DICLORAN (83)

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EXPLANATION

Dicloran, a fungicide, was first evaluated by the JMPR, for toxicology and residues, in 1974 and subsequently in 1977. The compound was again evaluated by the Meeting in 1998, for toxicology and residues, under the CCPR periodic Review Programme. The 1998 JMPR changed the ADI from 0-0.03 to 0-0.01 mg/kg body weight and concluded that an acute RfD was unnecessary. It recommended that the definition of the residue, for compliance with MRLs and for estimation of dietary intake, should be dicloran and indicated that the residue was fat-soluble. It also revised maximum residue levels for carrots and onions, bulb, while recommending withdrawal of the existing CXLs for grapes; lettuce, head; peaches; plums (including prunes); strawberries and tomatoes. The 33rd CCPR, in 2000, decided to retain for four years the MRLs which had been recommended for withdrawal in accordance with the Periodic Review Programme, pending the submission of additional data on these commodities.

The Meeting received data on rotational crops, an analytical method, and residue trials on plums, nectarines, peaches, grapes, strawberries, tomatoes and lettuce, and information on use patterns.

ENVIRONMENTAL FATE

Rotational crops

In order to estimate the uptake by various plants of dicloran present in soil, a study was conducted in Germany in 1998 (Fortsch, 1989). [Phenyl-U-^{14}C]dicloran was applied to loamy sand at an application rate of 2.59 mg a.i./kg dry soil, which was equivalent to a field application rate of 3 kg a.i./ha. The soil was stored outdoors, protected from rainfall and direct sunlight, and irrigated and turned over periodically. Samples were analyzed at intervals (Table 1). Lettuce, wheat and sugar beet were sown in the treated soil 33, 120 and 365 days after the treatment, at the start of 3 growing periods. Each crop was harvested at a mid-ripe stage (1st harvest) and maturity (2nd harvest) and the leaves and roots were analyzed for radioactive residues (Table 2).

Table 1. Extractable, unextractable and total radioactive residues (TRR) in extracts from soil treated with dicloran and then stored for up to 1 year (Fortsch, 1989).

Length of storage, days	Recovered TRR 1/, %	Unextracted radioactivity, % of TRR	Dicloran 2/		
			% of TRR	mg/kg in soil	% of the applied 3/
0	103.1	0.5	99	2.64	102
33	94.5	2.3	93	2.28	88
54	95.4	4.6	92	2.26	87
91	89.2	9.1	84	1.93	75
120	91.5	9.7	75	1.78	69
251	55.2	31.8	38	0.54	21
365	47.9	35.5	27	0.33	13

Results are the averages from analysis of two samples.
1/ Total of radioactivity in acetone, water, methanol Soxhlet and NaOH extracts, and in the unextractable fraction.
2/ Determined by the analysis of acetone, water and Soxhlet extracts by TLC.
3/ Applied radioactive dicloran, 2.59 mg/kg-soil.

During the storage period of 365 days, the total radioactivity recovered from the soil decreased to about a half of the applied radioactivity. Analysis of the acetone, water and Soxhlet extracts by TLC showed that unchanged dicloran decreased during the storage, to 13% after one year. Its half-life, calculated from the results, was about 140 days. On the other hand, unextracted radioactivity increased over time of storage, to 36% of the TRR after 365 days.

Plant materials were extracted with acetone, methanol/water, diluted hydrochloric acid, diluted sodium hydroxide solution, concentrated hydrochloric acid and concentrated sodium hydroxide solution. All extracts were analyzed by TLC. Plant material remaining after the extraction processes mentioned above was air-dried or lyophilized and aliquots were either combusted for measurement of radioactivity by LSC or analyzed by TLC.

Table 2. Uptake of radioactivity by lettuce, wheat and sugar beet, following pre-treatment of the soil with dicloran (Fortsch, 1989).

Growing period	Harvest	Total radioactive residue, expressed as dicloran						% of the radioactivity applied to the soil as dicloran		
		mg/kg			mg/plant					
		Leaves	Roots	Soil	Leaves	Roots	Soil 1/	Leaves	Roots	Soil
Lettuce										
1st	1st	2.33	6.26	2.31	0.45	0.08	13.9	2.9	0.5	89.4
	2nd	1.89	10.1	2.37	0.64	0.61	14.2	4.1	3.9	91.5
2nd	1st	0.16	1.11	1.18	0.02	0.01	7.08	0.1	<0.1	45.6
	2nd	0.07	0.44	1.23	0.03	<0.01	7.38	0.2	<0.1	47.5
3rd	1st	0.03	0.20	0.86	<0.01	<0.01	5.16	<0.1	<0.1	33.2
	2nd	0.04	0.36	0.76	0.01	<0.01	4.56	<0.1	0.1	29.3
Sugar beet										
1st	1st	3.88	0.55	1.68	1.45	0.40	25.2	3.7	1.0	64.9
	2nd	3.90	0.48	1.22	2.11	0.37	18.3	5.4	1.0	47.1
2nd	1st	0.12	0.11	0.79	0.08	0.05	11.9	0.2	0.1	30.5
	2nd	0.22	0.15	0.98	0.09	0.08	14.7	0.2	0.2	37.8
3rd	1st	0.33	0.09	0.77	0.03	<0.01	11.6	<0.1	<0.1	29.9
	2nd	0.16	0.09	0.88	0.05	0.01	13.2	0.1	<0.1	34.0
Wheat										
		Grain	Straw Roots	Soil	Grain	Straw Roots	Soil 2/	Grain	Straw Roots	Soil
1st	1st	0.22	35.8 88.6	2.06	<0.01	1.37 0.82	12.4	<0.1	8.8 5.3	79.8
	2nd	0.21	34.6 88.0	2.58	<0.01	1.09 0.60	15.5	<0.1	7.0 3.9	99.6
2nd	1st	0.15	0.59 na	1.20	<0.01	0.10 na	7.20	<0.1	0.6 na	0.7
	2nd	0.39	0.90 3.61	1.11	<0.01	0.10 0.02	6.66	<0.1	0.6 0.1	42.9
3rd	1st	0.05	0.10 0.53	0.84	<0.01	0.01 <0.01	5.04	<0.1	0.1 <0.1	32.4
	2nd	0.11	0.19 0.62	0.80	<0.01	0.01 <0.01	4.80	<0.1	0.1 <0.1	30.9

na: not analyzed.

1/ Total weight of soil used for calculation was 6 kg (volume of pots used was 5 l; soil bulk density was 1.29 g/cm^3).

2/ Total weight of soil used for calculation was 15 kg (volume of pots used was 12 l; soil bulk density was 1.29 g/cm^3).

Table 2 shows that total radioactive residues in crops and soil in the second and third growing periods (sown 120 and 365 days after soil treatment, respectively) were significantly lower than those found in the first growing period (sown at 33 days after soil treatment). Irrespective of the type of crop grown, most of the applied radioactivity remained in the soil.

In lettuce, TRR in leaves harvested at maturity after sowing 33 days after soil treatment was 1.89 mg/kg (expressed as dicloran), while the corresponding leaves of plants grown from the second and third sowings contained less than 0.1 mg/kg. In the case of sugar beet, the TRR in roots declined from 0.48 mg/kg (expressed in dicloran) from the first sowing, to less than 0.1 mg/kg in roots from the third sowing, when the TRR was less than 1% of the applied radioactivity. Sugar beet leaves showed higher TRR contents, ranging from 3.9 mg/kg in plants from the first sowing to slightly above 0.1 mg/kg in leaves from the later sowings. In wheat grain, only small amounts, equivalent to <0.1% of the applied radioactivity, were found in all cases. Wheat straw obtained from the first sowing showed relatively high TRR but straw obtained from the second sowing contained less than 1 mg/kg and that from the third sowing contained less than 0.2 mg/kg.

TLC analysis showed that less than 10% of the TRR was present as unchanged dicloran in extracts of plant samples harvested from the crops sown 33 days after soil treatment. Unidentified polar or polymeric degradation products were also found. Extracts of plant samples obtained after sowing 120 and 365 days after soil treatment contained mostly unidentified polar or polymeric degradation products.

Another study was conducted, in 1997, to establish the nature of the residues in rotational crops (O'Neal, 1997). [Phenyl-U-^{14}C]dicloran was applied to the surface of sandy loam soil, as a broadcast spray at a rate of 14.8 kg a.i./ha. Lettuce, turnip and wheat were planted 30, 120 and 365 days after treatment of the soil. Crops were grown according to normal agricultural practice and were harvested at maturity. A portion of wheat was also harvested at the immature stage.

Radioactive residues in each matrix were extracted with acetonitrile and dichloromethane. Unextracted ^{14}C-residues were subjected to hydrolysis with hydrochloric acid and sodium hydroxide and the hydrolysates were partitioned against dichloromethane. The remaining unextracted radioactive residues were characterized by sequential extraction and enzyme digestion. These plant extracts were concentrated and analyzed by HPLC and TLC, to identify radioactive compounds.

