### CARBARYL (008)

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### **EXPLANATION**

Carbaryl was evaluated for toxicology in 1963, 1965, 1966, 1967, 1969, 1973 and as part of the CCPR Periodic Review Programme in 1996. It was reviewed for residues in 1966, 1967, 1968, 1969, 1970, 1973, 1975, 1976, 1977, 1979 and 1984. It was reviewed again by the WHO Core Assessment Group in 2001. The 2001 toxicological evaluation established an ADI of 0.008 mg/kg body weight and an acute reference dose of 0.2 mg/kg bw but the scheduled evaluation of the residue aspects was postponed until the present Meeting. The present evaluation is within the CCPR Periodic Review Programme.

Data on the metabolism of farm animals and plants, environmental fate in soil and water, analytical methods, use patterns, residues in food in commerce or at consumption and national MRLs were reported to the Meeting by the manufacturer. Supervised trials on citrus, pome and stone fruits, grapes, olives, egg plant, tomato, sweet corn, pepper, lettuce, spinach, soya beans, carrots, garden and sugar beets, turnips, sweet potato, asparagus, maize (field corn), rice, sorghum, wheat and sunflower were reported together with processing studies on various crops and a cattle feeding study.

The Government of Australia submitted information on GAP and residues in food in commerce or at consumption, the Government of Thailand submitted information on GAP, summaries of analytical methods and the results of supervised trials on cabbages, chilli peppers, sweet corn, kale and dry soya beans, and the Government of The Netherlands submitted information on GAP and national MRLs.

### **IDENTITY**

Common name: carbaryl

CAS registry no.: 63-25-2

Chemical name

IUPAC: 1-naphthyl methylcarbamate

C.A. 1-naphthalenyl methylcarbamate

### Structural formula:

Molecular formula: C<sub>12</sub>H<sub>11</sub>NO<sub>2</sub>

Molecular weight: 201,2

# Physical and chemical properties

Pure active ingredient

Appearance: White to light tan solid (Anon., 1984)

log Pow

Melting point: 142°C

Octanol/water partition

coefficient

HPLC method 1.85 (Seymour, 1988)

Shake-flask method 2.36 at  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$  (Guyot and Loken, 1989)

Hydrolysis:

\* pH 5 (buffered) at 25°C Stable (Carpenter, 1990)

\* pH 7 (buffered) at 25°C half-life  $\sim$  12 days \* pH 9 (buffered) at 25°C half-life  $\sim$  3.2 hours

Photolysis: half-life 12 days (of 12 h irradiation per (Das, 1990b)

day) ( $k=-6.713 \times 10^{-2} \text{ days}^{-1}$ )

Dissociation constant: pK<sub>a</sub> 10.4 at 24°C (Carpenter, 1990)

**Technical Material** 

Minimum purity: 99% (990 g/kg) Water: 6% (6.0 g/kg)

Main impurities: 1-naphthol, 1-naphthyl 2,4-

dimethylallophanate, toluene

Vapour pressure:  $4.16 \times 10^{-7} \text{ hPa at } 23.5^{\circ}\text{C}$  (Hoffman, 1989)

Henry's Law constant: 7.3 x 10<sup>-10</sup> atm.m<sup>3</sup>.g<sup>-1</sup>.mol<sup>-1</sup> at 25°C (Scarborough, 1989)

Solubility:

Water (at 25°C) 113 mg/l (Andrawes and Bailey,

1978b)

Organic solvents (Long, 1987a)

 dichloromethane
 24.26 g/100 ml

 hexane
 0.0214 g/100 ml

 methanol
 7.96 g/100 ml

Relative density: 0.5646 (loose bulk density) (Long, 1987b)

0.7842 (packed bulk density)

Stability:

Thermal Stable when stored for 28 days under (Siemann, 1992)

ambient temperature ( $\sim 23$ °C) or elevated temperature ( $\sim 54$ °C)

Oxidising potential Stable for 28 days when mixed with

aluminium, iron and stainless steel metals; and mixed with iron (III), aluminium, zinc and copper ions

Flammability Autoflammability: 625°C

Flash point: 193°C

#### METABOLISM AND ENVIRONMENTAL FATE

### **Animal metabolism**

Rats. Four groups of male and female animals were dosed as follows.

Group A: Single intravenous (IV) low dose (1.02 mg/kg), 5 animals/sex

Group B: Single oral low dose (1.21 mg/kg), 5 animals/sex

Group C: Multiple oral low doses (14 daily unlabelled doses of 1.0 mg/kg followed by a single

labelled dose of 1.21 mg/kg on the 15th day), 5 animals/sex

Group D: Single oral high dose (48.0 mg/kg), specific activity of 8,435 dpm/µg, 5 animals/sex.

In a preliminary study two male and two female rats were treated with single oral low doses of [14C]carbaryl (0.966 mg/kg). No radioactivity was detected in the expired carbon dioxide and the organic volatiles trap contained <0.01% of the administered dose. In the main study organic volatile compounds and expired carbon dioxide were therefore not collected, but urine and faeces were collected from all animals at specified intervals. The rats were killed 7 days later and tissue samples analysed for total 14C residues.

Recoveries of <sup>14</sup>C from all groups ranged from 96.1% to 104% of the administered dose and most of the dose was excreted in the urine (Table 1), 70-80% of it within 12 hours from the low-dose groups and within 24 hours for the high-dose group.

	Table 1. Radioactivity recovered after the administration of	$\sum_{i=1}^{14} C$ carbaryl to rats (Struble, 1994a).
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Crown/Sov	% of radioactive dose <sup>1</sup>						
Group/Sex	Tissues	Residual Carcass	Faeces	Urine <sup>2</sup> (0-168 hour)	Total		
A/male	0.02	0.13	10.2	90.0	100		
A/female	0.02	0.34	8.71	88.6	97.7		
B/male	< 0.01	0.10	9.06	92.1	101		
B/female	0.01	0.23	8.40	91.5	100		
C/male	< 0.01	0.15	8.57	95.0	104		
C/female	< 0.01	0.21	7.68	95.0	103		
D/male	< 0.01	0.60	12.5	84.5	97.6		
D/female	0.01	0.90	6.98	88.2	96.1		

<sup>&</sup>lt;sup>1</sup> Means of residues in all animals for each group and sex.

