LINDANE (048)

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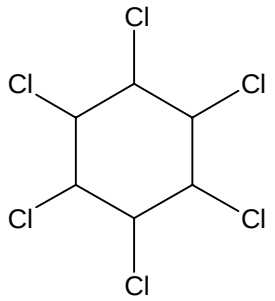
EXPLANATION

Lindane, a broad spectrum insecticide, which was originally evaluated by the JMPR in 1966 (under the name gamma-BHC) and re-evaluated for residues several times up to 1989, is included in the CCPR periodic review programme. At the 30th session of the CCPR (ALINORM 99/24), lindane was scheduled for review by the JMPR in 2003.

The basic manufacturer supplied information on identity, metabolism and environmental fate, residue analysis, use pattern, residues resulting from supervised trials on wheat and canola, fate of residues during storage or in processing, residues in animal commodities resulting from feeding, and residues in food in commerce or at consumption. In addition, GAP information and/or national MRLs were supplied by Australia, Germany, The Netherlands, Poland and the USA.

IDENTITY

ISO common name:	lindane
Chemical name	
IUPAC:	1α,2α,3β,4α,5α,6β-hexachlorocyclohexane
CA:	1α,2α,3β,4α,5α,6β-hexachlorocyclohexene
CAS Registry No:	58-89-9
CIPAC No:	488
Synonyms and trade names:	hexachlorocyclohexane (gamma-isomer); gamma-HCH, gamma-BHC (WHO, 1991)
Structural formula:	(WHO, 1991)



Molecular formula:	$C_6H_6Cl_6$
Molecular weight:	290.8 (290.9) (WHO, 1991)

Physical and chemical properties

Pure active ingredient

Property:	Value	Reference
Minimum purity:	99.5%.	(WHO, 1991)
Appearance:	Colourless, crystalline solid.	(WHO, 1991)
Vapour pressure:	4.34 mPa (3.26×10^{-5} mm Hg) at 20°C. 606 mPa (45.6×10^{-5} mm Hg) at 40°C.	(WHO, 1991)
Melting point:	112.8°C	(WHO, 1991)
Octanol/water partition coefficient:	3.2-3.7, pH and temperature not stated.	(WHO, 1991)
Solubility:	10 mg/l in water at 20°C 67 g/l in ethanol, temperature not stated slightly soluble in mineral oils, soluble in acetone and in aromatic and chlorinated solvents.	(WHO, 1991)
Relative density:	1.85 g/cm^3, temperature not stated	(WHO, 1991)

Property:	Value	Reference
Hydrolysis:	pH 5, stable, half-life 173.3 or 115.5 days, respectively, in 0.05 or 0.10 M buffer; 5% transformation after 30 days. pH 7, stable, half-life 309.4 or 281.7 days, respectively, in 0.05 or 0.10 M buffer; 5% transformation after 30 days. pH 9, unstable, half-life 36.3 or 35.4 days, respectively, in 0.05 or 0.10 M buffer; 43-44% transformation after 30 days.	(Mirfakhrae, 1986)
Photolysis:	Study 1, resistant to natural sunlight in water, ≥90% recovery after 28 days.	(Norris, 1986a)
	Study 2, resistant to natural sunlight in acetone-sensitized aqueous solution, ≥84% recovery after 28 days.	(Norris, 1986b)
	Study 3, resistant to simulated sunlight in water at pH 7 at 25°C, ≥92% recovery after 15 days.	(Willems, 1999)
Dissociation constant:	not applicable.	

Technical material

Property:	Value	Reference
Minimum purity:	99.0%	(WHO, 1991)
Main impurities:	Information on 5 impurities was made available to the Meeting	(EPA, 2001a)
Appearance:	Colourless, crystalline solid	(WHO, 1991)
Relative density:	1.85 g/cm^3, temperature not stated	(WHO, 1991)
Melting range:	>112°C	(WHO, 1991)
Stability:	Stable to light, air, heat, carbon dioxide and strong acids; dehydrochlorinated in the presence of alkali or on prolonged exposure to heat, with the formation of trichlorobenzenes, phosgene, and hydrochloric acid. Incompatible with strong bases, powdered metals (Fe, Zn, Al) and with oxidizing agents. Can undergo oxidation when in contact with ozone.	(WHO, 1991)
	Stable at 54 ± 1°C, ≥99.8% recovery after 14 days. Non-labelled lindane. After stability experiment, solid was dissolved in ethyl acetate and lindane quantified by GC-FID.	(Nunez, 2000) OPPTS guideline 830.6317, non-GLP

Formulations

The following formulations (g ai/kg or g ai/l) were identified from the information supplied by the manufacturer: DS (powders for dry seed treatment, 166, 187.5 and 250 g ai/kg); ES (emulsions for seed treatment, 120, 249 and 508 g ai/l); FS (flowable concentrates for seed treatment, 93, 353, 359, 400 and 479 g ai/l); LS (solutions for seed treatment, 69, 106 and 108 g ai/l). These formulations may contain one or more other pesticides (captan, carboxin, diazinon, mancozeb, maneb, metalaxyl, quintozene, thiabendazole, thiram).

FAO specifications for technical and formulated lindane have been published (Hauzenberger *et al*., 1990): lindane technical, 488/TC/S (1990); lindane wettable powder, 488/WP/S (1990); lindane emulsifiable concentrates, 488/EC/S (1990); lindane solutions, 488/OL/S (1990); lindane dustable powders, 488/DP/S (1990); and lindane aqueous suspension concentrates, 488/SC/S (1990).

Abbreviations used in the text for various chlorinated compounds

CP	chlorophenol
DCB	dichlorobenzene
DCP	dichlorophenol
HCB	hexachlorobenzene
HCCH	hexachlorocyclohexene
PCB	pentachlorobenzene
PCCH	pentachlorocyclohexene
PCP	pentachlorophenol
TCB	trichlorobenzene
TeCCH	tetrachlorocyclohexene
TCP	trichlorophenol
TeCB	tetrachlorobenzene
TeCP	tetrachlorophenol

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

The Meeting received information on lindane metabolism in lactating goats after oral and dermal application. Only the oral application studies were summarized, because dermal application studies are not relevant to this evaluation. In addition, the Meeting received information on metabolism in pheasants and laying hens. Metabolism in laboratory animals (mice, rats, rabbits, dogs) was summarized and evaluated by the WHO panel of JMPR in 2002.

Goats

Study 1. Three 1-3 year old lactating goats (Alpine, *Capra*) were treated orally with uniformly ^{14}C-labelled lindane (Wilkes *et al*., 1987a & 1987b). Two goats were dosed twice daily with lindane at 20 ppm, and one goat with lindane at 200 ppm, in the feed (dw) for four consecutive days (total of 8 doses). Dose rates corresponded to 1.2 mg ai/kg bw/d for goat 1, 1.5 mg ai/kg bw/d for goat 2 and 9.3 mg ai/kg bw/d for goat 3. Milk samples were collected twice daily. Urine and faeces were collected once a day. Goats were monitored for expired $^{14}CO_2$ by means of a modified indirect colorimeter chamber system. At 12-14 hrs after the last dose, goats were slaughtered and the following tissues were collected: liver, kidney, composite skeletal muscle and composite fat (subcutaneous, renal, visceral). In addition, the contents of stomach, small intestine, large intestine were collected. Samples of milk, faeces, urine, tissues and gut contents were stored at –20 °C until analysis (time not stated).

Total ^{14}C residue levels were determined by (combustion) LSC. Of the total administered radioactivity, 35-46% TAR was recovered in urine, 5.1-8.0% TAR in faeces, 4.0-4.4% TAR in tissues, 1.1-2.4% TAR in milk, 1.3-1.5% TAR in gut contents and 0.2-1.0% TAR was recovered as expired $^{14}CO_2$. From each goat, the total accountability of administered dose was low, being 51-59%. Studies with rumen fluid (Gemma *et al*., 1986 and Wilkes *et al*., 1987b) indicated that these losses were due to the formation of volatile ^{14}C-lindane metabolites that were not trapped in the trapping solutions used.

In milk, a plateau of excretion was achieved after 2-3 days, at 0.4 mg/kg eq and 3 mg/kg eq, in milk from the low- and high-dose goats, respectively. Assuming that the goat milk contained 4.1% milk fat, approximately 85% TRR in whole milk was localized in the milk fat.

In tissues from the low-dose goats, fat and liver samples contained 4 mg/kg eq, kidney 0.7 mg/kg eq and muscle <0.1 mg/kg eq. In tissues from the high-dose goats, fat contained 37 mg/kg eq, liver 20 mg/kg eq, kidney 6 mg/kg eq and muscle 5 mg/kg eq. Initially, tissues were analyzed for lindane (parent) content (Mulkey and Hallenbeck, 1987). Homogenized samples of fat, liver, kidney and muscle were extracted with ACN/water (80:20, v/v). Purified extracts were analyzed by LSC and GC. Details of the GC analysis were not submitted. Results are shown in Table 1. Essentially all of the ^{14}C activity contained in the fat and muscle was present as parent compound. In the liver, kidney and faeces, very little of the total ^{14}C activity was present as the parent compound.

Table 1. Proportion of lindane present in tissue extracts from goats treated orally with lindane (Mulkey and Hallenbeck, 1987).

Commodity	Dose rate (mg ai/kg feed)	Parent lindane, %TRR
fat, goat 1	20	84
fat, goat 2	20	82
fat, goat 3	200	86
muscle, goat 1	20	62
muscle, goat 2	20	90
muscle, goat 3	200	84
kidney, goat 3	200	4.7
liver, goat 3	200	0.4 [1/]
faeces, goat 1	20	5.2
faeces, goat 2	20	6.7
faeces, goat 3	200	3.1

[1/] Mean result of two analyses, 6 and 12 months after sampling.

The ^{14}C residues in tissues and faeces were characterized further by Mulkey and Hallenbeck, (1987). Homogenized samples of omental fat, muscle, kidney and faeces were extracted with hexane in the presence of sodium bisulfite and sodium sulfate. Hexane extracts were purified by liquid-liquid partitioning and clean-up on a Florisil column. Eluates were analyzed either directly or after methylation with diazomethane. Identification of residue components was by GC-ECD (DB-5 column), with the following reference standards: parent lindane; DCB (1,2-, 1,3- and 1,4- isomers); TCB (1,3,5-, 1,2,4- and 1,2,3- isomers); TeCB (1,2,3,4-, 1,2,3,5- and 1,2,4,5- isomers); PCB; HCB; DCP (2,3-, 2,4-, 2,5-, 2,6- and 3,4- isomers); TCP (2,3,5-, 2,3,6-, 2,4,5 and 2,4,6- isomers); TeCP (2,3,4,5-, 2,3,5,6- and 2,3,4,6- isomers); PCP; 2,3,4,5,6-PCCH; and 1,2,3,4,5,6-HCCH.

Additionally, the filter cakes from kidney and faeces, remaining after the hexane extraction, were extracted with MeOH/water (80:20, v/v). The extract was hydrolyzed with 6 M HCl (8 hrs of reflux), extracted with diethyl ether, purified and analyzed by GC-ECD. Homogenized liver samples were extracted directly with MeOH/water (80:20, v/v) and were treated further as for the filter cakes from kidney and faeces. All of the ^{14}C present in the fat samples was extracted with hexane and 76-90% TRR could be identified, see Table 2.

Of the ^{14}C present in the muscle samples, 81-90% TRR was extracted with hexane, except in goat 1 (62% TRR). Lindane (parent) was the only compound identified: 45-59% TRR, except in goat 1 (28% TRR), see Table 2.

Of the ^{14}C present in the kidney samples from goat 3, 26% TRR was extracted with hexane, an additional 54% TRR was extracted with MeOH/water and 15% TRR remained in the filter cake. Lindane (parent) was the only compound identified in the hexane extracts, see Table 2. Part of the MeOH/water-extracted residues could be hydrolyzed with 6 M HCl (20% TRR), 24% TRR remained in the aqueous solution and 10% TRR was lost. Due to extreme problems with interference from matrix co-extractives, no metabolites could be identified in diethyl ether extracts of the hydrolyzed MeOH/water extracts.

The majority of the ^{14}C present in the liver samples from goat 3 was extracted with MeOH/water (88% TRR) and only 17% TRR remained in the filter cake. Only a small amount of MeOH/water extracted residues could be hydrolyzed with 6 M HCl (19% TRR), 60% TRR remained in the aqueous solution and 9.3% TRR was lost. Due to extreme interference from matrix co-extractives, metabolite identification was difficult in the diethyl ether extracts containing the hydrolyzed metabolites. Only three metabolites were identified: 1,3,5-TCB, 2,6,-DCP and 2,3,4,5-TeCP (amounts not stated).

Of the ^{14}C present in the faeces samples from goat 3, 45% TRR was extracted with hexane, an additional 24% TRR was extracted with MeOH/water and 34% TRR remained in the filter cake. Lindane (parent) was the only compound identified in hexane extracts, see Table 2. Part of the MeOH/water extracted residues could be hydrolyzed with 6 M HCl (12% TRR), 8% TRR remained in the aqueous solution and 4% TRR was lost. Due to extreme interference from matrix co-extractives, no metabolites could be identified in the diethyl ether extracts containing the hydrolyzed metabolites.

Table 2. Identification of compounds in hexane extracts of tissues and faeces, obtained from goats dosed orally with lindane (Mulkey and Hallenbeck, 1987).

Commodity	Compound	Recovery of spike, % [1/]	Goat 1, low dose		Goat 2, low dose		Goat 3, high dose	
			% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Liver	na	na	na	na	na	na	na	na
Kidney	Parent lindane	64	na	na	na	na	4.5	0.27
Muscle	Parent lindane	91	28	0.024	59	0.044	45	0.24
Omental fat	1,2,4-TCB	81	4.9	0.13	5.3	0.25	3.6	1.3
	1,2,3,4-TeCB	76	-	-	-	-	<0.1	0.03
	1,2,3,5-TeCB	65	-	-	-	-	<0.1	<0.02
	1,2,4,5-TeCB	78	1.5	0.04	0.9	0.04	0.8	0.29
	1,2,3,4,5,6-HCCH	133	0.7	<0.02	0.4	<0.02	0.4	0.15
	Parent lindane	105, 122	83	2.2	73	3.4	71	26
	Total identified		90		80		76	
Faeces	Parent lindane	57	na	na	na	na	2.2	0.19

- Not detected.
na Not analyzed.
[1/] Results were not corrected for recovery of reference standards.

The ^{14}C residues in urine samples were characterized further by Mulkey and Hallenbeck, (1987). Urine samples were mixed with concentrated HCl, sodium bisulfite and mineral oil and were incubated for 2 hrs at 100°C. Samples were extracted sequentially with toluene and toluene/ethyl acetate (80:20, v/v). Extracts were purified, methylated with diazomethane and analyzed by GC-ECD (DB-17 column), against the following reference standards: DCP (2,4- and 2,6- isomers); TCP (2,3,5-, 2,3,6-, 2,4,5 and 2,4,6- isomers); and TeCP (2,3,4,5- and 2,3,5,6- isomers). The presence of parent lindane was not investigated.

Of the ^{14}C present in the hydrolyzed urine samples, 58-64% TRR was extracted with toluene and toluene/ethyl acetate and 26-34% TRR remained in the aqueous phase. Only 22-32% TRR could be identified in purified toluene/ethyl acetate extracts (see Table 3). Recoveries were generally bad to poor and the analytical method required modification.

Table 3. Metabolite identification in urine extracts from goats dosed orally with lindane (Mulkey and Hallenbeck, 1987).

Compound	Recovery of spiked compound, %	Goat 1, low dose		Goat 2, low dose		Goat 3, high dose	
		% TRR	mg/kg	% TRR	mg/kg	%T RR	mg/kg
2,4-DCP	lost	2.5	0.22	1.9	0.28	2.9	1.0
2,6-DCP	lost	0.2	0.02	0.3	0.04	0.3	0.10
2,3,5-TCP	lost	6.8	0.62	4.5	0.67	6.7	2.4
2,3,6-TCP	lost	0.3	0.03	0.2	0.02	0.2	0.08
2,4,5-TCP	52	12	1.0	7.3	1.1	6.4	2.3
2,4,6-TCP	53	9.0	0.81	6.8	0.99	9.8	3.5
2,3,4,5-TeCP	49	0.2	0.02	0.1	0.03	0.1	0.03
2,3,5,6-TeCP	78	0.7	0.07	0.6	0.08	0.5	0.19
Total identified		31		22		27	

The ^{14}C residues in milk were characterized further by Piznik and Wargo (1987). Whole milk was analyzed for lindane, according to the AOAC method (extraction of fat with diethyl ether/petroleum ether, followed by partition into ACN). The AOAC method was modified, such that evaporation steps were conducted at room temperature, to minimize losses of volatile metabolites (1,2,4-TCB). The Florisil column elution was modified to separate lindane and 1,2,4-TCB, which were both determined by GC-ECD (OV-17 column for lindane, DB-5 column for 1,2,4-TCB). The aqueous phase remaining after fat extraction was subjected to acid hydrolysis (16 hr reflux) and extracted with ethyl acetate. The ethyl acetate extracts were analyzed with TLC with autoradiography, against the following reference standards: 4-CP; DCP (2,3-, 2,4-, 2,5-, 2,6- and 3,4- isomers); TCP (2,3,5-, 2,3,6-, 2,4,5 and 2,4,6- isomers); TeCP (2,3,4,5-, 2,3,5,6- and 2,3,4,6- isomers); and PCP. The results are shown in Table 4.

Lindane (parent) was the major constituent of the residue in milk fat (55-77% TRR of whole milk), with 1,2,4-TCB as a secondary metabolite (up to 16% TRR of whole milk). The residue in skimmed milk consisted of conjugated material which, when hydrolyzed with acid, released 6 to 8 chlorophenols. These compounds, together, comprised 5.9-17% TRR of whole milk. In an extract of milk from the high-dose goat (sample 06), five of these chlorophenols were identified (% TRR in whole milk) as: 0.3% 4-CP, 0.5% 3,4-DCP, 1.1% 2,3,5-TCP, 1.5% 2,4,5-TCP and 2.8% 2,3,4,5-TeCP.

Table 4. Characterization/identification of residues in milk from goats dosed orally with lindane (Piznik and Wargo, 1987).

Whole milk samples	TRR [2] mg/kg eq	Diethyl ether/ petroleum ether extract			Ethyl acetate extracts, after acid hydrolysis, % TRR [2]	Aqueous remainder, % TRR [2]	Total, % TRR
		Total (LSC) % TRR [2]	parent (GC), % TRR [1]	1,2,4-TCB (GC), % TRR [1]			
Goat 1, 1x dose, 08, after 6th dose	0.41	91	65	9.0	5.9	2.7	99
Goat 1, 1x dose, 09, after 7th dose	0.25	89	77	16	7.5	3.1	116
Goat 2, 1x dose, 06, after 4th dose	0.26	87	74	nd	9.4	3.9	99
Goat 2, 1x dose, 07, after 5th dose	0.19	75	57	11	17	7.8	107
Goat 3, 10x dose, 06, after 4th dose	3.1	89	72	6.0	6.8 [3]	4.2	88
Goat 3, 10x dose, 07, after 5th dose	2.4	84	55	10	7.5	8.5	95

nd Not detected (detection limit 0.04 mg/kg as 1,2,4-TCB).
[1] Determined by GC-ECD (modified AOAC method).
[2] Determined by LSC and considered to be chlorophenols.
[3] Comprised of 0.3% 4-chlorophenol, 0.5% 3,4-DCP, 1.1% 2,3,5-TCP, 1.5% 2,4,5-TCP and 2.8% 2,3,4,5-TeCP.

Study 2. The fate of uniformly ^{14}C-labelled lindane was investigated in one 17 month old lactating goat (Saanen) (Willems and Pluijmen, 1999 and Curry *et al*., 1999a). Capsules were administered orally by means of a balling gun, once a day for 7 consecutive days. The actual dose level was 13 ppm dw feed, corresponding to 0.60 mg ai/kg bw/d. Milk was collected twice daily and urine and faeces once daily. Within 24 hrs after the last dose, the goat was slaughtered and the following tissues were collected: kidney, liver, fat and muscle. Fat samples were composites of renal and subcutaneous fat and muscle samples were composites of skeletal muscle from hind and fore legs. Samples were stored at –20°C or –70°C for up to 260 days.

Information on the relationships between TAR and TRR was not given. Of the total administered radioactivity, 49% TAR was recovered in excreta: 13% TAR in faeces, 34% TAR in urine and 3% TAR in the cage wash. The percentage of recovered radioactivity in milk, tissues and expired air was not stated. The highest concentrations of radioactive residues were found in the body fat (3.5 mg/kg eq), followed by liver (2.2 mg/kg eq), kidney (0.48 mg/kg eq) and muscle (0.20 mg/kg eq).

In milk, a plateau in excreted radioactivity was achieved after 2 days and the residues ranged from 0.10-0.29 mg/kg eq. Whole milk was fractionated into fat, proteins (curd) and whey by centrifugation, acetone precipitation and centrifugation. The majority of the radioactive residue in whole milk was associated with the milk fat (55-87% TRR), whilst whey contained 10-36% TRR and the protein fraction (curd) contained 2.4-8.6% TRR (or <0.01 mg/kg eq).

Solvent extracts of milk fat, whey, tissues and organs were analyzed by LSC. Selected extracts were analyzed by TLC, HPLC-UV, GC-ECD or GC-MS, with identification against the following external reference standards: DCB (1,2- and 1,4- isomers); TCB (1,3,5-, 1,2,4- and 1,2,3- isomers); 1,2,3,4-TeCB; DCP (2,3-, 2,4-, 2,5-, 2,6-, 3,4- and 3,5- isomers); TCP (2,3,4-, 2,3,5-, 2,3,6-, 2,4,5-, 2,4,6- and 3,4,5- isomers); TeCP (2,3,4,5-, 2,3,5,6- and 2,3,4,6- isomers); PCP; gamma-TeCCH; and gamma-PCCH.

A pooled whole milk sample (obtained from day 3 onwards), containing 0.18 mg/kg eq, was fractionated into milk fat (69% TRR), whey (17% TRR) and protein (<10% TRR or <0.01 mg/kg eq). The protein fraction (curd) was not further investigated. TRR in milk fat and whey were expressed on the basis of whole milk. Hexane extracted 69% TRR from the pooled milk fat, of which 66% TRR partitioned into ACN. Parent lindane, gamma-PCCH and 1,2,4-TCB were identified (see Table 5). DCM (neutral and at pH 2) extracted 9.0% TRR from the pooled whey, of which 6.2% TRR was identified as parent lindane. The DCM phase contained three other unidentified metabolite components. The aqueous phase, containing 6.7% TRR, was freeze-dried and dissolved in MeOH. The dissolved fraction contained three unidentified metabolite components. All unidentified metabolite components were ≤ 2.5% TRR (or ≤ 0.005 mg/kg eq) and required no further identification (see Table 5).

Hexane extracted 99% TRR from a composite fat sample, of which 93% TRR partitioned into ACN. A further 3.1% TRR and 0.3% TRR could be extracted with MeOH, at pH 2 and pH 10, respectively. Parent lindane was the main component and, in addition, gamma-PCCH and 1,2,4-TCB were identified (see Table 5).

MeOH extracted 93% TRR from a composite muscle sample, of which 90% TRR partitioned into DCM. Parent lindane, gamma-PCCH and 1,2,4-TCB were identified (see Table 5).

MeOH extracted 84% TRR from kidney, of which 46% TRR partitioned into DCM. In the DCM phase, parent lindane, gamma-TeCCH and, possibly, 1,2-DCB were identified (see Table 5). The aqueous phase remaining from the MeOH extract (37% TRR) contained 7 unidentified metabolite fractions. Upon further separation, these contained 1-2 components per fraction, resulting in a total of 12 sub-fractions. None of the components of the sub-fractions co-chromatographed with the reference standards and, because residue levels were ≤ 8.0% TRR (or 0.045 mg/kg eq), they required no further identification. Enzymatic deconjugation procedures and acid hydrolysis did not release any lindane-like exocons from these polar metabolites. A further 4.7% TRR and 0.26% TRR could be released from kidney by additional extraction with MeOH at pH 2 and pH 10, respectively. From the pH 2 extract, only 1.4% TRR partitioned into DCM and it contained a single unidentified metabolite. The unextractable radioactive material accounted for 9.6% TRR (or 0.046 mg/kg eq) and required no further characterization (see Table 5).

MeOH extracted 86% TRR from liver, of which 16% TRR partitioned into DCM. The DCM phase contained only parent lindane (see Table 5). The aqueous phase from the MeOH extract (54% TRR) contained 8 unidentified metabolite fractions. Upon further separation, six of these fractions contained 2-6 components per fraction, resulting in a total of 24 sub-fractions. None of the components of the sub-fractions co-chromatographed with the reference standards and residue levels were ≤ 2.0% TRR (or 0.045 mg/kg eq) and required no further identification, except for three components in fraction LiIII (2.3-2.9% TRR or 0.051-0.065 mg/kg eq). Fraction LiV (25% TRR, 0.57 mg/kg eq) and fraction LiVI (3.1% TRR, 0.069 mg/kg eq) contained only one unknown component. Enzymatic deconjugation procedures did not release any lindane-like exocons from these polar metabolites. Acid hydrolysis released only a very minor component (possibly 2,3,4-TCP) but the study authors did not state from which fraction. According to the study authors, the component in fraction LiV was a polar, water-soluble, non-lipophilic metabolite, which was almost certainly excreted in urine or faeces, without any potential for bio-accumulation. This metabolite is not of toxicological concern.