Table 3. Total radioactive residues (TRR, expressed as dicloran) in soil that had been treated with dicloran 30, 120 and 365 days before planting crops (O'Neal, 1997).

	Days after application	Average TRR as dicloran, mg/kg
30-day rotation	0	9.26
	90	6.96
	106	4.46
120-day rotation	0	11.21
	90	3.37
	181	6.08
	208	2.86
365-day rotation	0	12.31
	90	2.98
	412	2.20
	427	2.40
	587	1.95

The average TRR in soil on day 0 (day of sowing) was 10.92 mg/kg in the three simulated rotational periods. TRRs declined sharply in the first 90 days of growing period in all cases (Table 3).

Table 4 shows that the average TRR level in the harvested crops was the highest in the case of 120-day rotation, slightly higher than in the case of 30-day rotation. TRRs in the harvested crops from the 365-day rotation were significantly lower than those from the 30- and 120-day rotations. Combining the data from all three rotations, the proportion of TRR in plants that was acetonitrile-extractable averaged about 20%, although the values for different crops and plant parts were very variable (about 2-75%). Only a small proportion of the radioactivity (maximum 3.3% TRR) was dichloromethane-extractable. Averages of 19 and 21% TRR were acid- and alkali-extractable, respectively. An average of 30% of the TRR was unextractable. Sequential extraction and enzyme digestion showed that the majority of unextracted radioactive residues were associated with lignin, hemicellulose and cellulose.

Components of the TRR in the three crops are shown in Tables 5-7. Dicloran was observed in all plant materials harvested from the 30-, 120- and 365-day rotations, except for wheat grain from all rotations and wheat chaff from the 365-day rotation. In the 365-day rotation, dicloran (parent) residues in all crops were less than 0.02 mg/kg, except for wheat straw which contained 0.13 mg/kg. Dicloran metabolites which were found in crop samples throughout the course of the study included: 4-amino-2,6-dichlorophenol (DCAP), 4-amino-2,6-dichloroacetaniline (DCPD), 3,5-dichloro-4-hydroxyacetanilide (3,5-DCHA), 4-amino-3,5-dichloroacetanilide (DCAA), 2,6-dichloro-4-hydroxyaniline (DCHA), 2,6-dichloro-4-nitrophenol (DCNP), 2,6-dichlorophenol (2,6-DCP) and 2,6-dichloroaniline (2,6-DCA). The most frequently observed metabolite, 4-amino-2,6-dichlorophenol (DCAP), was observed in all rotations, although it was only observed in wheat straw in the 365-day rotation. While 12, 10 and 20 unidentified components were observed in samples from the 30-, 120- and 365-day rotations, respectively, none of them exceeded 10% TRR, or 0.05 mg/kg (expressed as dicloran) in the 365-day rotation. Based on their extraction and chromatographic behaviour, all unidentified components were polar in nature, with the exception of Unknown 23 (0.5% TRR; 0.01 mg/kg), which was slightly less polar than dicloran (see Table 6). In general, most of the unidentified components were observed in the aqueous phases of acid- and/or base-hydrolysates after partition with organic solvent. Thus they were probably either conjugates or natural components into which the radioactive carbon had been incorporated.

Of the unidentified components, Unknown 1 was the most frequently observed in the rotational crop matrices. This component is likely to be the same as the Unknown 1 found in the potato metabolism study (Evaluation of the 1998 JMPR, page 323), because both components displayed the same extraction and chromatographic behaviour and that in potatoes was observed to be very polar and comprised of multiple compounds, as demonstrated by reversed phase HPLC.

Metabolism of dicloran involved reduction of the nitro group and either acetylation of the amino group(s) or deamination and hydroxylation. Glutathione conjugation, substituting one or both of the chlorine atoms, also appears to occur. The minor metabolites are likely to be derived from the glutathione conjugation pathway (Evaluation of the 1998 JMPR, page 323). This study on rotational crops confirmed the metabolic pathway proposed by the 1998 JMPR (Evaluation of the 1998 JMPR, page 336).

Table 4. Extraction of total radioactive residues (TRRs) from rotational crops, grown in soil treated with dicloran at 30, 120 or 365 days before planting (O'Neal, 1997).

Matrix	DAA	TRR, mg/kg	Extraction used										Accountability of TRR	
			Acetonitrile		Dichloro-methane		0.1N HCI [1/]		0.1N NaOH [1/]		Non-extractable			
			%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR [2/]	mg/kg
30-day rotation														
Lettuce	106	1.38	25.8	0.36	2.6	0.04	21.8	0.30	20.1	0.28	23.5	0.32	93.8	1.29
Turnip foliage [3/]	90	0.88	28.5	0.25	2.3	0.02	11.6	0.10	21.6	0.19	28.4	0.25	92.5	0.81
Turnip root	90	0.52	74.9	0.39	1.6	<0.01	3.3	0.02	10.8	0.06	14.6	0.08	105.1	0.54
Immature wheat	90	1.04	35.8	0.37	1.6	0.02	32.5	0.34	17.6	0.18	8.0	0.08	95.5	0.99
Wheat straw	166	8.64	12.5	1.08	0.8	0.07	28.2	2.44	39.6	3.42	14.7	1.27	95.8	8.28
Wheat grain	166	0.19	1.7	<0.01	1.5	<0.01	34.8	0.07	24.5	0.05	21.9	0.04	84.4	0.16
Wheat chaff	166	0.91	5.6	0.05	0.6	<0.01	14.0	0.13	21.7	0.20	57.7	0.53	99.6	0.91
120-day rotation														
Lettuce	181	1.14	32.0	0.37	1.5	0.02	24.9	0.28	12.0	0.14	16.8	0.20	87.2	1.01
Turnip foliage	208	1.21	19.8	0.24	0.7	<0.01	31.3	0.38	13.2	0.16	20.4	0.25	85.4	1.03
Turnip root [1/]	208	0.84	28.0	0.24	1.1	<0.01	31.4	0.27	13.3	0.11	16.5	0.14	90.4	0.76
Immature wheat	181	1.94	37.3	0.72	1.4	0.03	24.9	0.48	21.4	0.42	5.2	0.10	90.2	1.75
Wheat straw	302	10.55	2.7	0.29	0.2	0.02	16.7	1.76	23.3	2.46	49.0	5.17	91.9	9.70
Wheat grain [3/]	302	1.38	1.9	0.03	2.0	0.03	34.7	0.48	25.5	0.35	22.1	0.31	86.2	1.20
Wheat chaff	302	1.71	2.0	0.3	0.7	0.01	11.5	0.20	18.2	0.31	56.1	0.96	88.8	1.51
365-day rotation														
Lettuce	412	0.11	44.1	0.05	3.3	<0.01	14.0	0.02	30.8	0.03	14.4	0.02	106.6	0.12
Turnip foliage	427	0.12	16.8	0.02	2.0	<0.01	4.9	<0.01	14.9	0.02	39.4	0.05	78.0	0.09
Turnip root	427	0.04	23.8	0.01	1.2	<0.01	11.1	<0.01	22.4	0.01	28.8	0.01	87.3	0.03
Immature wheat	412	0.30	17.0	0.05	1.9	<0.01	8.4	0.02	13.1	0.04	41.4	0.12	81.8	0.23
Wheat straw [3/]	587	1.79	5.1	0.09	2.8	0.05	6.4	0.11	24.3	0.43	62.2	1.11	100.8	1.79
Wheat grain	587	0.14	1.8	<0.01	2.4	<0.01	10.3	0.01	36.4	0.05	30.5	0.04	82.7	0.10

Matrix	DAA	TRR, mg/kg	Extraction used										Accountability of TRR	
			Acetonitrile		Dichloro-methane		0.1N HCl [1/]		0.1N NaOH [1/]		Non-extractable			
			%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR [2/]	mg/kg
Wheat chaff	587	0.18	4.6	<0.01	3.1	<0.01	13.2	0.02	13.7	0.02	57.1	0.10	91.7	0.14

[1/] Values are pre-partition percentages; the majority of radiocarbon remained in the aqueous phase after partition.
[2/] Sum of all fractionated TRR %.
[3/] Unextracted residues were characterized in these matrices.
Values for mg/kg are expressed as dicloran equivalents.
DAA = Days after application.

Table 5. Summary of identified and unidentified components in the total radioactive residue (TRR) in rotational crops grown 30 days after treating soil with dicloran (O'Neal, 1997).

