main source of entry of pesticides in animal body. Once the animal body system gets contaminated with pesticide residues, not only does it affect the animals directly but also exerts indirect effect on human health through food of animal origin like milk and meat. Therefore, unless residues in feed and fodder are controlled, pesticides are likely to accumulate in animal body tissues and then excrete in milk. Most of the residue-monitoring programmes are concentrated on food crops, fruits and vegetables. As evidenced from survey of literature, only a few reports about the status of pesticide residues in feed and fodder are available from India (Kaphalia and Seth 1982; Shastry 1983; Dikshith et al. 1989; Singh et al. 1997; Gupta et al. 2000; Prasad and Chhabra 2001; Kang et al. 2002) and abroad (Pierson et al. 1982; Lovell et al. 1996). Therefore, the present investigation was undertaken to analyse 533 feed samples collected from different areas of Bundelkhand region of India during the period of 2002–2005 for persistent OC residues.

Materials and methods

Sampling

Feed samples comprising of oilseed cakes of mustard (*Brassica* sp.), linseed (*Linum usitatissum*), sesamum (*Sesamum indicum*), cotton, pulse byproducts i.e. chunnies (powdered outer seed coat) of gram (*Cicer arietinum*), lentil (*Lens culinaris*), pea (*Pisum sativum*), urd (*Vigna* sp.), arhar (*Cajanus cajan*), cereal straw (wheat, paddy), cereal by products (chunnies of oat, rice, wheat flour) and pashu ahar™ (commercial concentrate mixture) were collected from different places like Jhansi, Banda, Gwalior, Jalaun, Tikamgar, Hamirpur, Chattarpur, Panna, Sagar and Bhind which cover the Bundelkhand region of India.

Analytical procedure

Extraction and clean-up

The analytical methods of Luke et al. (1975) and Nakamura et al. (1994) were followed with modifications. Samples ground to a fine powder (20 g) were extracted in soxhlet for 8 h continuously in hexane. The extract was dried, concentrated and cleaned up by partitioning and column chromatography using florisil.

Gas chromatographic analysis

The qualitative and quantitative determination was done in gas chromatography on a Varian CP-3800 equipment fitted with Ni^{63} electron capture detector. The column used was WCOT fused silica capillary having dimension of 30 m × 0.32 mm id × 0.25 μm film thickness (CP-SIL 5 CB). The operating conditions of GC were as follows:

Temperatures

Column – 180°C (1 min) $\xrightarrow{3^{o}C\,min^{-1}}$ 250°C (5 min)

Injector – 260°C, Split (1:10)

Detector – 300°C

Carrier gas: Nitrogen at a flow rate of 1 ml min^{-1} through column and 30 ml min^{-1} make up.

The identification of peaks and quantification of concentration was done based on the external standard solution injected initially and after every five samples.

The pesticides determined were α-HCH, β-HCH, γ-HCH, δ-HCH, op'DDE, pp'DDE, op' DDD, pp' DDD, op' DDT, pp' DDT, aldrin, heptachlor, α-endosulfan, β-endosulfan, endosulfan sulfate and dicofol. A working standard solution of mixture of all the above pesticides was prepared by combining aliquots of each individual stock solution of 100 μg/ml and diluting to a concentration of 1.0 μg min^{-1} with isooctane. The standard solution was stored in standard stoppered tubes at 4°C in refrigerator.

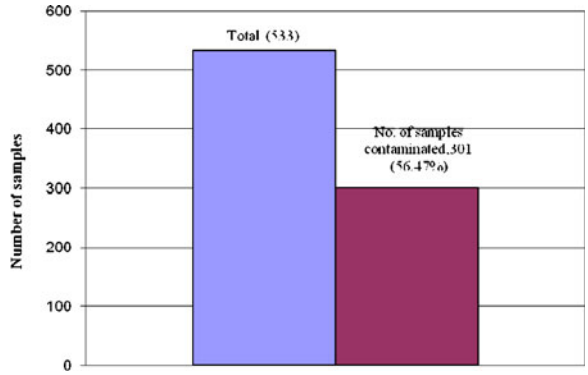

Fig. 1 Extended of contamination of feed samples with OC pesticide residue

Results and discussion

Method performance and validation

The detector linearity was tested by linear regression analysis of five-point response versus concentration calibration curve for each analyte. Linear regression equations were used to quantify analytes in samples. Calibration of gas chromatograph was done before sample analysis using the standards of pesticides obtained from authentic sources. Qualitative and quantitative analyses were performed by comparing the retention time and peak area of the samples, respectively, with those of the calibrated reference standards.