A further 8.1% TRR could be released from liver by additional extraction with MeOH at pH 2. After partitioning with DCM, the DCM phase (0.9% TRR) contained 4 unidentified components (see Table 5). The aqueous phase of the extract (7.1% TRR) contained 5 unidentified components. The unidentified components did not co-chromatograph with any of the reference standards and were all ≤ 1.3% TRR (or 0.029 mg/kg eq) and required no further identification, except for fraction LiXVI from the aqueous phase (2.8% TRR or 0.063 mg/kg eq). Surfactant extraction and refluxing in 6 M HCl released only small amounts of additional radioactivity from liver: 0.2% TRR and 1.5% TRR respectively. A final enzymatic extraction using gamma-glutamyltranspeptidase released only 0.08% TRR. The unextractable radioactive material accounted for 7.5% TRR. Overall

recovery of radioactivity from liver was 87% and, because apparent losses occurred during DCM partition of the initial neutral MeOH extract, it is possible that the proportion of parent lindane was higher than that recorded in Table 5. A separate report on identification of a liver metabolite (Stewart, 2000) was not summarized, partly because it was an unaudited, unsigned draft, but mainly because the metabolite to which the report referred was not clear.

Although storage stability of residues was verified by comparison of HPLC chromatograms, most results were not shown. HPLC chromatographic profiles were provided only for the MeOH extract of kidney, which showed no difference in HPLC patterns obtained from extracts prepared at 1 or 252 days after sampling (Pluijmen and Willems, 2000).

Table 5. Identification/characterization of metabolites in milk and tissues from a goat dosed orally with lindane (Willems and Pluijmen, 1999 and Curry *et al*., 1999a).

Matrix	Total residue, mg/kg eq (% TRR)	Identity or characterization	%TRR	mg/kg [9]
Pooled whole milk	0.18			
milk fat	0.14 (69)	parent lindane	56	0.11
		gamma-PCCH	4.7	0.008
		1,2,4-TCB	5.1	0.006
		hexane extractable	2.1	0.004 (eq)
		unextracted	2.8	0.005 (eq)
		total	71	-
whey	0.033 (17)	parent lindane	6.2	0.012
		DCM extractable	2.9 [1]	0.006 (eq)
		water phase, dissolved	3.8 [2]	0.008 (eq)
		water phase, undissolved	2.5	0.005 (eq)
		total	15	-
curd	<0.01 (<10)	-	-	-
Fat (composite sample)	3.5	parent lindane	85	2.9
		gamma-PCCH	5.8	0.18
		1,2,4-TCB	11	0.23
		unextracted	0.9	0.03 (eq)
		total	102	-
Muscle (composite sample)	0.20	parent lindane	81	0.16
		gamma-PCCH	3.5	0.006
		1,2,4-TCB	5.8	0.007
		MeOH extractable, aqueous phase	3.5	0.007 (eq)
		unextracted	5.1	0.010 (eq)
		total	99	-
Kidney	0.48	parent lindane	36	0.17
		gamma-PCCH	4.5	0.016
		1,2-DCB [3]	5.8	0.014
		MeOH extractable, aqueous phase	37 [4]	0.18
		MeOH, pH 2, DCM phase	1.4 [5]	<0.01
		MeOH, pH 2, aqueous phase	3.4	0.02
		MeOH, pH 10, extractable	0.26	<0.01
		unextracted	9.6	0.046
		total	97	-
Liver	2.2	parent lindane	16	0.36
		MeOH extractable, aqueous phase	54 [6]	1.2
		MeOH, pH 2, DCM phase	0.9 [7]	0.02
		MeOH, pH 2, aqueous phase	7.1 [8]	0.16
		0.1 M SDS extractable	0.2	<0.01
		6 M HCl extractable	1.5	0.03
		enzyme extractable	0.08	<0.002
		unextracted	7.5	0.17
		total	87	-

[1] Consisted of 3 fractions containing 0.9-1.0% TRR (or 0.002 mg/kg eq) each.
[2] Consisted of 3 fractions containing 0.4-2.5% TRR (or 0.001-0.005 mg/kg eq) each.
[3] Tentative identification, not confirmed by second method.
[4] Consisted of 7 fractions containing 1.0-9.4% TRR (or 0.005-0.045 mg/kg eq) each. Fractions were further separated into sub-fractions, each containing 1-2 components, resulting in 12 sub-fractions containing 1.0-8.0% TRR (or 0.005-0.038 mg/kg eq) each.
[5] Consisted of a single compound.

[6/] Consisted of 8 fractions containing 1.9-25% TRR (or 0.042-0.57 mg/kg eq) each. Six fractions were further separated into sub-fractions, each containing 2-6 components, resulting in 21 sub-fractions containing 0.2-2.0% TRR (or 0.005-0.045 mg/kg eq) and three sub-fractions of LiII containing 2.3-2.9% TRR (or 0.051-0.065 mg/kg eq). Fraction LiV (25% TRR, 0.57 mg/kg eq) and fraction LiVI (3.1% TRR, 0.069 mg/kg eq) each contained only one unknown component.

[7/] Consisted of 4 fractions, containing 0.15-0.36% TRR (or <0.01 mg/kg eq) each.

[8/] Consisted of 5 fractions, of which 4 fractions contained 0.9-1.3% TRR (or 0.020-0.029 mg/kg eq) each and one fraction contained 2.8% TRR (or 0.063 mg/kg eq).

[9/] Identified metabolite concentrations are expressed as metabolite, unidentified metabolite fractions are expressed as lindane equivalents.

Poultry

Study 1. A study on the nature of lindane residues in eggs, chicks and tissues from laying hen pheasants was conducted (Saha and Burrage, 1976, non-GLP). One group of pheasants was treated with 20 mg ai/animal uniformly ^{14}C-labelled lindane, in gelatine capsules. A second group of pheasants was fed with wheat seed, treated post-harvest with 100 g ai/t uniformly ^{14}C-labeled lindane. A third (control) group of pheasants was fed with untreated wheat seed. Each group consisted of 20 hens and 5 cocks and each group was treated for 15 days, once daily. Feed was provided at 80 g/bird/day. The specific activity of the undiluted radiolabel was 6.9 TBq/mg, with a radioactive purity >99%. Eggs were collected daily for about 70 days. From each 2-day collection, 12 eggs were taken at random for incubation and hatching (21 days at 37°C). The remaining eggs were stored at 5°C. Two chicks from each 2-day batch of eggs were killed the day they hatched and kept at –18°C. Five hen pheasants from each group were killed at 1, 16 and 181 days after the last treatment. Samples of breast muscle, liver, brain and fat were kept at –18°C prior to analysis. Sample storage times were not stated.

Due to stress, actual feed intake levels were low at the beginning of the experiment and reached a plateau of 55 g/bird/day after the 10th day of treatment. Actual feed intake levels for the pheasants fed treated wheat were 5-10 g/bird/day lower than those fed with untreated wheat (gelatine capsule-treated and untreated control groups). Pheasants fed with treated wheat consumed a dose of 24 mg ai/bird in a 15-day period, whereas the group fed with gelatine capsules consumed 20 mg ai/bird per day.

Eggs were separated into yolks and whites and the radioactivity measured using LSC. Almost all radioactivity was present in the yolks; all residues in whites were <0.2 mg/kg eq. Residues in yolks from the capsule-treated group were highly variable (e.g. 11-26 mg/kg eq at day 7), increased sharply in time and reached a mean maximum level of 19 mg/kg eq in 8 days. Thereafter, the level decreased gradually to <0.5 mg/kg eq in about 50 days. Residues in yolks from the hens fed with treated seed were even more variable than the capsule-treated group (e.g. 3-25 mg/kg eq at day 17), increased gradually in time and reached a mean maximum level of 17 mg/kg eq at 22 days. Thereafter the level decreased to <0.5 mg/kg eq in about 70 days.

Egg yolks from eggs collected on days 8, 15, 22, 31 and 34 from the capsule-treated group, and on days 9, 12, 23, 29 and 36 from the treated-seed group, were extracted with acetone/hexane (1:3, v/v). The extract was diluted with NaCl solution and extracted with hexane. The hexane extract was cleaned-up on a Florisil column and analyzed using GC-ECD and GC with Coulson conductivity detector (both with two different columns: 15% OV-101 at 148°C and 4% SE-30 plus 6% QF-1 at 135°C). About 89-95% TRR was extracted from the egg yolks and 41-79% TRR was identified as parent lindane. Chromatograms of the extract showed a further eight peaks, which were identified as: 1,2-DCB; 1,3,5-TCB; 1,2,4-TCB; 1,2,3-TCB; 1,2,3,5-TeCB or 1,2,4,5-TeCB; 1,2,3,4-TeCB; gamma-PCCH; and PCB. The identities of the metabolites were confirmed by GC-MS. Amounts were not stated.

Hatched chicks were homogenized and extracted with MeOH. Total radioactive residues were calculated from residues in extracts and remaining solids, as determined by (combustion) LSC. Residues in hatched chicks were significantly lower than those in the eggs. Residues in chicks from the gelatine capsule-treated group rose rapidly, to reach a mean maximum level of 7 mg/kg in chicks hatched from eggs collected on day 9. Residues in chicks from the treated-seed group increased

gradually, to reach a mean maximum level of 7 mg/kg in chicks hatched from eggs collected on day 24.

MeOH extracts of chicks, hatched from eggs collected on days 5/6, 13/14, 15/16, 17/18, 19/20, 23/24 and 27/28 from the gelatine capsule-treated group, were partitioned into petroleum ether. Extracts were analyzed by LSC. Extraction by MeOH was complete: 59-78% TRR partitioned into petroleum ether layer and 22-41% TRR remained the aqueous layer. The petroleum ether extract of chicks was cleaned-up on Florisil and found to contain 18% TRR parent (5.3 mg/kg) on day 5/6, decreasing to 0% on day 23/24 and day 27/28. GC analysis (see eggs) of the petroleum ether extracts showed a total of eight peaks, which were identified as parent lindane, 1,2-DCB; 1,2,4-TCB; 1,2,3,5-TeCB or 1,2,4,5-TeCB; 1,2,3,4-TeCB; gamma-PCCH; and PCB. The identity of the metabolites was not confirmed by GC-MS, due to their low concentrations. Amounts present were not stated. The aqueous MeOH extracts of chickens was acidified to pH 1 and extracted with diethyl ether. The remaining aqueous layer was refluxed for 2 hrs at pH 1 and was extracted again with diethyl ether. The two diethyl ether extracts were combined and treated with diazomethane, to convert phenolic metabolites to the anisoles. Extracts were cleaned up on Florisil. GC-ECD analysis showed the presence of several di-, tri- and tetrachloroanisoles but they could not be identified, due to their low concentrations and inadequate resolution.

Muscle and liver tissues from hen pheasants were homogenized and extracted with MeOH. Fatty tissues were dissolved in hexane. Total radioactive residues were calculated from measurements made on extracts and the remaining solids, as determined by (combustion) LSC. Results are shown in Table 6. The highest concentrations of residues were found in fatty tissues, which decreased to non-detectable levels (not stated) in 6 months. MeOH extracts of muscle and liver were further treated as described for the chick tissues. Residues in hexane extracts of fat were partitioned into ACN and transferred to petroleum ether. Extracts were cleaned-up on Florisil and analyzed for the presence of lindane, using GC-ECD. Results are shown in Table 6. Parent lindane was the major component of residues in all tissues tested. Metabolites were not investigated.

Table 6. Radioactive residues in tissues from pheasants treated with radio-labelled lindane (mean ± s.d.) (Saha and Burrage, 1976).

Treatment	Day	Muscle		Liver		Fat	
		TRR mg/kg eq	parent % TRR	TRR mg/kg eq	parent % TRR	TRR mg/kg eq	parent % TRR
20 mg ai/bird/d, in capsules	1	1.0 ± 0.20	64 ± 6.8	3.1 ± 0.65	41 ± 8.2	52 ± 5.6	78 ± 8.2
20 mg ai/bird/d, in capsules	16	0.57 ±0.15	57 ± 7.2	0.09 ± 0.03	30 ± 4.2	21 ± 3.2	55 ± 4.9
20 mg ai/bird/d, in capsules	181	ND	ND	ND	ND	ND	ND
seed treated with 100 g ai/t	1	0.15 ± 0.04	74 ± 7.7	0.55 ± 0.07	46 ± 5.1	6.4 ± 1.5	80 ± 9.2
seed treated with 100 g ai/t	16	0.56 ± 0.31	70 ± 6.3	1.9 ±0.65	42 ± 4.6	29 ±8.7	79 ± 8.5
seed treated with 100 g ai/t	181	ND	ND	ND	ND	ND	ND

ND = Not detected (detection limit not stated).

Study 2. A study on the nature of lindane residues was conducted in White Leghorn laying hens (Merricks, 1987a, (GLP); Curry and Brookman, 1997a & 1997f). Four groups of hens were dosed with uniformly ^{14}C-labelled lindane, administered once daily *via* gelatine capsule, as follows.

Group 1 (4 hens): 1.3 ppm feed, 0.12 mg/kg bw, for 6 days, necropsy within 12 hrs after final dose.
Group 2 (6 hens): 1.3 ppm feed, 0.12 mg/kg bw, for 6 days, necropsy after 6 days withdrawal.
Group 3 (2 hens): 1.2 ppm feed, 0.12 mg/kg bw, for 4 days, necropsy within 12 hrs after final dose.
Group 4 (2 hens): 120 ppm feed, 11 mg/kg bw, for 4 days, necropsy within 12 hrs after final dose.

Groups 1 and 2 hens served to obtain a material balance and to study the depletion of residues during a withdrawal (depuration) period. Groups 3 and 4 hens had been intended for metabolite identification and quantification but only group 4 hens were used for these purposes. A control group of four hens received empty capsules. The hens consumed 150 g feed per day and had bodyweights of 1.4-1.7 kg. The specific activity of the undiluted radiolabel was 4.2 MBq/mg and the radioactive purity was >98%. Eggs and excreta were collected daily. Eggs were removed from the shells and separated into whites and yolks. All samples were stored frozen.

Table 7 summarizes the total recovered radioactive residues (TRR) in all collected matrices, as measured by combustion LSC. Total recovery of the administered radioactive dose was above 90% in groups 1 and 2. In group 1, 44% TAR was recovered in excreta, 2.6% in eggs and 47% in tissues and organs. In group 2, 63% was recovered in excreta, 9.0% in eggs and 20% in tissues and organs. During the dosing period (4-6 days), residue levels in eggs did not reach a plateau. Lindane residues tend to accumulate in fatty tissues, such as fat and egg yolks and the lower residues in breast muscle, compared with thigh muscle which contains a higher proportion of fat, support this general finding. The proportion of the dose found in tissues and organs declined by approximately half during depuration, indicating continued clearance of residues *via* excreta and eggs.

Table 7. Residues in hen matrices after feeding radiolabelled lindane (Merricks, 1987a).

Matrix	Group 1 (A), 1.3 ppm feed, 0 days depuration		Group 2 (B), 1.3 ppm feed, 6 days depuration		Group 3 (low) 1.2 ppm feed		Group 4 (high) 120 ppm feed	
	mg/kg eq	% TAR	mg/kg eq	% TAR	mg/kg eq	parent, % TRR	mg/kg eq	parent, % TRR
Liver	0.39	0.97	0.15	0.43	0.32	49	12	51
Kidney	0.28	0.26	0.10	0.09	0.20	ND	11	-
Gizzard (washed)	0.12	0.20	0.04	0.08	0.13	ND	7.9	-
Skin	0.92	10	0.41	5.5	0.72	54	50	86
Breast muscle	0.05	0.50	0.02	0.20	0.019	ND	1.4	100
Thigh muscle	0.22	3.0	0.11	1.8	0.16	91	12	71
Fat	2.8	13	1.0	5.5	1.3	123	97	85
Heart	0.12	0.05	0.04	0.02	0.24	104	21	100
Blood	0.05	0.27	0.02	0.14	-	-	-	-
Abdominal content [3/]	0.76	18	0.23	6.3	-	-	-	-
Excreta (mean)	-	44	--	63	0.75 (day 4) 38% TAR	11	69 (day 3) 30% TAR 114 (day 4)	80
Eggs (total)	1.7	2.6	<1.3 [1/] <5.2 [2/]	9.0	-	-	-	-
Egg yolks, days 1-11	1: <0.01 2: 0.04 3: 0.15 4: 0.24 5: 0.45 6: 0.83	-	1: <0.01 2: 0.05 3: 0.16 4: 0.23 5: 0.36 6: 0.54 7: 0.69 8: 0.82 9: 0.72 10: 0.90 11: 0.74	-	1: <0.01 2: 0.03 3: 0.10 4: 0.19	101%	1: <0.01 2: 1.5 3: 5.1 4: 11	94
Egg whites, days 1-11	<0.01 (all days)	-	<0.01 (all days)	-	<0.01 (all days)	ND	1: <0.01 2: 0.11 3: 0.16 4: 0.21	100
Total	-	93	-	92	-	-		-

[1/] On last day of dosing.
[2/] After six days of depuration.
[3/] Not defined by the study author.
- Not analyzed.
ND Matrices contained insufficient radioactivity for analysis.

Tissues and eggs from group 3 and 4 were examined for identification of metabolites. Matrices from group 3 hens were extracted with ethyl acetate, vacuum filtered, then the extracts were evaporated to dryness and re-dissolved in hexane. Extracts were cleaned up using a Florisil column and analyzed by TLC and GC-ECD. Identification attempts using TLC were unsuccessful, because lindane and possible metabolites did not show any UV absorption and volatile compounds were lost during TLC plate development. Extraction (without clean-up steps) was nearly complete (68-152% TRR). However, upon GC-ECD analysis, it became evident that volatile metabolites had been lost

during the extract evaporation steps. Therefore, only lindane was quantified in the solutions from the group 3 hens. Table 7 shows the levels of lindane (parent) in matrices from group 3 hens.

A different extraction scheme was used for metabolite identification and quantification in matrices from the group 4 hens. Tissues (except skin and fat), eggs and excreta were extracted with hexane and then with ethyl acetate. The hexane extracts were not evaporated before column clean-up. Ethyl acetate extracts were evaporated to dryness and re-dissolved in hexane. Both extracts were cleaned-up using Florisil columns. Skin and fat were extracted with hexane and the residues were partitioned into ACN. The ACN was saturated with NaCl and residues were partitioned back into hexane. Solutions containing sufficient radioactivity were examined by GC-ECD, against authentic standards of: HCCH; 2,3,4,5,6-PCCH; TeCCH; hexachlorocyclohexane isomer mixture; HCB; PCB; TeCB (1,2,4,5-, 1,2,3,4- and 1,2,3,5- isomers); TCB (1,2,3-, 1,2,4- and 1,3,5- isomers); DCB (1,2-, 1,3- and 1,4- isomers); 2,6-dichlorobenzenethiol; DCP (2,3-, 2,4-, 2,5-, 2,6- and 3,4- isomers); TCP (2,3,5-, 2,4,5- and 2,4,6- isomers); TeCP (2,3,4,5- and 2,3,4,6- isomers); and PCP.

Extraction (without clean-up steps) was apparently complete (89-141% TRR), although the accountability sums for breast muscle and fat indicate that certain measurements were not particularly accurate. For example, TRR in breast muscle and fat exceeded 120%. All individual extractable residue components, present at 0.01 mg/kg or above, were identified in the poultry eggs and edible tissues. Table 8 shows the identity and distribution of metabolites in poultry tissues and eggs. The residue consisted mainly of parent lindane in all tissues. In liver, the main metabolite was 1,2,4-TCB and, in thigh muscle, the main metabolite was TeCB. Contrary to expectations, hexachlorobenzene was detected in liver but not in fat.

Table 8. Identified residue components in group 4 hens, following four daily doses of 120 ppm in feed (Merricks, 1987a).

Compound or measurement	Units	Egg Yolk	Egg White	Thigh muscle	Breast muscle	Liver	Fat
TRR	(mg/kg)	11	0.21	12	1.4	12	97
Unextracted (solids)	(% TRR)	3.6	nr	1.6	20	6.	1.6
Extracted	(% TRR)	96	nr	106	90	89	141
Total identified	(% TRR)	121	67	109	147	76	102
Parent lindane	(% of identified)	94	100	71	100	51	85
DCBs	(% of identified)	ND	ND	ND	ND	9.5	ND
1,3,5-TCB	(% of identified)	ND	ND	ND	ND	6.4	ND
1,2,4-TCB	(% of identified)	0.63	ND	2.0	ND	19	3.5
1,2,3-TCB	(% of identified)	ND	ND	0.33	ND	0.87	0.60
1,2,4,5- or 1,2,3,4-TeCB	(% of identified)	ND	ND	18	ND	2.3	3.1
1,2,3,4-TeCB or TeCCH	(% of identified)	ND	ND	4.4	ND	4.4	0.97
2,3,4,5,6-PCCH	(% of identified)	4.2	ND	4.6	ND	3.8	6.1
1,2,3,4,5-PCB	(% of identified)	0.13	ND	0.02	ND	ND	0.10
HCCH	(% of identified)	0.62	ND	0.19	ND	0.53	0.60
HCB	(% of identified)	ND	ND	ND	ND	1.4	ND

ND = not detected; nr = not reported.

Plant metabolism

The Meeting received information on the fate of lindane in plants grown from seed treated with lindane. The Meeting also received information on the fate of lindane after post-emergence spray application to spinach and cucumber plants and after pre-harvest spray application to apples (fruit, leaves).

Plants grown from lindane-treated seeds

Study 1. Wheat seeds (var. Chanthatch) were coated with uniformly ^{14}C-labelled lindane in solution, at an actual dose rate of 480 g ai/t (Balba and Saha, 1974, non-GLP). The treated seeds were planted in the field (location not stated) in a medium- to heavy-textured soil (details not stated). Crops were sampled 19 and 100 days after planting. Samples were stored at –18°C until analysis (time not stated).

Recovery of total applied radioactivity was not determined. Residues in grain were analyzed by dry combustion only. Residues in seedlings, roots, straw and chaff were extracted with MeOH. The MeOH extract was concentrated, diluted with 1% sodium carbonate and residues were partitioned into diethyl ether The diethyl ether layer, containing non-acidic compounds, was purified and analyzed by LSC and GC-ECD (for lindane). The remaining aqueous carbonate layer was then acidified with HCl to pH 1 and heated under reflux for 30 min before being extracted with diethyl ether. This diethyl ether extract (containing acidic compounds) and the aqueous phase (containing highly polar compounds) were analyzed by LSC. Results are shown in Table 9.

Significant amounts of residues were present in the seedlings and in the mature plants, indicating that lindane and/or its degradation products are readily translocated into the growing wheat plants. Extraction of the plant tissues with MeOH recovered more radioactive material from the seedlings than any part of the mature plants, indicating that the ^{14}C was more strongly bound in the mature plant tissues. Only 26% TRR in the seedlings was hydrophilic and was comprised of 12.7% TRR acidic compounds and 13.6% TRR highly polar compounds. The proportion of hydrophilic compounds increased in the mature plant parts: 27.4% TRR in the roots, 53% TRR in the straw and 34% TRR in the chaff.

Metabolites in root extracts were identified further. Non-acidic compounds were identified by GC-ECD (3 columns), GC with a Coulson conductivity detector and GC-MS, against external reference compounds. Reference standards were: parent lindane, 1,3,5-TCB, 1,2,4-TCB, 1,2,3-TCB, 1,2,3,5-TeCB, 1,2,4,5-TeCB, 1,2,3,4-TeCB, gamma-PCCH, PCB, o-DCB, m-DCB and p-DCB. Acidic compounds were analyzed by 2D-TLC with autoradiography and co-chromatography with reference standards (not stated which). In roots, eight non acidic compounds (chlorobenzenes) were identified and were present in the following proportions (expressed as % TRR): 5.7% as 1,2,4-TCB; 1.9% as 1,3,5-TCB; 1.9% as 1,2,3-TCB; 1.9% as 1,2,3,5-TeCB and/or 1,2,4,5-TeCB; 0.63% as 1,2,3,4-TeCB; 0.63% as gamma-PCCH; 0.63% as PCB; and 0.63% m-DCB and/or p-DCB. In roots, four acidic compounds (chlorophenols) were also identified as (i) PCP; (ii) 2,3,5,6-TeCP and/or 2,3,4,6-TeCP; (iii) 2,3,4-TCP, 2,4,5-TCP, 2,3-DCP and/or 2,4-DCP; (iv) 2,4,6-TCP.

Table 9. Distribution and characterization of residues in wheat grown from lindane-treated seed (Balba and Saha, 1974).

Wheat plant part	DAT	TRR, mg/kg eq	solids, % TRR	MeOH extractable, % TRR	Identity/character of MeOH extractables			
					Lindane, %TRR	non acidic compounds, % TRR	acidic compounds, % TRR	highly polar compounds, % TRR
Seedlings	19	0.55	9	91	36	29	13	14
Roots	100	2.3	37	63	21	14	14	14
Straw	100	0.11	33	67	5.4	8.7	21	32
Chaff	100	0.02	66	34	nd	nd	4.1	30
Grain	100	nd	-	-	-	-	-	-

DAT = days after treatment.
nd = not detected, detection limit not stated.
- = not determined.

Study 2. Radish, sugar beet, spinach, mustard, field corn, sweet corn, and spring wheat seeds were treated with uniformly ^{14}C-labelled lindane, applied as a diluted EC (209.2 g ai/l) formulation (Piznik, 1987a, non-GLP). Actual dose rates were 369-2288 g ai/t (see Table10 and 11). Treated seeds were planted outdoors, under a clear protective roof (Columbus, NJ, USA). Five different soils were used: four loamy sand soils (1.6-1.9% om, pH 5.4-6.2, CEC 5.3-5.9 and 7.2-9.2% clay) and one silt loam soil (2.0% om, pH 5.7, CEC 7.0 and 25% clay). Each day, the soil was sprinkled with water, simulating 6.35 mm rainfall per day. Crops were sampled at regular intervals and were stored frozen for a maximum of one week (exact temperature not stated). Results for sugar beet are from plants which did not reach full maturity.

Recovery of total applied radioactivity was not determined. Measurable residues (>0.01 mg/kg eq) were found in all crops parts, except corn cobs and corn grain (see Tables 10 and 11). Seed produced by mustard was not investigated. Residues from selected samples were extracted

with ACN and analyzed for the presence of parent lindane, using the AOAC method. Exact extraction conditions, GC detection conditions and validation results for this method were not stated. The average lindane (parent) content was <0.1 mg/kg for root crops, <0.04 mg/kg for leafy crops and forage and <0.01 mg/kg for cereal grains (see Table 12). In all crops (except mustard foliage), the percentage of characterized and/or identified residues was less than 75%.