	Turnip foliage		Turnip root		Lettuce		Immature wheat		Wheat chaff		Wheat grain		Wheat straw	
TRR, as dicloran	0.88 mg/kg		0.52 mg/kg		1.38 mg/kg		1.04 mg/kg		0.91 mg/kg		0.19 mg/kg		8.64 mg/kg	
Component	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Unknown 1	22.33	0.20	2.25	0.02	23.74	0.32	29.07	0.31	5.93	0.06	nd	nd	7.28	0.63
Unknown 2	2.17	0.02	nd	nd	1.17	0.02	nd	nd	2.85	0.02	20.60	0.04	nd	nd
Unknown 3	nd	nd	1.21	0.01	nd	nd	0.7	0.01	nd	nd	nd	nd	2.43	0.21
Unknown 4	nd	nd	0.15	<0.01	nd	nd	nd	nd	1.89	0.02	nd	nd	nd	nd
Unknown 5	nd	nd	1.39	0.01	nd	nd	nd	nd	1.00	0.01	nd	nd	nd	nd
Unknown 6	0.64	0.01	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Unknown 8	nd	nd	0.45	<0.01	0.94	0.01	nd	nd	nd	nd	nd	nd	nd	nd
Unknown 9	nd	nd	nd	nd	0.86	0.01	nd	nd	nd	nd	nd	nd	nd	nd
Unknown 10	nd	nd	nd	nd	1.02	0.01	nd	nd	nd	nd	nd	nd	nd	nd
Unknown 14	0.36	<0.01	nd	nd	nd	nd	nd	nd	3.51	0.03	nd	nd	nd	nd
Unknown 19	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	8.43	0.73
Unknown 22	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	11.93	1.03
Unidentified sub-total	25.50	0.23	5.45	0.04	27.73	0.37	29.78	0.32	15.18	0.14	20.60	0.04	30.07	2.60
DCAP	0.14	<0.01	5.70	0.03	1.66	0.02	3.49	0.04	7.58	0.07	nd	nd	10.72	0.93
DCPD	0.42	<0.01	nd	nd	nd	nd	nd	nd	9.49	0.09	nd	nd	4.77	0.41
3,5-DCHA	0.67	0.01	nd	nd	nd	nd	4.51	0.04	0.95	0.01	nd	nd	3.40	0.29
DCA	0.47	<0.01	2.00	0.01	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
DCHA	nd	nd	nd	nd	nd	nd	2.54	0.03	nd	nd	nd	nd	3.28	0.28
DCNP	0.07	<0.01	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	1.24	0.11
2,6-DCP	0.31	<0.01	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
2,6-DCA	0.42	<0.01	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	3.98	0.35
Dicloran	29.94	0.26	75.54	0.39	31.12	0.44	33.28	0.34	0.22	<0.01	nd	nd	12.35	1.07
Identified sub-total	32.44	0.27	83.24	0.43	32.78	0.46	43.82	0.45	18.24	0.17	nd	nd	39.74	3.44
Unextracted	28.4	0.25	14.6	0.08	23.5	0.32	8.0	0.08	57.7	0.53	21.9	0.04	14.7	1.27
Total	86.34	0.75	103.29	0.55	84.01	1.15	81.60	0.85	91.12	0.84	42.50	0.08	84.51	7.31

nd = not detected.

Table 6. Summary of identified and unidentified components in the total radioactive residue (TRRs) in rotational crops grown 120 days after treating soil with dicloran (O'Neal, 1997).

	Turnip foliage		Turnip root		Lettuce		Immature wheat		Wheat chaff		Wheat grain		Wheat straw	
TRR, as dicloran	1.21 mg/kg		0.84 mg/kg		1.14 mg/k		1.94 mg/kg		1.71 mg/kg		1.38 mg/kg		10.55 mg/kg	
Component	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Unknown 1	8.22	0.10	20.16	0.17	6.42	0.07	15.96	0.31	22.40	0.38	14.60	0.20	7.17	0.76
Unknown 2	13.58	0.16	16.83	0.14	nd	nd	nd	nd	0.80	0.01	10.12	0.14	8.62	0.91

Unknown 4	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	30.38	0.42	nd	nd
Unknown 9	nd	nd	nd	nd	1.91	0.02	4.38	0.09	nd	nd	nd	nd	nd	nd
Unknown 11	nd	nd	nd	nd	nd	nd	2.19	0.04	nd	nd	nd	nd	11.83	1.24
Unknown 13	nd	nd	nd	nd	4.41	0.05	0.57	0.01	nd	nd	nd	nd	2.56	0.27
Unknown 14	nd	nd	nd	nd	nd	nd	0.64	0.01	nd	nd	nd	nd	7.23	0.76
ND	nd	nd	nd	nd	nd	nd	0.42	0.01	nd	nd	nd	nd	nd	nd
Unknown 20	nd	nd	nd	nd	nd	nd	1.28	0.02	nd	nd	nd	nd	1.08	0.11
Unknown 23	nd	nd	nd	nd	nd	nd	nd	nd	0.49	0.01	nd	nd	nd	nd
Unidentified sub-total	21.80	0.26	36.99	0.31	12.74	0.14	25.44	0.49	23.69	0.40	55.10	0.76	38.49	4.05
DCPD	3.86	0.05	1.24	0.01	4.02	0.05	10.63	0.20	nd	nd	nd	nd	nd	nd
DCAP	nd	nd	nd	nd	8.57	0.09	10.96	0.22	nd	nd	nd	nd	nd	nd
3,5-DCHA	nd	nd	nd	nd	0.97	0.01	7.53	0.15	nd	nd	nd	nd	7.13	0.75
DCHA	nd	nd	nd	nd	1.09	0.01	nd	nd	nd	nd	nd	nd	1.26	0.13
DCAA	1.24	0.02	nd	nd	1.67	0.02	1.33	0.03	nd	nd	nd	nd	0.31	0.03
2,6-DCP	7.76	0.09	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
2,6-DCA	4.50	0.05	0.68	0.01	0.48	0.01	3.53	0.07	nd	nd	nd	nd	nd	nd
Dicloran	18.94	0.23	28.89	0.25	23.06	0.26	15.89	0.31	0.71	0.01	nd	nd	0.88	0.10
Identified sub-total	36.3	0.44	30.81	0.27	39.86	0.45	49.87	0.98	0.71	0.01	nd	nd	9.58	1.01
Unextracted	20.4	0.25	16.5	0.14	16.8	0.20	5.2	0.10	56.1	0.96	22.10	0.31	49.0	5.17
Total	56.70	0.95	84.30	0.72	69.40	0.79	80.51	1.57	80.50	1.37	77.20	1.07	97.07	10.23

nd = not detected. ND = an unnumbered component, different from all other identified or unidentified components.

Table 7. Summary of identified and unidentified components in the total radioactive residue (TRR) in rotational crops grown 365 days after treating soil with dicloran (O'Neal, 1997).

	Turnip foliage		Turnip root		Lettuce		Immat. wheat		Wheat chaff		Wheat grain		Wheat straw	
TRR as dicloran	0.12 mg/kg		0.04 mg/kg		0.11 mg/kg		0.30 mg/kg		0.18 mg/k		0.14 mg/kg		1.78 mg/kg	
Component	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Unknown 1	nd	nd	nd	nd	nd	nd	5.32	0.02	nd	nd	nd	nd	nd	nd
Unknown 2	19.02	0.03	42.84	0.02	24.26	0.03	14.08	0.05	10.66	0.02	nd	nd	6.50	0.12
ND	nd	nd	nd	nd	2.24	<0.01	nd	nd	16.00	0.03	nd	nd	nd	nd
Unknown 4	0.67	<0.01	nd	nd	1.48	<0.01	nd	nd	1.34	<0.01	nd	nd	nd	nd
Unknown 5	0.66	<0.01	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Unknown 6	0.67	<0.01	nd	nd	5.95	0.01	nd	nd	nd	nd	nd	nd	1.71	0.03
Unknown 7	0.52	<0.01	nd	nd	1.96	<0.01	nd	nd	nd	nd	nd	nd	nd	nd
Unknown 8	0.72	<0.01	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	1.13	0.02
Unknown 9	1.38	<0.01	nd	nd	0.42	<0.01	nd	nd	nd	nd	nd	nd	1.71	0.03
Unknown 10	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	1.72	0.03
Unknown 11	1.09	<0.01	2.50	<0.01	6.45	0.01	8.34	0.03	nd	nd	nd	nd	2.87	0.02
Unknown 12	1.43	<0.01	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Unknown 13	nd	nd	nd	nd	16.21	0.02	nd	nd	nd	nd	nd	nd	1.86	0.03
Unknown 14	1.16	<0.01	nd	nd	3.61	<0.01	3.73	0.01	nd	nd	nd	nd	nd	nd
Unknown 15	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	4.06	0.07
Unknown 16	0.94	<0.01	nd	nd	nd	nd	nd	nd	nd	nd	9.82	0.01	nd	nd
Unknown 17	0.37	<0.01	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Unknown 18	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	22.06	0.03	nd	nd
Unknown 20	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.98	0.02
Unknown 21	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	6.62	0.01	nd	nd
Unidentified sub-total	28.63	0.03	45.34	0.02	62.58	0.07	31.47	0.11	28.00	0.05	38.50	0.05	22.54	0.37
DCAP	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	1.50	0.03
DCPD	nd	nd	nd	nd	4.75	0.01	nd	nd	nd	nd	nd	nd	nd	nd
3,5-DCHA	nd	nd	2.90	<0.01	nd	nd	nd	nd	nd	nd	nd	nd	2.05	0.04
DCHA	nd	nd	nd	nd	3.00	<0.01	nd	nd	nd	nd	nd	nd	nd	nd
DCAA	nd	nd	nd	nd	7.67	0.01	nd	nd	nd	nd	nd	nd	nd	nd
Dicloran	3.08	<0.01	6.85	<0.01	8.00	0.01	6.52	0.02	nd	nd	nd	nd	7.34	0.13
ND	3.08	<0.01	9.75	<0.01	23.42	0.03	6.52	0.02	nd	nd	nd	nd	10.89	0.20
Unextracted	39.40	0.05	28.8	0.01	14.4	0.02	41.4	0.12	57.1	0.10	31.8	0.04	62.2	1.11
Total	71.11	0.08	83.89	0.03	100.40	0.12	79.39	0.25	85.1	0.15	70.30	0.09	95.63	1.68