A maximum of 0.02% of the dose was recovered from tissues of the low-dose groups, with concentrations of <0.01 mg/kg as carbaryl. All the high-dose tissue samples contained <0.06 mg/kg except the carcass (0.264 mg/kg males and 0.436 mg/kg females), kidneys (0.188 mg/kg males and 0.333 mg/kg females) and blood (0.103 mg/kg males and 0.170 mg/kg females).

Metabolites were identified by co-elution with reference standards using HPLC and two-dimensional TLC and/or by positive or negative electrospray LC-MS. The profiles of radioactivity in composite urine and faeces samples of males and females in all groups indicated that the metabolism of [\frac{14}{C}]carbaryl was similar regardless of the route of administration, dose level or sex. The main metabolite in the faeces was 5,6-dihydro-5,6-dihydroxycarbaryl. A small amount of (apparently unabsorbed) carbaryl was found in the faeces of the high-dose rats.

Composite urine samples from all groups were extracted to isolate intact conjugated metabolites and extractable products after enzymatic or acid hydrolysis for quantification. Free carbaryl represented only 0.2 % of the administered dose and 5-hydroxycarbaryl, 1-naphthol and their conjugates were the main metabolites (Table 2).

Table 2. Summary of <sup>14</sup>C-labelled metabolites isolated from rat group D male urine.

Compound		Percentage of administered dose			
		Free metabolites	netabolites Conjugated metabolites		Total
			Enzyme-hydrolysed	Acid-hydrolysed	
carbaryl		0.2	Not found	2.7	2.9
Dihydro dihydroxycarbaryl	5,6-dihydro-5,6- dihydroxycarbaryl	1.7	6.5	Not found	8.2
isomers	3,4-dihydro-3,4- dihydroxycarbaryl	0.9	Not found	Not found	0.9

<sup>&</sup>lt;sup>2</sup> Includes cage rinse, wash (1% trisodium phosphate solution) and wipe

Compound		Percentage of administered dose				
	Free metabolites	Conjugated n	netabolites	Total		
		Enzyme-hydrolysed	Acid-hydrolysed			
1-naphthyl sulfate	0.9	Not found Not found		0.9		
N (hydroxymethyl)hydroxycarbaryl	1.3	4.4	4.4 Not found			
1,5-dihydroxynaphthalene	0.3	Not found 2.7		3		
hydroxy-napthylcarbamate	Not found	Not found 0.5		0.5		
5-hydroxycarbaryl	4.3	2.1	6.4	12.8		
4-hydroxycarbaryl	0.9	3.1 2.3		6.3		
1-naphthol	0.3	12.6 1.6		14.5		
dimer of 1 ,4-naphthoquinone <sup>1</sup>	Not found	Not found	1.5	1.5		
	10.8	28.7	17.7	57.2		

<sup>&</sup>lt;sup>1</sup> Artefact formed from 1,4-naphthoquinone during sample preparation.

It appears that three routes of biotransformation take place in rats: hydroxylation of the *N*-methyl group to yield (*N*-hydroxymethyl)carbaryl; hydrolysis of the carbamate ester to form 1-naphthol, and epoxidation at the 3,4- or 5,6-positions of the naphthalene ring with subsequent metabolism to dihydrodihydroxycarbaryl and conjugation with glutathione (Figure 1).

<u>Dairy cattle</u>. No full studies of the metabolism of carbaryl in dairy cattle were reported. A review of published papers and a summary of studies reported to previous JMPRs is therefore included here.

In two early studies carbaryl at 450 ppm in the diet was fed to dairy cattle for 2 weeks and carbaryl, 1-naphthol or conjugates of 1-naphthol were not present in the milk (Gyrisco *et al.*, 1960; Whiterhurst *et al.*, 1963). In another study cattle were fed a diet containing 200 ppm of carbaryl for 27 days and their tissues were reported to be free of carbaryl residues (Claborn *et al.*, 1963).

The colorimetric method used in these studies to detect carbaryl and 1-naphthol was later shown to be inadequate for the detection of other metabolites in meat and milk (Dorough, 1967). Dorough and Cassida (1964) demonstrated that the residues in the milk of a treated goat consisted mainly of conjugated carbamate metabolites. Several unconjugated metabolites were also isolated, the main one later identified as 5,6-dihydro-5,6-dihydroxycarbaryl (Leeling and Casida, 1966).

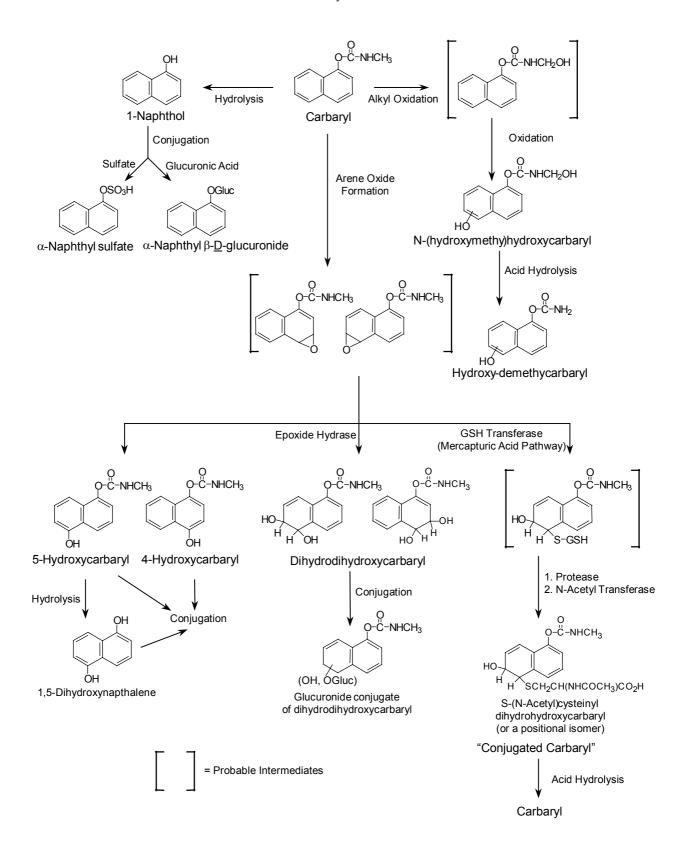


Figure 1. Proposed metabolic pathways of carbaryl in rats.