Recovery experiment was conducted by spiking the feed samples with all the pesticide standards taken for analysis at 0.1 and 0.5 mg kg^{-1} level to see the efficiency of extraction and analytical procedure. The mean average recovery varied from 88–95% with standard deviation being less than 8 U indicating good repeatability of the method. The limit of detections were 0.001 mg kg^{-1} for HCH (α, γ, δ), 0.002 mg/kg for endosulfan (α, β, sulfate), 0.003 mg/kg DDT (op$^{|}$ and pp$^{|}$ isomers of DDE, DDD and DDT) and 0.005 for β-HCH and dicofol.

Residues in the samples

The overall and relative contamination of different feed samples is shown in Figs. 1 and 2, respectively. Out of 533 samples analysed for

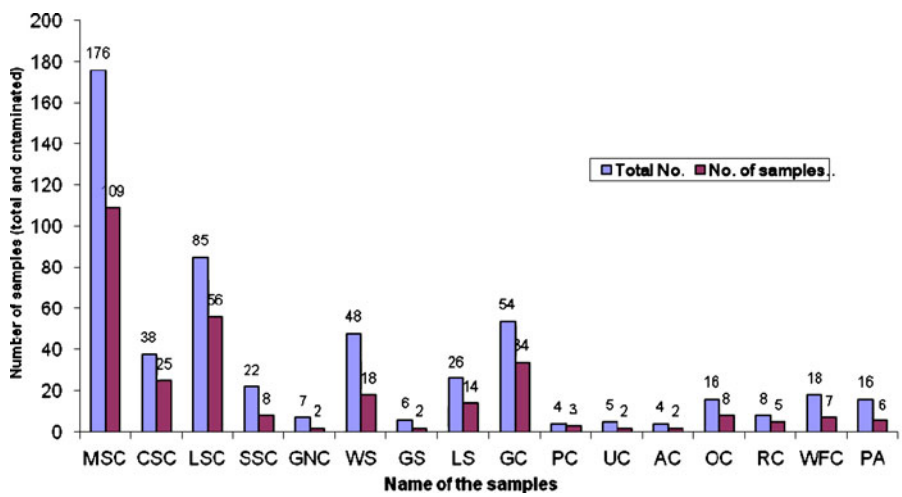

Fig. 2 Level of contamination of different feed samples with OC pesticide residues