Table 10. Total radioactive residues in fast-growing crops grown from seed treated with radio-labelled lindane (Piznik, 1987a).

Crop, part	Seed treatment, actual dose rate, g ai/t [1/]	Total recovered radioactive residue (mg/kg eq)					
DAT		7	14	21	22	42	46
spinach, leaves	817	-	-	0.10	-	0.020	-
mustard, roots	586	-	-	-	-	-	0.055
mustard, foliage	586	2.0	-	-	0.021	-	<0.01
radish, roots	381	1.2	-	-	0.056	-	-
radish, leaves	381	2.9	0.13	-	0.040	-	-

DAT days after treatment.
- not analyzed.
[1/] Dose rate was expressed as ppm: assuming 1 t = 1000 kg, 1 ppm =1 g ai/t.

Table 11. Total radioactive residues in slow growing crops grown from seed treated with radio-labelled lindane (Piznik, 1987a).

Crop, part	Seed treatment, actual dose rate, g ai/t [1/]	Total recovered radioactive residue (mg/kg eq)					
DAT		32	60	74	90	113	150
sugar beet, roots [2/]	2288	-	1.0	0.95	0.54	0.84	0.30
sugar beet, foliage [2/]	2288	-	0.34	0.17	0.28	0.36	0.18
field corn, roots	1774	1.2	0.67	-	0.84	-	-
field corn, foliage	1774	0.11	0.063	-	0.064	-	-
field corn, grain	1774	-	-	-	<0.01	-	-
field corn, cobs	1774	-	-	-	<0.01	-	-
field corn, husks	1774	-	-	-	0.025	-	-
sweet corn, roots	1444	1.0	-	0.16	0.36	-	-
sweet corn, foliage	1444	0.22	-	<0.01	0.051	-	-
sweet corn, grain	1444	-	-	<0.01	<0.01	-	-
sweet corn, cobs	1444	-	-	-	<0.01	-	-
sweet corn, husks	1444	-	-	<0.01	<0.01	-	-
spring wheat, roots	369	0.47	2.6	4.8	2.7	4.2	-
spring wheat, foliage	369	8.6	0.58	0.80	0.92	2.2	2.9
spring wheat, grain	369	1.6	0.059	0.033	0.090	0.097	0.052

DAT days after treatment.
- not analyzed.
[1/] Dose rate was expressed as ppm: assuming 1 t = 1000 kg, 1 ppm =1 g ai/t.
[2/] Sugar beet did not reach full maturity.

Table 12. Residue characterization (LSC) and lindane quantification (GC) in crops grown from seed treated with radio-labelled lindane (Piznik, 1987a).

Crop, part	DAT	TRR, mg/kg eq	ACN extracts, % TRR (mg/kg eq)	Solids, %TRR (mg/kg eq)	Total, % TRR [2/]	Lindane (parent) in ACN extracts, % TRR (mg/kg)
Mustard, foliage	22	0.021	57.1 (0.012)	14.3 (0.003)	71.4	81.0 (0.017) [1/]
Radish, roots	22	0.056	67.9 (0.038)	8.9 (0.005)	76.8	53.6 (0.030)
Sugar beet, roots	150	0.30	58.9 (0.18)	13.8 (0.041)	72.7	30.0 (0.090)
Sugar beet, foliage	150	0.18	96.1 (0.17)	59.1 (0.094)	148	19.3 (0.035)
Field corn, roots	90	0.84	36.7 (0.31)	14.8 (0.12)	51.5	19.7 (0.16)
Field corn, foliage	90	0.064	25.0 (0.016)	64.1 (0.041)	89.1	12.5 (0.008)
Sweet corn, foliage	90	0.051	118 (0.060)	27.5 (0.014)	145	23.5 (0.012)
Spring wheat, foliage	150	2.9	4.6 (0.14)	109 (3.2)	113	0.5 (0.016)
Spring wheat, grain	150	0.052	0.0 (<0.01)	217 (0.11)	217	3.8 (0.002)

[1/] The mass balance between %TRR in ACN extracts (LSC detection) and % lindane in ACN extracts (GC detection) was evidently erroneous but the study author gave no explanation.
[2/] Where the sum of % extracted and % solids was below 80% or above 120%, the mass balances were evidently erroneous but the study author gave no explanation.

Apple, foliar treatment to fruit and leaves

A single Red Delicious apple tree was treated, once, with 1 kg ai/ha uniformly ^{14}C-labelled lindane as an EC formulation, just prior to petal fall, using a hand-held compressed air sprayer (Gemma, 1987; Curry and Brookman, 1997b). Specific activity of the lindane was 4.6 MBq/mg, with a radioactive purity >96%. Samples of foliage were taken 1 hr and 1, 8, 14, 21, 28, 57, 84, 117, and 131 days after the application. Immature fruit were collected 28, 57, 84, and 117 days after application. Mature fruit were collected 131 days after application. At each sampling interval, foliage and fruit were taken from all sections of the tree. Samples were stored frozen for 37-45 days until analysis.

Samples were homogenized and analyzed by (combustion) LSC. Total radioactive residues are shown in Table 13. Lindane residues were present in both leaves and fruit, following application just prior to petal fall. The presence of lindane residues in the fruit indicates that it was distributed throughout the tree and transferred into the maturing fruit from leaves and twigs. Total radioactive residues declined during the maturation period. Levels of radioactivity in apple fruit were considerably lower at each collection than the corresponding levels in foliage.

Table 13. TRR and extractability of residues in apple foliage and fruit from a tree treated with lindane at 1 kg ai/ha (Gemma, 1987 ; Curry and Brookman, 1997b).

DAT	Foliage				Fruit			
	TRR, mg/kg eq	ACN/water % TRR	HCl/MeOH % TRR	Unextracted % TRR	TRR, mg/kg eq	ACN/water % TRR	HCl/MeOH % TRR	Unextracted % TRR
0	124 [1/]; 129 [2/]	99	0.6	0.4	-	-	-	-
1	51 [1/]; 56 [2/]	99	0.7	0.2	-	-	-	-
8	7.4 [1/]; 7.1 [2/]	81	5.9	13	-	-	-	-
14	3.8 [1/]; 3.7 [2/]	75	13	12	-	-	-	-
21	2.3 [1/]; 2.3 [2/]	66	34	0	-	-	-	-
28	1.8 [1/]; 1.4 [2/]	61	14	24	0.38 [1/]; 0.49 [2/]	69	31	0
57	0.66 [1/]; 0.97 [2/]	42	58	0	0.12 [1/]; 0.15 [2/]	87	-	13
84	0.95 [1/]; 0.78 [2/]	59	22	19	0.07 [1/]; 0.07 [2/]	86	-	14
117	4.2 [1/]; 2.3	52	4.8	43	0.06 [1/]; 0.08 [2/]	75	-	25
131	0.81 [1/]; 0.72 [2/]	61	6.9	32	0.04 [1/]; 0.08 [2/]	75	-	25

[1/] Values measured by combustion LSC in original samples.
[2/] Values calculated from ACN/water + HCl/MeOH + unextracted residues.

Homogenized samples were extracted with 10% water in ACN. The filter cake from initial extraction was extracted by refluxing with 2% HCl in MeOH for 1 hr. After vacuum filtration, MeOH was removed by evaporation and the remaining aqueous fraction was partitioned into ethyl acetate. The remaining aqueous fraction was adjusted to 6 M with respect to HCl and was refluxed for 1 hr, cooled, and partitioned into ethyl acetate. The ethyl acetate fractions were evaporated to dryness and re-dissolved in acetone. Extracts and solids were analyzed by (combustion) LSC. Extractability of the radioactive residues in/on foliage ranged from 99% at 0 DAT to 68% at 131 DAT, while the extractability of the radioactive residues in/on fruit ranged from 100% at 28 DAT (immature) to 75% at 131 DAT (see Table 14).

ACN/water extracts were further cleaned-up for metabolite identification. ACN was removed from the extract by evaporation and the remaining aqueous fraction was partitioned into petroleum ether. The petroleum ether fraction was evaporated to dryness and re-dissolved in acetone. The aqueous fraction remaining after partition with petroleum ether was acidified to pH 3 and partitioned into ethyl acetate. The aqueous fraction from ethyl acetate partition was made basic (pH 10) and partitioned into ethyl acetate. Both the acid-partition and the base-partition ethyl acetate fractions were evaporated to dryness and re-dissolved in acetone. Metabolites in the final acetone solutions (from filter cake and ACN/water extracts) were characterized by TLC (silica gel; four solvent systems) and co-chromatography with reference standards. The identity of parent lindane and PCP was confirmed by GC-ECD. Tables 14 and 15 present information on the identity and levels of lindane residues in apple fruit and leaves, respectively. Lindane was the primary extractable component of residues in fruit and foliage.

At harvest (131 DAT), foliage consisted of parent lindane (3.2% TRR), minute quantities of chlorinated phenols (2.0% TRR), TLC-origin material (19% TRR), water-soluble material (40% TRR) and unextracted residues (32% TRR). At harvest (131 DAT), residues in fruit consisted of parent lindane (11% TRR), PCP (14% TRR), minute quantities of two other chlorinated phenols (0.6% TRR), TLC-origin material (12% TRR), water-soluble material (38% TRR) and unextracted residues (25% TRR). In mature apple fruit, the absolute levels of unextracted residues and residues in the aqueous layer were too low (<0.02 mg/kg eq) to justify further investigation.

Table 14. Metabolite identification in apple fruits, expressed as % TRR (Gemma, 1987; Curry and Brookman, 1997b).

Compound	28 DAT	57 DAT	84 DAT	117 DAT	131 DAT
TRR mg/kg eq	0.38	0.12	0.07	0.06	0.04
Unextracted, % TRR	0.00	13	14	25	25
Aqueous layer , % TRR [1/]	17	33	43	38	38
Parent, % TRR [2/]	20	23	35	15	11
PCP, % TRR [2/]	0.9	0.9	1.5	9.5	14
2,3-DCP, % TRR [2/]	-	-	-	-	-
2,4-DCP, % TRR [2/]	-	-	-	-	-
2,3,5-TCP, % TRR [2/]	-	-	-	-	-
2,4,5-TCP, % TRR [2/]	0.9	-	1.5	0.1	0.3
2,4,6-TCP, % TRR [2/]	-	-	-	-	-
2,3,4,5-TeCP, % TRR [2/]	1.2	1.3	2.5	0.1	0.3
2,3,5,6-TeCP, % TRR [2/]	-	-	-	-	-
TLC origin, % TRR [2/]	34	3.5	11	12	12
Other polar metabolites, % TRR [2/]	-	-	-	-	-
Other unknowns, % TRR [2/]	3.2	3.6	5.6	-	-
Total recovery, % TRR	78	79	114	100	100

DAT = days after treatment.
[1/] Extracted residues remaining in the aqueous phase after partitioning against petroleum ether or ethyl acetate.
[2/] Extracted residues partitioned into petroleum ether or ethyl acetate and characterized/identified by TLC.

Table 15. Metabolite identification in apple leaves, expressed as % TRR (Gemma, 1987; Curry and Brookman, 1997b).

Compound	0 DAT	1 DAT	8 DAT	14 DAT	21 DAT	28 DAT	57 DAT	84 DAT	117 DAT	131 DAT
TRR mg/kg eq	124	51	7.4	3.8	2.3	1.8	0.66	0.95	4.2	0.81
Unextracted, % TRR	0.40	0.20	13	12	0	24	0	19	43	32
Aqueous layer, % TRR [1/]	0.00	0.04	15	27	44	36	33	37	37	40
Parent, % TRR [2/]	77	78	39	28	3.8	8.2	2.4	-	5.1	3.2
PCP, % TRR [2/]	-	-	-	-	0.2	1.0	0.8	-	0.8	1.8
2,3-DCP, % TRR [2/]	-	-	0.2	0.3	-	-	0.2	0.4	-	-
2,4-DCP, % TRR [2/]	-	-	-	-	-	0.1	-	-	-	-
2,3,5-TCP, % TRR [2/]	-	-	0.2	0.3	-	-	0.2	0.4	-	-
2,4,5-TCP, % TRR [2/]	-	-	0.3	0.5	-	1.0	0.8	1.2	0.1	0.1
2,4,6-TCP, % TRR [2/]	-	-	-	-	-	0.1	-	-	-	-
2,3,4,5-TeCP, % TRR [2/]	-	-	-	-	-	1.3	1.3	-	0.1	0.1
2,3,5,6-TeCP, % TRR [2/]	-	-	0.3	0.4	-	-	0.2	0.6	-	-
TLC origin, % TRR [2/]	1.6	0.8	14	24	34	26	52	40	16	19
Other polar, % TRR [2/]	7.4	9.1	8.3	6.9	3.8	0.7	1.3	1.6	0.1	-
Other unknowns, % TRR [2/]	13	12	7.2	6.1	2.9	3.2	3.6	-	-	-
Total recovery	99	99	97	100	89	102	96	100	102	97

DAT = days after treatment.
[1/] Extracted residues left in the aqueous phase after partitioning against petroleum ether or ethyl acetate.
[2/] Extracted residues partitioned into petroleum ether or ethyl acetate and characterized/identified by TLC.

Cucumber, post-emergence application

Cucumber plants (variety Fenumex) received three foliar applications of uniformly ^{14}C-labelled lindane, each at a rate of 0.71 kg ai/ha as an EC formulation (England and Savage, 1987; Curry and Brookman, 1997c). The first application was at the 2-leaf stage, and the second and third were 1 and 2 weeks after the first application. The nominal spray volume was 540 l/ha and the spray concentration 0.13 kg ai/hl. Actual spray volumes were 532-550 l/ha. The specific activity of the

undiluted radiolabel was 0.95 GBq/mmol and the radioactive purity was >99%. Cucumber plants were grown in loamy sand (USDA classification, pH 6.7, 1.1% om, 5% clay, CEC 6.6) in pots in plastic-covered tunnels in Ongar, Essex, UK. Watering was from the bottom or by drenching at soil level; overhead watering was not performed.

Separate material balance studies were conducted on 3 plants grown in aerated plastic enclosures and one plant grown in an aerated glass enclosure. Volatiles were collected in a trap for CO_2 (ethanolamine/ethoxyethanol, 25:75, v/v) and a trap for volatile organic compounds (polyurethane foam plugs, *iso*-octane and ethylene glycol). Plants, soil and traps were analyzed for radioactivity, at 7 or 21 DAT, from the glass- and plastic-enclosed plants, respectively. Recovery of radioactivity from the plants maintained in plastic enclosures was low (19.2% and 38.5%) and the results were not considered further. The recovery of radioactivity from plants in the glass enclosure study was quantitative (104%). Radioactivity in the glass-enclosed plant, at 7 DAT, was found in leaves (41% TAR), stems (3.9% TAR), roots (0.9% TAR), soil (14% TAR) and traps (17% TAR) or was associated with tanks/tubes/pots (27% TAR). After each treatment, plants from the plastic tunnels were collected at 0, 1, 3, and 7 days for autoradiography. Autoradiography indicated that radioactivity spread rapidly throughout the plant. Radioactivity in the stems and roots, detected immediately following treatment, disappeared after 24 hrs and, by 7 days, the majority of the radioactivity had disappeared from the leaves.

Whole cucumber plants were collected at 1, 2, 4, 7, 14, 28 and 61 DAT and were separated into leaves, stems, roots and, where applicable, into fruit. Separate mature fruit were taken at 39, 42, 46, 53, 61, 64, 70 and 83 DAT. Leaves, stems and roots were subsequently extracted with acetone and MeOH. Cucumber fruit were extracted with acetone on the day they were collected. Analysis of fruit extracts were completed within 64 days after collection. Extracts and solids were analyzed by (combustion) LSC and the results are shown in Table 16. Most of the radioactivity was in the leaves and the extractable radioactivity declined rapidly. Growth dilution of the residue appeared to play an important role in reduction of the residue on a weight/weight basis, as is shown in Table 16 (see mg/kg values for day 7 versus day 61). Residues in cucumber fruit were 0.00009-0.0032 mg/kg eq and were therefore not investigated further.

Initial plant extracts that contained sufficient radioactivity were examined by HPLC-UV with radioactivity detection (ODS-2 column, with isocratic or gradient elution). No radioactive components co-eluted with potential metabolite reference materials, including chlorinated benzenes, cyclohexanes, or phenols (not stated which were used as reference standards). Lindane was the only residue component identified. Post-extraction solids were subjected to acid hydrolysis (MeOH/2 M HCl, 1:1, v/v, 2 hrs reflux) and the hydrolysate was examined by HPLC. Hydrolysis released additional radioactivity, which was shown to consist of multiple, low-level radioactive components. Aliquots of extracts were methylated using diazomethane. The initial acetone extract, the MeOH extract, and the methylated acetone extract from leaves were examined by GC-MS. Again, only lindane was identified.

Table 16. Extractability of residues in cucumber plants treated 3x with radio-labelled lindane at 0.71 kg ai/ha, post-emergence (England and Savage, 1987; Curry and Brookman, 1997c).

DAT	Leaves				Stems				Roots			
	TRR [1/], µg eq	TRR, mg/kg eq	extract, % TRR	solids, % TRR	TRR [1/], µg eq	TRR, mg/kg eq	extract, % TRR	solids, % TRR	TRR [1/], µg eq	TRR, mg/kg eq	extract, % TRR	solids, % TRR
0	196 [2/]	-	98	2.4	-	-	-	-	-	-	-	-
1	120	-	94	5.6	6.6	-	98	1.5	0.41	-	93	7.3
4	36	-	70	30	1.3	-	76	24	0.13	-	38	62
7	27	2.7	60	40	0.34	0.14	26	74	0.70	0.057	80	20
14	23	-	53	47	7.3	-	98	2.3	1.9	-	45	55
28	12	-	25	75	0.92	-	54	46	0.51	-	41	59
61	15	0.085	30	70	0.86	0.004	45	55	0.27	0.015	3.7	96

[1/] Sum of extracted and solid residues.
[2/] Combined leaves and stems.

Spinach, post-emergence application

Spinach plants, at the 2-leaf stage, received a single foliar application of uniformly ^{14}C-labelled lindane as an EC formulation, at dose rates of 0.9 or 1.5 kg ai/ha, for varieties Viroflay and Perpetual, respectively (England, 1987, non-GLP; Curry and Brookman, 1997d). The specific activity of the undiluted radiolabel was 0.95 GBq/mmol and the radioactive purity was >99%. The diluted formulation was applied to the leaves using a Hamilton syringe. The nominal spray volume was 540 l/ha; actual spray rates were 540 and 538 l/ha. Spinach plants were grown in clay-loam (USDA classification, pH 6.1; 3.0% om; 28% clay; CEC 13.59) in pots, kept outside in a net-covered enclosure in Ongar, Essex, UK. Plants were watered from the bottom; no overhead watering was performed.

Separate material balance studies were conducted on 6 plants of each variety, grown in aerated plastic enclosures, and 1 plant of each variety, grown in an aerated glass enclosure. Volatiles were collected in a trap for CO_2 (ethanolamine/ethoxyethanol, 25:75, v/v) and a trap for volatile organic compounds (*iso*-octane and ethylene glycol). Plants, soil and traps were analyzed for radioactivity at 7 DAT.

Recovery of radioactivity from plants maintained in plastic enclosures was low (6-12% TAR) and the results were not considered further. The recovery of the glass enclosure study was found to be nearly quantitative (70-102% TAR). Radioactivity applied to the glass-enclosed plants, at 7 DAT, was distributed over the leaves (1% TAR), roots (0% TAR), soil (4-61% TAR) and traps (0-4%) or was associated with the tanks/tubes/pots (34-66% TAR) or was lost (30% TAR), probably because the soil was dried instead of being extracted.

Whole plants were collected at 0, 1, 3 and 7 DAT for autoradiography, which showed that residues were translocated rapidly throughout the spinach plant. At 1 DAT, there was no radioactivity associated with the roots and, at 7 DAT, the greater part of the residue had disappeared from the leaves.

The Viroflay variety was sampled at 0, 3, 7 and 14 DAT and the study was terminated at 28 DAT, because the plants had started flowering. The Perpetual variety was sampled at 0, 1, 3, 7, 14, 28, 60 and 92 DAT and separated into leaves and roots. Immediately after separation, the leaves and roots were extracted with acetone. Extracts were stored at 4°C for up to 1 month. Extracts and solids were subjected to (combustion) LSC. Extracts that contained sufficient radioactivity were examined by HPLC-UV, with radioactivity detection (ODS-2 column, isocratic elution). LSC analysis showed that total radioactive residues (TRR) had declined markedly by day 7, to <1% TAR (Table 17). By the time the plants had matured (60-92 DAT, treatment 1.5 kg ai/ha) the TRR was, at maximum, 0.0004 mg/kg eq, which was too low to allow identification of metabolites (see Table 17). In acetone extracts of immature plants harvested at 0, 1 and 3 DAT, parent lindane was the only radioactive component observed.

Table 17. Radioactive residues in spinach (Perpetual variety), treated once at 1.5 kg ai/ha (England, 1987; Curry and Brookman, 1997d)

DAT	Leaves, % TAR	Leaves, TRR, mg/kg eq	Leaves, acetone, % TRR	Leaves, solids, % TRR	Roots, TRR, mg/kg eq
0 [1/]	100	-	98	1.9	-
1	11	-	97	3.3	-
3	5.7	-	94	6.2	-
7	0.79	0.41	67	33	0.25
14	0.59	0.11	53	47	0.059
28	0.67	0.012	50	50	0.034
60	0.01	0.0001	ND	100	0.0004
92	0.09	0.0004	100	ND	0.0003

DAT = days after treatment.
ND = not detected, level not stated.
[1/] Whole plants, plants were too small to separate into leaves and roots.

Proposed metabolic pathways in livestock and in plants

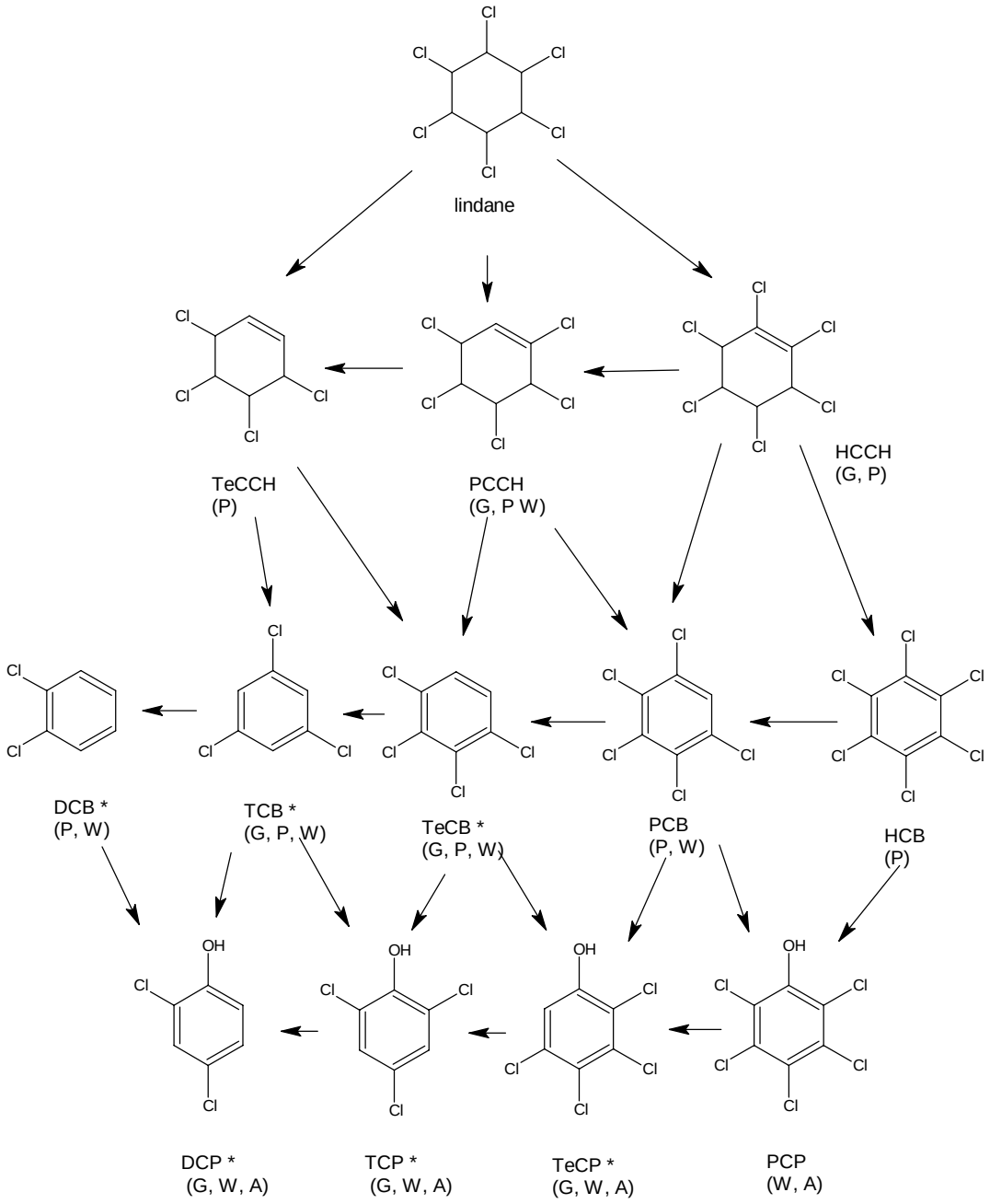


Figure 1. Metabolism of lindane in livestock (G = goat, P = poultry) and in plants (W = wheat roots, A = apple fruit/leaves).

Environmental fate in soil

The Meeting received information on rotational crop field studies.