In another study, [1-naphthyl-<sup>14</sup>C]carbaryl was fed to lactating cows at levels of 0.15, 0.43 and 1.35 mg/kg body weight (equivalent to 10, 30 and 100 ppm in the feed) for 14 days (Dorough, 1971). Equilibrium between intake and elimination was reached within 2 days. At each feeding level, approximately 0.2% of the dose was secreted in the milk at each sampling day with <sup>14</sup>C concentrations equivalent to 1/400 of those in the diet. The major metabolites in the milk were 5,6-dihydro-5,6-dihydroxycarbaryl, 1-naphthyl sulfate and the sulfate conjugate of 5-methoxy-6-hydroxycarbaryl (Table 3).

Table 3. Metabolites in the milk of dairy cows after feeding with [1-naphthyl-<sup>14</sup>C]carbaryl at 100 ppm for 14 days (Dorough, 1971).

Compound	mg/kg as carbaryl	% of total in milk
carbaryl	0.017	6
5,6-dihydro-5,6-dihydroxycarbaryl	0.094	34
3,4-dihydro-3,4-dihydroxycarbaryl	0.013	5
5-hydroxycarbaryl	0.003	1
5,6-dihydro-5,6-dihydroxy-1-naphthol	0.009	3
α-naphthyl sulfate	0.072	26
Sulfate conjugate of 5-methoxy-6-hydroxycarbaryl	0.063	23
5-methoxy-1,6-naphthalenediol	0.007	2
Total	0.278	0.22% of TRR

Cows were slaughtered 18 hours after the last dose and the distribution of labelled residues (Table 4) and the nature of residues found in tissues and organs (Table 5) were determined. The highest residues at all dose levels were in the kidneys, where the main identified metabolite was naphthyl sulfate. The highest levels of carbaryl were in muscle and liver. 5,6-dihydro-5,6-dihydroxycarbaryl was the main identified metabolite in muscle and heart (Table 5).

Table 4. Total <sup>14</sup>C in the tissues of cows fed [1-naphthyl-<sup>14</sup>C]carbaryl for 14 days.

Tissues	mg/kg [14C]carbaryl equivalents					
Tissues	10 ppm	30 ppm	100 ppm			
Kidney	0.095	0.531	1.003			
Liver	0.033	0.100	0.411			
Lung	0.020	0.064	0.207			
Muscle	0.009	0.031	0.104			
Heart	0.012	0.038	0.095			
Fat	0.000	0.015	0.025			
Blood	0.008	0.036	0.141			

Table 5. Radioactive residues in tissues of a cow fed 100 ppm [1-naphthyl-<sup>14</sup>C]carbaryl in the diet for 14 days (Dorough, 1971).

Commont	Percentage of total radioactivity in sample						
Compound	Kidney	Liver	Lung	Muscle	Heart	Blood	
carbaryl	3.3	9.2	2.1	17.0	3.7	0	
5,6-dihydro-5,6-di-hydroxycarbaryl	4.5	3.0	8.8	38.6	31.3	22.0	
5,6-dihydro-5,6-dihydroxy-1-naphthol	1.8	4.1	0	0	4.9	2.0	
α -naphthyl sulfate	29.3	4.1	27.3	0	4.0	51.8	
Water-soluble unknowns	43.2	32.9	47.5	30.6	41.8	7.1	
Unextractable unknowns	17.9	46.7	14.3	13.8	14.3	17.1	

Hens. Struble (1994b) dosed laying hens orally with [1-naphthyl-¹⁴C]carbaryl at a rate of 1.32 mg/animal/day twice a day for 7 consecutive days, equivalent to approximately 8.8 ppm in the diet (group 2, with 2 hens) and 10.5 ppm (group 3, with 10 hens) based on food consumption during the test period. Ten control hens (group 1) were given capsules containing cellulose and acetone (evaporated). Eggs from group 3 were collected twice daily and hens were killed approximately 6 hours after the last dose for collection of tissues and gastrointestinal (GI) tract contents. Expired ¹⁴CO₂ and volatile organic compounds were collected from group 2 hens every 24 hours, and excreta and pan paper washes/wipes from all groups once daily.

Most of the radioactivity in both treated groups was recovered from the excreta (average 97.7% of the TRR), none was detected as  $^{14}\text{CO}_2$  or expired volatile organic compounds from the 2 hens of group 2. Total radioactivity in the tissues was only 0.17% of the administered dose. The TRR in group 2 was highest in the kidneys and liver and lowest in abdominal fat, breast and thigh muscle (Table 6).

Table 6. Distribution of radioactivity after feeding [14C]carbaryl to laying hens for 7 days at 10.5 ppm.

Sample	% of TRR	μg of [14C]carbaryl equivalents/g
Fat (abdominal)	< 0.01	0.014
Kidneys	0.03	0.268
Liver	0.08	0.187
Muscle (breast)	0.02	0.014
Muscle (thigh)	0.01	0.016
Skin with fat	0.03	0.055
Total in tissues	0.17	-
Blood	-	0.184
Egg whites	0.02	0.005 to 0.009 <sup>1</sup>
Egg yolks	0.1	0.002 to 0.176 <sup>1</sup>
Excreta	94.62 <sup>2</sup>	-
Total recovered	95.1	

<sup>&</sup>lt;sup>1</sup> during the 7-day dosing period.

Residues in yolks, liver, thigh muscle and abdominal fat were extracted with organic solvent. Glucuronide or sulfate conjugates in liver and muscle were first hydrolysed with  $\beta$ -glucuronidase/aryl

<sup>&</sup>lt;sup>2</sup> Includes pan paper wash and GI tract contents.

sulfatase and the hydrolysate extracted with organic solvents. Remaining solids or aqueous residues containing >10% of the TRR in a sample or >0.01 mg/kg [\frac{14}{C}]carbaryl equivalents were subjected to acid or base hydrolysis and further extracted. Radioactive metabolites in the extracts were characterized by high-performance liquid chromatography (HPLC) and one- and two-dimensional TLC (Table 7).

Table 7. <sup>14</sup>C residues in the eggs and tissues of laying hens fed with [<sup>14</sup>C]carbaryl in the diet for 7 days.