Table 1 Concentration of HCH isomers and total HCH (mg kg^{-1}) in feed samples

Samples	α-HCH	β-HCH	γ-HCH	δ-HCH	Σ-HCH
Mustard seed cake	Tr–0.04 (0.008 ± 0.008)	Tr–0.46 (0.058 ± 0.092)	Tr–0.092 (0.007 ± 0.013)	Tr–0.117 (0.013 ± 0.027)	Tr–0.478 (0.053 ± 0.085)
Linseed cake	Tr–0.06 (0.011 ± 0.011)	Tr–0.13 (0.02 ± 0.032)	Tr–0.033 (0.006 ± 0.006)	Tr–0.115 (0.011 ± 0.024)	Tr–0.1697 (0.028 ± 0.038)
Cottonseed cake	Tr–0.07 (0.011 ± 0.018)	Tr–0.916 (0.367 ± 0.38)	Tr–0.091 (0.016 ± 0.027)	Tr–0.043 (0.015 ± 0.015)	Tr–0.921 (0.1677 ± 0.285)
Sesamum seed cake	Tr–0.023 (0.009 ± 0.01)	Tr–0.012 (0.006 ± 0.004)	Tr–0.024 (0.007 ± 0.009)	0.002–0.005 (0.0033 ± 0.002)	0.003–0.035 (0.013 ± 0.013)
Groundnut cake	–	–	–	–	–
Wheat straw	Tr–0.007 (0.004 ± 0.002)	Tr–0.047 (0.018 ± 0.018)	Tr–0.008 (0.004 ± 0.003)	Tr–0.024 (0.007 ± 0.008)	Tr–0.06 (0.018 ± 0.018)
Lentil straw	0.005–0.095 (0.021 ± 0.029)	0.012–0.062 (0.037 ± 0.0246)	0.004–0.0284 (0.015 ± 0.009)	0.002–0.034 (0.017 ± 0.013)	0.01–0.21 (0.055 ± 0.062)
Gram straw	0.0073		0.0084	0.0293	0.045
Oat chunni	0.002–0.043 (0.011 ± 0.012)	0.01–0.052 (0.029 ± 0.015)	0.004–0.069 (0.025 ± 0.027)	0.009–0.034 (0.023 ± 0.009)	0.01–0.161 (0.053 ± 0.043)
Urd chunni	0.013–0.029 (0.021 ± 0.008)	–	0.006–0.012 (0.009 ± 0.003)	0.003–0.005 (0.004 ± 0.001)	0.023–0.046 (0.034 ± 0.011)
Gram chunni	0.002–0.646 (0.162 ± 0.227)	0.025–0.516 (0.196 ± 0.176)	0.002–0.328 (0.077 ± 0.094)	Tr–0.157 (0.028 ± 0.041)	0.006–1.573 (0.306 ± 0.451)
Arhar chunni	Tr–0.004 (0.002 ± 0.001)	Tr–0.006 (0.005 ± 0.002)	0.002–0.004 (0.003 ± 0.006)	0.004–0.005 (0.0045 ± 0.001)	0.01–0.019 (0.015 ± 0.004)
Pea chunni	0.017	–	–	–	0.017
Rice chunni	Tr–0.006 (0.003 ± 0.002)	Tr	0.002–0.008 (0.005 ± 0.002)	0.004	0.007–0.0122 (0.01 ± 0.002)
Wheat flour chunni	Tr–0.042 (0.021 ± 0.02)	0.0173	0.018	0.004–0.012 (0.008 ± 0.004)	0.001–0.089 (0.031 ± 0.041)
Compound feed	0.002–0.018 (0.007 ± 0.006)	Tr	0.002–0.02 (0.009 ± 0.008)	0.002–0.006 (0.004 ± 0.002)	0.002–0.04 (0.019 ± 0.012)

different organochlorinated pesticides, 301 samples were positive containing residues of HCH isomers, DDTs, endosulfan and dicofol. About 60% samples were found contaminated in case of mustard seed cake (MSC), cotton seed cake (CSC), linseed cake (LSC), gram chunni (GC), pea chunni and rice chunni (RC). For the rest of the samples, the extent of contamination was 30–40%.

The concentrations of different HCH isomers viz., α, β, γ and δ and that of total HCH in different feed samples are given Table 1. Apart from the most active isomer i.e. γ-isomer (lindane), other isomers like α, β and δ were also detected in most of the feed samples. It was observed that among different HCH isomers, the concentration of predominantly accumulating, metabolically stable and most persistent β-HCH was highest in most of the samples. The mean total HCH concentration, which is the summation of all individual isomers, varied from 0.01 to 0.306 mg kg^{-1} in different samples, and this was found lesser than the earlier studies (Kaphalia and Seth 1982; Dikshith et al. 1989; Singh et al. 1997; Gupta et al. 2000).

Endosulfan, one of the cyclodiene groups of chlorinated insecticides, measured as its two active isomers α and β and their toxic metabolite endosulfan sulphate were detected in all types of samples except groundnut cake (GNC), gram straw (GS) and RC where no residue of endosulfan was found. The mean total endosulfan residues comprising of α and β isomers and sulphate varied from 0.009 to 0.237 mg/kg in different kinds of feed samples (Table 2). In earlier instances, endosulfan was not included normally in the monitoring programme for OCPs of feed and

Table 2 Concentration of endosulfan isomers and sulfate (mg kg^{-1}) in feed samples