nd = not detected. ND = an unnumbered component, different from all other identified or unidentified components.
Immat. wheat = immature wheat.

The third study was conducted to determine the magnitude of dicloran residues in or on rotational crops, under actual field-use conditions following an application to bare ground (Mickelson, 2000). Dicloran was applied at an application rate of 4.4 kg a.i./ha on two test sites, one in California and the other in North Carolina in the USA. The treated plot was divided into sub-plots for three rotational crops, planted 30, 120 and 360 days after the application. In California, lettuce, radish, wheat and sorghum were grown but wheat was grown only in the 120-day rotation and sorghum only in the 30- and 360-day rotations. In North Carolina, mustard, radish, wheat and sorghum were grown but wheat was grown only in the 30- and 120-day rotations and sorghum was grown only in the 360-day rotation. The samples were extracted with dichloromethane and the extracts evaporated using a rotary evaporator. Extracts were re-dissolved in *n*-hexane, partitioned into acetonitrile and the residues were quantified using LC-MS/MS (Table 8).

Dicloran (parent) concentrations in the rotational crops were below the limit of quantification (<0.05 mg/kg) in all crops grown on treated soil. with exception of lettuce (0.104 and 0.071 mg/kg), radish tops (0.227 and 0.187 mg/kg) and radish roots (0.278 and 0.243 mg/kg) grown in the 30-day rotation in California. Dicloran residues were not found at or above the LOQ in any of the control samples grown on untreated soil. Procedural recovery of dicloran was 65-122% from lettuce, mustard greens, radish tops and roots, wheat forage, hay, grain and straw and sorghum forage, grain and stover.

Table 8. Dicloran (parent) residues found in rotational crops grown in California and North Carolina, 30, 120 and 360 after field soils with dicloran (Mickelson, 2000).

Crop/commodity	Residue range, dicloran mg/kg [1/]
Lettuce	<0.05-0.104
Mustard greens	<0.05
Radish top	<0.05-0.227
Radish roots	<0.05-0.278
Wheat forage	<0.05
Wheat hay	<0.05
Wheat grain	<0.05
Wheat straw	<0.05
Sorghum forage	<0.05
Sorghum grain	<0.05
Sorghum stover	<0.05

[1/] LOQ = 0.05 mg/kg.

RESIDUE ANALYSIS

Analytical methods

The validity of a method used for the determination of dicloran residues in egg white and chicken muscle from laying hens and goat milk and goat fat from lactating goats in metabolism studies was checked (Teeter, 1998; JMPR, 1999). Egg white and chicken muscle samples were fortified, if necessary, and extracted with acetone/water (6:1) and the extracts then partitioned with a mixture of hexane/ethyl acetate (9:1). Milk and fat tissue samples underwent an initial extraction with acetone/water (7:1) followed by an extraction of the aqueous layer with hexane and partition into acetonitrile. After an additional clean-up step, including a diol solid-phase extraction, dicloran was eluted with toluene. Dicloran was determined by gas chromatography and an electron capture detector.

Each matrix was tested with 3 samples from the metabolism study, together with 1 control and 2 fortified samples. Recovery from the freshly fortified samples was 93.0-106.7% (see Table 9). These results show that the method tested is valid for the determination of dicloran in egg white, chicken muscle, goat milk and goat fat.

Table 9. Recovery of dicloran from hen and goat matrices (Teeter, 1998; JMPR, 1999).

Sample matrix	Fortification, mg/kg	Recovery, %	Residue in metabolism study, mg/kg	Recovery ± RSD [%] [1/]
Egg white	0.15	102.7, 106.7	1.36	88.2 ± 3.0
Chicken muscle	0.05	94.0, 100.0	0.058	82.2 ± 11.6
Goat milk	0.699	94.4, 93.0	0.701	47.8 ± 6.3
Goat fat	0.97	100.0, 96.9	1.0	81.7 ± 7.3

[1/] Compared with the determination by radio-analysis.

Average recovery of dicloran from samples produced in the metabolism studies was higher than 80%, with exception of the goat milk sample. In the milk samples from the goat metabolism study, the average level of dicloran measured by the method was 47.8% of the amount determined radiochemically but no explanation was given.

USE PATTERN

Information on the use of dicloran on grapes, lettuce, nectarines, peaches, plums, strawberries and tomatoes in Argentina, Chile, Italy and the USA, for the control of fungi of the genera *Sclerotinia*, *Monilinia*, *Rhizopus*, *Botrytis* and others, was provided by the manufacturer. The information available to the Meeting on registered uses is summarized in Table 10. Labels officially approved in Argentina, Chile, Italy and the USA were provided to the Meeting.

Table 10. Registered uses of dicloran on grapes, lettuce, nectarines, peaches, plums, strawberries and tomatoes.

Crop	Country	Formulation		Application				PHI, days
		Type	Conc. a.i. g/kg	Method	Spray conc., kg a.i./hl	Rate kg a.i./ha,	No.	
Grapes	Argentina	WP	750	Spraying	0.19		[6/]	7
Grapes	Chile	WP	750	Spraying	0.19-0.26	2.3	[2/]	1
Grapes	USA	DP	60	Dusting		2.0	[13/]	1
Grapes	USA	WP	750	Spraying		1.7-3.9	[14/]	
Lettuce, leaf only (greenhouse)	USA	WP	750	Spraying		2.2	2 [20/]	14
Lettuce	Argentina	WP	750	Spraying	0.28		[7/]	10
Lettuce	Chile	WP	750	Spraying	0.23-0.30	1.9-3.0	[4/]	14
Lettuce	Italy	WP	475	Spraying	0.07-0.12		[8/]	21
Lettuce	Italy	WP	500	Spraying	0.08-0.10	0.08-013 [9/]	2-3 [9/]	20
Lettuce	Italy	WP	450	Spraying	0.07-0.09			20
Lettuce	Italy	SC	460	Spraying	0.08-0.10		2-3 [12/]	20
Lettuce, head & leaf	USA	WP	750	Spraying		[17/]	2	14
Lettuce, head & leaf	USA	WP	750	Spraying		2.8-4.5	1 [18/]	14
Lettuce, head & leaf	USA	WP	750	Spraying		2.2	[19/]	14
Nectarines	Chile	WP	750	Fine spraying	0.13-0.15	2.6-3.0	[1/]	1
Nectarines (post-harvest)	Chile	WP	750	Dipping or immersion in the packing line	0.09; for industrial processing, 0.13			-
Nectarines	USA	WP	750	Spraying		1.1-4.5	2-4 [15/]	10
Peaches	Argentina	WP	750	Spraying	0.15		3 [5/]	1
Peaches (post-harvest)	Argentina	WP	750	Spraying or immersion	0.09-0.11			-
Peaches	Chile	WP	750	Fine spraying	0.13-0.15	2.6-3.0	[1/]	1
Peaches (post-harvest)	Chile	WP	750	Dipping or immersion in the packing line	0.09; for industrial processing, 0.13			-
Peaches	USA	WP	750	Spraying		1.1-4.5	2-4 [15/]	10
Plums	Argentina	WP	750	Spraying	0.15		3 [5/]	1
Plums (post-harvest)	Argentina	WP	750	Spraying or immersion	0.09-0.11			-
Plums	Chile	WP	750	Fine spraying	0.13-0.15	2.6-3.0	[1/]	1
Plums	USA	WP	750	Spraying		1.1-4.5	2	[16/]
Strawberries	Chile	WP	750	Spraying	0.23-0.26	2.6	4 [3/]	1
Strawberries	Italy	WP	475	Spraying	0.12-0.14		[8/]	21
Strawberries	Italy	WP	500	Spraying	0.08-0.10		[11/]	20
Strawberries	Italy	WP	450	Spraying	0.07-0.09			20
Strawberries	Italy	SC	460	Spraying	0.08-0.10		[11/]	20
Tomatoes	Italy	WP	475	Spraying	0.07-0.12		[8/]	21
Tomatoes	Italy	WP	500	Spraying	0.08-0.10		2-3 [10/]	20
Tomatoes	Italy	SC	460	Spraying	0.08-0.10		2-3 [12/]	20
Tomatoes	Italy	WP	450	Spraying	0.07-0.09			20