Compound	Egg ye	olks	Live	er	Thigh n	nuscle	Abdominal	fat
	% of TRR	mg/kg <sup>1</sup>	% of TRR	mg/kg <sup>1</sup>	% of TRR	mg/kg <sup>1</sup>	% of TRR	mg/kg <sup>1</sup>
Carbaryl	1.7	0.003	0.8	0.001			26.9	0.004
N-hydroxymethylcarbaryl	3.6	0.006						
α-Naphthyl sulfate	44.2	0.078						
α-Naphthyl-β-D- glucuronide	2.5	0.004						
Demethylcarbaryl			9.2	0.017	0.7	< 0.001		
1-Naphthol	NQ <sup>2</sup>	NQ					39.1	0.005
S-(5-hydroxynaphthyl)cysteine			4.6	0.009				
Unknown 1			4.2	0.008	8.4	0.001	3.7	0.001
Unknown 2	13.5	0.024						
Unknown 3					19.4	0.003		
Unknown 4	0.7	0.001						
Unknown 5			1.8	0.003				
Unknown 6			6.8	0.013				
Unknown 7			8.2	0.015				
Other Unknowns (<10% of TRR)	26.6 (19) <sup>4</sup>	0.047	9.9 (4)	0.019	3.9 (7)	< 0.001	13.7 (7)	0.002
Extracts <sup>5</sup>	9.1 (5)	0.016	24.8 (4)	0.046	28.7 (5)	0.005	9.6 (3)	0.001
Insoluble			15.2 (1)	0.028	37.5 (1)	0.006	8.7 (2)	0.001
Total identified or characterized	101.9	0.178	85.5	0.158	98.6	0.015	101.7	0.004

<sup>&</sup>lt;sup>1</sup> As carbaryl

[14C]carbaryl was highest in extracts of abdominal fat (0.004 mg/kg [14C]carbaryl equivalents or 26.9% of the TRR), and was present in egg yolks and liver. The metabolite 1-naphthol was the major component in fat (39.1% of the TRR) and as sulfate and glucuronide conjugates in egg yolks. An *N*-demethylated metabolite, demethylcarbaryl, was present in the liver and muscle. Liver contained *S*-(5-hydroxynaphthyl)cysteine, likely to be a product of conjugation of carbaryl or 1-naphthol with glutathione (GSH) via the mercapturic acid pathway with subsequent secondary metabolism in the tissue resulting in the cysteine conjugate. Unidentified polar metabolites were found in the liver and thigh muscle, probably formed from the catabolism of metabolites in mercapturic acid pathway. A non-polar mixture of a maximum of 10 metabolites (Unknown 1), was found in fat, liver and muscle, probably formed by reactions of 1,4-naphthoquinone (from 1,4-dihydroxynaphthalene) with other compounds, either *in vivo* or as an artefact during the sample extraction, hydrolysis, or isolation. Egg yolks and liver contained other non-polar metabolites

<sup>&</sup>lt;sup>2</sup> NQ: not quantified

<sup>&</sup>lt;sup>3</sup> Totals for chromatographically isolated unknowns were reported but not quantification of individual components

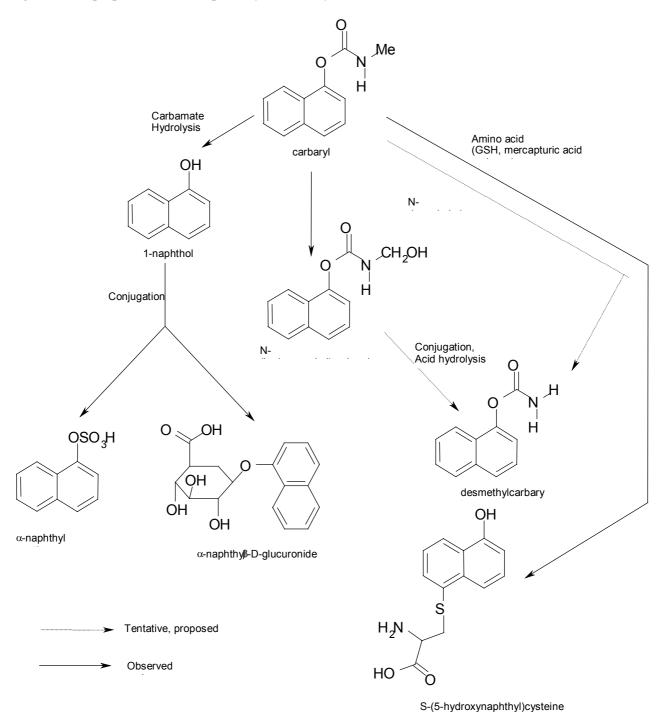
<sup>&</sup>lt;sup>4</sup> Numbers in parentheses are minimum number of components in the fractions

<sup>&</sup>lt;sup>5</sup> In extracts after 6M NaOH hydrolysis and extraction with methyl *tert*-butyl ether, attempts at HPLC analysis were unsuccessful owing to large amounts of co-extracted material

(Unknowns 2, 4 and 5). Unextractable radioactivity in the liver accounted for 15.2% of the TRR after enzymic, acid and base hydrolyses; no parent compound or 1-naphthol could be found after the exhaustive hydrolyses.

The metabolic pathways of  $[^{14}C]$  carbaryl in hens have routes that include carbamate hydrolysis, hydroxylation, N-demethylation, formation of sulfate and glucuronide conjugates and conjugation with GSH resulting in the formation of S-(5-hydroxynaphthyl)cysteine (Figure 2).

Figure 2. The proposed metabolic pathways of carbaryl in hens.



#### Plant metabolism

The metabolism of [1-naphthyl-<sup>14</sup>C]carbaryl was studied in radish, lettuce, and soya bean plants and apples after multiple foliar applications. Extracts were radioassayed by LSC and analysed by TLC, and organosoluble residues by HPLC. <sup>14</sup>C residues in the initial aqueous extract were separated into major fractions by preparative HPLC and these fractions subjected to HCl and β-glucosidase hydrolysis for analysis by HPLC confirmed by TLC. Unhydrolysed aqueous fractions were analysed by comparing UV and radiometric chromatograms, and some fractions were analysed by MS.

Radish. Greenhouse-grown radishes were treated five times at 9-11 day intervals with [\frac{14}{C}]carbaryl (radiochemical purity >98%; mixed with unlabelled carbaryl to a final specific activity of 29,145 dpm/\(\pi\)g) at a rate of 2.0 kg/ha per application (Harsy, 1994b). Plants were harvested 7 days after the last application and separated into roots and tops. Tops were rinsed with acetone/water (50:50). Rinsed tops and roots were extracted, partitioned, and the total radioactivity in the water-soluble, organosoluble and unextractable internal residues determined (Table 8). Residues in the roots represented only 2.5% of external and internal residues in the tops.