Rotational crop studies in the field

A rotational crop study was conducted in the field, in Madera, California, USA (Hurshman and Xiao, 1991, GLP). Soil was treated with uniformly ^{14}C-labelled lindane, prior to planting of crops, at a treatment rate of 0.85 kg ai/ha, as an EC formulation and a spray volume of 700 l/ha. Lindane was incorporated to depths of 5 and 10 cm, on 7 September 1988. The specific activity of the undiluted radiolabel was 1.2 MBq/mmol and the radiochemical purity was >99%. Plots were irrigated with an overhead sprinkler system and fertilized when necessary. Plots were protected with plastic mesh cages against damage from insects, rodents and birds. Plots were covered with plastic sheets between

November 7, 1988 and February 3, 1989, when frost damage was possible. No additional chemical treatments were applied to the plots.

Soil cores (30 cm depth) were collected before, and immediately after, the incorporation of lindane. Similarly, cores were collected at each crop planting and when the plants or plant commodities were gathered. All cores were stored below –20 °C (storage time not stated). Subsequently, each core was cut in half, resulting in sub-cores from depths of 0-15 and 15-30 cm. Characteristics of the sandy loam soil for the 0-15 cm and 15-30 cm sub-cores were, respectively: 0.4% and 0.3% om; pH 6.3 and 6.7; CEC 3.3; and 2.8 and 8 % clay.

Lettuce (variety Walmann's Green Leaf), carrots (variety Goldmine), and barley (variety BB882) were planted at 30, 121 and 365 days after soil incorporation of lindane. Immature and mature samples of each crop were gathered from each planting. Lettuce samples were taken by cutting the plants just above the ground. Carrots were separated into roots and foliage. Barley was harvested as forage, straw and grain. Samples were stored below -20°C (storage time not stated).

The TRR in each dried soil sub-core was determined by (combustion) LSC, on a dry weight basis. In control soil samples, no residues were found. Results from treated soils are shown in Table 18. With one exception, the 15-30 cm sub-cores contained TRRs of ≤ 0.01 mg/kg eq.

Only the 0-15 cm sub-cores were extracted with acetone. Extracts were examined by HPLC-UV (two sequential columns, NovaPak Phenyl and NovaPak C18, with gradient elution) by comparing retention times against lindane and potential metabolites (1,3,4,5,6-PCCH, 2-CP, 2,4-DCP, 2,4,5-TCP, 2,3,4,6-TeCP and PCP). Lindane was the only residue component which was extractable from soil (see Table 18). This finding suggested that lindane and soil-bound (unextractable) residues were the materials to which the rotational crops would be exposed. Lindane residues in soil slowly declined over time, showing that it is rather persistent. At 240 DAT, 73% of the lindane present 2 hrs after treatment was still present.

Post-extraction solids from two 0-15 cm sub-cores were hydrolyzed, in an attempt to release additional radioactivity. The 267 DAT sub-core was hydrolyzed with 9 N H_2SO_4, in a sealed tube for 24 hrs at 55°C, and an additional 9.1% TRR was released. The 279 DAT sub-core was hydrolyzed with 6 N HCl for 24 hrs, under reflux, and an additional 18% TRR was released. Both aqueous fractions were extracted with ethyl acetate and examined by HPLC, as described above. Lindane was the only component identified in the ethyl acetate extracts: an additional 8.3% TRR in the 267 DAT sub-core and an additional 1.6% TRR in the 279 DAT sub-core.

Table 18. Radioactive residues in soil treated with lindane (Hurshman and Xiao, 1991).

Soil sampled, DAT	Planting interval, DAT	15-30 cm soil core	0-15 cm soil core	0-15 cm, acetone extract		0-15 cm, unextracted
		TRR, mg eq/kg dw	TRR, mg eq/kg dw	Parent lindane, % TRR	Unidentified, % TRR	% TRR
2 hrs	-	<LOQ	0.091	68	14	4.8
30	30	0.012	0.096	64	17	8.9
153	30	0.0079	0.088	61	4.6	9.1
183	30	<LOQ	0.092	61	3.6	10
212	30	0.0032	0.10	58	7.2	23
240	30	<LOQ	0.078	58	0.0	20
267	30	0.0030	0.067	28	2.7	60
121	121	0.0023	0.052	88	2.1	16
222	121	<LOQ	0.090	51	5.9	23
237	121	0.0074	0.055	56	6.9	31
249	121	0.0017	0.041	63	4.2	27
279	121	<LOQ	0.090	63	0.0	36
288	121	0.0059	0.044	28	3.0	25
308	121	<LOQ	0.040	57	7.6	33
365	365	0.0018	0.047	44	6.8	28
429	365	<LOQ	0.053	29	3.8	47
470	365	0.0016	0.028	30	6.4	64
491	365	0.0040	0.035	28	4.3	47
572	365	0.0038	0.030	-	34	72

Soil sampled, DAT	Planting interval, DAT	15-30 cm soil core	0-15 cm soil core	0-15 cm, acetone extract		0-15 cm, unextracted
		TRR, mg eq/kg dw	TRR, mg eq/kg dw	Parent lindane, % TRR	Unidentified, % TRR	% TRR
607	365	0.0053	0.041	-	25	64

- = Not analyzed.
LOQ = 25-35 dpm/g, not stated as mg/kg.

The TRR in each commodity was determined by (combustion) LSC and the results are shown in Table 19. The TRR in crop commodities did not appear to be strongly correlated with the TRR in soil. An approximately linear relationship between crop and TRR levels in soil was observed only in mature barley grain and straw.

Crop tissues were extracted with acetone, the acetone was evaporated from the extract, which was then extracted with hexane and analyzed by HPLC, as described above for soil. The identity of lindane and PCCH was confirmed by GC-MS (SE-54 column, positive EI), after derivatization of the extracts with MSTFA (*N*,*O*,-bis(trimethylsilyl)trifluoroacetamide). The identity of chlorophenolic metabolites was confirmed by GC-ECD (DB-5 column) after clean-up of the extracts using HPLC. In a single exception to the above procedure, mature barley grain from the 121-day planting was extracted with MeOH/water (70/30, v/v). The MeOH was removed by evaporation and the remaining aqueous extract was partitioned with ethyl acetate. Otherwise, the two procedures were identical. Table 19 lists the concentrations of parent and metabolites found in various commodities. In many cases, part of the organic extractable ^{14}C-residue was lost by evaporation during the concentration step, before HPLC analysis.

Table 19. Parent and metabolites [1/] of lindane in rotational crops, grown in soil treated at 0.85 kg ai/ha (Hurshman and Xiao, 1991).

Matrix	DAT	Planting interval DAT	TRR, mg/kg eq	Un-extracted, % TRR	Acetone extracts [2/]							
					Parent, % TRR	PCCH, % TRR	CP, % TRR	DCP, % TRR	TCP, % TRR	TeCP, % TRR	Unk % TRR	Lost, % TRR
Barley forage, immature	153	30	0.099	52	16	ND	ND	2.4	3.0	ND	3.6	13
	222	121	0.39	39	26	ND	ND	1.0	2.	4.4	0.05	18
	429	365	0.11	41	3.2	ND	ND	ND	ND	ND	9.3	56
Barley straw, mature	267	30	0.39	76	0.36	ND	ND	ND	0.42	ND	2.6	8.2
	279	121	0.93	78	2.4	ND	0.90	2.3	1.1	ND	1.7	10
	607	365	0.10	68	-	-	-	-	-	-	34 [3/]	-
Barley grain, mature	267	30	0.048	100	-	-	-	-	-	-	0 [3/]	-
	279	121	0.085	71	-	-	-	-	-	-	32 [3/]	-
	607	365	0.032	112	-	-	-	-	-	-	0 [3/]	-
Lettuce, immature	153	30	0.021	32	138	ND	ND	ND	ND	ND	ND	0
	237	121	0.042	25	26	ND	ND	ND	ND	ND	5.7	37
	470	365	0.012	36	-	-	-	-	-	-	80 [3/]	-
Lettuce, mature	212	30	0.043	35	43	ND	7.6	ND	7.0	4.3	5.4	0
	288	121	0.022	34	-	-	-	-	-	-	53 [3/]	-
	491	365	0.0091	36	-	-	-	-	-	-	59 [3/]	-
Carrot root, immature	183	30	1.3	1.1	58	ND	ND	ND	ND	ND	4.5	24
	249	121	0.72	1.4	94	4.6	ND	ND	ND	ND	1.0	0
	572	365	0.55	0.88	85	4.5	ND	ND	ND	ND	4.1	21
Carrot foliage, immature	183	30	0.14	6.0	88	ND	ND	ND	ND	ND	10	0
	249	121	0.33	6.2	67	ND	ND	ND	ND	ND	8.0	2.2
	572	365	0.038	24	-	-	-	-	-	-	74 [3/]	-
Carrot root, mature	240	30	0.44	1.0	48	ND	ND	ND	ND	ND	2.4	34
	308	121	0.41	2.5	83	5.6	ND	ND	ND	ND	3.2	0.05
	607	365	0.40	0.90	89	3.2	ND	ND	ND	ND	2.8	0
Carrot foliage, mature	240	30	0.092	13	69	ND	ND	ND	ND	ND	14	0
	308	121	0.19	10	91	ND	ND	ND	ND	ND	2.8	3.4
	607	365	0.064	29	18	ND	ND	ND	ND	ND	37	8.5

- = Not analyzed; imm = immature; mat = mature; unk = unknown fractions.
ND = not detected, <0.0014 mg/kg.
[1/] Identified metabolites were 1,3,4,5,6-PCCH, 2-CP, 2,4-DCP, 2,4,5-TCP and 2,3,4,6-TeCP.
[2/] Barley grain was extracted with MeOH/water.
[3/] Organic extracts were not further characterized.

Only the barley samples contained high proportions of unextractable residues. Direct hydrolytic extraction (9 N H_2SO_4), acid hydrolysis (24 hrs reflux with 6 N HCl) and base hydrolysis (3 N KOH in MeOH) were conducted on barley, to compare the various hydrolysis procedures. While none of the hydrolytic techniques produced a substantial release of additional organic extractable material, base hydrolysis was the most successful of the methods employed.

Post-extraction solids, from the acetone extraction of barley straw from the 121-day planting, were refluxed for 24 hrs with 6N HCl. The aqueous hydrolysate was partitioned with ethyl acetate, then adjusted to pH 12 and extracted again with ethyl acetate. This procedure released an additional 7.4% TRR, which was not further investigated.

Post-extraction solids, from acetone extraction of mature barley straw and grain from the 30-day planting, were hydrolyzed by refluxing for 24 hrs with 3N KOH in MeOH. MeOH was removed from the hydrolysate by evaporation. The resulting aqueous fraction was extracted with ethyl acetate. The remaining aqueous fraction was acidified to pH 2 and extracted with ethyl acetate. Hydrolysis released an additional 11% TRR organo-extractable residues from straw and 23% TRR from grain. Additional residue components in straw were identified as 4-CP (17% TRR), 2,4-DCP (3.8% TRR) and 2,4,5-CP (1.4% TRR), making a 202% recovery. Additional residue components in grain were identified as 4-CP (8.8% TRR), 2,4-DCP (2.1% TRR) and 2,4,5-TCP (3.4% TRR), leaving 9.2% TRR as unidentified.

Analysis of soil at each planting and crop sampling interval during the study revealed that lindane metabolites, if present in soil, were at levels too low to quantify. Thus, during the rotational crop study, lindane was available for uptake but its soil metabolites were not. Metabolites present in crop commodities therefore arose from metabolism of lindane within the plants. The metabolites identified in crop commodities from the rotational crop study are among those identified in the seed-treatment metabolism study on wheat. Thus, the rotational crop study confirms that degradation of lindane taken up by roots proceeds by hydroxylation and successive losses of chlorine, consistent with the proposed metabolic pathway in Figure 1.

Metabolite profiles in the 2 hr-treated soil sample and the 30-day rotational immature barley straw sample, analyzed at the beginning and the end of the analytical phase of the rotational crop study (Hurshman and Xiao, 1991), were compared and showed no major differences. Although this shows that the residues were stable in these matrices during storage for at least 20 weeks, no information was available on the period between sampling and the start of the analysis phase.

Environmental fate in water/sediment systems

The Meeting received information on hydrolysis and photolysis in water.

Hydrolysis and photolysis in water

Hydrolysis in water was determined, without adhering to a guideline (Mirfakhrae, 1986, non-GLP). Lindane was uniformly ^{14}C-labelled, with a specific activity of 131 MBq/mmol and a radiochemical purity of 97.9%. Solutions of 1 mg/l at pH 5, 7 and 9, at two buffer concentrations, were kept in the dark at 25 ± 1°C for 30 days. Single samples were taken at 0, 3, 7, 14 and 30 days and the compounds present were identified by TLC-autoradiography after hexane extraction, confirmation by GC-ECD and identification of degradation products by GC-MS.

At pH 5, lindane was stable, with half-lives of 173.3 and 115.4 days at buffer concentrations of 0.05 and 0.10 M, respectively. After 30 days, 5% of lindane had been degraded.

At pH 7, lindane was stable, with half-lives of 309.4 and 281.7 days at buffer concentrations of 0.05 and 0.10 M, respectively. After 30 days, 5% of lindane had been degraded.

At pH 9, lindane was unstable, with half-lives of 36.3 and 35.4 days at buffer concentrations of 0.05 and 0.10 M, respectively. After 30 days, 43-44% of lindane had been degraded, forming 7% 2,3,4,5,6-pentachlorocyclohexene and 4% trichlorobenzenes (1,2,4-trichlorobenzene and 1,2,3-trichlorobenzene) and 32-33% unaccountable.

In a first study, photolysis in water was determined according to EPA guideline 161-2 (Norris, 1986a, non-GLP). Lindane was uniformly ^{14}C-labelled, with a specific activity of 131 MBq/mmol and a radiochemical purity of 97.8%. Solutions of 0.64 mg/l lindane in water were irradiated, outside, with natural sunlight for 28 days in New Jersey, USA, latitude 40°25'N, longitude 74°30'E (day temperatures 12-44°C, period August-September). Duplicate samples were taken at 0, 7, 14, 21 and 28 days and the compounds present were identified by ethyl acetate extraction followed by GC-ECD and TLC-autoradiography, against reference standards. Lindane was resistant to photolysis by natural sunlight. After 28 days, ≥90% recovery was found (dark control at 25°C ≥92% recovery). No degradation products were observed.

In a second study, photolysis in water was determined under the same conditions as in study 1, except that 1.3 mg/l lindane in water plus 1.8% (v/v) acetone was used and the experiment was conducted in September at a day temperature 12-44 °C (Norris, 1986b, non-GLP). In acetone-sensitized aqueous solution, lindane was resistant to natural sunlight. After 28 days, ≥84% recovery was found (dark control data not available). No degradation products were observed.

In a third study, photolysis in water was determined according EPA guideline 161-2 (Willems, 1999, GLP). Lindane was uniformly ^{14}C-labelled, with a specific activity of 1032 MBq/mmol and a radiochemical purity of 99.6%. Solutions of 2.6 mg/l lindane in water plus 0.2% (v/v) ACN (not a sensitizer), buffered at pH 7 (KH_2PO_4/NaOH) was irradiated with simulated sunlight (1.5 kW Xenon lamp, ≥290 nm, 700 W/m^2) continuously for 15 days at 25.4 ± 1.5°C (standard deviation). The irradiation was equivalent to 44.5 days of natural sunlight at 40°N in summer, assuming 12 hrs light per day. Single samples were taken at 0, 1, 3, 7, 10 and 15 days and the compounds present were identified by ethyl acetate extraction followed by HPLC-LSC and TLC-autoradiography, against reference standards. In water at pH 7 and 25°C, lindane was resistant to simulated sunlight. After 15 days, ≥92% recovery was found (dark control ≥91% recovery). No degradation products were observed.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The meeting received analytical methods proposed as enforcement method and methods used in supervised residue trials, storage stability studies, processing studies and feeding studies. Analytical methods for use on spinach and cucumbers were not summarized because no corresponding residue trials data were submitted.

Enforcement methods

The Meeting was informed by the government of The Netherlands about the existence of a multi-residue enforcement method for fruit and vegetables, consisting of an extraction method for non-fatty matrices and detection by GC-ITD, with an LOQ of 0.03 mg/kg (Netherlands, 1996). Further details were not given.

The AOAC method was published as a multi-residue method for the determination of organochlorine and organophosphorus pesticide residues (AOAC, 1984) in non-fatty foods (including barley, corn meal and silage, hay, mustard greens, oats, popcorn, wheat), dairy products and whole eggs. Homogenized samples were extracted with acetonitrile, ACN, (high-water samples) or aqueous ACN (low-water samples or high-sugar foods). Fat was extracted from fatty foods (milk, cheese, butter, fish, fats and oils) and partitioned between petroleum ether and ACN. ACN extracts were diluted with water and the residues partitioned into petroleum ether. Petroleum ether extracts were purified by chromatography on a Florisil column from which residues were eluted by a mixture of petroleum ether and diethyl ether. Lindane in the concentrated eluates was quantified by GC (DC-200 or OV-101 column) with ECD. Complete recovery (>80%) was reported but details were not shown (McMahon and Burke, 1978).

Methods used for plant commodities

Method 109 was used in supervised trials, storage stability studies and processing studies on wheat and canola. Method 109, 04/98 was intended for the determination of lindane (parent) residues in

wheat (Willard, 1999). Homogenized samples (20 g) were extracted with ACN/water. The water was salted out, the dried ACN extract was washed with hexane to remove oils and other hexane-soluble co-extractives, followed by an additional solid-phase clean-up.. Quantification was by GC-MS (fused silica column coated with RTX-1 or RTX-200; *m/z* 219). Calibration was by external standards in toluene, using four levels in the range 0.006-0.08 mg/l, equivalent to 0.003-0.04 mg/kg. Extracts from recovery performed at 0.05 mg/kg were diluted before analysis. Validation results are shown in Table 20. Linearity was verified: r≥0.998 (2 curves). Matrix effects (on the slope of the calibration curve) were not investigated.

Method 109 was revised for the analysis of wheat and canola (Willard, 1999 and 2000). Revision #2, 03/99, incorporated additional clean-up steps, to obtain a cleaner extract: a hexane partition, gel permeation chromatography, a DCM/salt-water partition and solid-phase extraction. Validation results are shown in Table 20. Linearity was verified: r≥0.9994 (4 curves). Matrix effects (on the slope of the calibration curve) were not investigated. Because of matrix interferences, the LOQ should be increased to at least 0.0037/0.3 = 0.01 mg/kg in wheat hay.

Table 20. Validation data for method 109 (Willard 1999 and 2000).

Commodity	LOQ, reported	Spike, mg/kg	n	% rec., mean	% rec., range	RSD_R [1/]	Controls, mg/kg	Reference, method version
Wheat, forage	0.005	0.005 0.05	6 6	80 78	70-90 77-81	9.2% 1.8%	<0.0017 (5)	Willard, 1999, GLP M 109, original
Wheat, forage	0.005	0.005 0.05	5 6	94 91	81-109 89-99	13% 3.9%	<0.0017 (10)	Willard, 1999, GLP M 109, rev. #2
Wheat, hay	0.005	0.005 0.05	11 11	97 90	87-107 72-101	7.3% 8.7%	<0.0017-0.0037 (17)	Willard, 1999, GLP M 109, rev. #2
Wheat, straw	0.005	0.005 0.05	12 12	103 95	82-121 65-122	15% 14%	<0.0017 (17)	Willard, 1999, GLP M 109, rev. #2
Wheat, grain	0.005	0.005 0.01	11 11	99 107	90-111 99-118	7.4% 8.0%	<0.0017 (18)	Willard, 1999, GLP M 109, rev. #2
Canola, seed	0.005	0.005 0.05	7 7	82 84	71-90 74-106	6.6% 13%	<0.0017 (11)	Willard, 2000, GLP M 109, rev. #2
Canola, meal	0.005	0.005 0.05	4 4	97 90	89-110 77-102	9.8% 11%	<0.0017 (4)	Willard, 2000, GLP M 109, rev. #2
Canola, refined oil	0.005	0.005 0.01 0.05	4 1 4	95 81 92	80-119 - 79-109	17% - 15%	<0.0017 (5)	Willard, 2000, GLP M 109, rev. #2
Canola, edible oil	0.005	0.005 0.02	1 1	78 76	- -	- -	<0.0017 (1)	Willard, 2000, GLP M 109, rev. #2

[1/] Within-laboratory reproducibility.

Methods used for animal commodities

Modified AOAC methods were used in feeding studies on poultry, cows, sheep and swine and in storage stability studies on animal commodities.

Modified AOAC method, ASD 87/241, 1987-1988. For the storage stability study on tissues, eggs and milk (Piznik *et al*., 1988), the AOAC method was modified and renamed as ASD 87/241. Homogenized tissues and eggs (10 g) were extracted with ACN. Milk (20 g) was extracted according to the AOAC method. The extracts were filtered and evaporated to dryness. The residual material was re-dissolved in toluene/ethyl acetate (1:3 v/v) and cleaned-up by GPC. The eluates were evaporated to dryness and re-dissolved in hexane. Lindane was determined by GC-ECD (3% OV-17). Residues were quantified using a single external standard, at 0.004 mg/l (mg/kg equivalents were not stated). Method validation results are shown in Table 21 (Piznik, 1987b). Linearity of the calibration curve was not shown and matrix effects (on the slope of the calibration curve) were not investigated. Due to matrix interferences, the LOQ should be increased to at least 0.0017/0.3 = 0.006 mg/kg for milk and 0.002/0.3 = 0.007 mg/kg for eggs.

Modified AOAC method, unnamed, 1987-1988. For the storage stability study on milk and the feeding studies on cows and poultry (Merricks, 1987b and 1988; Curry and Brookman, 1997f; Curry *et al*., 1999a), the AOAC method was modified. Milk (10-20 g) was extracted with hexane and lindane was partitioned into ACN. Tissues (10 g) and eggs (10 g) were extracted with ACN. The

extract was mixed with saturated NaCl solution and lindane was partitioned into hexane. Lindane was determined by GC-ECD (3% OV-17). A separate method validation study was not conducted. Validation results in the form of procedural recoveries are shown in Table 21. Information on calibration was not given. Results above 0.6 mg/kg in milk were not considered to be valid, because of poor precision in procedural recovery (48%-142%). Due to matrix interferences, the LOQ should be increased to at least 0.024/0.3 = 0.08 mg/kg for eggs, 0.0048/0.3 = 0.02 mg/kg for milk, 0.01/0.3 = 0.04 mg/kg for muscle (hen, cow), 0.013/0.3 = 0.05 mg/kg for liver (hen, cow) and 0.016/0.3 = 0.06 mg/kg for kidney (hen, cow).

Modified AOAC method, SBL SOP 81.00-87, 1988. For the feeding study on sheep (Billings, 1988b), the AOAC method was modified and renamed as SBL SOP 81.00-87. Tissues (5-20 g) were extracted with ACN in the presence of Celite. Fat (3 g) was warmed to liquefy it, diluted with petroleum ether and the lindane partitioned into ACN. The ACN extract was mixed with saturated NaCl solution and lindane was partitioned into petroleum ether, for further clean-up on a Florisil column. The lindane was eluted with 6% ethyl ether in petroleum ether and was determined by GC-ECD (3% SP-2250 on Supelcoport 100/200 mesh). Lindane was quantified using 8 single standards in petroleum ether, in the range 0.01-0.80 mg/l. Validation results are shown in Table 21. Linearity of the calibration curve was not demonstrated and matrix effects (on the slope of the calibration curve) were not investigated. Due to matrix interferences, the LOQ should be increased to at least 0.033/0.3 = 0.1 mg/kg for fat (sheep).

Modified AOAC method, SBL SOP 81.01-88, 1988. For the feeding study on pigs (Billings, 1988a), the AOAC method was modified and renamed as SBL SOP 81.01-88. The procedure was the same as for SBL SOP 81.00-87, except that fat (3 g) was homogenized in dry ice and was extracted cold with ACN. A separate validation study was not performed.