Crop	Country	Formulation		Application				PHI, days
		Type	Conc. a.i. g/kg	Method	Spray conc., kg a.i./hl	Rate kg a.i./ha,	No.	
Tomatoes (greenhouse)	USA	WP	750	Spraying		0.84	4	10

1/ At flowering: apply from the flower bud stage onward. Repeat at 8-10 day intervals; pre-harvest: apply every 15 days.
2/ First application at pre-flowering stage. Second application when 100% of the flowers have opened.
3/ Begin application at 2-4 open leaves. Repeat every 10 days during flowing and/or when conditions favour disease.
4/ If the ambient conditions are favourable for the development of disease, repeat every 7 days.
5/ Apply 18, 10 and 1 day before harvest.
6/ Apply at the beginning of flowering, before closing of the raceme, and as weather conditions require.
7/ Treat after the first true leaves appear.
8/ Not to apply before 15 days have passed after transplanting and at least the second true leaf appears.
9/ Treatment on bare ground, both in seed beds and outdoors.
10/ At intervals of 3-4 weeks. The young plants must have at least four complete leaves (2 true leaves and 2 cotyledons) and survived 10-15 days after transplanting.
11/ Carry out the first treatment when flowering starts and subsequent treatments every 10-15 days, as necessary.
12/ Use for treatment on bare ground before sowing or transplanting and/or during vegetation, 2-3-treatments with an application interval of 20-30 days. Apply on plants which have developed 2-4 true leaves after surviving 15 days after transplanting.
13/ Follow after cluster tightening with the dust application and repeat at 2-week intervals. Use limited to grapes grown west of the Rocky Mountains only.
14/ Begin applications when disease is anticipated. Repeat applications at 7-day intervals or as needed. Up to 4.5 kg/ha may be applied per season. Use is limited to grapes grown west of the Rocky Mountains only.
15/ For blossom blight, apply at pink bud and full bloom. For fruit decay, apply 18 days and 10 days before harvest.
16/ Apply at popcorn and full bloom.
17/ Apply 1.7 kg/ha (in 190-470 l of water/ha) pre-emergence, in a 10-15 cm band over the transplant or seed row. Make a second application of 2.8 kg/ha (in 470-940 l of water) immediately after thinning.
18/ Apply immediately after thinning.
19/ Apply prior to thinning (2-6 leaf stage) and repeat immediately after thinning.
20/ Apply 7 days following transplanting. Repeat application when plants are half-mature.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information from the manufacturer on supervised field trials on:

Table 11	Nectarines	Australia, Brazil and USA;
Table 12	Peaches	Brazil, Canada and USA;
Table 13	Plums	Brazil and USA;
Table 14	Grapes	Brazil, Mexico and USA;
Table 15	Strawberries	Brazil and USA;
Table 16	Tomatoes	Italy and USA;
Table 17	Lettuce	Brazil and USA.

Information provided to the 1998 JMPR (JMPR,, 1999) and relevant to the present evaluation is also included in Table 11-17. The results of trials included in the Monograph of the 1998 JMPR but with an indication of unavailability of recovery data, or of the lack of critical information, were not included in these tables. Where both pre- and post-harvest treatments were applied, the pre-harvest intervals are shown in parentheses. Residues data were rounded to two significant figures. The residues derived from trials matching GAP conditions are double-underlined.

Table 11. Residues of dicloran in nectarines from supervised trials in Australia, Brazil and USA

Location, year, (variety); reference(s)	Application				Days after application	Residues, mg/kg
	Form	kg a.i./ha	kg a.i./hl	No.		
Pre-harvest application						
USA, 1968, (variety not stated); R204 [2/]; JMPR, 1999	WP 75 or 50%		0.12	1	1	0.3
Post-harvest application						
Piedara/SP, Brazil, 2000, (Sun Rype); 632-3275	WP 73.2%		0.089 (12 sec)	1	0 C	5.5 <1.0
Videira/SC, Brazil, 2001, (Ferronate); 632-3275	WP 73.2%		0.087 (12 sec)	1	0 C	2.3 <1.0
Manua da Serra/PR, Brazil, 2001, (Sun Gold); 632-3275	WP 73.2%		0.087 (12 sec)	1	0 C	2.3 <1.0
USA, 1968, (variety not stated); R185;	WP 50%		0.24	1 [1/]	0	2.0

Location, year, (variety); reference(s)	Application				Days after application	Residues, mg/kg
	Form	kg a.i./ha	kg a.i./hl	No.		
JMPR, 1999			0.24	1 [1/]	0	0.88
			0.24	1 [1/]	0	0.50
USA, 1968, (variety not stated); R204 [2/]; JMPR, 1999	WP 50%		0.24	1 [1/]	0	2.0
			0.24	1 [1/]	0	0.95
Australia, 1973, (variety not stated); R422; JMPR, 1999	SC 50%		0.075	1	1 2 4 6 8 10 12 14	2.3 (surface wash) 4.0 (surface wash) 3.3 (surface wash) 2.0 (surface wash) 4.6 (surface wash) 2.8 (surface wash) 4.6 (surface wash) 2.5 (surface wash)
Combination of pre- and post-harvest applications						
USA, 1968, (variety not stated); R204 [2/]; JMPR, 1999	WP 75 or 50%		0.12 0.24	1 (pre-) 1(post-) [1/]	(1) 0	4.4 (with wax)
			0.12 0.24	1 (pre-) 1(post-) [1/]	(1) 0	4.8 (with wax)
			0.12 0.34	1 (pre-) 1(post-) [1/]	(1) 0	5.1 (with wax)
			0.12 0.24	1 (pre-) 1(post-) [1/]	(1) 0	0.4 (with wax)
			0.12 0.24	1 (pre-) 1(post-) [1/]	(1) 0	0.5 (with wax)
	DP 6% WP 75%	3.4 (DP)	0.24 (WP)	3 (pre-) 1 (post-) [1/]	(1) 0	1.5

[1/] Treated with Decco wax applicator.
[2/] Limit of detection not reported.
C = control samples. 12 sec = the dipping time, prior to draining and drying.

Table 12. Residues of dicloran in peaches from supervised trials in Brazil, Canada and the USA.

Location, year, (variety); reference(s)	Application				Days after application	Residues, (mg/kg)
	Form	kg a.i./ha	kg a.i./hl	No.		
Pre-harvest application						
Piedara/SP, Brazil, 2001, (Eldorado); 632-3274	WP 73.2%	1.168	0.146	3	1 [1/] C	2.7 <1.0
Manua da Serra/PR, Brazil, 2001, (Bint); 632-3274	WP 73.2%	1.168	0.146	3	1 [1/] C	4.8 <1.4
Canada, 1964, (variety not stated); R107; JMPR, 1999	WP 50%	6.7		7	0	3.9
USA, 1966, (variety not stated); R174-7; JMPR, 1999	WP 75%		0.12	2	4	0.09
Post-harvest application						
Canada, 1966, (variety not stated); R174-8; JMPR, 1999	WP 70%		0.36	1 [3/]	0	2.1
USA, 1966, (variety not stated); R174-2; JMPR, 1999	WP 75%		0.036	1 [2/]	0	1.5
			0.054	1 [2/]	0	1.9
			0.06	1 [2/]	0	2.1
USA, 1966, (variety not stated); R174-6; JMPR, 1999	WP 50%		0.15	1 [3/]	0	2.4
Australia, 1973, (variety not stated); R420; JMPR, 1999	WP 75%		0.075	1 [4/]	1 3 6 9	5.5 (surface wash) 4.1 (surface wash) 3.5 (surface wash) 2.7 (surface wash)