Of the radioactive components in the plants, including external, organosoluble and water-soluble residues, carbaryl was the only component in the external rinse of the tops and the internal organosoluble extracts of the roots. The major identified metabolites were conjugates of carbaryl and 4-hydroxycarbaryl, the former tentatively identified as 5,6-dihydro-5-(S-cysteinyl)-6-hydroxycarbaryl or a positional isomer by MS and MS-MS analysis.

Table 8. Total <sup>14</sup>C residues in radish plants.

	Te	ops	Roots		
Fraction	% of TRR	mg/kg as carbaryl	% of TRR	mg/kg as carbaryl	
External rinse	37.9	57.05	-	-	
Internal water-soluble	11.0	16.55	17.0	0.63	
Internal organosoluble	42.8	64.5	36.3	1.34	
Unextractable	8.3	12.48	43.4	1.61	
Compounds in the external rinse and internal extracts					
Carbaryl	80.70	121.55	36.34	1.343	
Carbaryl conjugate	1.00	1.51	2.05	0.076	
1 -Naphthol glycoside	0.54	0.81			
4-Hydroxycarbaryl glycoside (hexose)	1.31	1.97			
Hydroxymethylcarbaryl glycoside	0.36	0.54			
Polar metabolite			1.40	0.052	
Unidentified glycoside			1.65	0.061	
Radioactivity unaccounted for <sup>1</sup>	7.81	11.79	15.20	0.562	
Total	100.00	150.65	100.00	3.699	

<sup>&</sup>lt;sup>1</sup> Includes losses in multiple extraction steps

The unextractable residues were submitted to a series of buffer extractions and enzyme treatments, then separated into cellulose and lignin fractions (Table 9). The main radioactivity was in the cellulose fraction in the roots and in the protease and cellulose fractions in the tops. Protease was the most effective of the enzyme treatments in releasing radioactivity. In most of the extracts,

concentration resulted in precipitation of radioactivity and HPLC analysis was not possible. When HPLC analysis was successful, most chromatograms showed a polar, early-eluting component, but no peak was identified.

Table 9. Analysis of unextractable residues in radish plants.

Treatment	Tops			Roots
Treatment	% of TRR	mg/kg as carbaryl	% of TRR	mg/kg as carbaryl
NaOAc buffer	0.24	0.355	0.71	0.026
Cellulase	0.40	0.605	1.60	0.059
NaH <sub>2</sub> PO <sub>4</sub> buffer	0.26	0.396	0.81	0.030
Amylase	0.64	0.971	1.85	0.068
Protease	2.19	3.295	2.80	0.104
Lignin (oil and precipitate)	1.10	1.66	2.02	0.075
Cellulose	2.17	3.27	18.80	0.694
Soluble organics	1.36	2.05	7.52	0.278
Water-soluble species	0.35	0.528	0.99	0.037

<u>Leaf lettuce</u> (*Latuca sativa*, var. Royal Green) was treated at 10-day intervals with four applications of [1-naphthyl-<sup>14</sup>C]carbaryl (>98% radiochemical purity, specific activity 29,145 dpm/µg) at 1.96 kg/ha and the plants harvested 8 days after the last application (Harsy, 1994a). Residues in acetone/water (50:50) rinses of the leaves were shown to be unchanged carbaryl. Most of the radioactivity in the tissue extracts was found in the organosoluble fraction, also representing unchanged carbaryl. The water-soluble fraction showed major fractions by preparative HPLC, which were subjected to hydrolysis with B-glucosidase and HCl (Table 10).

Table 10. <sup>14</sup>C residues in lettuce.

Fraction	% of TRR	mg/kg as carbaryl
External rinse	67.0	23.5
Internal water-soluble	3.2	1.1
Internal organosoluble	29.4	10.3
Unextractable	2.2	0.77
Compounds in the external rinse and internal extracts		
Carbaryl	96.4	33.85
α-Naphthyl glycoside <sup>2</sup>	0.70	0.25
5-Hydroxycarbaryl glycoside	0.43	0.15
4-Hydroxycarbaryl glycoside	0.36	0.13
hydroxymethylcarbaryl hexose conjugate	0.40	0.14
Carbaryl conjugate	0.04	0.02
Metabolites not identified <sup>3</sup>	1.00	0.35
Radioactivity unaccounted for	0.67	0.24
Total <sup>1</sup>	100	35.13

<sup>&</sup>lt;sup>1</sup> Sum of 1-naphthyl glucoside in two fractions

<sup>&</sup>lt;sup>2</sup> Includes all water-soluble radioactivity not counted in assigned peaks. The largest single metabolite in this group accounted for 0.09% of the TRR.

Soya bean plants (*Glycine max*, var. S23-03) were treated four times at 14-day intervals with  $[^{14}\text{C}]$  carbaryl (radiochemical purity >98%, diluted with unlabelled carbaryl to a final specific activity of 28,750 dpm/ $\square$ g) at 1.7-2.1 kg/ha. The forage and mature plants (seed and hay) were harvested 7 and 47 days respectively after the last foliar application (Harsy, 1994c). The forage was surface-rinsed with acetone/water (50:50) and the rinsed forage, hay and beans extracted.

Carbaryl accounted for 96.3% of the combined radioactivity in the external rinse and organosoluble extract of forage, 94.5% in the organic extracts of hay and 85.4% of the TRR in the organic extract of beans (Table 11). A hexose conjugate of hydroxymethylcarbaryl was the major metabolite in forage and hay and an important metabolite in beans. A conjugate of 1-naphthol, suspected to be a malonyl glycoside, was the major metabolite in the beans (Table 11).

The unextractable residue contained a significant amount of radioactivity in all samples (Table 12), with the highest concentration in the cellulose fraction. Protease was the most effective enzyme treatment in releasing radioactivity. In most of the extracts, concentration resulted in precipitation of radioactivity and HPLC analysis was not possible; when it was successful, the only compound that could be identified was carbaryl. Most chromatograms showed mainly a polar, early-eluting component.

Table 11. Carbaryl residues in soya bean plants (Harsy, 1994c).