Samples	α-Endosulfan	β-Endosulfan	Endosulfan sulfate	Σ-Endosulfan
Mustard seed cake	Tr–0.04 (0.007 ± 0.008)	Tr–0.166 (0.018 ± 0.032)	Tr–0.223 (0.031 ± 0.064)	Tr–0.389 (0.03 ± 0.061)
Linseed cake	Tr–0.307 (0.017 ± 0.054)	Tr–0.083 (0.016 ± 0.021)	Tr–0.148 (0.034 ± 0.053)	Tr–0.307 (0.035 ± 0.064)
Cottonseed cake	Tr–0.031 (0.012 ± 0.009)	Tr–0.032 (0.008 ± 0.008)	Tr–0.024 (0.009 ± 0.01)	Tr–0.048 (0.018 ± 0.014)
Sesamum seed cake	Tr	0.048	0.008–0.046 (0.027 ± 0.019)	0.008–0.094 (0.051 ± 0.043)
Groundnut cake	–	–	–	–
Wheat straw	Tr–0.025 (0.008 ± 0.008)	Tr–0.022 (0.007 ± 0.007)	Tr–0.029 (0.016 ± 0.01)	Tr–0.044 (0.019 ± 0.016)
Lentil straw	Tr–0.326 (0.101 ± 0.132)	Tr–0.012 (0.008 ± 0.005)	0.006	0.001–0.326 (0.087 ± 0.122)
Gram straw	–	–	–	–
Oat chunni	0.003–0.198 (0.072 ± 0.077)	Tr–0.858 (0.198 ± 0.333)	Tr–0.305 (0.104 ± 0.142)	Tr–1.287 (0.237 ± 0.435)
Urd chunni	0.0168	–	0.005–0.011 (0.008 ± 0.003)	0.005–0.028 (0.016 ± 0.011)
Gram chunni	Tr–0.074 (0.015 ± 0.019)	Tr–0.049 (0.012 ± 0.013)	0.003–0.0615 (0.020 ± 0.018)	0.003–0.12 (0.029 ± 0.033)
Arhar chunni	0.009–0.022 (0.015 ± 0.009)	0.0396	–	0.009–0.061 (0.035 ± 0.026)
Pea chunni	–	0.0092	–	0.017
Rice chunni	0.004–0.009 (0.006 ± 0.002)	0.0036	0.0031	0.008–0.01 (0.009 ± 0.001)
Wheat flour chunni	–	–	0.031–0.065 (0.048 ± 0.016)	0.031–0.065 (0.048 ± 0.016)
Compound feed	0.01–0.023 (0.017 ± 0.006)	0.023–0.046 (0.035 ± 0.011)	0.003	0.046–0.06 (0.053 ± 0.007)