Location, year, (variety); reference(s)	Application				Days after application	Residues, (mg/kg)
	Form	kg a.i./ha	kg a.i./hl	No.		
Australia, 1973, (variety not stated); R422; JMPR, 1999	SC 50%		0.075	1 [4/]	1 2 4 6 8 10 12 14	6.3, 6.0 (2 trials)(surface wash) 7.5, 8.2 (2 trials)(surface wash) 8.7, 6.2 (2 trials)(surface wash) 4.9, 3.9 (2 trials)(surface wash) 4.6, 6.4 (2 trials)(surface wash) 2.3, 2.3 (2 trials)(surface wash) 8.1, 8.8 (2 trials)(surface wash) 4.1, 9.7 (2 trials)(surface wash)
Combination of pre- and post-harvest applications						
USA, 1966, (variety not stated); R174-7; JMPR, 1999	WP 75%		0.12 0.018	2(pre-) 1(pos-t) [2/]	(4) 0	0.8
						2.5 (after cold water dump tank)
						0.8 (after wet brush defuzzing)
						0.6 (after grading and packing)
						0.7 (after chlorine wash)
			0.12 0.018, 0.015	2(pre-) 2(post-) [2/]	(4) 0	1.0
USA, 1988, (variety not stated); R414; JMPR, 1999	WP 75%	4.5	0.09	3(pre-) 1(post-)	(1), 0	16.3 (dip)
					(1), 0	15.4 (hydrocooler + antifoam)
					(1), 0	11.9 (hydrocooler)
		3.4	0.09	3(pre-) 1(post-)	(1), 0	11.1 (dip)
					(1), 0	18.7 (hydrocooler + antifoam)
					(1), 0	8.3 (hydrocooler)
USA, 1996, (variety not stated); 95007; JMPR, 1999	WP 75%	4.5	0.09	4(pre-) 1(post-) [4/]	(10) 0	5.8
		4.5	0.09	5(pre-) 1(post-) [4/]	(10) 0	5.3

[1/] Fruit at ripening stage.
[2/] Treated with hydrocooler.
[3/] Sprayed.
[4/] Dip.
C = control samples.

Table 13. Residues of dicloran in plums from supervised trials in Brazil and the USA.

Location, year, (variety); reference(s)	Application				Days after application	Residues, mg/kg
	Form	kg a.i./ha	kg a.i./hl	No.		
Pre-harvest application						
Piedade/SP, Brazil, 2001, (Rubi Mel); 632-3310	WP 73.2%	1.168	0.146	3	1 [2/] C [1/]	1.4 <1.0
Manua da Serra/PR, Brazil, 2001; (Reubennell), 632-3310	WP 73.2%	1.168	0.146	3	1 C [1/]	<1.0 <1.0
Passa Quatro/MG, Brazil, 2001, (Santa Rita); 632-3310	WP 73.2%	1.464	0.146	3	1 C [1/]	1.1 <1.0
USA, 1995, (variety not stated); 95011; JMPR, 1999	WP 75%	4.5		4	10	0.17
Combination of pre- and post-harvest applications						
USA, 1986, (variety not stated); R415; JMPR, 1999	WP 75%	2.8	8.7	2(pre-) 1(post-) [3/]	(112) 0	14.0 (2.3 l/hr spray) (with wax)
		2.8	0.24	2(pre-) 1(post-) [4/]	(88) 0	6.1 (110 l/hr spray) (with wax)
USA, 1995, (variety not stated); 95008; JMPR, 1999	WP 75%	4.5	0.24	2(pre-) 1(post-) [4/]	(154) 0	1.9 (with wax) (1 kg/ai/25 t)
		4.5	1.1	2(pre-) 1(post-) [3/]	(154) 0	2.1 (with wax) (1 kg/56 t)

[1/] Control samples.
[2/] Fruits ripening stage; for other trials, mature fruits.
[3/] Low-volume applicator.
[4/] Conventional applicator.

Table 14. Residues of dicloran in grapes from supervised trials of pre-harvest applications in Brazil, Mexico and the USA.

Location, year, (variety); reference(s)	Application				Days after application	Residues, (mg/kg)
	Form	kg a.i./ha	kg a.i./hl	No.		
São Sebastião da Amoreira/PR, Brazil, 2001, (Italia); 632-4011	WP 73.2%	2.562	0.256	5	1 [1/] C	1.1 <1.0
Louveira/SP, Brazil, 2001, (Niagara Rosado); 632-4011	WP 73.2%	2.565	0.256	5	1 [1/] C	5.2 <1.0
Jaiba/MG, Brazil, 2001, (Niagara Rosado); 632-4011	WP 73.2%	2.565	0.256	5	1 [1/] C	4.0 <1.0
Sonora, Mexico, 2001, (Thompson seedless); 632-4010	WP 75.6%	2.25		3	1 C	1.5 0
Sonora, Mexico, 2001, (Thompson seedless); 632-4010	WP 75.6%	2.25		3	1 C	0.36 <0.02
Baja California, Mexico, 2001, (Thompson seedless); 632-4010	WP 75.6%	2.25		3	1 C	0.83 <0.02
Baja California, Mexico, 2001, (Thompson seedless); 632-4010	WP 75.6%	2.25		3	1 C	0.95 <0.02
California, USA, 1967, (variety not stated); R180 [2/]; JMPR, 1999	WP 75% DP 6%	2.2 x 1 (WP) 2.0 x 2 (DP)		3	1	6.0
California, USA, 1984, (variety not stated); 66007; JMPR, 1999	WP 75% DP 6%	2.2 x 1 (WP) 2.0 x 3 (DP)		4	1	0.29
		2.2 x 2 (WP) 0.2 x 1 (DP) 2.0 x 7 (DP)		10	1	0.62
California, USA, 1995, (variety not stated); 95012; JMPR, 1999	WP 75%	4.5		2	3	1.0

[1/] Fruit at ripening stage.
[2/] Limit of detection not reported.
C = control samples.

Table 15. Residues of dicloran in strawberries from supervised pre-harvest trials in Brazil and the USA.

Location, year, (variety); reference(s)	Application				Days after application	Residues (mg/kg)
	Form	kg a.i./ha	kg a.i./hl	No.		
Valinhos/SP, Brazil, 2001, (Oro Grande); 632-4113	WP 73.2%	2.048	0.256	4	1 [1/] C	4.3 <1.0
Louveira/SP, Brazil, 2001, (Campiniero); 632-4113	WP 73.2%	2.048	0.256	4	1 [1/] C	4.5 <1.0
Loudrina/PR, Brazil, 2001, (Dover); 632-4113	WP 73.2%	2.048	0.256	11	1 [1/] C	<1.0 <1.0
USA, 1963, (variety not stated); R52; JMPR, 1999	WP 50%		0.12	9	1	2.6
			0.12	10	5	1.6
			0.12	9	1	2.9
			0.12	10	5	1.9
	WP 75%		0.09	1	1	5.5 (with cap)
USA, 1963, (variety not stated); R54; JMPR, 1999	WP 50%		0.04	1	2	1.2
			0.09	2	1	0.73 (with cap)
	WP 75%		0.09	1	0 1 3 5	6.3 5.2 5.6 5.0
USA, 1963, (variety not stated); R151; JMPR, 1999	WP 75%	1.7		4	11	0.2
		1.7		3	9	1.24
		1.7		4	0	0.45
			0.16	4	11	0.13

[1/] Fruit at ripening stage.
C = control samples.

Table 16. Residues of dicloran in tomatoes from supervised trials in Italy, the UK and the USA.

Location, year, (variety); reference(s)	Application				Days after application	Residues (mg/kg)
	Form	kg a.i./ha	kg a.i./hl	No.		
Pre-harvest application						
Monterotondo (RM), Italy, 2001; 633-2010, 633-2011	WP 47.7%	0.570	0.095	3	0 4 12 20 25 C	0.15 0.04 <0.01 <0.01 <0.01 <0.01
Possuolo del Fruili (UD), Italy, 2001, (Brigade); 633-2013, 633-2012	WP 47.7%	0.474 (1st, 2nd) 0.570 (3rd)	0.095	3	0 5 12 20 25 C	0.58 0.23 0.10 0.04 0.01 <0.01
Ronchis (UD), Italy, 2001, (Imeas); 633-2013, 633-2012	WP 47.7%	0.665 (1st, 2nd) 0.950 (3rd)	0.095	3	0 5 12 20 25 C	0.56 0.31 0.13 0.08 0.08 <0.01
USA, 1962, (glasshouse); R24; JMPR, 1999	WP 75%		0.07	1	0	0.91
					5	0.70 0.20 (washed)
					10	0.41 0.18 (washed)
			0.13	1	0	1.40
					5	0.78 0.26 (washed)
					10	0.50 0.22 (washed)
			0.27	1	0	3.0
					5	3.2 0.54 (washed)
					10	2.0 0.84 (washed)
UK, 1972, (glasshouse); R269; JMPR, 1999	FU	1.3		1	4 h 6 h 8 h 10 h 12 h 1 2-10	0.67 (surface wash) 0.53 (surface wash) 0.42 (surface wash) 0.30 (surface wash) 0.26 (surface wash) 0.07 (surface wash) <0.05 (surface wash)
USA, 1963, (variety not stated); R71; JMPR, 1999	WP 50%	1.1		2	15	1.3
		1.7		2	0	0.47
		1.1, 1.2		2	0	0.31
		2.7		1	5	0.64
		5.4		1	14	0.59
	DP 8%	4.5		2	0	0.25
	DP 4%	1.6		1	0	0.39
USA, 1963, (variety not stated); R73; JMPR, 1999	WP 50%	1.1		3	8	0.17
		1.1		4	0	1.2
		1.1		5	4	1.5
		1.1		6	0 15	2.4 1.4
		1.1		7	0	1.9
USA, 1995, (variety not stated); 95010; JMPR, 1999	WP 75%	0.84		4	3	0.2
Post-harvest application						
USA, 1963, (variety not stated); TOM95; JMPR, 1999	WP 75%		0.9 [1/]	1	0	1.12, 1.56, 2.23, 2.74 2.83, 2.84, 3.39 (7 trials) (1 l of solution/2.4 t)
			0.9 [1/]	1	0	2.48, 2.76, 2.81, 2.93, 4.24, 4.35 (6 trials) (1 l of solution/1.8 t)

Location, year, (variety); reference(s)	Application				Days after application	Residues (mg/kg)
	Form	kg a.i./ha	kg a.i./hl	No.		
			1.2 [1/]	1	0	2.8, 4.5 (2 trials) (1 l of solution/2.4 t)
			1.8	1	0	3.68, 4.11, 4.49, 5.73 (4 trials) (1 l of solution/2.4 t)

[1/] Application by controlled droplet applicator. Solution included wax.
C = control samples.