Compound	Plant fraction	Fora	ige	Hay		Bea	ın
Compound	Plant fraction	% of TRR	mg/kg <sup>1</sup>	% of TRR	ppm	% of TRR	mg/kg <sup>1</sup>
	external rinse	38.1	106.39	-	-	-	-
	internal water-soluble	13.5	37.71	36.7	151.18	83.2	18.25
	internal organosoluble	19.4	54.14	38.5	158.60	4.8	1.05
	unextractable	14.50	40.5	25.60	105	15.6	3.42
Carbaryl	external and organic	55.37	156.3	36.40	149.9	4.1	0.9
Carbaryl conjugate	aqueous			1.30	5.21		
1 -Naphthol	organic	0.20	0.379				
α -Naphthyl malonyl glycoside <sup>3</sup>	aqueous					26.1	5.72
5-Hydroxycarbaryl glycoside	aqueous					9.1 <sup>3</sup>	2
4-Hydroxycarbaryl glycoside	aqueous			2.00	8.24		
Hydroxymethylcarbaryl	organic	0.20	0.509	3.4 <sup>2</sup>	14		
Hydroxymethylcarbaryl hexose conjugate	aqueous	7.30	20.4	12.20	50.3	16.9	3.71
5,6-dihydro-5,6,- dihydroxycarbaryl	organic	0.20	0.525				
Demethylcarbaryl conjugate	aqueous partition			2.70	11.1		

Compound	Diama Constitution	Fora	Forage			Bean	
Compound	Plant fraction	% of TRR	mg/kg <sup>1</sup>	% of TRR	ppm	% of TRR	mg/kg <sup>1</sup>
HCl-resistant metabolite	aqueous partition	1.60	4.5	1.30	5.21	4.5	0.987
Radioactivity unaccounted for <sup>4</sup>		20.70	56.2	15.10	62.9	23.7	5.19
Total extractable		100.0	279.3	100.0	411.9	100.0	21.93

<sup>&</sup>lt;sup>1</sup> As carbaryl

Table 12. Analysis of unextractable residues in soya beans.

T	Fora	age	Н	ay	Bea	an
Treatment or fraction	% of TRR	mg/kg <sup>1</sup>	% of TRR	ppm	% of TRR	mg/kg <sup>1</sup>
NaOAc buffer	0.43	1.20	2.0	8.24	0.4	0.088
Cellulase	0.62	1.73	1.2	4.94	0.4	0.088
NaH <sub>2</sub> PO <sub>4</sub> buffer	0.50	1.40	1.2	4.94	0.2	0.044
Amylase	0.87	2.43	1.5	6.18	0.7	0.154
Protease	2.35	6.56	5.0	20.6	1.7	0.373
Lignin (oil and precipitate)	1.18	3.27	1.6	6.59	0.2	0.044
Cellulose	5.39	15.1	7.8	32.1	3.1	0.680
Soluble organics	1.30	3.63	2.4	9.89	1.3	0.285
Water-soluble species	0.37	1.03	0.9	3.71	0.6	0.132

<sup>&</sup>lt;sup>1</sup> As carbaryl

Apples. Golden Delicious apples were surface-treated with [1-naphthyl- $^{14}$ C]carbaryl (specific activity 6.56 mCi/mM) in a 50:50 acetone/water solution (Chancey, 1974). There were three regimes: treatments I and III with one application of 10  $\mu$ Ci/apple and apples harvested after 53 and 28 days respectively; treatment II with two applications of 10  $\mu$ Ci/apple and harvest after 53 days. The apples were first washed with acetone/water (80:20) to remove surface residues, then some were peeled and the peel and pulp analysed. Tissues were extracted with acetone/water (90:10) and the extracts partitioned into organosoluble and water-soluble fractions. The surface residues and the organosoluble fraction were analysed by two-dimensional TLC and the water-soluble fraction was subjected to enzyme and acid hydrolyses and the resulting aglycones separated by two-dimensional TLC.

Carbaryl constituted on average 93.6% of the surface residues from all treatments and the remainder consisted of traces of 1-naphthol, hydroxymethylcarbaryl and one minor unidentified component. Metabolites in the peel and pulp extracts were qualitatively similar. Free carbaryl and conjugates of 4-hydroxycarbaryl, 5-hydroxycarbaryl and 1-naphthol were the predominant constituents of the identified residues (Table 13).

<sup>&</sup>lt;sup>2</sup> Sum of metabolite in aqueous and organic fractions

<sup>&</sup>lt;sup>3</sup> Tentative assignment

<sup>&</sup>lt;sup>4</sup> Includes all radioactivity not counted in identified compounds in the extracts and external rinse, as well as losses in multiple extraction steps.

Table 13. Distribution of <sup>14</sup>C in apples treated with [<sup>14</sup>C]carbaryl.

			<sup>14</sup> C, % o	f applied		
	Trea	tment I	Treatm	nent II	Treatn	nent III
	1 x 10 μCi, 53 days		2 x 10 μCi, 28 days		1 x 10 μCi, 28 days	
Compound	Peel	Pulp	Peel	Pulp	Peel	Pulp
Carbaryl (free)	9.5	20.1	14.0	31.7	21.9	45.8
1 –Naphthol conjugated	5.3	3.2	4.1	3.1	1.9	2.1
5,6-Dihydrodiol zone <sup>1</sup>	0.9	0.4	0.4	0.5	0.3	0.3
4-Hydroxycarbaryl conjugated	8.3	3.7	5.7	3.2	3.8	1.8
5-Hydroxycarbaryl conjugated	5.4	3.7	3.1	3.5	2.0	2.1
7-Hydroxycarbaryl conjugated	1.2	0.2	0.8	0.2	0.4	0.1
Hydroxymethylcarbaryl conjugated	0.8	1.9	1.0	2.1	0.7	2.1
Origin of TLC	7.3	5.6	4.4	7.0	2.4	3.5
Unknowns	0.1	0.1	0.1	0.1	-	-
Unhydrolysed	6.0	5.9	3.3	3.8	2.0	1.8
Unextracted	5.8	5.9	5.0	3.4	4.0	1.0

<sup>&</sup>lt;sup>1</sup> 5,6-Dihydro-5,6-dihydroxycarbaryl and 5 other components

The proposed metabolic pathways of carbaryl in plants are shown in Figure 3 and include methyl- and ring-hydroxylation, carbamate ester hydrolysis and *N*-demethylation. There is also conjugation of the hydroxy-compounds to form water-soluble glycosides.