Table 21. Validation results for modified AOAC methods

Commodity	LOQ, reported	Spike, mg/kg	n	% rec., mean	% rec., range	RSD_r or RSD_R [1/]	Controls, mg/kg (n)	Reference
Liver (beef)	0.01	0.01	2	91	84-98	-	ND (6)	Piznik, 1987b ASD 87/241, storage stability study
		0.05	1	99	-	-		
		0.1	1	84	-	-		
		1	1	100	-	-		
		5	1	83	-	-		
Kidney (beef)	0.01	0.01	2	91	84-98	-	ND (6)	Piznik, 1987b ASD 87/241, storage stability study
		0.05	1	101	-	-		
		0.1	1	91	-	-		
		1	1	103	-	-		
		5	1	88	-	-		
Muscle (beef)	0.01	0.01	2	108	100-115	-	ND (6)	Piznik, 1987b ASD 87/241, storage stability study
		0.05	1	110	-	-		
		0.10	1	94	-	-		
		1.0	1	88	-	-		
		10	1	89	-	-		
Fat (beef)	0.01	0.01	2	106	97-115	-	<0.003 (6)	Piznik, 1987b ASD 87/241, storage stability study
		0.05	1	91	-	-		
		0.1	1	92	-	-		
		1	1	99	-	-		
		2	1	98	-	-		
Milk	0.001	0.001	3	72	67-80	9.4%	0.0003-0.0017 (5)	Piznik, 1987b ASD 87/241, storage stability study
		0.005	2	88	78-98	-		
		0.01	3	85	74-103	18%		
		0.1	1	68	-	-		
		0.5	2	80	77-82	-		
		1	2	86	85-88	-		
		2	1	85	-	-		
Chicken liver	0.01	0.01	1	93	-	-	ND (6)	Piznik, 1987b ASD 87/241, storage stability study
		0.05	1	93	-	-		
		0.1	1	101	-	-		
		0.2	1	106	-	-		
		0.5	1	93	-	-		

Commodity	LOQ, reported	Spike, mg/kg	n	% rec., mean	% rec., range	RSD_r or RSD_R [1/]	Controls, mg/kg (n)	Reference
		1	1	93	-	-		
Chicken muscle	0.01	0.01	1	81	-	-	ND (6)	Piznik, 1987b ASD 87/241, storage stability study
		0.05	1	100	-	-		
		0.1	1	99	-	-		
		0.2	1	100	-	-		
		0.5	1	94	-	-		
		1	1	91	-	-		
Eggs	0.005	0.005	3	96	79-109	16%	<0.0015-0.002	Piznik, 1987b ASD 87/241, storage stability study
		0.05	2	93	92-93	-		
		0.1	2	96	92-100	-		
		0.5	2	98	97-100	-		
		1	2	98	97-99	-		
Milk (cow)	0.001	0.001	1	88	-	-	ND-0.0048 (14)	Merricks, 1987b, modified method, (cow feeding study and milk storage stability)
		0.05	2	85	82-88	-		
		0.5	4	86	74-100	17%		
		0.6	1	94	-	-		
		1.0	4	85	58-133	39%		
		2.0	3	93	54-117	37%		
		2.5	1	109	-	-		
		5.0	4	86	55-115	29%		
		10.0	3	98	48-142	48%		
Liver (cow)	0.01	0.01	1	104	-	-	0.007-0.015 (2)	Merricks, 1987b, modified method, (cow feeding study and milk storage stability)
		0.1	1	89	-	-		
		10	1	114	-	-		
Kidney (cow)	0.01	0.1	1	100	-	-	ND (1)	Merricks, 1987b, modified method, (cow feeding study and milk storage stability)
		10	1	106	-	-		
Muscle (cow)	0.01	0.01	1	91	-	-	0.0058 (1)	Merricks, 1987b, modified method, (cow feeding study and milk storage stability)
		0.1	1	87	-	-		
		10	1	97	-	-		
Fat (cow)	0.01	0.01	1	112	-	-	0.0053-0.011 (2)	Merricks, 1987b, modified method, (cow feeding study and milk storage stability)
		0.1	1	111	-	-		
		200	1	116	-	-		
Eggs	0.005	0.005	6	102	62-127	25%	<0.005 – 0.024 (12)	Merricks, 1988, modified method, (poultry feeding study)
		0.01	2	85	75-95	-		
		0.05	8	96	69-123	16%		
Liver (hen)	0.01	0.05	1	84	-	-	<0.005-0.013	Merricks, 1988, modified method, (poultry feeding study)
		0.5	1	87	-	-		
		5	1	75	-	-		
Kidney (hen)	0.01	0.5	1	88	-	-	<0.01-0.016 (2)	Merricks, 1988, modified method, (poultry feeding study)
		5	1	100	-	-		
Muscle (hen)	0.01	0.05	1	111	-	-	<0.01-0.01 (4)	Merricks, 1988, modified method, (poultry feeding study)
		0.1	1	78	-	-		
		0.5	1	90	-	-		
		5	1	107				
Fat (hen)	0.01	1.0	1	90	-	-	<0.003 (2)	Merricks, 1988, modified method, (poultry feeding study)
		100	1	91				
Muscle (sheep)	0.01	0.1	9	92	69-108	15%	ND (3)	Billings, 1988b SBL SOP 81.00-87, sheep feeding study
		1	9	85	72-100	10%		
		10	9	89	80-94	5.0%		
Fat (sheep)	0.01	0.1	8	101	84-114	10%	0.030-0.033 (3)	Billings, 1988b SBL SOP 81.00-87, sheep feeding study
		1	9	105	101-117	5.8%		
		10	9	104	99-119	5.9%		

ND = not determined, detection limit not stated.

[1/] Data from Piznik (1987b) and Merricks (1987b) represent within-laboratory repeatability, RSD_r, whereas those from Merricks (1988) and Billings (1988b) represent within-laboratory reproducibility, RSD_R.

Stability of pesticide residues in stored analytical samples

The Meeting received data on the stability of residues in wheat (forage, hay, straw, grain), canola (seed, meal, refined oil), spinach and cucumbers, stored frozen. Storage stability data on spinach and cucumbers were not summarized, because no corresponding residue trials were available. In addition, the Meeting received data on the stability of residues in animal commodities (tissues, eggs, milk).

Wheat

Willard, 1999 & 2001 (GLP) fortified wheat samples (forage, hay, straw, grain) with 0.05 mg/kg lindane. Samples were kept frozen at –20 ± 5°C and were analyzed for lindane (parent), using revision #2 of Method 109 (see Table 22). Results were not corrected for concurrent method recovery (76-114%), nor for matrix interferences (<0.005 mg/kg). Results are shown in Table 22. Lindane was stable in wheat matrices for the time periods investigated.

Table 22. Storage stability in wheat matrices (n = 2) fortified with lindane at 0.05 mg/kg (Willard, 1999 & 2001).

Commodity	Storage time, days	Lindane remaining, %
Wheat, forage	0	75-77 [1/]
	434	101-102
Wheat, hay	0	92-92
	187	75-83
	553	75-77
Wheat, straw	0	88-100
	185	80-81
	550	79-83
Wheat, grain	0	95-101
	188	83-94
	546	82-84

[1/] Sample was analyzed using the original version of method 109 (04/98).

Canola

Willard, 2000 (GLP) fortified canola samples (seed, meal, refined oil, edible oil) with 0.05 mg/kg lindane. Samples were kept frozen at –20 ± 5°C and were analyzed for lindane (parent) using revision #2 of Method 109 (see Table 23). Results were not corrected for concurrent method recovery (76-119%) but were corrected for matrix interferences (<0.005 mg/kg). Results are shown in Table 23. Lindane was stable in canola matrices for the time periods investigated.

Table 23. Storage stability in canola matrices (n = 2) fortified with lindane at 0.05 mg/kg (Willard, 2000).

Commodity	Storage time, days	Lindane remaining, %
Canola, seed	0	87-89
	31	66-78
	198	70-77
Canola, meal	0	96-104
	29	84-88
	47	81-82
Canola, refined oil	0	94-99
	29	87-88
	56	106-108

Animal commodities

Merricks, 1987b, fortified cow milk with 1.0-5.0 mg/kg lindane. Samples were kept frozen at –15°C for 265 days and were analyzed for lindane, using a modified AOAC method. Results were not corrected for concurrent method recovery (103-117%). Matrix interferences were not stated. Results are shown in Table 24. Lindane was stable in milk for at least 265 days.

Table 24. Storage stability in milk (n = 2) fortified with lindane

Commodity	Storage time, days	Lindane remaining, %	
		Fortified at 1.0 mg/kg	Fortified at 5.0 mg/kg
Cow milk	0	97-105	101-103
	64	94-96	75-85
	265	86-98	83-92

Beef and chicken tissues, eggs and milk were fortified at 0.5 mg/kg lindane (Piznik *et al*., 1988, non-GLP; Curry and Brookman, 1997e; Curry *et al*., 1999b). All commodities were purchased in local retail food stores. Samples were kept frozen at –18 °C for 12 months and were analyzed for lindane, using a modified AOAC method, method ASD 87/241. Procedural recoveries were determined in eggs at 1-3 months and in beef muscle at 1-6-months but no procedural recovery data were provided for the 9 and 12-month intervals. Results were not corrected for concurrent method recovery (96-106%). Results from control samples were not reported. Results for the 4-month storage period for beef were not considered to be valid, because of a poor procedural recovery (208%). Results (n=1) are shown in Table 25. Lindane was stable for 9 months in tissues and for at least 12 months in milk and eggs.

Table 25. Stability of lindane in animal commodities spiked at 0.5 mg/kg and stored at –18°C (Piznik *et al*., 1988; Curry and Brookman, 1997e; Curry *et al*., 1999b).

Months	Lindane % remaining								Procedural recovery, %	
	Chicken			Beef						
	Muscle	Liver	Eggs	Muscle	Fat	Liver	Kidney	Milk	Eggs	Beef muscle
1	NA	NA	92	NA	92	NA	NA	82	96	94
2	98	86	76	92	78	86	102	78	100	98
3	86	98	86	88	84	96	100	96	98	98
4	90	78	-	46	32	78	86	86	--	208
5	64	90	80	80	84	76	46	80	--	106
6	66	84	82	48	56	72	72	20	--	90
9	84	118	80	98	98	88	84	102	--	--
12	68	94	106	82	84	84	100	108	--	--

NA = Not analyzed.
-- = Not determined.

USE PATTERN

Lindane is a broad spectrum insecticide, used for the protection of seeds and seedlings against wireworms, seed corn maggots, flea beetles, seed corn beetles, and/or white grubs. It is also used in the field for control of white grubs, symphilids, and/or wireworms and is used against horn-flies, lice, fleas, ticks and/or mange mites on livestock.

Lindane is authorised for use as a seed treatment in the USA and Canada on cereal grains (barley, corn, oats, rye, sorghum, wheat) and in Canada on oilseeds (mustard). Lindane is authorised for pre-harvest treatments in Australia on pineapple. Lindane is not authorised for use in Germany and The Netherlands.

Because the basic manufacturer wished to support seed treatment uses on canola and cereal grains, only the uses on oilseeds and cereal grains are summarized in Tables 26 and 27. For all uses, except canola, original labels were available. For canola, only a pending label was available and therefore this information is not included in Table 26. Labels warn against use of oilseeds or cereal grains as food, feed or for oil production and, for this reason, some of the formulations contain a dye to identify the seeds as inedible. For calculation purposes, 1 tonne of cereal grains was assumed to weigh 1000 kg.

Table 26. Registered pre-planting seed treatments of lindane on oilseeds.

Crop	Country	Form g ai/l or g ai/kg	Application				Remarks
			Method	Rate, g ai/t	Spray conc, kg ai/hl	No.	
Mustard	Canada	FS 400 [1]	Liquid seed-treating equipment	11200	na	1	≤ 4 months before sowing

na Not applicable. Formulation is used directly on seed, no dilution with water needed.
[1] Liquid combination formulation of 40.0% (w/w) lindane with 1.6% thiabendazole and 4.8% thiram.

Table 27. Registered pre-planting seed treatments of lindane on cereal grains.

Crop	Country	Form g ai/l or g ai/kg [1]	Application				Remarks
			Method	Rate, g ai/t	Spray conc, kg ai/hl	No.	
Barley	Canada	DS 187.5 [17]	Mixing with seed in drill or planter box or any standard dry seed treating equipment or by shovel method	488	na	1	Immediately before sowing
	Canada	LS 108 [18]	On-farm or commercial use with liquid closed seed treating system	428	na	1	Immediately before sowing
	USA [2]	DS 166 [12]	Mixing with seed in planter box	415	na	1	Immediately before sowing
	USA [2]	DS 187.5 [6]; DS 187.5 [9]; DS 187.5 [14]	Mixing with seed in drill or planter box	488 [3]	na	1	Immediately before sowing
	USA [2]	DS 250; DS 250 [8]	Mixing with seed in drill or planter box or any commercial or barrel treater	625	na	1	≤ 6 weeks before sowing
	USA [2]	DS 250	Mixing with seed in planter box	2601 [3]	na	1	Immediately before sowing
	USA [2]	LS 69 [4,13]	Mixing with seed in hopper or drill box or on-farm seed treatment machine	375 [3]	na	1	Immediately before sowing
	USA [2]	FS 93 [4,11]	ns	364	na	1	-
	USA [2]	LS 106 [4,7]	Mixing with seed in drill or planter box	432 [3]	na	1	Immediately before sowing
	USA [2]	ES 120 [4,10]	ns	627	na	1	-
	USA [2]	ES 249 [4]	Seed treating equipment	263	na	1	≤ 6 weeks before sowing
	USA [2]	FS 359 [4]	Mixing with seed in liquid or slurry treaters	316	na	1	Immediately before sowing
	USA [2]	FS 353 [4]	Mixing with seed in liquid or slurry treaters	311	na	1	Immediately before sowing
	USA [2]	ES 508 [4]	Mixing with seed in liquid or slurry treaters	332	na	1	Immediately before sowing
	USA [2]	FS 479 [4]	Commercial seed treatment facilities with closed transfer systems	313	na	1	-
Corn (maize)	Canada	DS 250 [15]	Mixing with seed in drill or planter box	500-551	na	1	At or near planting time
	Canada	DS 166 [19]	Mixing with seed	564	na	1	-
	Canada	DS 166 [19]	Mixing with seed in slurry machine	523	4.15	1	-
	Canada	DS 166 [19]	Hand mixing as slurry	558	2.79	1	-
	USA [2]	DS 166 [12]	Mixing with seed in planter box	830	na	1	Immediately before sowing
	USA [2]	DS 187.5 [6]; DS 187.5 [9]; DS 187.5 [14]	Mixing with seed in drill or planter box	576-628 [3]	na	1	Immediately before sowing
	USA [2]	DS 250 [5]; DS 250 [16]	Mixing with seed in drill or planter box	558-562	na	1	Immediately before sowing
	USA [2]	DS 250 [8]	Mixing with seed in drill or planter box or any commercial or barrel treater	625-1250	na	1	Immediately before sowing
	USA [2]	DS 250	Mixing with seed in planter box or any commercial or barrel treater	625	na	1	≤ 6 weeks before sowing

Crop	Country	Form g ai/l or g ai/kg [1]	Application				Remarks
			Method	Rate, g ai/t	Spray conc, kg ai/hl	No.	
	USA [2]	DS 250	Mixing with seed in planter box	2232 [3]	na	1	Immediately before sowing
	USA [2]	LS 106 [4,7]	Mixing with seed in drill or planter box	494 [3]	na	1	Immediately before sowing
	USA [2]	ES 249 [4]	Seed treating equipment	650	na	1	≤ 6 weeks before sowing
	USA [2]	FS 359 [4]	Mixing with seed in liquid or slurry treaters	780-1563	na	1	Immediately before sowing
Maize: field corn	USA [2]	DS 250 [15]	Mixing with seed in planter box	558 [3]	na	1	Immediately before sowing
Maize: sweet corn	USA [2]	DS 250 [15]	Mixing with seed in planter box	558 [3]	na	1	Immediately before sowing
Oats	Canada	DS 187.5 [17]	Mixing with seed in drill or planter box or any standard dry seed treating equipment or by shovel method	690	na	1	Immediately before sowing
	Canada	LS 108 [18]	On-farm or commercial use with liquid closed seed treating system	596	na	1	Immediately before sowing
	USA [2]	DS 166 [12]	Mixing with seed in planter box	208	na	1	Immediately before sowing
	USA [2]	DS 187.5 [6]; DS 187.5 [9]; DS 187.5 [14]	Mixing with seed in drill or planter box	733 [3]	na	1	Immediately before sowing
	USA [2]	DS 250; DS 250 [8]	Mixing with seed in drill or planter box or any commercial or barrel treater	312	na	1	≤ 6 weeks before sowing
	USA [2]	DS 250	Mixing with seed in drill box	625	na	1	-
	USA [2]	DS 250	Mixing with seed in planter box	3910 [3]	na	1	Immediately before sowing
	USA [2]	LS 69 [4,13]	Mixing with seed in hopper or drill box or on-farm seed treatment machine	563 [3]	na	1	Immediately before sowing
	USA [2]	FS 93 [4,11]	Ns	304	na	1	-
	USA [2]	LS 106 [4,7]	Mixing with seed in drill or planter box	649 [3]	na	1	Immediately before sowing
	USA [2]	ES 249 [4]	Seed treating equipment	163-325	na	1	≤ 6 weeks before sowing
	USA [2]	FS 359 [4]	Mixing with seed in liquid or slurry treaters	328	na	1	Immediately before sowing
	USA [2]	FS 353 [4]	Mixing with seed in liquid or slurry treaters	323	na	1	Immediately before sowing
	USA [2]	ES 508 [4]	Mixing with seed in liquid or slurry treaters	332	na	1	Immediately before sowing
	USA [2]	FS 479 [4]	Commercial seed treatment facilities with closed transfer systems	313	na	1	-
Rye	Canada	DS 187.5 [17]	Mixing with seed in drill or planter box or any standard dry seed treating equipment or by shovel method	420	na	1	Immediately before sowing
	Canada	LS 108 [18]	On-farm or commercial liquid closed seed treating systems	281	na	1	Immediately before sowing
	USA [2]	DS 166 [12]	Mixing with seed in planter box	208	na	1	Immediately before sowing
	USA [2]	DS 187.5 [6]; DS 187.5 [14]	Mixing with seed in drill or planter box	419 [3]	na	1	Immediately before sowing
	USA [2]	DS 250; DS 250 [8]	Mixing with seed in drill or planter box or any commercial or barrel treater	312	na	1	≤ 6 weeks before sowing
	USA [2]	DS 250	Mixing with seed in planter box	2232 [3]	na	1	Immediately before sowing

Crop	Country	Form g ai/l or g ai/kg [1]	Application				Remarks
			Method	Rate, g ai/t	Spray conc, kg ai/hl	No.	
Rye	USA [2]	LS 69 [4,13]	Mixing with seed in hopper or drill box or on-farm seed treatment machine	322 [3]	na	1	Immediately before sowing
	USA [2]	LS 106 [4,7]	Mixing with seed in drill or planter box	371 [3]	na	1	Immediately before sowing
	USA [2]	ES 249 [4]	Seed treating equipment	293	na	1	≤ 6 weeks before sowing
	USA [2]	FS 359 [4]	Mixing with seed in liquid or slurry treaters	328	na	1	Immediately before sowing
	USA [2]	FS 353 [4]	Mixing with seed in liquid or slurry treaters	323	na	1	Immediately before sowing
	USA [2]	ES 508 [4]	Mixing with seed in liquid or slurry treaters	332	na	1	Immediately before sowing
	USA [2]	FS 479 [4]	Commercial seed treatment facilities with closed transfer systems	313	na	1	-
Sorghum	USA [2]	DS 166 [12]	Mixing with seed in planter box	556 [3]	na	1	Immediately before sowing
	USA [2]	DS 187.5 [14]	Mixing with seed in drill or planter box	628 [3]	na	1	Immediately before sowing
	USA [2]	DS 250; DS 250 [8]	Mixing with seed in drill or planter box or any commercial or barrel treater	625	na	1	≤ 6 weeks before sowing
	USA [2]	DS 250	Mixing with seed in planter box	2232 [3]	na	1	Immediately before sowing
	USA [2]	ES 249 [4]	Seed treating equipment	650	na	1	≤ 6 weeks before sowing
	USA [2]	FS 359 [4]	Mixing with seed in liquid or slurry treaters	626-937	na	1	Immediately before sowing
Wheat	Canada	DS 187.5 [17]	Mixing with seed in drill or planter box or any standard dry seed treating equipment or by shovel method	390	na	1	Immediately before sowing
	Canada	LS 108 [18]	On-farm or commercial use with liquid closed seed treating system	337	na	1	Immediately before sowing
	USA [2]	DS 166 [12]	Mixing with seed in planter box	208	na	1	Immediately before sowing
	USA [2]	DS 187.5 [6]; DS 187.5 [9]; DS 187.5 [14]	Mixing with seed in drill or planter box	391 [3]	na	1	Immediately before sowing
	USA [2]	DS 250; DS 250 [8]	Mixing with seed in drill or planter box or any commercial or barrel treater	312	na	1	≤ 6 weeks before sowing
	USA [2]	DS 250	Mixing with seed in planter box	2085 [3]	na	1	Immediately before sowing
	USA [2]	LS 69 [4,13]	Mixing with seed in hopper or drill box or on-farm seed treatment machine	300 [3]	na	1	Immediately before sowing
	USA [2]	FS 93 [4,11]	ns	304	na	1	-
	USA [2]	LS 106 [4,7]	Mixing with seed in drill or planter box	346 [3]	na	1	Immediately before sowing
	USA [2]	ES 120 [4,10]	ns	313	na	1	-
	USA [2]	ES 249 [4]	Seed treating equipment	163-325	na	1	≤ 6 weeks before sowing
	USA [2]	FS 359 [4]	Mixing with seed in liquid or slurry treaters	328	na	1	Immediately before sowing
	USA [2]	FS 353 [4]	Mixing with seed in liquid or slurry treaters	323	na	1	Immediately before sowing
	USA [2]	ES 508 [4]	Mixing with seed in liquid or slurry treaters	332	na	1	Immediately before sowing
	USA [2]	FS 479 [4]	Commercial seed treatment facilities with closed transfer systems	313	na	1	-

na = Not applicable. Formulation is used directly on seed, no dilution with water needed.
ns = Not specified.
1/ Formulation concentration in g ai/kg for solid formulations (DS) and in g ai/l for liquid formulations (ES, FS, LS).
2/ GAP information provided/confirmed by national government.
3/ Dose rate expressed as oz/bushel or fl. oz/bushel. One bushel of grain was assumed to be 21.8 kg for barley, 25.4 kg for corn, 14.5 kg for oats, 25.4 kg for rye, 25.4 kg for sorghum and 27.2 kg for wheat.
4/ Labels on fluid formulations contained only w/w information; concentrations in g ai/L were obtained separately from the basic manufacturer.
5/ Combination formulation of 25.00% (w/w) lindane with 14.67% captan, 15.52% diazinon and 1.00% metalaxyl.
6/ Combination formulation of 18.75% (w/w) lindane with 50.00% maneb.
7/ Combination formulation of 8.6% (w/w) lindane with 25.6% maneb.
8/ Combination formulation of 25.0% (w/w) lindane with 12.20-12.23% captan.
9/ Combination formulation of 18.75% (w/w) lindane with 20.00% carboxin and 35.00% maneb.
10/ Combination formulation of 10.53% (w/w) lindane with 17.68% pentachlorobenzene.
11/ Combination formulation of 8% (w/w) lindane with 14% carboxin and 12% thiram.
12/ Combination formulation of 16.6% (w/w) lindane with 32.75% captan.
13/ Combination formulation of 6.60% (w/w) lindane with 20.00% mancozeb (= 4.00% Mn^{2+}, 0.50% Zn^{3+} and 15.50% ethylenebisdithiocarbamate ion).
14/ Combination formulation of 18.75% (w/w) lindane with 50.00% mancozeb (= 10.00% Mn^{2+}, 1.25% Zn^{3+} and 38.75% ethylenebisdithiocarbamate ion)
15/ Combination formulation of 25.00% or 25% (w/w) lindane with 14.67% or 15% captan and 15.00% or 15% diazinon.
16/ Combination formulation of 25% (w/w) lindane with 14% carboxin and 15% diazinon.
17/ Combination formulation of 18.75% (w/w) lindane with 37.5% maneb.
18/ Combination formulation of 10.8% (w/w) lindane with 32.3% maneb.
19/ Combination formulation of 16.6% (w/w) lindane with 11.0% diazinon and 33.5% captan.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised trials of crops grown from seed treated with lindane for the following:

Cereal grain	Table 28	Wheat grain
	Table 29	Wheat forage, hay and straw
Oilseeds	Table 30	Canola.

Double underlined residue data are from treatments according to GAP and were used to estimate maximum residue levels.

Wheat

Wheat seed (6.8-34 kg) was treated with a water-diluted FS formulation, using commercially available laboratory-scale treatment equipment (Willard, 1999). Winter wheat varieties were treated in October 1997, spring wheat varieties in April 1998. Wheat was sown 6-30 days after treatment and was grown in 7 different States in the USA (10 x winter wheat; 5 x spring wheat), using field plots of 139 m^2. The seeding rates, combined with the seed treatment rate, resulted in field rates of 0.156-0.272 kg ai/ha. Duplicate samples were collected from each plot. Wheat forage samples were collected at or near the jointing stage. Hay samples were collected at early flower to soft dough stage. Grain and straw were collected at normal harvest maturity. Sample weights were 0.5-5 kg. Samples were stored at -20 ± 5 °C until analysis: 56-469 days for forage, 312-416 days for hay, 302-390 days for grain and 320-401 days for straw. Wheat forage samples were analyzed using the original version of method 109, whereas the other wheat matrices were analyzed using version #2 of method 109. Residue concentrations in forage, hay, grain and straw (Tables 28 and 29) are expressed on an as received basis (fresh weight). Results were not corrected for concurrent method recovery (65-122%), nor for the results obtained from control samples (<0.0017 in all matrices except wheat hay, with results up to 0.0037 mg/kg). Because of matrix interference, the reported LOQ for wheat hay should be increased to 0.01 mg/kg.

Table 28. Residues of lindane (parent) in wheat grain, grown from seed treated with lindane (Willard, 1999).

Location, year, variety	Application				DAT	Plant part	Lindane (parent), mg/kg
	Form	No.	g ai/t	kg ai/hl			
USA, LA1, 1997, winter wheat, Coker 9835	FS 305 1/	1	328.5	2.8	20 224	sowing grain	- <0.005 (2), mean ≤0.005

Location, year, variety	Application				DAT	Plant part	Lindane (parent), mg/kg
	Form	No.	g ai/t	kg ai/hl			
USA, TX1, 1997, winter wheat, Tam 202	FS 305 [1/]	1	328.5	2.8	26 252	sowing grain	- <0.005 (2), mean ≤0.005
USA, OK1, 1997, winter wheat, Karl 92	FS 305 [1/]	1	328.5	2.8	15 244	sowing grain	- <0.005 (2), mean ≤0.005
USA, OK2, 1997, winter wheat, Karl 92	FS 305 [1/]	1	328.5	2.8	17 246	sowing grain	- <0.005 (2), mean ≤0.005
USA, NC1, 1997, winter wheat, Coker 9803	FS 305 [1/]	1	328.5	2.8	22 251	sowing grain	- <0.005 (2), mean ≤0.005
USA, IL1, 1997, winter wheat, Pioneer Brand 2571	FS 305 [1/]	1	328.5	2.8	7 261	sowing grain	- <0.005 (2), mean ≤0.005
USA, KS1, 1997, winter wheat, Karl 92	FS 305 [1/]	1	328.5	2.8	16 258	sowing grain	- <0.005 (2), mean ≤0.005
USA, KS2, 1997, winter wheat, Karl 92	FS 305 [1/]	1	328.5	2.8	17 266	sowing grain	- <0.005 (2), mean ≤0.005
USA, NE1, 1997, winter wheat, Karl 92	FS 305 [1/]	1	328.5	2.8	6 275	sowing grain	- <0.005 (2), mean ≤0.005
USA, IA1, 1997, winter wheat, Pioneer Brand 2571	FS 305 [1/]	1	328.5	2.8	7 285	sowing grain	- <0.005 (2), mean ≤0.005
USA, NE2, 1998, spring wheat, Butte 86	FS 305 [1/]	1	328.5	2.8	12 110	sowing grain	- <0.005 (2), mean ≤0.005
USA, ND1, 1998, spring wheat, Russ	FS 305 [1/]	1	328.5	2.8	30 134	sowing grain	- <0.005 (2), mean ≤0.005
USA, ND2, 1998, spring wheat, Russ	FS 305 [1/]	1	328.5	2.8	29 128	sowing grain	- <0.005 (2), mean ≤0.005
USA, SD1, 1998, spring wheat, Butte 86	FS 305 [1/]	1	328.5	2.8	17 118	sowing grain	- <0.005 (2), mean ≤0.005
USA, ID1, 1998, spring wheat, Penewawa	FS 305 [1/]	1	328.5	2.8	27 140	sowing grain	- <0.005 (2), mean ≤0.005

- Seed at sowing was not analyzed for residues.
[1/] In combination with carboxin and thiram, which were also applied to the control wheat.