Table 17. Residues of dicloran in lettuce from supervised pre-harvest trials in Brazil and the USA.

Location, year, (variety); reference(s)	Application				Days after application	Residues (mg/kg)
	Form	kg a.i./ha	kg a.i./hl	No.		
Ibipora/PR, Brazil, 2001, (Taina, crisp-head type); 633-4225	WP 73.2%	2.343	0.293	2	14 [1/] C	<u>≤0.10</u> <0.10
Arthur Nogueira/SP, Brazil, 2001, (Lisa Sem Rival, head type); 633-4225	WP 73.2%	2.928	0.293	2	14 [1/] C	<u>0.16</u> <0.10
Iracemapolis/SP, Brazil, 2001, (Lisa Sem Rival, head); 633-4225	WP 73.2%	2.048	0.293	2	14 [1/] C	<u>0.51</u> <0.10
USA, 1972, (glasshouse) (season not reported); R268 [4/]; JMPR, 1999	FU	1.6		1	0.5 1 2 3 4	16.7 22.0 13.5 10.5 [2/] 8.0 [3/]
USA, 1964, (variety not stated); R112; JMPR, 1999	WP 75%		0.36	2	25	0.05
		3.4		1	26	0.13

[1/] Half-mature.
[2/] One of three replicate samples showed <6.0 mg/kg and, for the calculation of the mean, a value of 6.0 was used.
[3/] Two of three replicate samples showed <6.0 mg/kg and, for the calculation of the mean, a value of 6.0 was used.
[4/] LOQ = 6.0 mg/kg.
C = control samples.

NATIONAL MAXIMUM RESIDUE LIMITS

National MRLs were reported by the Netherlands (Table 18). However, it was reported that dicloran is not authorized for use on agricultural crops in the Netherlands.

Table 18. National MRLs for dicloran.

Country	Commodity	MRL, mg/kg
Netherlands	Apricots	10
	Beans (with pods)	2
	Blackberries	5
	Broad-leaf endive	3
	Carrots	10
	Cherries	15
	Currants (red, black and white)	5
	Gherkins	0.5
	Grapes (table and wine)	10
	Kiwi fruits	10
	Luttuce	3
	Nectarines	10
	Other fruiting vegetables (except sweet corn)	0.3
	Peaches	15
	Plums	10
	Raspberries	10
	Strawberries (other than wild)	10
	Tomatoes	0.5
	Witloof	1
	Other	0.01*

* MRL set at or about the LOQ. The Netherlands reported that the analytical method used, based on determination of residues by GC-ITD, has LOQs of 0.03-0.05 mg/kg for most of the commodities identified.

APPRAISAL

Dicloran, a fungicide, was first evaluated for toxicology and residues in 1974 and subsequently in 1977. The compound was again evaluated by the Meeting in 1998 for toxicology and residues under the CCPR Periodic Review Programme. The 1998 JMPR changed the ADI from 0-0.03 to 0-0.01 mg/kg body weight and concluded that an acute RfD was unnecessary. It recommended that the definition of the residue for compliance with MRLs and for the estimation of dietary intake should be dicloran and indicated that the residue was fat-soluble. It also estimated revised maximum residue levels for carrots and bulb onions, while recommending withdrawal of the existing CXLs for grapes, head lettuce, peaches, plums (including prunes), strawberries and tomatoes. The 33rd CCPR, in 2000, decided to retain for four years the MRLs recommended for withdrawal in accordance with the procedures of the Periodic Review Programme, pending the submission of additional data on these commodities.

The Meeting received information on residues in rotational crops, an analytical method, use patterns, and residue trials on nectarines, peaches, plums, grapes, strawberries, tomatoes and lettuce.

Rotational crops

When dicloran was applied to loamy sand at a rate equivalent to 3 kg ai/ha, the applied radioactivity remained mainly in the soil. Both the TRR and dicloran were significantly higher in the first growing period than the second and third in all crops. The highest residues were found in leaves or straw of lettuce, sugar beet and wheat but the TRRs decreased sharply after the first growing period. Unchanged dicloran found in plant extracts was less than 10% of the TRR in the first growing period and was at or below the limit of quantification in the second and third periods.

When dicloran was sprayed on the surface of sandy loam soil at a rate of 14.8 kg ai/ha, the average TRR in harvested crops (lettuce, turnip and wheat) was highest in the 120-day rotation and lowest in the 365-day rotation. On average, acetonitrile extracts, dichloromethane extracts and acid and alkaline extracts contained about 20%, 3% and 20% of the TRR, respectively. Unextracted fractions contained about 30% of the TRR, which was found to be associated with cell wall components. Dicloran was observed in most samples from days 30, 120 and 365, at generally decreasing levels. The presence of dicloran metabolites was confirmed throughout the course of the study: 4-amino-2,6-dichlorophenol, 4-amino-2,6-dichloroacetanilide, 3,5-dichloro-4-hydroxyacetanilide, 4-amino-3,5-dichloroacetanilide, 2,6-dichloro-4-hydroxyaniline, 2,6-dichloro-4-nitrophenol, 2,6-dichlorophenol and 2,6-dichloroaniline were identified.

In a study of the uptake of dicloran from soil by rotational crops under actual field conditions, dicloran was applied at a rate of 4.4 kg ai/ha and lettuce, mustard, radish, wheat and sorghum were planted 30, 120 and 360 days after the application. In most crops, dicloran concentrations were below the limit of quantification (<0.05 mg/kg). The highest dicloran concentrations were found in radish roots of the 30-day rotation at 0.278 and 0.243 mg/kg.

Analytical methods

The validity of the method used for the determination of dicloran in milk, eggs and tissues from goats and hens in metabolism studies was checked. Egg white and chicken muscle samples, with and without fortification, were extracted with acetone/water (6:1), and partitioned with hexane/ethyl acetate (9:1). Milk and fat samples were extracted with acetone/water (7:1) followed by extraction of the aqueous layer with hexane and partition into acetonitrile. After a clean-up step including a solid-phase extraction on a diol column, dicloran was eluted with toluene and determined by gas chromatography with an electron-capture detector. Recovery from the fortified samples was 93.0-106.7%, showing that the method can be satisfactorily used for the determination of dicloran in these samples. However, comparison of the analytical results with those from radio-analysis indicated that while the average recoveries from egg white, chicken muscle and goat fat were above 80%, that from goat milk was 47.8%.

Residues from supervised trials

The Meeting received the results of supervised trials on nectarines, peaches, plums, strawberries and lettuce in Brazil, grapes in Brazil and Mexico and tomatoes in Italy, all conducted after the last evaluation by the 1998 JMPR. The Meeting also considered the results of supervised trials on these crops which had been evaluated by the 1998 JMPR, against GAP reported to the current Meeting.

Nectarines. Three new post-harvest trials were conducted in Brazil in 2001. The conditions (in 0.087-0.089 kg ai/hl dip) were in accordance with GAP in Chile for post-harvest application to nectarines (dip or immersion in 0.09 kg ai/hl). The residues were 2.3, 2.3 and 5.5 mg/kg.

The 1998 JMPR evaluated pre-harvest, post-harvest, and combined pre- and post-harvest trials carried out in the USA in 1968. One pre-harvest trial (PHI 1 day) did not comply with GAP in the USA which requires a PHI of 10 days. The post-harvest trials and combined pre- and post-harvest trials were not in compliance with any GAP.

One Australian post-harvest trial (0.075 kg ai/hl, SC) complied with Chilean post-harvest GAP but only the surface residue was measured and could not be used to estimate a maximum residue level.