Figure 3. Proposed metabolic pathways of carbaryl in plants.

#### **Environmental fate in soil**

<u>Photolysis</u>. The degradation of [1-naphthyl- $^{14}$ C]carbaryl was studied after surface application to a sandy loam soil (Das, 1990a). The test substance was applied to 1-mm thick soil layers (68% sand, 24% silt, 8% clay, 0.1% organic carbon, pH 7.5 and 12.97 Meq/100 g cation exchange capacity (CEC)) on glass plates at a rate of 9.8  $\pm$  0.3 mg/kg based on soil dry weight, equivalent to approximately 11.2 kg ai/ha.

The soil plates were exposed to artificial sunlight (approximately 12 h light and 12 h dark per day) for 30 days. The light intensities at the beginning and at the end of the study were 504.1 and 501.0 W/m² respectively, comparable to a natural sunlight value of 545.8 W/m². The spectral distribution of the xenon arc lamp was comparable to that of natural sunlight, especially in the effective range of 300-450 nm. The soil plates were positioned 23 cm beneath the light source in the photolysis chamber and the temperature of the soil was  $25 \pm 1$ °C. Control samples treated in the same manner were incubated in the dark.

The soil plates were sampled in duplicate at 0, 1, 3, 7, 14, 21 and 30 days and extracted immediately. The extracts were analysed by high-performance liquid chromatography for the parent and photoproducts, with identification confirmed by LC-MS. Evolution of volatile compounds was monitored with appropriate traps.

The parent concentrations under the unirradiated conditions did not change significantly within the range 96.9%-100.3% and under irradiation gradually declined from 97.5% to 58.5% by the end of the 30 days, with a calculated half-life of 41 days. No major metabolites (>10% of the TRR) were detected. Bound  $^{14}$ C residues representing 32.4% of the applied dose at 30 days were released from soil with acidic solvent and contained a mixture of unidentified highly polar materials. The recovery of  $^{14}$ C was  $102.1 \pm 1.2\%$  and  $98.8 \pm 3.1\%$  under unirradiated and irradiated conditions respectively.

<u>Aerobic degradation</u>. In a degradation study with a sandy loam soil (77% sand, 10% silt, 13% clay, 0.8% organic carbon, pH 6.7, 1.9 Meq/100 g CEC, 1.43 g/ cm<sup>3</sup> bulky density) Miller (1993a) treated the soil with [1-naphthyl- $^{14}$ C]carbaryl at approximately 11.2 mg/kg based on the soil dry weight, equivalent to 12.5 kg ai/ha. The soil moisture was adjusted to 75% of the field moisture capacity (0.33 bar). The test vessels were maintained in the dark at 25 ± 1°C. Evolution of organic volatiles and production of  $^{14}$ CO<sub>2</sub> were monitored. Three test vessels were sampled at 0, 1, 2, 4, 7 and 14 days after treatment.

Soil samples were extracted with various solvent systems and most of the radioactivity was found in the methanol/water extract. Carbaryl was degraded with a calculated half-life of 4.0 days, when  $^{14}\text{CO}_2$  formation reached 53.3% of the applied radioactivity (Table 14). In the extractable residues, 1-naphthol was the only significant product identified. Unextracted residues reached a maximum of 17.7% by day 14, from which base hydrolysis released an average of 60% of the residue. At the end of the experiment, carbaryl accounted for 1/10 of the radioactivity of  $^{14}\text{CO}_2$ . The average recovery of  $^{14}\text{C}$  was 91.0  $\pm$  3.7% (Table 14).

Table 14. Radioactivity in sandy loam soil treated with [1-naphthyl-14C]carbaryl (% of TRR).

~		Solvent syst	em					
, ,	Sampling Methanol/water, 9:1		0.5 M phosphoric acid	Acetone	$CO_2$	Unextractable	Total	
day	Carbaryl <sup>1</sup>	1-naphthol <sup>1</sup>	in acetone/water, 7:3	110000110				
0	69.7 (0.70 )	18.1 (0.18)	0.6	ND	-	0.1	88.5	
1	52.8 (0.53)	34.5 (0.35)	3.8	0.1	0.1	1.1	92.4	
2	64.4 (0.64 )	2.8 (0.03)	5.9	0.8	15.4	11.0	100.3	

~		Solvent syst	em					
Sampling	Methanol/water, 9:1		0.5 M phosphoric acid	Acetone	$CO_2$	Unextractable	Total	
day	Carbaryl <sup>1</sup>	1-naphthol <sup>1</sup>	in acetone/water, 7:3	110000110				
4	23.1 (0.23)	ND	6.1	1.0	53.3	12.0	95.5	
7	22.1 (0.22)	0.2 (<0.01)	7.2	1.3	42.7	15.6	89.1	
14	6.0 (0.06)	ND	7.9	1.5	59.7	17.7	92	

<sup>&</sup>lt;sup>1</sup>% of TRR (mg/kg as carbaryl)

ND: Not detected

Adsorption/desorption. The adsorption and desorption of [1-naphthyl-<sup>14</sup>C]carbaryl were determined by Skinner (1994) in four soils and a sediment, whose characteristics are shown in Table 15. A preliminary trial demonstrated that a soil to test solution ratio of 1 to 5 (7 g of soil and 35 ml of test solution) was optimal and that adsorption and desorption equilibria were reached after 4 and 13 hours of shaking respectively.

Table 15. Physicochemical properties of soils and sediment (Skinner, 1994).