Table 3 Concentration of DDTs and Dicofol (mg kg^{-1}) in feed samples

Samples	op DDD	pp DDD	op DDE	pp DDE	op DDT	pp DDT	Σ-DDT	Dicofol
Mustard seed cake	Tr–0.228 (0.034 ± 0.05)	Tr–0.123 (0.025 ± 0.033)	Tr–0.392 (0.052 ± 0.11)	Tr–0.104 (0.017 ± 0.022)	Tr–0.963 (0.164 ± 0.328)	Tr–0.406 (0.093 ± 0.143)	Tr–1.691 (0.093 ± 0.235)	Tr–0.268 (0.088 ± 0.095)
Linseed cake	Tr–0.019 (0.009 ± 0.006)	Tr–0.534 (0.059 ± 0.146)	Tr–0.78 (0.054 ± 0.176)	Tr–0.043 (0.007 ± 0.009)	Tr—-0.014 (0.004 ± 0.005)	Tr–0.011 (0.006 ± 0.003)	Tr–1.315 (0.058 ± 0.221)	0.006–0.148 (0.045 ± 0.046)
Cottonseed cake	0.016–0.092 (0.054 ± 0.037)	Tr–0.038 (0.016 ± 0.016)	Tr–0.092 (0.026 ± 0.038)	Tr–0.157 (0.063 ± 0.062)	0.264–0.612 (0.438 ± 0.174)	Tr–0.246 (0.09 ± 0.110)	Tr–0.742 (0.174 ± 0.257)	Tr–0.312 (0.118 ± 0.138)
Sesamum seed cake	–	0.037	0.587	Tr	–	0.013–0.052 (0.032 ± 0.019)	Tr–0.6 (0172 ± 0.247)	
Groundnut cake	–	–	–	0.016	–	–	0.016	
Wheat straw	0.006–0.008 (0.007 ± 0.001)	0.014–0.066 (0.04 ± 0.025)	Tr–0.06 (0.021 ± 0.021)	Tr–0.02 (0.007 ± 0.006)	Tr–0.482 (0.128 ± 0.204)	Tr–0.015 (0.009 ± 0.005)	Tr–0.607 (0.092 ± 0.195)	0.008–0.663 (0.336 ± 0.327)
Lentil straw	–	0.177	0.005–0.336 (0.153 ± 0.142)	0.006–0.019 (0.012 ± 0.005)	0.013	Tr–0.037 (0.015 ± 0.014)	0.006–0.336 (0.112 ± 0.12)	0.017–0.059 (0.038 ± 0.021)
Oat chunni	Tr–0.012 (0.008 ± 0.004)	0.009–0.206 (0.072 ± 0.080)	Tr–0.104 (0.041 ± 0.039)	0.006–0.389 (0.102 ± 0.165)	–	0.042	0.018–0.389 (0.141 ± 0.137)	0.327
Urd chunni	0.013	Tr	–	0.005–0.023 (0.014 ± 0.009)	–	0.016	0.02–0.039 (0.03 ± 0.009)	
Gram chunni	Tr–0.082 (0.03 ± 0.032)	Tr–0.472 (0.01 ± 0.162)	Tr–0.301 (0.063 ± 0.119)	Tr–0.127 (0.017 ± 0.029)	0.01–0.12 (0.046 ± 0.052)	0.006–1.152 (0.207 ± 0.361)	Tr–1.302 (0.146 ± 0.297)	0.013–0.055 (0.031 ± 0.015)
Arhar chunni	0.015–0.026 (0.021 ± 0.006)	0.0344	–	0.01–0.039 (0.024 ± 0.015)	–	–	0.024–0.1 (0.062 ± 0.037)	
Pea chunni			0.04				0.04	
Rice chunni	Tr	Tr	Tr–0.01 (0.004 ± 0.003)	Tr–0.076 (0.043 ± 0.031)	0.0084	Tr–0.012 (0.005 ± 0.004)	0.006–0.096 (0.043 ± 0.036)	
Wheat flour chunni	0.009	0.047	0.032	–	–	–	0.088	–
Compound feed	–	0.012	–	0.102	0.005	0.011	0.118	–

other samples except in rare cases as observed through scanning of literature. The focus of monitoring was mainly concentrated on DDTs and HCHs. But Prasad and Chhabra (2001) found that endosulfan constituted 8% of total OCPRs in overall feed and fodder samples collected and analysed at Karnal, India. Deka et al. (2004) also detected β-endosulfan and endosulfan sulphate in concentrated feed samples at Jorhat, Assam (India) while in Ludhiana (Punjab), India endosulfan could be detected in one sample out of 17 total feed concentrate samples (Kang et al. 2002).

DDT residues represented by op' and pp' isomers of DDD, DDE and DDT, ether singly or in different combinations, were found present in different types of samples except GS where they were totally absent. In samples of MSC, CSC, LSC, wheat straw (WS), lentil straw (LS) and GC all the components of total DDT were detected. In samples of GNC, only pp' DDE could be found. In general, it was observed that the pp' isomers of DDE, DDD and DDT were more frequently encountered than their op' isomers. The mean total DDT concentration in different feed samples varied from 0.016 to 0.118 mg kg^{-1} (Table 3) which was less than those reported by Kaphalia and Seth (1982), Dikshith et al. (1989) and Battu et al. (1996). However, Sharma et al. (2005) detected only pp' DDT in the range of 0.007 ± 0.005 μg g^{-1} in concentrate feed samples collected from Haryana (India).

Dicofol, a DDT analogue and an acaricide, was found present in the samples of only MSC, LSC, CSC, WS, LS, oat chunni (OC) and GC. In others, it could not be detected. The mean concentration ranged between 0.0305 and 0.3356 mg kg^{-1} (Table 3). In other monitoring works on feed and fodder samples for OCPs carried out at different places, the presence or absence of dicofol residues was not mentioned excepting Kang et al. (2002) who detected dicofol in two samples out of a total of 17 having mean concentration of 0.26 mg kg^{-1}.