Peaches. Two new pre-harvest supervised trials were conducted in Brazil in 2001. The conditions (0.146 kg ai/hl, 3 applications at an interval of 15 days, 1-day PHI) were in accordance with GAP in Argentina (0.15 kg ai/hl, 3 applications, 1-day PHI). The residues were 2.7 and 4.8 mg/kg.

A pre-harvest trial, and post-harvest and combined pre- and post-harvest trials conducted in the USA in 1966, 1988 and 1996 were evaluated by the 1998 JMPR. The pre-harvest trial with a PHI of 4 days was not in compliance with US GAP which requires a PHI of 10 days. Four combined trials in 1988 and 1996 with a post-harvest rate of 0.09 kg ai/hl were in accordance with post-harvest GAP in Argentina (0.09-0.11 kg ai/hl spray or immersion) and in Chile (0.09 kg ai/hl dip or immersion) but in two of these trials the last pre-harvest application was made one day before harvest while US pre-harvest GAP requires 10 days. The residues from two other trials in conformity with US GAP (pre-harvest) and Argentinean and Chilean GAP (post-harvest) were 5.3 and 5.8 mg/kg. Of two combined trials and four post-harvest trials in 1966, a post-harvest trial at an application rate of 0.15 kg ai/hl and another at 0.06 kg ai/hl approximated GAP in Argentina. The residues were 2.1 and 2.5 mg/kg.

Three Australian post-harvest trials (1973) reported only surface residues and could not be used for estimating a maximum residue level, and one pre-harvest (1964) and one post-harvest trial (1966) in Canada did not comply with any GAP.

The residues data from post-harvest trials on nectarines and peaches were mutually supportive. The combined residues data (median underlined) were 2.1, 2.3, 2.3, 2.5, 2.7, 4.8, 5.3, 5.5 and 5.8 mg/kg. The Meeting therefore estimated a maximum residue level of 7 mg/kg Po and an STMR of 2.7 mg/kg for nectarines and peaches.

Plums. Three new pre-harvest trials were carried out in Brazil in 2001. The conditions (0.146 kg ai/hl, 3 applications at an interval of 15 days, 1-day PHI) were in compliance with pre-harvest GAP in Argentina (0.15 kg ai/hl, 3 applications, 1-day PHI). The residues were <1.0, 1.1 and 1.4 mg/kg.

Four combined and one pre-harvest trial in the USA in 1986 and 1995 were not in accordance with any GAP reported to the current Meeting.

The Meeting concluded that there were too few valid trials to estimate a maximum residue level.

Grapes. Three new trials in Brazil and four in Mexico in 2001 were reported to the Meeting. Although trials were conducted in Brazil (0.256 kg ai/hl, 5 applications with an interval of 1 month, 1-day PHI), no GAP was reported for Brazil and they did not comply with GAP in Argentina (0.19 kg ai/hl, 7-day PHI).

Trials carried out in Sonora and Baja California, Mexico (2.25 kg ai/ha, 3 applications at an interval of 15 days, 1-day PHI) were in accordance with GAP in the USA (1.7-3.9 kg ai/ha for WP or 2.0 kg ai/ha for DP, 1-day PHI; applicable to grapes grown west of the Rocky Mountains only). The residues were 0.36, 0.83, 0.95 and 1.5 mg/kg.

The 1998 JMPR evaluated data from US supervised trials in California in 1967, 1984 and 1995. One trial in 1967 and two trials in 1984 with a WP application at 2.2 kg ai/ha followed by 1-3 dust applications at 2.0 kg ai/ha with 1-day PHI were comparable with US GAP (1.7-3.9 kg ai/ha for WP or 2.0 kg ai/ha for DP, 1-day PHI). The residues were 0.29, 0.62 and 6.0 mg/kg.

One USA trial in 1995, with a WP application at 4.5 kg ai/ha and 3-day PHI, approximated US GAP. The residue was 1.0 mg/kg.

The combined residues from Mexican trials and valid US trials were (median underlined) 0.29, 0.36, 0.62, 0.83, 0.95, 1.0, 1.5 and 6.0 mg/kg. The Meeting estimated a maximum residue level of 7 mg/kg and an STMR of 0.89 mg/kg.

Strawberries. Three new trials were conducted in Brazil in 2001 at a rate of 0.256 kg ai/hl, with 4 (2 trials) or 11 applications (1 trial) and a PHI of 1 day. No GAP for strawberries was reported for Brazil or Argentina.

A number of trials were carried out in the USA in 1963 but no GAP for strawberries was reported for the USA.

The Meeting concluded that there were insufficient data to estimate a maximum residue level.

Tomatoes. Three new trials were conducted in Italy in 2001 at a rate of 0.095 kg ai/hl which complied with GAP in Italy (0.07-0.12 kg ai/hl). The residues at a PHI of 20 days were <0.01, 0.04 and 0.08 mg/kg.

Trials conducted in the USA in 1962 (greenhouse) and in 1963 and 1995 (field) were not in compliance with US GAP (0.84 kg ai/ha, 4 applications, PHI of 10 days). One trial in the UK in 1972 (greenhouse) reported only residues in the surface wash.

The Meeting concluded that there were too few valid trials to estimate a maximum residue level.

Lettuce. Three new pre-harvest trials were conducted in Brazil in 2001. The conditions (0.293 kg ai/hl, 2 applications at an interval of 7 days, 14-day PHI) were in accordance with pre-harvest GAP in Argentina (0.28 kg ai/hl, 10-day PHI). The residues were <0.10, 0.16 and 0.51 mg/kg.

Three field trials and one greenhouse trial were conducted in the USA in 1964 and 1972 respectively. While US GAP requires a PHI of 14 days, samples were taken only up to 4 days or at 25-26 days.

The Meeting concluded that there were too few valid trials to estimate a maximum residue level.

Processing studies

The 1998 JMPR estimated processing factors for grapes to be 1.1 to juice, 1.5 to wet pomace and 0 to sun-dried grapes, so STMR-Ps were calculated to be 0.98 mg/kg for grape juice, 1.3 mg/kg for wet pomace and 0 mg/kg for sun-dried grapes.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed in Table 19 are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with MRLs: *dicloran.*
Definition of the residue for estimation of dietary intake: *dicloran.*
The residue is fat-soluble.

Table 19. Summary of recommendations.

Commodity		Recommended MRL, mg/kg		STMR or STMR-P, mg/kg
CCN	Name	New	Previous	
FB 0269	Grapes	7	W	0.89
FS 0245	Nectarines	7 Po	-	2.7
FS 0247	Peaches	7 Po	W	2.7
JF 0269	Grape juice			0.98

Commodity		Recommended MRL, mg/kg		STMR or STMR-P, mg/kg
CCN	Name	New	Previous	
	Grape pomace, wet			1.3
	Grapes, sun-dried			0

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) were calculated for the five GEMS/Food regional diets, using STMRs for 3 commodities estimated by the current Meeting and 2 estimated by the 1998 JMPR, and STMR-Ps for two processed commodities (Table 20). The ADI allocated by the 1998 JMPR was 0-0.01 mg/kg bw. The calculated IEDIs were 0-30% of the maximum ADI. The Meeting concluded that the intake of residues of dicloran resulting from the uses considered by the 1998 JMPR and the current JMPR was unlikely to present a public health concern.

Table 20. International Estimated Dietary Intakes (IEDIs) of dicloran for the five GEMS/Food regional diets (ADI = 0-0.01 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
VR 0577	Carrot	6.11	2.8	17.1	2.5	15.3	0.0	0.0	6.3	38.5	22.0	134.4
FB 0269	Grapes (fresh, wine, excluding dried grapes)	0.89	15.8	14.1	1.0	0.9	0.0	0.0	1.3	1.2	13.8	12.3
DF 0269	Grapes, dried (= currants, raisins and sultanas)	0	0.3	0.0	0.0	0.0	0.0	0.0	0.3	0.0	2.3	0.0
JF 0269	Grape juice	0.98	-	-	-	-	-	-	-	-	-	-
FS 0245	Nectarine	2.7	1.3	3.4	0.3	0.7	0.0	0.0	0.4	1.1	6.3	16.9
VA 0385	Onion, bulb	0.1	23.0	2.3	11.5	1.2	7.3	0.7	13.8	1.4	27.8	2.8
FS 0247	Peach	2.7	1.3	3.4	0.3	0.7	0.0	0.0	0.4	1.1	6.3	16.9
Total intake (µg/person)=				40.2		18.7		0.7		43.2		183.2
Bodyweight per region (kg bw) =				60		55		60		60		60
ADI (µg/person)=				600		550		600		600		600
% ADI=				6.7		3.4		0.1		7.2		30.5
Rounded % ADI=				7		3		0		7		30

Short-term intake

The 1998 JMPR agreed that an acute RfD was unnecessary for dicloran. The Meeting therefore concluded that the short-term intake of dicloran residues was unlikely to present a public health concern.

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