Table 29. Residues of lindane (parent) in wheat animal fodder, grown from seed treated with lindane (Willard, 1999).

Location, year, variety	Application				DAT	Plant part	Lindane (parent), mg/kg
	Form	No.	g ai/t	kg ai/hl			
USA, LA1, 1997, winter wheat, Coker 9835	FS 305 [1/]	1	328.5	2.8	125 180 224	forage hay straw	<0.005 (2), mean ≤0.005 ≤0.005 <0.005 (2), mean ≤0.005
USA, TX1, 1997, winter wheat, Tam 202	FS 305 [1/]	1	328.5	2.8	173 205 252	forage hay straw	<0.005, 0.0056, mean ≤0.005 0.0052, 0.0057, mean 0.0054 <0.005 (2), mean ≤0.005
USA, OK1, 1997, winter wheat, Karl 92	FS 305 [1/]	1	328.5	2.8	174 216 244	forage hay straw	<0.005 (2), mean ≤0.005 <0.005 (2), mean ≤0.005 <0.005 (2), mean ≤0.005
USA, OK2, 1997, winter wheat, Karl 92	FS 305 [1/]	1	328.5	2.8	171 215 246	forage hay straw	<0.005 (2), mean ≤0.005 0.0079, 0.0085, mean 0.0082 <0.005 (2), mean ≤0.005
USA, NC1, 1997, winter wheat, Coker 9803	FS 305 [1/]	1	328.5	2.8	181 231 251	forage hay straw	<0.005, 0.0053, mean ≤0.005 <0.005 (2), mean ≤0.005 <0.005 (2), mean ≤0.005
USA, IL1, 1997, winter wheat, Pioneer Brand 2571	FS 305 [1/]	1	328.5	2.8	182 226 261	forage hay straw	<0.005 (2), mean ≤0.005 0.0075, 0.0080, mean 0.0077 <0.005 (2), mean ≤0.005
USA, KS1, 1997, winter wheat, Karl 92	FS 305 [1/]	1	328.5	2.8	198 243 258	forage hay straw	0.032, 0.039, mean 0.036 <0.005, 0.0057, mean ≤0.005 <0.005 (2), mean ≤0.005
USA, KS2, 1997, winter wheat, Karl 92	FS 305 [1/]	1	328.5	2.8	200 243 266	forage hay straw	0.0086; 0.0087, mean 0.0087 <0.005 (2), mean ≤0.005 <0.005 (2), mean ≤0.005

Location, year, variety	Application				DAT	Plant part	Lindane (parent), mg/kg
	Form	No.	g ai/t	kg ai/hl			
USA, NE1, 1997, winter wheat, Karl 92	FS 305 [1/]	1	328.5	2.8	205 244 275	forage hay straw	0.012, 0.029, mean 0.021 0.021, 0.025, mean 0.023 <0.005 (2), mean ≤0.005
USA, IA1, 1997, winter wheat, Pioneer Brand 2571	FS 305 [1/]	1	328.5	2.8	208 238 285	forage hay straw	0.014 (2), mean 0.014 <0.005 (2), mean ≤0.005 <0.005 (2), mean ≤0.005
USA, NE2, 1998, spring wheat, Butte 86	FS 305 [1/]	1	328.5	2.8	55 80 110	forage hay straw	0.0096, 0.0097, mean 0.0097 0.0089, 0.010, mean 0.0094 <0.005 (2), mean ≤0.005
USA, ND1, 1998, spring wheat, Russ	FS 305 [1/]	1	328.5	2.8	70 110 134	forage hay straw	<0.005 (2), mean ≤0.005 <0.005 (2), mean ≤0.005 <0.005 (2), mean ≤0.005
USA, ND2, 1998, spring wheat, Russ	FS 305 [1/]	1	328.5	2.8	71 100 128	forage hay straw	<0.005 (2), mean ≤0.005 0.0051, 0.0079, mean 0.0065 <0.005 (2), mean ≤0.005
USA, SD1, 1998, spring wheat, Butte 86	FS 305 [1/]	1	328.5	2.8	47 84 118	forage hay straw	0.031, 0.033, mean 0.032 <0.005 (2), mean ≤0.005 <0.005 (2), mean ≤0.005
USA, ID1, 1998, spring wheat, Penewawa	FS 305 [1/]	1	328.5	2.8	80 107 140	forage hay straw	0.017, 0.018, mean 0.017 <0.005 (2), mean ≤0.005 <0.005 (2), mean ≤0.005

[1/] In combination with carboxin and thiram, which were also applied to the control wheat.

Canola

Canola seeds (0.24-10.8 kg) were treated in Alberta (Canada) with an FS 400 formulation, using commercially available laboratory-scale treatment equipment, in June 1998 (Willard, 2000). Canola seeds were sown 36-41 or 136 days after treatment and were grown in 5 different States in the USA, in plots of 139 m^2. The seeding rates, combined with the seed treatment rate, resulted in field rates of 0.156-0.272 kg ai/ha. Duplicate samples were collected from each plot. Canola seed was collected at normal harvest maturity. Sample weights were 1.0-1.6 kg, except at ID1 (0.5 kg), where frost stopped the development of immature canola seed. From the ND1 test site, additional canola seed was collected for processing (25 kg). Samples were stored at –20 ± 5°C until analysis, in 35-175 days. Samples were analyzed using method 109 rev #2. Residues concentrations were expressed on an as received basis (fresh weight). Results were not corrected for concurrent method recovery (71-106%), nor for the results obtained from control samples (<0.0017 mg/kg).

Table 30. Residues of lindane (parent) in canola, grown from seed treated with lindane (Willard, 2000).

Location, year, variety	Application				DAT	Plant part	Lindane (parent), mg/kg
	Form, g ai/kg	No.	g ai/t	kg ai/hl			
USA, ID1, 1998, Springfield	FS 399 [1/]	1	8620	na	38 172	sowing seed [2/]	- 0.0065; 0.0078; mean 0.0072
USA, WA1, 1998, Cavalier	FS 399 [1/]	1	8620	na	36 125	sowing seed	- <0.005; 0.016; mean 0.010
USA, MN1, 1998, Hyola 401	FS 399 [1/]	1	8620	na	41 155	sowing seed	- 0.0051; 0.012; mean 0.0086
USA, ND2, 1998, Hyola 401	FS 399 [1/]	1	8620	na	37 154	sowing seed	- <0.005 (2); mean < 0.005
USA, ND1, 1998, Hyola 401	FS 399 [1/]	1	8620	na	38 148	sowing seed	- <0.005 (3); mean <0.005
USA, NC1, 1998, unknown	FS 399 [1/]	1	8620	na	136 359	sowing seed	- <0.005 (2); mean <0.005

- Seeds at sowing were not analyzed for residues.

[1/] In combination with thiabendazole and thiram.

[2/] Only 0.5 kg of mature seed collected, because frost stopped the development of immature canola seeds.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No data were available.

In processing

Canola seed (0.24-10.8 kg) was treated with an FS 400 formulation, using commercially available laboratory-scale treatment equipment, in June 1998 (Willard, 2000). The canola seed was sown and grown in the USA (for details, see supervised residue trials on ND1 site, Table 30). Harvested samples were stored at –20 ± 5°C until processing. Canola seed was processed, on a small scale, into meal, refined oil and edible oil. Canola seed (20.8 kg) with incurred residues was dried to a moisture content of 7-10% at 55-71°C. Non-seed impurities were removed, resulting in 15.8 kg of dry clean seed. A sub-sample of the whole seed (6.80 kg) was flaked in a flaking roll, with a gap setting of 0.038-0.051 cm. The flakes were heated at 82-99°C for 10-15 min. Flakes were pressed in an expeller, to liberate most of the crude oil. Because only a small amount of oil was released from the seed, the oil was added back to the remaining press cake. The press cake was extracted in a batch extractor, with hexane at 43-52°C (3x 30 min; 2x 15 min). Finally, the press cake was toasted, resulting in 3.76 kg of meal. The extracted crude oil was heated to 75-90°C, to remove the hexane, resulting in 2.62 kg of crude oil. To a sub-sample of 1.65 kg crude oil, NaOH was added, to neutralize the fatty acids. The oil was mixed for 20 min at 40-45°C and 10 min at 65-70°C. The oil was then allowed to settle for 1 hr at 60-65°C. Afterwards, the oil was decanted and filtered, resulting in 0.352 kg soapstock and 1.38 kg refined oil. To a sub-sample of 0.908 kg refined oil, activated bleaching earth was added. The oil was bleached under vacuum for 10-15 min at 85-100°C and filtered, resulting in 0.851 kg bleached oil. A sub-sample of 0.830 kg bleached oil was vacuum steam-distilled at 235-250°C for 45-60 min. Finally, a citric acid solution was added, resulting in 0.827 kg edible oil. Processed samples were stored at –20 ± 5°C until analysis, 6-56 days later. Samples were analyzed using method 109, rev #2. Results were not corrected for concurrent method recovery (71-119%) nor for results obtained from the control samples (<0.0017 mg/kg). Results are presented in Table 31.

When fraction amounts were corrected for sub-sampling, mass fractions (mass of processed product relative to mass of RAC) were: 77.0% for dry clean seed, 40.8% for meal, 29.6% for crude oil, 24.7% for refined oil and 23.0% for edible oil. During processing, residues were concentrated in crude oil but, in edible oil, no lindane was found because it was degraded during the bleaching and deodorizing processes. Because no residues were found in the canola seed, processing and transference factors cannot be calculated. Additional processing studies, with higher levels of incurred residues, are needed to estimate a processing factor for oil.

Table 31. Residues of lindane (parent) in canola, grown from seed treated with lindane (Willard, 2000).

Location, year, variety	Form., g ai/kg	No.	g ai/t	DAT	Commodity	Lindane, mg/kg	P-factor	T-factor
USA, ND1, 1998, Hyola 401	FS 399 [1/]	1	8620	38	sowing	-	-	-
				148	seed	<0.005	-	-
					meal	<0.005	-	-
					refined oil	0.013	-	-
					edible oil	<0.005	-	-

P-factor = processing factor = residue in processed commodity / residue in RAC.
T-factor = transfer factor = P-factor x mass of processed commodity / mass of RAC.
[1/] In combination with thiabendazole and thiram.

Residues in the edible portion of food commodities

No data available.

RESIDUES IN ANIMAL COMMODITIES

Direct animal treatments

No data were available and are not applicable.

Farm animal feeding studies

Dairy cattle

A study of the transfer of lindane from feed items into milk and tissues from dairy cattle was conducted (Merricks, 1987b, GLP; Hemingway *et al*., 1999), in which 13 milking Holstein cows were assigned to 4 groups: 3 dosed groups of 4 and a single control cow. Body weights were 480-610 kg. Oral dosing levels were equivalent to 20, 60, and 200 ppm in the feed and dosing took place for 28 days, after each morning milking (by gelatine capsule, using a balling gun). Three of the four cows in each group received both oral and direct dermal treatments of lindane and one of the four cows in each group received only the oral dose. Because direct dermal application to livestock is no longer supported, the results discussed below are those obtained using cows that received only oral dosing.

Milk samples were taken on several days and the morning and evening milk was analyzed separately. Animals were sacrificed 20 hrs after the last dosing and tissue samples were taken of: liver; kidney; composite of leg and loin muscle; and a composite of omental and peripheral fat. Milk samples were kept frozen for 30-79 days at –15°C, and tissue samples for 120-142 days at -15°C, prior to analysis. Samples were analyzed using the modified AOAC method. Results were not corrected for concurrent method recovery (48-142% from milk, 87-116% from tissues), nor for residue levels detected in control samples (up to 0.0048 mg/kg in milk and up to 0.015 mg/kg in tissues). Results for lindane which exceeded 0.6 mg/kg were not considered to be valid, because of poor procedural recoveries (48-142%), but the other results were considered to be valid.

Table 32 shows the lindane levels in tissues and milk, at the three oral dose rates. Lindane residues in milk reached a plateau at day 7, in all dose groups. Lindane residues in the tissues were highest in fat and successively lower in muscle, kidney and liver.

Table 32. Lindane concentrations (in mg/kg) in milk and tissues from cows dosed orally with lindane (Merricks, 1987b; Hemingway *et al*., 1999).

Matrix	Study day	Cow 4, 20 ppm in feed	Cow 8, 60 ppm in feed	Cow 10, 200 ppm in feed
Whole milk [1/]	1	0.12	0.68	2.1
Whole milk [1/]	3	0.19	1.1	2.2
Whole milk [1/]	7	0.54	0.94	3.9
Whole milk [1/]	14	0.16	0.74	3.2
Whole milk [1/]	21	0.26	0.86	6.9
Whole milk [1/]	25	0.34	1.0	5.8
Whole milk [1/]	28	0.56	1.5	10
Whole milk	Mean for days 7-28	0.37	1.0	6.0
Liver	28	0.10	0.19	0.72
Kidney	28	0.34	1.1	4.9 [2/]
Muscle	28	0.97	1.8	8.8
Fat	28	12	20	158

[1/] Each result is the mean of results for the afternoon milk of the study day indicated and the morning milk of the following day.
[2/] Average of two determinations.

Sheep

A study of the transfer of lindane from feed items into sheep tissues was conducted (Billings, 1988b, GLP) in which 20 Hampshire cross-bred feeder lambs were assigned to 4 groups, 3 dosed and 1 control. Each dosed group consisted of 6 lambs, 3 of each sex. At the beginning of dosing, lambs were 18 weeks of age, with body weights of 36-49 kg. At the end of dosing, body weights had increased to 45-63 kg. Oral dosing levels for the 3 groups were equivalent to 17.5, 52.5 and 175 ppm in the feed and dosing took place for 28 days (by gelatine capsule, using a balling gun). Four of the 6 lambs in each group, 2 males and 2 females, received both oral and direct dermal treatments of lindane and 2 lambs in each group (1 male, 1 female) received only oral treatment. Because direct dermal application of livestock is no longer supported, the results discussed below are those obtained using lambs that received only the oral treatments.

Animals were sacrificed 10-12 hrs after the last dosing and the following tissue samples were taken: liver (whole but without gallbladder); kidney; muscle from various muscle groups; and a composite fat sample, taken from subcutaneous, perirenal and omental fat. Tissue samples were stored for 155-212 days at -20°C prior to analysis. Samples were analyzed using a modified AOAC method, SBL SOP 81.00-87. Results were not corrected for concurrent method recovery (72-127%), nor for results obtained from control samples (up to 0.048 mg/kg). Results below 0.2 mg/kg are not considered to be valid because of the matrix interferences observed in control samples. The 2 control animals contained lindane residues in the fat (0.18-0.19 mg/kg). The control animals were housed in the same pen as the animals which were dip-treated with lindane and the study author believed that the lindane residues in the control animals were caused by exposure to lindane in the air. Consequently, sheep fat was purchased at a local grocery for method validation.

Table 33 shows the lindane levels in each tissue at the three oral dose rates. Lindane residues were highest in fat and successively lower in muscle, kidney and liver.

Table 33 Residues [1/] of lindane (parent) in tissues from sheep orally dosed with lindane at three levels (Billings, 1988b).

Dose (ppm in feed)	Lamb no.	Sex	Liver lindane, mg/kg	Kidney lindane, mg/kg	Muscle lindane, mg/kg	Fat lindane, mg/kg
17.5	240	M	0.02	0.55	0.43	17
	329	F	0.02	0.93	1.0	21
		mean	0.02	0.74	0.73	19
52.5	220	M	0.03	2.3	1.9	43
	328	F	0.01	1.2	1.3	43
		mean	0.02	1.8	1.6	43
175	234	M	0.14	5.6	9.1	173
	339	F	0.11	4.0	6.3	223
		mean	0.12	4.8	7.7	198

[1/] Each value represents the mean of triplicate determinations.

Pigs (swine)

A study of the transfer of lindane from feed items into pig tissues was conducted (Billings, 1988a, GLP), in which 20 Yorkshire/Landrace cross-bred pigs were assigned to 4 groups, 3 dosed and 1 control. Each dosed group consisted of 6 pigs, 3 of each sex. Animals were 16-20 weeks old when dosing began and had bodyweights of 63-80 kg. Oral dosing levels for the 3 groups were equivalent to 7.0, 21 and 70 ppm in the feed and dosing took place for 28 days (by gelatine capsule, using a balling gun). Four of the 6 pigs in each group, 2 males and 2 females, received both oral and direct dermal treatment of lindane and 2 pigs in each group (1 male, 1 female) received only oral treatment. Because direct dermal application to livestock is no longer supported, the results discussed below are those obtained using pigs that received only oral treatment.

Animals were sacrificed 6-10 hrs after the last dose and the following tissue samples were taken: liver (whole but without gallbladder); kidney; muscle from various muscle groups; and a composite fat sample taken from subcutaneous and perirenal fat. Tissue samples were stored for 124-174 days at -20°C, prior to analysis. Samples were analyzed using a modified AOAC method, SBL SOP 81.01-88. Results were not corrected for concurrent method recovery (84-115%; n = 5-7 for each matrix), nor for the levels in detected control samples (<0.02 mg/kg). Four concurrent method recovery data (for fat, liver and muscle) were rejected for either very low (10%) or very high recoveries (1900%), at spike levels of 0.1-1 mg/kg.

Table 34 shows the lindane levels in each tissue at the 3 oral dose rates. Lindane residues were highest in fat and successively lower in muscle, kidney and liver.

Table 34. Residues [1/] of lindane in tissues of pigs, orally dosed with lindane at three levels (Billings, 1988a).

Dose (ppm in feed)	Pig no.	Sex	Liver lindane, mg/kg	Kidney lindane, mg/kg	Muscle lindane, mg/kg	Fat lindane, mg/kg
7	119	M	<0.02	0.048	0.059	1.7
	124	F	<0.02	0.050	0.11	1.7
		mean	<0.02	0.049	0.086	1.7
21	139	M	<0.02	0.28	0.33	6.3
	127	F	<0.02	0.14	0.15	5.0
		mean	<0.02	0.21	0.24	5.6
70	133	M	<0.02	0.24	0.71	16
	126	F	<0.02	0.54	0.85	17
		mean	<0.02	0.39	0.78	16

[1/] Each value represents the mean result of triplicate analyses.

Poultry

A study of the transfer of lindane from feed items into poultry tissues was conducted (Merricks, 1988, GLP; Curry *et al.*, 1997), in which 60 Leghorn laying hens were assigned to 12 treatment groups and 2 control groups. Each dosed group consisted of 4 hens and each control group consisted of 6 hens. The birds had bodyweights of 1.4-1.9 kg. Oral dosing levels were equivalent to 1.5, 4.5 and 15 ppm in the feed (by gelatine capsule). Dosing took place for 28 days or 60 days (by gelatine capsule) after the morning egg collection.

Eggs from the 4 hens in each group were removed from the shells, composited by dose group and homogenized by shaking. Only eggs collected on the days indicated in Tables 35, 36 and 37 were analyzed. Two groups of hens at each dose level were sacrificed after the 28th dose. The birds were sacrificed 20 hrs after the last dosing and the following tissue samples were taken: liver, kidney, breast muscle, thigh muscle and fat. Samples were composited by group (4 hens). Homogenized egg samples and tissues were stored at –15°C, prior to analysis. Although the exact storage time was not stated, it was estimated from the data to be at least 100-140 days. Samples were analyzed using a modified AOAC method. Results were not corrected for concurrent method recovery (62-127% in eggs and 75-111% in tissues), nor for the residue levels in control samples (up to 0.024 mg/kg in eggs and up to 0.013 mg/kg in tissues). Results below 0.08 mg/kg in eggs and below 0.05 mg/kg in tissues were not considered to be valid, because of matrix interferences in the control samples.

Tables 35, 36 and 37 show the levels of lindane residues in eggs obtained from the hens fed at low, medium and high doses, respectively. Residues in eggs reached a plateau by day 14, in all dose groups. No differences in residue levels were observed between hens dosed for 28 days and hens dosed for 60 days, at any dose level. Very little lindane was transferred into any poultry tissue, except fat, as shown in Table 38.

Table 35. Residues of lindane (mg/kg parent) in eggs from hens orally dosed with lindane at 1.5 ppm (Merricks, 1988; Curry *et al.*, 1997).

Day	Group 3, hens 13-16	Group 4, hens 17-20	Group 5, hens 21-24	Group 6, hens 25-28
Days dosed	28	28	60	60
0	<0.005	<0.005	<0.005	<0.005
1	<0.005	<0.005	<0.005	<0.005
3	0.018	0.021	0.027	0.025
7	0.097	0.092	0.12	0.13
14	0.23	0.20	0.20	0.24
21	0.20	0.14	0.17	0.22
25	0.19	0.16	0.18	0.22
28	0.22	0.16	0.20	0.24
35	[1/]	[1/]	0.20	0.25
42			0.20	0.25
49			0.20	0.31
56			0.24	0.30

Day	Group 3, hens 13-16	Group 4, hens 17-20	Group 5, hens 21-24	Group 6, hens 25-28
Days dosed	28	28	60	60
60			0.25	0.35
Mean of days 14-60	0.21	0.17	0.21	0.26
Mean all groups	0.21			

[1/] Hens were sacrificed after the 28th day of dosing.

Table 36. Residues of lindane (mg/kg parent) in eggs from hens orally dosed with lindane at 4.5 ppm (Merricks, 1988; Curry *et al*., 1997).

Day	Group 7, hens 29-32	Group 8, hens 33-36	Group 9, hens 37-40	Group 10, hens 41-44
Days dosed	28	28	60	60
0	<0.005	<0.005	<0.005	<0.005
1	<0.005	<0.005	<0.005	<0.005
3	0.046	0.054	0.051	0.033
7	0.21	0.22	0.30	0.30
14	0.62	0.52	0.66	0.64
21	0.58	0.63	0.63	0.57
25	0.63	0.72	0.78	0.56
28	0.58	0.54	0.65	0.59
35	[1/]	[1/]	0.71	0.68
42			0.32	0.66
49			0.44	0.47
56			0.48	0.54
60			0.53	0.57
Mean of days 14-60	0.60	0.60	0.58	0.59
Mean all groups	0.59			

[1/] Hens were sacrificed after the 28th day of dosing.

Table 37. Residues of lindane (mg/kg parent) in eggs from hens orally dosed with lindane at 15 ppm (Merricks, 1988; Curry *et al*., 1997).

Day	Group 11, hens 45-48	Group 12, hens 49-52	Group 13, hens 53-56	Group 14, hens 57-60
Days dosed	28	28	60	60
0	<0.005	<0.005	<0.005	<0.005
1	<0.005	<0.005	<0.005	<0.005
3	0.14	0.18	0.24	0.12
7	0.78	0.99	0.90	0.84
14	2.0	2.3	2.3	2.0
21	1.5	3.1	2.7	2.2
25	1.7	2.3	2.1	2.2
28	2.2	2.6	2.4	2.3
35	[1/]	[1/]	2.6	1.7
42			2.1	2.1
49			2.0	2.0
56			2.3	2.4
60			2.4	2.6
Mean of days 14-60	1.8	2.6	2.3	2.1
Mean of all groups	2.2			

[1/] Hens were sacrificed after the 28th day of dosing.

Table 38. Lindane residues in tissues from poultry fed lindane at 1.5-15 ppm (Merricks, 1988; Curry *et al*., 1997).

Dose level (ppm)	Group	Days dosed	Liver, lindane, mg/kg	Kidney, lindane, mg/kg	Thigh muscle, lindane, mg/kg	Breast muscle, lindane, mg/kg	Fat, lindane, mg/kg
1.5	3	28	0.14	0.19	0.19	0.03	2.5
	4	28	0.10	0.15	0.18	0.03	2.5
	5	60	0.10	0.15	0.15	0.04	2.4
	6	60	0.11	0.21	0.18	0.03	2.7
	Mean		0.11	0.18	0.18	0.03	2.5
4.5	7	28	0.46	0.38	0.35	0.07	7.0
	8	28	0.55	0.71	0.37	0.12	8.5

Dose level (ppm)	Group	Days dosed	Liver, lindane, mg/kg	Kidney, lindane, mg/kg	Thigh muscle, lindane, mg/kg	Breast muscle, lindane, mg/kg	Fat, lindane, mg/kg
4.5	9	60	0.33	0.41	0.60	0.08	9.7
	10	60	0.17	0.45	0.43	0.08	8.1
	Mean		0.38	0.49	0.44	0.09	8.3
15	11	28	0.83	2.5	1.2	0.40	27
	12	28	0.72	1.6	1.5	0.32	28
	13	60	0.86	2.2	1.6	0.33	29
	14	60	0.95	2.0	1.4	0.34	27
	Mean		0.84	2.0	1.4	0.35	28

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The Meeting received information from Australia (national government) and the USA (manufacturer).