The maximum residue limit or tolerance limit of pesticides in feed materials have not been set in India. However, the limiting value (which may be defined as the concentration of a pesticide in feed and fodder if fed to lactating animals daily, the likely residues in milk will be less than their MRL values and safe for human consumption) of a pesticide in feed can be derived on the basis of its legal permissible limit in milk and its rate of transference from feed to milk. Based on short-term feeding experiments on buffaloes, the transfer coefficients of HCH isomers and DDT

Table 4 Number of samples exceeding the limiting value of HCH isomers and total DDT

Samples	Pesticides with their limiting value and no. of samples exceeding them				
	α-HCH (0.12)	β-HCH (0.02)	γ-HCH (0.12)	δ-HCH (0.07)	Total DDT (0.10)
Mustard seed cake	Nil	19 (10.79%)	Nil	3 (1.7%)	12 (6.82%)
Linseed seed cake	Nil	4 (4.7%)	Nil	1 (1.17%)	2 (2.35%)
Cotton seed cake	Nil	4 (10.52%)	Nil	Nil	3 (7.89%)
Sesamum seed cake	Nil	Nil	Nil	Nil	1 (4.54%)
Groundnut cake	–	–	–	–	Nil
Wheat straw	Nil	2 (4.16%)	Nil	Nil	1 (2.08%)
Gram straw	Nil	–	–	–	–
Lentil straw	Nil	1 (3.85%)	Nil	Nil	3 (11.54%)
Gram chunni	8 (14.81%)	10 (18.52%)	5 (9.26%)	2 (3.7%)	7 (12.96%)
Pea chunni	Nil	–	–	–	Nil
Urd chunni	Nil	–	Nil	Nil	Nil
Arhar chunni	Nil	Nil	Nil	Nil	Nil
Oat chunni	Nil	3 (18.75%)	Nil	Nil	3 (18.75%)
Rice chunni	Nil	Nil	Nil	Nil	Nil
Wheat flour chunni	Nil	Nil	Nil	Nil	Nil
Pashu ahar	Nil	Nil	Nil	Nil	1 (6.25%)

Figure in parenthesis denote percentage of total samples analysed; (–) indicates that no sample was found containing residue of that particular compound

complex have been recommended (Kalra et al. 1986; Kapoor and Kalra 1988, 1993). Using these values, the limiting values of different pesticides in feed were calculated. Thus, the limiting values of α-HCH, β-HCH, γ-HCH, δ-HCH and total DDT were found to be 0.12, 0.02, 0.12, 0.07 and 0.10 mg/kg, respectively (Kang et al. 2002). We have compared the residue data in our samples with respect to the above limiting values (Table 4). In case of α- and γ-HCH, only eight (14.81%) and five (9.26%) samples, respectively, of GC had concentration higher than the limiting value, and in all other feed samples, the level was below the threshold limit. For β-HCH 19 (10.79%) samples of MSC, four each of LSC (4.7%) and CSC (10.52%), two (4.16%) of WS, one (3.85%) of LS, 10 (18.52%) of GC and three (18.75%) of OC contained residues higher than its limiting value of 0.02 mg kg^{-1}. Only three samples (1.7%) of MSC, one (1.17%) of LSC and two (3.7%) of GC had δ-HCH concentration above its limiting value. The limiting value of total DDT (0.10 mg kg^{-1}) was exceeded in 12 (6.82%) samples of MSC, two of LSC (2.35%), three (7.89%) of CSC, one (4.54%) of sesamum seed cake, one (2.08%) of WS, three (11.54%) of LS, seven (12.96%) of GC, three (18.75%) of OC and one (6.25%) of pashu ahar samples.

From the results of the present study, it has been revealed that feeds meant for animal consumption are still containing residues of OC pesticides to some extent though the levels of contamination are not much and number of samples exceeding the limiting value are also very less. However, regular monitoring of feed samples is necessary for checking the status of residues since contaminated feed and fodder act as the main source of entry of pesticides in animal body. Based on the results of the monitoring programme, appropriate measures should be taken so that animal products which are free from residues can be produced.