Lindane was included in the Australian Total Diet Survey of 1996 (ATDS 18). Lindane was not detected and estimated dietary intakes of lindane were less than 0.01% of the ADI, see Table 39.

In the residue monitoring data from the Australian Residue Survey results of 2001-2002, lindane residues were not detected in any samples, see Table 40.

Table 39. Estimated daily dietary exposure to lindane, expressed as percentages of the ADI for sub-populations in Australia (ANZFA, 2001).

Study	ADI, mg/kg bw	% ADI					
		Adult males 25-34 years	Adult females 25-34 years	Boys 12 years	Girls 12 years	Toddlers 2 years	Infants 9 months
ATDS 18	0.002	7.0	6.5	9.4	7	15.5	11.0

Table 40. Residue monitoring data from the Australian Residue Survey results of 2001-2002 (AFFA, 2002).

Commodity	LOR (mg/kg)	EMRL (mg/kg)	No. samples	No. with residues	No. with residues exceeding EMRL
Apples	0.05	2	232	0	0
Barley	0.01	0.5	290	0	0
Bran	0.01	0.5	37	0	0
Canola	0.01	0.05	62	0	0
Cattle (fat)	0.1	2	616	0	0
Chickpea	0.01	not set	11	0	0
Deer (fat)	0.1	2	32	0	0
Eggs	0.1	0.1	21	0	0
Field peas	0.01	not set	50	0	0
Flour	0.01	0.5	36	0	0
Game pig (fat)	0.1	2	60	0	0
Goat (fat)	0.1	2	104	0	0
Honey	0.02	not set	196	0	0
Horse (fat)	0.1	2	20	0	0
Kangaroo (fat)	0.1	2	67	0	0
Lupine	0.01	not set	54	0	0
Oats	0.01	0.5	41	0	0
Onions	0.05	2	162	0	0
Ostrich (fat)	0.1	0.7	23	0	0
Pears	0.05	0.5	74	0	0
Pig (fat)	0.1	2	308	0	0
Poultry (fat)	0.1	0.7	52	0	0
Sheep (fat)	0.1	2	469	0	0
Sorghum	0.01	0.5	106	0	0
Wheat	0.01	0.5	832	0	0

LOR: limit of reporting.

In the residue monitoring data from the United States Department of Agriculture's Pesticide Data Program (PDP) in 2001, lindane residues were not detected in any samples, except in beef adipose and broccoli (Curry, 2003). In beef adipose 0.0033 mg/kg lindane was found twice and in broccoli 0.003 mg/kg lindane was found once. No confirmatory analysis was conducted but the level

of lindane was barely above the detection limit and well below the tolerance limit. The tolerance limit was, however, not stated.

Table 41. Residue monitoring data for lindane from the Pesticide Data Program, 2001, (USA) (Curry, 2003).

Commodity	Detection limit range (mg/kg)	Tolerance limit (mg/kg)	No. samples	No. with residues	No. with residues exceeding tolerance limit
Apples	0.003		736	0	0
Beef adipose	0.002		291	2	0
Bananas	0.002-0.003		702	0	0
Broccoli	0.001-0.002		720	1	0
Celery	0.002-0.004		376	0	0
Cherries	0.002-0.003		286	0	0
Carrots	0.002-0.003		739	0	0
Sweet corn, canned	0.002-0.006		181	0	0
Green beans	0.002-0.004		707	0	0
Grapes	0.002		705	0	0
Lettuce	0.002-0.006		554	0	0
Mushrooms	0.002-0.003		184	0	0
Nectarines	0.001-0.002		359	0	0
Oranges	0.001-0.002		745	0	0
Poultry adipose	0.002		155	0	0
Peaches	0.003		529	0	0
Pineapple	0.002		730	0	0
Potatoes	0.002-0.006		733	0	0
Sweet peas, canned	0.002		185	0	0
Rice	0.008		669	0	0
Tomato paste	0.003		369	0	0
Water, drinking	1×10^{-8}		154	0	0

NATIONAL MAXIMUM RESIDUE LIMITS

MRLs for Australia, The Netherlands and Poland were provided by the national governments, see Table 42. Canadian MRLs, EU MRLs and proposed U.S. tolerances were provided by the manufacturer.

Table 42. National maximum residue limits for food crops and animal commodities. Residue is defined as lindane (no metabolites).

Country	Codex code	Food commodity	MRL, mg/kg
Australia		Apples	E 2
		Cereal grains	E 0.5
		Cherries	E 0.5
		Cranberry	E 3
		Crustaceans	E 1
		Edible offal (mammalian)	E 2
		Eggs	E 0.1
		Fish	E 1
		Grapes	E 0.5
		Molluscs (including cephalopods)	E 1
		Oilseed [except peanut]	E 0.05
		Peaches	E 2
		Peanuts	E 0.05
		Plums (including prunes)	E 0.5
		Poultry, edible offal of	E 0.7
		Poultry meat (in the fat)	E 0.7
		Strawberries	E 3
		Sugar cane	E 0.002*
		Vegetables	E 2
		Primary feed commodities	E 0.1
Canada		Meat and meat by-products of cattle, goat, sheep, pig	2.0
		Fat of cattle, goat, sheep and pig	2.0

Country	Codex code	Food commodity	MRL, mg/kg
Canada		Meat and meat by-products of poultry	0.7
		Butter, cheese, milk and other dairy products	0.2
EU [1/]		Cereals (barley, buckwheat, maize, millet, oats, rye, sorghum, triticale, wheat, other cereals)	0.01
		Meat (bovine, bovine frozen, swine, sheep, goats, horses, asses, mules, ….)	0.02
		Edible commodities of bovines, swine, sheep, goats…	0.02
		Fat of pig and poultry	0.02
		Meat, offal and blood (sausage and similar and other meat offal or blood)	0.02
		Meat and edible offal (edible flours and means)	0.02
		Meat and edible offal (others)	0.02
		Meat and edible offal of poultry	0.7
		Dairy products (milk and cream, butter, other fats, oils, cheese and curd)	0.001
		Eggs (in shell, not in shell, yolks)	0.1
Netherlands		Meat of chicken	0.7
		Other meat	0.02
		Milk	0.001 *
		Eggs	0.1
		Tea	0.05*
		Hops	0.05*
		Other	0.01*
Poland		Fruits and vegetables	0.1
	GC 0080	Cereal grains	0.01
	MM 0095	Meat and meat products	1.0
	ML 0106	Milk and milk products (up to 4% fat)	0.008
	ML 0106	Milk and milk products (more than 4% fat)	0.2
	PE 0112	Eggs	0.1
	DT 1114	Tea	0.1
USA		Cereal grain (excluding rice and wild rice)	T 0.005
		Cereal straw (excluding rice and wild rice)	T 0.005
		Cereal hay (excluding rice and wild rice)	T 0.02
		Cereal forage (excluding rice and wild rice)	T 0.05
		Cereal stover (excluding rice and wild rice)	T 0.05
		Canola seed	T 0.01
		Fat of meat of poultry, cattle, goats, horses, sheep, swine	T 0.01
		Milk	T 0.001

* MRL set at or about the LOQ

E Extraneous residue limit. It refers to a pesticide residue arising from environmental sources (including former agricultural uses) other than the use of the pesticide directly or indirectly on the commodity.

T Proposed tolerances, based on results of supervised field trials on cereal grains and transfer of lindane from animal feed items to animal commodities. Establishment of final tolerances awaits GLP compliant metabolism data on treated cereal grain seed.

[1/] Lindane is not authorized for use in the EU, therefore all tolerances are set at the LOQ, except for meat and edible offal of poultry and eggs, because these values were harmonized with Codex CXLs.

APPRAISAL

Lindane, a broad-spectrum insecticide, was originally evaluated by the JMPR in 1966 (under the name gamma-BHC) and re-evaluated for residues several times up to 1989. Lindane was scheduled for residue review by the 2003 JMPR under the Periodic Review Programme by the 30th Session of the CCPR (ALINORM 99/24).

At the 31st Session of the CCPR several delegations preferred revocation of the MRLs for lindane as (1) the TMDI greatly exceeded the temporary ADI; (2) it had been banned in many countries; (3) it has limited uses and (4) its last evaluation was in 1989. It was decided that at its next Session the Committee would consider the revocation of the existing CXLs, "(except those accompanied by the letter F)", if not supported. The company confirmed support for cereals, sugar beet, maize and oil seeds (sunflower and canola).

The Meeting received information on identity, metabolism and environmental fate, residue analysis, use pattern, residues resulting from supervised trials on wheat and canola, fate of residues during processing, animal feeding studies, and residues in food in commerce or at consumption. In

addition, information on GAP and/or national MRLs was supplied by the governments of Australia, Germany, The Netherlands, Poland and the USA.

Animal metabolism

The Meeting received information on metabolism in orally-dosed lactating goats, laying pheasants and laying hens. All studies were with uniformly-labelled [^{14}C]lindane.

Studies on metabolism in mice, rats, rabbits and dogs evaluated by the WHO Core Assessment Group of the 2002 JMPR indicated that lindane undergoes extensive metabolism in animals. Stepwise dehydrogenation, dechlorination, and dehydrochlorination may be followed by conjugation with sulfate or glucuronide. Lindane itself was considered to be the toxicologically significant compound.

Lactating goats dosed orally with [^{14}C]lindane at doses of 13, 20 or 200 ppm in the feed excreted 34-46% of the administered ^{14}C in the urine, 5.1-13% in the faeces, and 1.1-2.4% in milk, and 4.0-4.4% was found in the tissues. The highest radioactive residues were found in body fat, followed by liver, kidney and muscle. Only 0.2-1.0% of the administered radioactivity was recovered as expired $^{14}CO_2$. Total recoveries for each goat were low (51-59%), probably owing to loss of volatile ^{14}C-metabolites untrapped by the solutions.

In fat and muscle, ^{14}C was mainly in the parent compound (73-85% and 28-81% of the TRR, corresponding to about 3 mg/kg and 0.02-0.16 mg/kg, respectively, at the lower doses). The metabolites identified in fat were 1,2,4-trichlorobenzene (3.6-11%), γ-pentachlorocyclohexene (5.8%), 1,2,4,5-tetrachlorobenzene (0.8-1.5%), 1,2,3,4,5,6-hexachlorocyclohexene (0.4-0.7%), and 1,2,3,4-tetrachlorobenzene and 1,2,3,5-tetrachlorobenzene (each <0.1%), and in muscle 1,2,4-trichlorobenzene and γ-pentachlorocyclohexene (5.8% and 3.5% of the TRR, respectively).

In liver and kidney the parent compound constituted only about 16% and 4.5-36% of the TRR, respectively, corresponding to about 0.36 mg/kg and 0.17 mg/kg at the 13 ppm feed level. In liver all individual components were ≤1.3% of the TRR (or 0.029 mg/kg eq). Identified, although not quantified, were 1,3,5-trichlorobenzene, 2,6-dichlorophenol and 2,3,4,5-tetrachlorophenol, and tentatively 2,3,4-trichlorophenol. In kidney, γ-tetrachlorocyclohexene and possibly 1,2-dichlorobenzene were identified at 4.5% and 5.8% of the TRR, respectively.

In milk residues reached a plateau after 2-3 days. Approximately 55-87% of the TRR in whole milk was in the fat. Lindane was the major constituent in milk fat (55-77% of the TRR of whole milk; 0.11 mg/kg at the 13 mg ai/kg feed level) with 1,2,4-trichlorobenzene forming up to 16% of the TRR and γ-pentachlorocyclohexene at about 5% of the TRR of whole milk. Skimmed milk contained 6-8 conjugated chlorophenols (5.9-17% of the TRR of whole milk).

In a study on laying hens, 44-63% of the total administered radioactivity (TAR) was recovered from the excreta, 2.6-9.0% from eggs and 20-47% from tissues and organs. Total recovery of the administered radioactive dose was above 90%. The highest concentration of residues was found in the fat (5.5-13% of the TAR), followed by skin (5.5-10% of the TAR), thigh muscle (1.8-3.0% of the TAR), liver (0.43-0.97% of the TAR), breast muscle (0.2-0.5% of the TAR) and kidney (0.09-0.26% of the TAR).

Lindane was the main residue component in all tissues analyzed: fat 85%, thigh muscle 71%, liver 51%, breast muscle 100%, egg yolk 94%, egg white 100% of the identified residues. In breast muscle and egg whites, no radioactive residue other than the parent was identified, whereas in fat and egg yolks the main metabolite was 2,3,4,5,6-pentachlorocyclohexene (6.1% and 4.2% of the identified residues respectively), and in liver 1,2,4-trichlorobenzene (19% of the identified residues) while dichlorobenzenes were present at 9.5% and 1,3,5-trichlorobenzene at 6.4%. In thigh muscle the main metabolite was 1,2,4,5- or 1,2,3,4-tetrachlorobenzene (18% of the identified residues). All other metabolites identified were ≤5% of the TRR. During the dosing period (4-6 days) no plateau was reached in eggs.

Laying hen pheasants were dosed for 15 days. Eggs were collected daily for about 70 days for analysis. Residues in the yolks were highly variable, increasing sharply and reaching a mean

maximum level in 8 days in a group dosed with capsules and gradually, in a group fed treated seed, reaching a mean maximum in 22 days. Thereafter levels decreased gradually to <0.5 mg/kg eq in both groups (in about 50 days in the former and about 70 days in the latter).

Metabolism of lindane in laboratory animals was qualitatively similar to that in farm animals.

Plant metabolism

The Meeting received information on the fate of lindane in plants grown from lindane-treated seeds, in spinach and cucumber plants after post-emergence spray applications and in apples after pre-harvest spray applications. The studies were conducted with uniformly-labelled [^{14}C]lindane.

Wheat seeds coated with lindane at an actual rate of 480 g ai/t were planted in the field and the resulting crops sampled 19 and 100 days after treatment. Significant amounts of residues were present in the seedlings and in the mature plants, indicating that lindane and/or its degradation products are readily translocated into growing plants. Extraction of the plant tissues with MeOH recovered more radioactive residues from the seedlings (91% of the TRR) than from any part of the mature plants (63% of the TRR from roots, 67% from straw, 34% from chaff), indicating that radioactive residues are more strongly bound in mature plant tissues.

No radioactive residue could be detected in grain. The parent was found at 36% of the TRR (0.2 mg/kg eq) in seedlings, 21% (0.48 mg/kg eq) in roots and 5.4% (0.006 mg/kg eq) in straw, and was undetected in chaff. In the seedlings, 26% of the TRR (0.14 mg/kg eq) was hydrophilic and 29% (0.16 mg/kg eq) hydrophobic. The proportion of the hydrophilic compounds increased in the mature plants: 27.4% of the TRR (0.63 mg/kg eq) in the roots, 53% (0.06 mg/kg eq) in the straw and 34% (0.007 mg/kg eq) in the chaff. Roots contained eight non-acidic compounds (chlorobenzenes), each ≤5.7% of the TRR (0.13 mg/kg eq), and four acidic compounds (chlorophenols).

Radishes, sugar beet, spinach, mustard, maize, sweet corn, and spring wheat seeds were coated with uniformly-labelled [^{14}C]lindane (actual dose rates 380, 2290, 820, 590, 1770, 1440 and 370 g ai/t, respectively) and planted outdoors under a clear protective roof. Sugar beet plants did not reach full maturity. Significant residues (>0.01 mg/kg eq) were found in all crop parts, except maize cobs and grains. The highest levels of radioactivity were found in spring wheat samples (foliage > grain > roots at 32 days). Mustard seeds were not analyzed.

Residues were extracted with acetonitrile (ACN) and analyzed for the presence of lindane. In the ACN extracts of fast-growing crops, 81% of the TRR (mustard foliage) and 54% of the TRR (radish roots) was identified as the parent. In the ACN extracts of slow-growing crops, lindane constituted 30% of the TRR (0.09 mg/kg) in sugar beet roots, 19% (0.04 mg/kg) in sugar beet foliage, 20% (0.16 mg/kg) in maize roots, 12% (0.008 mg/kg) in maize foliage, 24% (0.012 mg/kg) in sweet corn foliage, 0.5% (0.016 mg/kg) in spring wheat foliage and 3.8% (0.002 mg/kg) in spring wheat grain. It should be noted that virtually all of the radioactivity in spring wheat foliage (109%; 3.2 mg/kg) and grain (217%; 0.11 mg/kg eq) was unextracted. (The author's figures are quoted although clearly in error; no explanation was suggested.)

A single Red Delicious apple tree was treated once with 1 kg ai/ha uniformly-labelled [^{14}C]lindane, just before petal fall. Lindane and its metabolites were found in both leaves and fruit. The presence of lindane in the fruit indicates that it was distributed throughout the tree and transferred to the maturing fruit from the leaves and twigs. Total radioactive residues decreased during the maturation period and those in the apples were about one fifth of the levels in the foliage at each collection. The amount of unextracted residue increased with time from about zero initially to 30-40% of the TRR in the foliage and 25% of the TRR in the mature fruit.

At harvest at 131 days, the radioactive residue in foliage consisted of 3.2% of the TRR as lindane, minute quantities of chlorinated phenols (2.0% of the TRR), TLC-origin material (19% of the TRR), water-soluble material (40% of the TRR) and unextracted residues (32% of the TRR). Residues in the fruit consisted of 11% of the TRR as parent, 14% as pentachlorophenol, minute quantities of two other chlorinated phenols (0.6% of the TRR), TLC-origin material (12% of the TRR), water-soluble material (38% of the TRR) and unextracted residues (25% of the TRR). From

extracts of mature apples, the levels of unextracted residues and residues in the aqueous layer were too low (<0.02 mg/kg eq) to justify further investigation.

Fenumex cucumber plants were treated three times with EC foliar applications of [^{14}C]lindane at a rate of 0.71 kg ai/ha each. Autoradiography indicated that radioactivity spread very rapidly throughout the plant. Radioactivity detected in the stem and root immediately after treatment disappeared after 24 h and, after seven days, most had disappeared from the leaves.

Most of the radioactivity was in the leaves and extractable radioactivity decreased rapidly. Growth dilution appeared to be important in reducing the residue on a weight/weight basis. Residues in cucumber fruits ranged from 0.00009-0.0032 mg/kg eq and were therefore not further investigated. In the initial plant extracts that contained sufficient radioactivity, no radioactive components co-eluted with potential metabolites, including chlorinated benzenes, cyclohexanes, and phenols (it was not stated which were used as reference standards). Lindane was the only residue component identified. Hydrolysis of the post-extraction solids released additional radioactivity consisting of multiple low-level components.

Separate recovery studies were conducted on a cucumber plant grown in an aerated glass enclosure. Volatiles were collected in traps for CO_2 and volatile organic compounds. Radioactivity at 7 days was distributed among leaves (41% of the TAR), stems (3.9% of the TAR), roots (0.9% of the TAR), soil (14% of the TAR) and traps (17% of the TAR) or remained stuck to the tanks, tubes and pots (27% of the TAR).

Perpetual spinach plants at the two-leaf stage were treated with a single foliar application of uniformly-labelled [^{14}C]lindane at a rate of 1.5 kg ai/ha. Autoradiography showed that lindane was translocated rapidly throughout the plants. At 1 day no radioactivity was associated with the roots and at 7 days the greater part of the residue had disappeared from the leaves. Total radioactive residues (TRR) had decreased markedly by day 7 to below 1% of the TAR. By the time the plants matured (60-92 days) the TRR was at most 0.0004 mg/kg eq, too low to allow identification of metabolites. In acetone extracts of immature plants at 0, 1 and 3 days, lindane was the only radioactive component observed.

Lindane is intended for use as a seed treatment on oilseeds and cereal grains. After seed treatment, significant amounts of residues were present in the seedlings and in the mature plants, indicating that lindane and/or its degradation products are readily translocated. Extraction efficiency decreased when plants matured indicating that radioactive residues are more strongly bound in the mature plant tissues. This is also consistent with the fact that lindane was a major component of residues in fast-growing crops, but its contribution decreased in slow-growing crops. In one study on wheat plants grown from seeds treated with lindane according to GAP, no radioactivity could be detected in the grain at harvest. In a second study, only 0.052 mg/kg eq radioactive residue was found in the wheat grain, of which virtually all (217% according to the author's figures) was unextractable. Of the very small amount extracted, 3.8% of the TRR (corresponding to 0.002 mg/kg eq) was the parent compound. In wheat foliage at harvest, only about 5% of the radioactive residue was extractable, corresponding to 0.14 mg/kg equivalents. Of this, 10% (0.016 mg/kg) was identified as the parent compound.

All metabolites found in plants were also characterized in animal metabolism studies.

Environmental fate in soil

In a field rotational crop study, soil was treated with [^{14}C]lindane at a rate of 0.85 kg ai/ha as an EC formulation in a spray volume of 700 l/ha before planting. Lindane was incorporated to a depth of 5 or 10 cm. Soil cores (30 cm in depth) were collected before and immediately after treatment, at each crop planting and at harvest. Subsequently each core was divided into 0-15 and 15-30 cm sub-cores. Walmann's Green Leaf lettuce, Goldmine carrots, and BB882 barley were planted 30 days, 121 days and 365 days respectively after spraying.

With one exception, the 15-30 cm sub-cores contained TRRs of $\leq$0.01 mg/kg eq, so only the 0-15 cm sub-cores were extracted with acetone. Lindane was the only component found to be

extractable, at 28-88% of the TRR, suggesting that the rotational crops were exposed only to lindane and soil-bound (unextracted) residues. Lindane was found to be rather persistent in soil: 73% of the parent compound found 2 h after treatment was still present 240 days later. The TRR in crops did not appear to be strongly related to the TRR in soil. An approximately linear relationship between crop and soil TRR levels was observed only in mature barley grain and straw. In mature lettuce plants, radioactive residues decreased with increasing plant-back intervals, from 0.04 mg/kg eq at 30 days to 0.009 mg/kg eq at 365 days. Of the TRR, 43% was identified as lindane, 19% as 3 different chlorophenols and 35% was unextracted.

In mature carrot roots the amount of radioactive residue remained constant at the different plant-back intervals, at 0.4 mg/kg equivalents. Approximately 86% of the TRR was identified as lindane, 4.4% as 1,3,4,5,6-pentachlorocyclohexene, 3% was unidentified and 1.7% was unextracted.

In barley forage, 0.10-0.4 mg/kg eq radioactive residue was found at the different plant-back intervals, of which 16-26% of the TRR was lindane, about 9% was 3 different chlorophenols, 39-52% was unextracted and 17-18% was unidentified. In barley grain, radioactivity (0.05-0.09 mg/kg eq) was either unextracted (71-112% of the TRR) or uncharacterized (0-32% of the TRR). About half of the uncharacterized radioactivity was later attributed to 3 different chlorophenols. In barley straw, 68-78% of the TRR (0.1-0.9 mg/kg eq) was unextracted, 11-34% was uncharacterized, 0.36-2.4% was lindane and about 4% was present as 3 different chlorophenols.

At each sampling lindane degradation products, if present in soil, were too low to quantify so only lindane was available for uptake, and the degradation products in crops arose from metabolism of lindane within the plants. The metabolites identified in crops from the rotational study are among those identified in the seed-treatment metabolism study on wheat, confirming that degradation of lindane taken up by roots proceeds by hydroxylation and successive losses of chlorine.

Environmental fate in water-sediment systems

Solutions of 1 mg/l uniformly-labelled [^{14}C]lindane at pH 5, 7 and 9 at two ionic strengths were kept in the dark at 25 ± 1°C for 30 days. At pH 5 and 7, lindane was stable with half-lives of 115-173 and 282-309 days, respectively, with 5% transformation after 30 days. At pH 9, lindane was unstable with a half-life of 35-36 days. After 30 days, 43-44% transformation was found with 7% 2,3,4,5,6-pentachlorocyclohexene and 4% trichlorobenzenes (1,2,4- and 1,2,3-), and 32-33% unaccountable.

In water, as well as in an acetone-sensitized aqueous solution, [^{14}C]lindane was resistant to natural sunlight. After 28 days recovery was comparable to the dark control and no degradation products were observed. Lindane was also resistant to simulated sunlight. After 15 days irradiation equivalent to 44.5 days of natural sunlight at 40° N in summer, in water at pH 7 at 25°C, recovery was $\geq$92% (dark control $\geq$91% recovery). Again no degradation products were observed.

It was concluded that lindane is resistant to hydrolysis (except at high pH) and photolysis.

Methods of analysis

Analytical methods proposed as enforcement methods and those used in supervised residue trials, storage stability studies, processing studies and feeding studies were reported.

Two enforcement methods were reported. A Dutch multi-residue method for fruit and vegetables involved extraction by a method for non-fatty samples and GC with ion-trap detection, with an LOQ of 0.03 mg/kg. The AOAC multi-residue method for organochlorine and organophosphorus pesticide residues is suitable for non-fatty foods, dairy products and whole eggs. After extraction lindane was quantified by GC with ECD. The LOQ was not stated.

Method 109, a GC-MS method, was used in supervised trials, storage stability studies and processing studies on wheat and canola. The reported LOQ was 0.005 mg/kg in wheat and canola commodities.

Modified AOAC methods were used in feeding studies on poultry, cows, sheep and pigs and storage stability studies on animal commodities. Reported LOQs were 0.01 mg/kg for liver, kidney,

muscle and fat, 0.001 mg/kg for milk and 0.005 mg/kg for eggs, but the validated LOQs were liver and kidney 0.05 mg/kg, muscle 0.02 or 0.03 mg/kg, milk 0.005-0.2 mg/kg, and eggs 0.1 mg/kg.

Stability of residues in stored analytical samples

The Meeting received data on the stability of residues in wheat forage, hay, straw and grain, in canola seed, meal and refined oil, and in animal tissues, eggs and milk stored frozen.

Lindane residues in wheat and canola were stable at –20°C for the time tested (wheat forage 14 months, wheat hay, wheat straw and wheat grain 18 months, canola seed 6 months, canola meal 1.5 months, refined canola oil 1.8 months). Lindane was stable at –18°C for 9 months in animal tissues and for at least 12 months in milk and eggs.

Definition of the residue

In goat fat and muscle, the residue was mainly present as the parent compound (82-86% and 62-90% of the TRR respectively). In goat liver and kidney, the parent compound was present at 0.4-16% and 4.7-36% of the TRR respectively. Approximately 55-87% of the TRR in whole milk was associated with the milk fat where lindane was the major constituent (55%-77% of the TRR of whole milk). In laying hens, lindane was the major radioactive residue in all tissues tested: fat 87%, thigh muscle 77%, liver 39%, breast muscle >100%, egg yolk >100%, egg white 67% of the TRR. In breast muscle and egg white, no radioactive residue besides lindane was identified.

As the parent compound is the major residue in all animal commodities, and since the remaining residue is made up not of one single component but of a wide range of chlorocyclohexenes, chlorobenzenes and chlorophenols, the Meeting agreed that the parent is a suitable marker molecule for enforcement in animal commodities and is also the compound of interest for dietary risk assessment.

The log K_{ow} of lindane is 3.2–3.7. Taking into account results from farm animal feeding studies, the Meeting concluded that lindane should be classified as fat-soluble.

Lindane is intended for use as a seed treatment on oilseeds and cereal grains. As discussed in the section on plant metabolism, after pre-planting seed treatment virtually all the radioactive residue in wheat grain (>100% of the TRR) and wheat foliage (95% of the TRR) at harvest is unextractable. In view of the low levels of radioactive residue present at harvest (0.052 mg/kg eq in grain), the lack of an alternative marker molecule, the fact that lindane was considered to be the toxicologically significant compound, and the fact that the existing definition of the residue is lindane, the Meeting agreed that the definition of the residue for compliance with MRLs and for estimation of dietary intake should be:

lindane, for both plant and animal commodities.

The residue is fat-soluble.

Supervised residue trials

Trials data were reported on wheat and canola.

Root and tuber vegetables (group 016). There is a current CXL for carrots of 0.2 E mg/kg, which stems from 1977 and was based on a rotational crop study. The rotational crop study described above confirms that carrots can take up relatively large amounts of the parent compound. As neither information on GAP nor supervised trials on carrots were reported, the Meeting decided to recommend withdrawal of the existing CXL for carrots.

Neither GAP information nor supervised trials data on sugar beet were provided and therefore the Meeting decided to withdraw the current recommendations, for both sugar beet and sugar beet leaves or tops, of 0.01 mg/kg.

Cereal grains (group 020). Lindane is registered in Canada and the USA for use on barley, maize, sweet corn, oats, rye, sorghum and wheat, as DS, LS, FS and ES formulations with a single treatment of the seeds immediately before sowing. Fifteen residue trials on wheat were conducted in the USA (1997, 1998) using an application rate of 328 g ai/t. In the USA, there are 13 labels for use on wheat.

On 12 of these the application rate ranges from 163 to 391 g ai/t but, on one label, the application rate is 2085 g ai/t and therefore the trials cannot be considered to be at the maximum US GAP.

In Canada two labels for use on wheat are registered; the critical label specifies an application rate of 390 g ai/t, so all 15 US trials were according to maximum Canadian GAP. Residues in wheat grain were all <0.005 mg/kg.

From the rotational crop study, it was clear that the behaviour of lindane in barley is comparable to that in wheat. The metabolism study with various lindane-treated seeds showed that wheat grain and foliage contained more lindane than maize grain and foliage. Therefore the Meeting decided to extrapolate the results from the supervised trials on wheat to all registered pre-planting seed treatments of lindane to cereal grains, i.e. barley, maize, sweet corn, oats, rye and sorghum.

The Meeting estimated a maximum residue level of 0.01* mg/kg, in grain of wheat, barley, maize, sweet corn, oats, rye and sorghum, an STMR of 0.005 mg/kg and an HR of 0.005 mg/kg.

Oilseeds (group 023). Lindane is registered for use on mustard in Canada. Six residue trials on canola were conducted in the USA (1998) but no relevant GAP was available for evaluation of the data.

The Meeting decided to recommend withdrawal of the current CXL for rape seed of 0.05* mg/kg.

Straw, fodder and forage of cereal grains and grasses (group 051). The wheat trials that were evaluated for grain residues were also evaluated for residues in wheat forage, hay and straw. The results were again extrapolated to barley, maize, sweet corn, oats, rye and sorghum.

Residues in wheat forage were <0.005 (8), 0.0087, 0.0097, 0.014, 0.017, 0.021, 0.032 and 0.036 mg/kg.

The Meeting estimated a highest residue level of 0.036 mg/kg in wheat, barley, maize, sweet corn, oat, rye and sorghum forage, and an STMR of 0.005 mg/kg.

Because of matrix interferences in wheat hay up to 0.0037 mg/kg, the reported LOQ for wheat hay should be increased to 0.01 mg/kg. Taking this into account, residues in wheat hay were <0.01 (14) and 0.023 mg/kg.

The Meeting estimated a highest residue level of 0.023 mg/kg in wheat, barley, maize, sweet corn, oat, rye and sorghum forage, and an STMR of 0.01 mg/kg.

Residues in wheat straw were <0.005 (15) mg/kg.

The Meeting estimated a maximum residue level of 0.01* mg/kg in the straw and fodder (dry) of cereal grains, with an STMR and HR of 0.005 mg/kg.

Fate of residues during processing

The Meeting received information on the fate of residues during the processing of canola seed to oil.

Canola seed with incurred residues were processed on a small scale into meal, refined oil and edible oil. In the seed, meal and edible oil no residues were detected but, in the refined oil, a residue of 0.013 mg/kg was found. Because no residues were found in the canola seed, processing and transference factors could not be calculated.

Farm animal dietary burdens

The Meeting estimated the dietary burden of lindane residues in farm animals from the diets listed in Appendix IX of the FAO Manual (FAO, 2002). One feed commodity only from each Codex Commodity Group was used. Calculation from the MRLs or HR values provided the concentrations in feed suitable for estimating MRLs for animal commodities, while calculation from the STMRs for feed was suitable for estimating STMRs for animal commodities. In the case of processed commodities, the STMR-Ps were used for both calculations.

Table 43. Maximum farm animal dietary burden estimation.

Commodity	Group	Residue, mg/kg	Basis	% dry matter	Residue, on dry wt mg/kg	% of diet			Residue contribution, mg/kg		
						Beef cattle	Dairy cattle	Poultry	Beef cattle	Dairy cattle	Poultry
Wheat grain	GC	0.01	MRL	89	0.011	50		80	0.006		0.009
Wheat forage	AF	0.036	HR	25	0.144	25	60		0.036	0.086	
Wheat hay	AS	0.023	HR	88	0.026	25	40		0.006	0.01	
						Maximum dietary burden:			0.05	0.1	0.009

Table 44. Median farm animal dietary burden estimation.

Commodity	Group	Residue, mg/kg	Basis	% dry matter	Residue, on dry wt mg/kg	% of diet			Residue contribution, mg/kg		
						Beef cattle	Dairy cattle	Poultry	Beef cattle	Dairy cattle	Poultry
Wheat grain	GC	0.005	STMR	89	0.006	50	40	80	0.003	0.002	0.005
Wheat forage	AF	0.005	STMR	25	0.02	25	60		0.005	0.012	
Wheat hay	AS	0.01	STMR	88	0.011	25			0.003		
						Median dietary burden:			0.011	0.014	0.005

Farm animal feeding studies

Animal feeding studies were reported for dairy cattle, sheep, pigs and chickens. In all the studies, only the parent compound was determined.

Three milking Holstein cows were dosed orally with lindane at levels equivalent to 20, 60 or 200 ppm in the feed. Dosing took place for 28 days after the morning milking (by gelatin capsule using a balling gun). Milk samples were taken on days 1, 3, 7, 14, 21, 25 and 28. Animals were slaughtered 20 h after the last dosing. Residues in fat were 12, 20 and 158 mg/kg for the 20, 60 and 200 ppm cows, respectively. Residues in muscle were 0.97, 1.8 and 8.8 mg/kg, in liver 0.10, 0.19, and 0.72 mg/kg and in kidney 0.34, 1.1 and 4.9 mg/kg. Residues in milk were 0.37, 1.0 and 6.0 mg/kg (mean for day 7-28) but results above 0.6 mg/kg lindane in milk were considered invalid because of bad procedural recoveries (48%-142%).

Hampshire cross-bred feeder lambs (one male and one female per group) were dosed orally with lindane at levels equivalent to 17.5, 52.5 and 175 ppm in the feed for 28 days as before and slaughtered 10-12 h after the last dosing. No difference between residues in male and female lambs was observed, so results are given as the mean for each group. Results below 0.2 mg/kg were considered invalid because of matrix interferences in control samples. Residues in fat were 19, 43 and 198 mg/kg in lambs fed at 17.5, 52.5 and 175 ppm, respectively. Residues in muscle were 0.73, 1.6 and 7.7 mg/kg, in liver 0.02, 0.02 and 0.12 mg/kg and in kidney 0.74, 1.8 and 4.8 mg/kg.

Yorkshire/Landrace cross-bred pigs (one male and one female per group) were dosed orally with lindane at levels equivalent to 7.0, 21 and 70 ppm in the feed for 28 days and slaughtered 6-10 hrs after the last dosing. No difference between residues in males and females was observed, so results are given as the mean for each group. Residues in fat were 1.7, 5.6 and 16 mg/kg, in muscle 0.086, 0.24 and 0.78 mg/kg, in liver <0.02, <0.02, and <0.02 mg/kg and in kidney 0.049, 0.21 and 0.39 mg/kg, respectively for the 3 feeding levels.

Leghorn laying hens (four birds per group) were dosed orally by gelatin capsule with lindane at levels equivalent to 1.5, 4.5 and 15 ppm in the feed, for 28 or 60 days after the morning egg collection (two groups of 4 hens at each level for each period). Eggs from days 0, 1, 3, 7, 14, 21, 25, 28, 35, 42, 49, 56 and 60 were analyzed. Birds were killed 20 h after the last dosing. Samples were composited by group. Residues were similar in the four groups at each dose level. Mean residues in fat were 2.5, 8.3 and 28 mg/kg for the hens fed at 1.5, 4.5 and 15 ppm, respectively. Corresponding mean residues in thigh muscle were 0.18, 0.44 and 1.4 mg/kg, in breast muscle 0.03, 0.09 and 0.35 mg/kg, in liver 0.11, 0.38 and 0.84 mg/kg, and in kidney 0.18, 0.49 and 2.0 mg/kg. The highest individual residues in birds fed at 1.5, 4.5 and 15 ppm were 2.7 mg/kg, 9.7 mg/kg and 29 mg/kg in fat, 0.19 mg/kg, 0.60 mg/kg and 1.6 mg/kg in thigh muscle, 0.04 mg/kg, 0.12 mg/kg and 0.40 mg/kg in breast muscle, 0.21 mg/kg, 0.71 mg/kg and 2.5 mg/kg in kidney, and 0.14 mg/kg, 0.55 mg/kg and

0.95 mg/kg in liver. The mean residues in eggs were 0.21, 0.59 and 2.2 mg/kg, and the highest residues in individual group composites were 0.35, 0.68 and 2.6 mg/kg, at the three dose levels.

Residues in animal commodities

The estimated maximum dietary burdens for beef and dairy cattle were 0.05 and 0.1 mg/kg feed, respectively, so the dairy cattle burden represents the worst case. In the feeding study with dairy cows, the lowest dosing level was 20 mg ai/kg feed. The resulting residues in tissues and milk were calculated by applying the transfer factors at this level to the dietary burdens (transfer factor = residue level in sample ÷ feeding level). The dietary burden for poultry was 0.009 ppm, which was lower than the lowest feeding level in the feeding study (1.5 ppm), so the resulting residues in eggs and poultry tissues were calculated by applying the appropriate transfer factors at this feeding level. Residues in pork commodities were similarly calculated from the lowest level fed in the pig feeding study (7 ppm).

The highest individual residues in tissues and eggs from the lowest levels administered in the feeding studies were used in conjunction with the maximum dietary burdens to calculate the highest likely residue levels and, in conjunction with the STMR dietary burdens to estimate the STMRs, in commodities derived from cattle, poultry and pigs.

Table 45. Calculation of MRLs and STMRs for animal commodities.

Feeding level (ppm) *(Extrapolated)* Actual [2]		Lindane residues, mg/kg [1]											
		Milk	Muscle		Fat		Liver		Kidney		Eggs	
		Mean [3]	High [4]	Mean [3]	High [4]	Mean [3]	High [4]	Mean [3]	High [4]	Mean [3]	High [4]	Mean [3]
MRL, dairy	*0.1* 20	*(0.002)* 0.37	*(0.005)* 0.97		*(0.06)* 12		*(0.0005)* 0.1		*(0.002)* 0.34			
MRL, poultry	*0.009* 1.5		*(0.001)* 0.19		*(0.016)* 2.7		*(0.0008)* 0.14		*(0.001)* 0.21		*(0.002)* 0.35	
STMR, dairy	*0.014* 20	*(0.0003)* 0.37	*(0.0007)* 0.97		*(0.008)* 12		*(0.00007)* 0.1		*(0.0002)* 0.34			
STMR, poultry	*0.005* 1.5			*(0.0006)* 0.18		*(0.008)* 2.5		*(0.0004)* 0.11		*(0.0006)* 0.18		*(0.0007)* 0.21

[1] Values in italics are the estimated dietary burdens. Values in normal font are the lowest feeding levels in feeding studies.

[2] Residue values in parentheses in italics are extrapolated to the dietary burdens from the lowest feeding levels used in the feeding studies and the residues found in those studies.

[3] Mean is estimated mean animal tissue (or milk) residue in the relevant feeding group.

[4] High is the estimated highest individual animal tissue residue in the relevant feeding group.

The Meeting estimated: maximum residue levels of 0.1 mg/kg (fat) for meat from mammals other than marine mammals, 0.01* mg/kg for edible offal and 0.01* mg/kg for milks; and STMRs of 0.0007 mg/kg and 0.008 mg/kg in muscle and fat from mammals other than marine mammals, respectively, 0.0002 mg/kg for edible offal and 0.0003 for milks; and HRs of 0.005 mg/kg and 0.06 mg/kg in muscle and fat from mammals other than marine mammals, respectively, and 0.002 mg/kg for edible offal.

CXLs exist for eggs (0.1 mg/kg E) and poultry meat (0.7 mg/kg (fat) E). These recommendations stem from 1968/1969 and 1973, when maximum residue levels for animal commodities were defined as EMRLs. Currently, this designation is reserved for pesticide residues arising from environmental sources other than the use directly or indirectly on the commodity.

The Meeting recommended MRLs of 0.05 mg/kg for lindane in poultry meat (fat), 0.01* mg/kg in edible offal of poultry and 0.01* mg/kg in eggs, to replace the current CXLs of 0.1 mg/kg E for eggs and 0.7 mg/kg (fat) E for poultry meat, and estimated STMRs of 0.0006 mg/kg and 0.008 mg/kg for poultry muscle and fat, 0.0004 mg/kg in edible offal of poultry and 0.0007 mg/kg in eggs, and HRs of 0.001 mg/kg and 0.016 mg/kg in poultry muscle and fat, 0.001 mg/kg in edible offal of poultry and 0.002 mg/kg in eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed in Table 46 are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: *lindane.*

The definition applies to plant and animal commodities. The residue is fat-soluble.

Table 46. Summary of recommendations.

Commodity		Recommended MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New	Current		
GC 0640	Barley	0.01 (*)		0.005	0.005
VR 0577	Carrots	W	0.2 E		
MO 0105	Edible offal (mammalian)	0.01*		0.0002	0.002
PE 0112	Eggs	0.01*	0.1 E	0.0007	0.002
GC 0645	Maize	0.01 (*)		0.005	0.005
MM 0095	Meat (from mammals other than marine mammals)	0.1 (fat)		muscle 0.0007, fat 0.008	muscle 0.005, fat 0.06
ML 0106	Milks	0.01*		0.0003	
GC 0647	Oats	0.01 (*)		0.005	0.005
PO 0111	Poultry, edible offal of	0.01*		0.0004	0.001
PM 0110	Poultry meat	0.05 (fat)	0.7 (fat) E	muscle 0.0006, fat 0.008	muscle 0.001, fat 0.016
SO 0495	Rape seed	W	0.05 (*)		
GC 0650	Rye	0.01 (*)		0.005	0.005
GC 0651	Sorghum	0.01 (*)		0.005	0.005
AS 0081	Straw and fodder (dry) of cereal grains	0.01 (*)		0.005	0.005
VR 0596	Sugar beet	W	0.1		
AV 0596	Sugar beet leaves or tops	W	0.1		
VO 1275	Sweet corn (kernels)	0.01 (*)		0.005	0.005
GC 0654	Wheat	0.01 (*)		0.005	0.005
	Wheat, barley, maize, oat, rye, sweet corn, sorghum forage			0.005	0.036
	Wheat, barley, maize, oat, rye, sweet corn, sorghum hay			0.01	0.023

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes of lindane, based on the STMRs estimated for 13 commodities in the five GEMS/Food regional diets, were in the range of 0 to 1% of the maximum ADI of 0.005 mg/kg bw (Table 47). The Meeting concluded that the long-term intake of residues of lindane resulting from its uses that have been considered by JMPR is unlikely to present a public health concern.

Table 47. International Estimated Dietary Intakes (IEDIs) of lindane for the five GEMS/Food regional diets (ADI = 0-0.005 mg/kg bw/day).

Code	Commodity	STMR or STMR-P mg/kg	Diets: g/person/day. Intake = daily intake: µg/person									
			Mid-East		Far-East		African		Latin American		European	
			diet	intake	diet	intake	diet	intake	diet	intake	diet	intake
GC 0640	Barley (fresh)	0.0	1.0	0.0	3.5	0.0	1.8	0.0	6.5	0.0	19.8	0.1
MO 0105	Edible offal (mammalian)	0.0	4.2	0.0	1.4	0.0	2.8	0.0	6.1	0.0	12.4	0.0
PE 0112	Eggs	0.0	14.6	0.0	13.1	0.0	3.7	0.0	11.9	0.0	37.6	0.0
GC 0645	Maize (fresh)	0.1	16.5	0.1	0.0	0.0	0.0	0.0	1.5	0.0	0.0	0.0

Code	Commodity	STMR or STMR-P mg/kg	Diets: g/person/day. Intake = daily intake: µg/person									
			Mid-East		Far-East		African		Latin American		European	
			diet	intake	diet	intake	diet	intake	diet	intake	diet	intake
MM 0095	Meat from mammals other than marine mammals: 20% as fat	0.1	7.4	0.1	6.6	0.1	4.8	0.0	9.4	0.1	31.1	0.2
MM 0095	Meat from mammals other than marine mammals: 80% as muscle	0.0	29.6	0.0	26.2	0.0	19.0	0.0	37.6	0.0	124.4	0.1
ML 0106	Milks	0.0	116.9	0.0	32.1	0.0	41.8	0.0	160.1	0.0	289.3	0.1
GC 0647	Oats	0.0	0.0	0.0	0.0	0.0	0.2	0.0	0.8	0.0	2.0	0.0
PM 0110	Poultry meat: 10% as fat	0.0	3.1	0.0	1.3	0.0	0.6	0.0	2.5	0.0	5.3	0.0
PM 0110	Poultry meat: 90% as muscle	0.0	27.9	0.0	11.9	0.0	5.0	0.0	22.8	0.0	47.7	0.0
PO 0111	Poultry, edible offal of	0.0	0.1	0.0	0.1	0.0	0.1	0.0	0.4	0.0	0.4	0.0
GC 0650	Rye	0.0	0.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	1.5	0.0
GC 0651	Sorghum	0.0	2.0	0.0	9.7	0.0	26.6	0.1	0.0	0.0	0.0	0.0
VO 1275	Sweet corn (kernels)	0.0	0.0	0.0	0.0	0.0	3.3	0.0	0.0	0.0	6.2	0.0
GC 0654	Wheat	1.6	327.3	1.6	114.8	0.6	28.3	0.1	116.8	0.6	178.0	0.9
Total intake (µg/person)=				1.9		0.8		0.4		0.8		1.6
Bodyweight per region (kg bw) =				60		55		60		60		60
ADI (µg/person)=				300		275		300		300		300
% ADI=				0.6		0.3		0.1		0.3		0.5
Rounded % ADI=				1		0		0		0		1

Short-term intake

The International Estimated Short Term Intake (IESTI) for lindane was calculated for 13 food commodities for which maximum residue levels were estimated and for which consumption data were available. The results are shown in Tables 48 and 49.

The IESTI represented 0% of the acute RfD for the general population and 0% of the acute RfD for children. The Meeting concluded that the short-term intake of residues of lindane resulting from its uses that have been considered by JMPR is unlikely to present a public health concern.

Table 48. Assessment of risk to the general population from the short-term dietary intake of residues of lindane (acute RfD = 0.06 mg/kg bw, i.e. 60 µg/kg bw/day).

Codex Code	Commodity	STMR or STMR-P mg/kg	HR or HR-P, mg/kg	Large portion diet			Unit weight			Variab-ility factor	Case	IESTI µg/kg bw/day	% acute RfD rounded
				Coun-try	Body wt (kg)	Large portion, g/person	Unit wt, g	Coun-try	Unit wt, edible portion, g				
GC 0640	Barley (fresh)	0.005	-	-	-	-	-	-	-	-	3	-	-
PE 0840 [1/]	Chicken eggs	-	0.002	FRA	62.3	219	-	-	-	-	1	0.01	0
MO 0105	Edible offal (mammalian)	-	0.002	FRA	62.3	277	-	-	-	-	1	0.01	0
GC 0645	Maize (fresh)	0.005	-	-	-	-	-	-	-	-	3	-	-
MM 0095	Meat from mammals other than marine mammals: 20% as fat	-	0.06	AUS	67.0	104	-	-	-	-	1	0.09	0

Codex Code	Commodity	STMR or STMR-P mg/kg	HR or HR-P, mg/kg	Large portion diet			Unit weight			Variab-ility factor	Case	IESTI µg/kg bw/day	% acute RfD rounded
				Coun-try	Body wt (kg)	Large portion, g/person	Unit wt, g	Coun-try	Unit wt, edible portion, g				
MM 0095	Meat from mammals other than marine mammals: 80% as muscle	-	0.005	AUS	67.0	417	-	-	-	-	1	0.03	0
ML 0106	Milks	0.0003	-	USA	65.0	2466	-	-	-	-	3	0.01	0
GC 0647	Oats	0.005	-	FRA	62.3	305	-	-	-	-	3	0.02	0
PM 0110	Poultry meat: 10% as fat	-	0.016	AUS	67.0	43	-	-	-	-	1	0.01	0
PM 0110	Poultry meat: 90% as muscle	-	0.001	AUS	67.0	388	-	-	-	-	1	0.01	0
PO 0111	Poultry, edible offal of	-	0.001	USA	65.0	248	-	-	-	-	1	0.00	0
GC 0650	Rye	0.005	-	NLD	63.0	77	-	-	-	-	3	0.01	0
GC 0651	Sorghum	0.005	-	USA	65.0	18	-	-	-	-	3	0.00	0
VO 1275	Sweet corn (kernels)	0.005	-	-	-	-	-	-	-	-	3	-	-
GC 0654	Wheat	0.005	-	USA	65.0	383	-	-	-	-	3	0.03	0

[1/] Because of lack of information on large portion size of PE 0112, eggs, the calculation was made for PE 0840 chicken eggs.

Table 49. Assessment of risk to children up to 6 years from the short-term dietary intake of residues of lindane (acute RfD = 0.06 mg/kg bw, i.e. 60 µg/kg bw/day).

Codex Code	Commodity	STMR or STMR-P mg/kg	HR or HR-P, mg/kg	Large portion diet			Unit weight			Variab-ility factor	Case	IESTI µg/kg bw/day	% acute RfD rounded
				Coun-try	Body wt (kg)	Large portion, g/person	Unit wt, g	Coun-try	Unit wt, edible portion, g				
GC 0640	Barley (fresh)	0.005	-	-	-	-	-	-	-	-	3	-	-
PE 0840 [1/]	Chicken eggs	-	0.002	FRA	17.8	134	-	-	-	-	1	0.02	0
MO 0105	Edible offal (mammalian)	-	0.002	FRA	17.8	203	-	-	-	-	1	0.02	0
GC 0645	Maize (fresh)	0.005	-	-	-	-	-	-	-	-	3	-	-
MM 0095	Meat from mammals other than marine mammals: 20% as fat	-	0.06	AUS	19.0	52	-	-	-	-	1	0.16	0
MM 0095	Meat from mammals other than marine mammals: 80% as muscle	-	0.005	AUS	19.0	208	-	-	-	-	1	0.05	0
ML 0106	Milks	0.0003	-	USA	15.0	1286	-	-	-	-	3	0.03	0

Codex Code	Commodity	STMR or STMR-P mg/kg	HR or HR-P, mg/kg	Large portion diet			Unit weight			Variability factor	Case	IESTI µg/kg bw/day	% acute RfD rounded
				Country	Body wt (kg)	Large portion, g/person	Unit wt, g	Country	Unit wt, edible portion, g				
GC 0647	Oats	0.005	-	USA	15.0	62	-	-	-	-	3	0.02	0
PM 0110	Poultry meat: 10% as fat	-	0.016	AUS	19.0	22	-	-	-	-	1	0.02	0
PM 0110	Poultry meat: 90% as muscle	-	0.001	AUS	19.0	201	-	-	-	-	1	0.01	0
PO 0111	Poultry, edible offal of	-	0.001	USA	15.0	37	-	-	-	-	1	0.00	0
GC 0650	Rye	0.005	-	NLD	17.0	37	-	-	-	-	3	0.01	0
GC 0651	Sorghum	0.005	-	-	-	-	-	-	-	-	3	-	-
VO 1275	Sweet corn (kernels)	0.005	-	-	-	-	-	-	-	-	3	-	-
GC 0654	Wheat	0.005	-	USA	15.0	151	-	-	-	-	3	0.05	0

[1/] Because of lack of information on large portion size of PE 0112, eggs, the calculation was made for PE 0840 chicken eggs.

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