Acknowledgements The authors are highly thankful to the Indian Council of Agricultural Research (ICAR), New Delhi for providing the financial support and sponsoring the research work. The authors are also grateful to the Head, PAR Division and Director, IGFRI, Jhansi, INDIA for their encouraging support.

References

Battu, R. S., Singh, B., Chahal, K. K., & Kalra, R. L. (1996). Contamination of animal feed with residues of HCH and DDT. *Pesticide Research Journal, 8*(2), 172–175.

Deka, S. C., Barman, N., & Baruah, A. A. L. H. (2004). Monitoring of pesticide residues in feed, fodder and butter in Assam. *Pesticide Research Journal, 16*(1), 86–89.

Dikshith, T. S. S., Kumar, S. N., Raizada, R. B., & Srivastava, M. K. (1989). Organochlorine insecticide residues in cattle feed. *Bulletin of Environmental Contamination and Toxicology, 43*, 691–696.

Gupta, A., Singh, B., Parihar, N. S., & Bhatnagar, A. (2000). Monitoring of HCH and DDT residues in certain animal feed and feed concentrates. *Pestology, 24*(12), 47–49.

Kalra, R. L., Chawla, R. P., Joia, B. S., & Tiwana, M. S. (1986). Excretion of DDT residues into milk of the Indian buffalo, *Bubalus bubalis* (L.) after oral and dermal exposure. *Pesticide Science, 17*, 128–134.

Kang, B. K., Singh, B., Chahal, K. K., & Battu, R. S. (2002). Contamination of feed concentrate and green fodder with pesticide residues. *Pesticide Research Journal, 14*(2), 308–312.

Kaphalia, B. S., & Seth, T. D. (1982). Organochlorine pesticides contamination in some species of fodder grasses. *Environmental Pollution Series B, 3*, 231–237.

Kapoor, S. K., & Kalra, R. L. (1988). Residues of HCH in milk after its oral administration or dermal application to Indian buffaloes, *Bubalus bubalis* (L.). *Pesticide Science, 24*, 193–204.

Kapoor, S. K., & Kalra, R. L. (1993). Comparative excretion of DDT analogues into milk of Indian buffaloes, *Bubalus bubalis* (L.) following their oral administration. *Pesticide Science, 37*, 261–266.

Lovell, R. A., McChesney, D. G., & Price, W. D. (1996). Organohalogen and organophosphorus pesticides in mixed feed rations: Findings from FDA's domestic surveillance during fiscal years 1989–1994. *Journal-Association of Official Analytical Chemists International, 79*(2), 544–549.

Luke, M. A., Froberg, J. E., & Masumoto, H. T. (1975). Extraction and clean up of organochlorine, organophosphate, organonitrogen and hydrocarbon pesticides in produce for determination by gas liquid chromatography. *Journal-Association of Official Analytical Chemists, 58*(5), 1020–1026.

Nakamura, Y., Tonogai, Y., Sekiguchi, Y., Tsumura, Y., Nishida, N., Takakura, K., et al. (1994). Multiresidue analysis of 48 pesticides in agricultural products by capillary gas chromatography. *Journal of Agricultural and Food Chemistry, 42*(11), 2508–2518.

Pierson, D. A., Hoffman, J. S., Nord, P. J., Gebhart, J. E., & Frank, C. W. (1982). Distribution of chlorinated pesticides in animal feed components and finished feeds. *Journal of Agricultural and Food Chemistry, 30*(1), 187–189.

Prasad, K. S. N., & Chhabra, A. (2001). Organochlorine pesticide residues in animal feed and fodders. *Indian Journal of Animal Science, 71*(12), 1178–1180.

Sharma, V., Wadhawa, B. K., & Stan, H. J. (2005). Multiresidue analysis of pesticides in animal feed concentrate. *Bulletin of Environmental Contamination and Toxicology, 74*, 342–349.

Shastry, M. S. (1983). Monitoring of pesticide residues in animal feeds and animal products. *Pesticides, 10*, 36–38.

Singh, R., Kumar, B., Madan, V. K., Kumar, R., & Kathpal, T. S. (1997). Monitoring of HCH residues in animal feeds. *Indian Journal of Animal Science, 67*(3), 250–252.