Properties	Silty clay loam	Sandy loam	Loamy sand	Sediment (clay loam)	Silt loam
рН	6.7	5.3	7.2	7.5	6.7
CEC (meq/100 g)	13.00	2.94	2.02	15.26	16.80
Organic matter (%)	3.38	1.43	0.05	1.40	2.411
Organic carbon (%)	1.99 <sup>2</sup>	$0.84^{2}$	$0.03^{2}$	$0.82^{2}$	1.42
WHC(%) @1/3 Bar	29.21	10.88	5.01	32.14	29.0
WHC (%) @ 15 Bar	14.25	4.61	2.79	13.59	not done
Sand (%)	14.8	73.2	86.8	33.2	17
Silt (%)	53.6	13.6	3.6	37.6	62
Clay (%)	31.6	13.2	9.6	29.2	21
Bulk Density (g/cc)	1.18	1.69	1.46	1.18	1.61
Source	Madison Co., KY	Wake Co., NC	Santa Cruz Co., CA	San Joaquin Co., CA	Madison Co., KY

WHC: water-holding capacity

In the definitive study, aqueous test solutions of [1-naphthyl-<sup>14</sup>C]carbaryl in 0.01 M calcium chloride (CaC1<sub>2</sub>) were prepared at concentrations of 0.27, 1.02, 2.51, 5.01 and 10.0 mg/l. During the adsorption phase these were maintained at 24-26 °C for 4 hours in a shaking water bath with 7.0 g of air-dried sieved soil or sediment plus 35 ml of test solution. In the desorption phase sufficient fresh 0.01 M CaC1<sub>2</sub> was added to the tubes containing the drained soil pellets to make a final volume of 35 ml and the tubes shaken for 4 hours. Carbaryl was found to be stable during the adsorption and desorption phases as determined by TLC and HPLC analysis. The overall mean recovery of <sup>14</sup>C from all soils at all concentrations was 101.1 + 1.5%.

No measurable adsorption occurred to the loamy sand soil with a very low organic matter content (0.05%). The Freundlich adsorption coefficients (K values) were in the range 1.74 to 3.52 and correlated well (correlation coefficient 0.95) with the organic matter (Table 16) and for desorption ranged from 6.72 to 7.66, average 7.01. Carbaryl is predicted to have medium mobility ( $K_{oc}$  between 150 and 500) to high mobility in the systems tested.

<sup>&</sup>lt;sup>1</sup> Calculated from determined organic carbon (%) value using conversion factor 1.7 (Lyman et al., 1990).

<sup>&</sup>lt;sup>2</sup> Calculated from determined organic matter (%) value using conversion factor 0.588 (reciprocal of 1.7).

Soil	Organic	Adsorption				Desorption			
5011	matter (%)	K	Koc	1/n	r <sup>2</sup>	K	Koc	1/n	r <sup>2</sup>
Silty Clay Loam	3.38	3.52	177	0.797	0.999	7.66	385	0.858	0.997
Silt Loam	2.41	3.00	211	0.784	1.000	6.89	485	0.861	0.998
Sandy Loam	1.43	1.74	207	0.837	0.997	6.72	800	1.016	0.999
Sediment (clay loam)	1.40	2.04	249	0.835	0.999	6.78	827	0.949	1.000
Average		2.57	211			7.01	624		

Table 16. Freundlich equation parameters and  $K_{oc}$  for adsorption and desorption of carbaryl.

Another carbaryl adsorption study was conducted with sand, sandy loam, silt loam and silty clay loam soils as well as an aquatic sediment by equilibrating [1-naphthyl-<sup>14</sup>C]carbaryl (purity 97.1%; specific activity of 21 mCi/mM) and unlabelled carbaryl (purity 99.8%) at 1, 2, 3, 4 and 5 mg/kg in 5 g of air-dried soil for two hours (Chib, 1985). Carbaryl adsorption to soils and the sediment increased with an increase in carbaryl concentration in solution in all the soils investigated. The highest adsorption value at each concentration was with the silt loam soil (5.3% organic matter) and the lowest with sand (0.4% organic matter).

Mobility. A study using soil thin-layer plates was conducted with [1-naphthyl-<sup>14</sup>C]carbaryl (purity 97.1%; specific activity of 21 mCi/mM) and unlabelled carbaryl (purity 99.8%) on Norfolk sandy loam, silt loam, silty clay loam and loam (Chib and Andrawes, 1985). Rf values varied from 0.11 to 0.23 (Table 17).

Table 17. S	Soil characteri	stics and mo	bility on thin	-layer plates	, USA.
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			Characterist	ics		
Soil (location)	Sand (%)	Silt (%)	Clay (%)	Organic matter (%)	pН	Rf
Sandy loam (Raleigh, NC)	73.6	10.8	15.6	1.0	5.3	0.17
Sandy loam (Clayton, NC)	83.0	15.0	2.0	0.8	5.8	0.17
Silt loam (Newton, Iowa)	16.8	58.4	24.8	5.3	5.0	0.11
Silty clay loam (Newton, Iowa)	4.8	62.4	32.8	3.6	6.3	0.23
Loam (Nebraska)	24.8	48.4	26.8	3.0	5.2	0.17

The same authors conducted an aged-residues column leaching study on moist sandy loam soil. A mixture of  $^{14}$ C-labelled and unlabelled carbaryl (5,000,000 dpm, 714 µg), equivalent to 3 kg/ha, was placed on top of a 30 cm glass column (5.2 cm i.d.) packed with 847 g of soil, the top was covered with a cotton swab and the system allowed to age for 30 days at 23°C. The column was then eluted daily with the equivalent of  $\frac{1}{2}$  ha-cm of water per day for 46 days (ca. 58 cm of rainfall) and the radioactivity of the eluate determined. Only 0.61% of the applied radioactivity was eluted with the leachate after 46 days. The column was then divided into 2.5 cm slices, the soil samples dried in a Petri dish overnight and submitted to combustion analysis. Only 28.7% of the applied radioactivity remained in the soil by the end of the study, most of which (18.9%) remained in the top 5 cm of the column. It was suggested that the remaining  $^{14}$ C (71.3%) was lost as volatile degradation products.

Residues in confined rotational crops. Harsy (1995) applied [1-naphthyl-<sup>14</sup>C]carbaryl to a sandy loam soil at exaggerated rates of 17.8, 18.0 and 17.3 kg/ha, determined by subtracting the radioactivity remaining in each spray bottle from the radioactivity before spraying. The plots were aged for 30, 120, or 365 days before being planted with lettuce, radishes and wheat. Radioactivity in the soil was monitored in each box by taking soil cores at 7.5 cm intervals for analysis.

The 0 to 7.5 cm layer showed the highest level of  $^{14}$ C residues before planting (15.2 to 21.1 ppm) which decreased to < 5 ppm with time. Levels at 30 and 120 days were <0.2 ppm in the 7.5–15 cm layer (Table 18).

Table 18. Radioactive residues in soil plots treated with [14C]carbaryl.

Planting time	No. of samples <sup>1</sup>	Days after	Radi	oactivity, mg/kg as ca	arbaryl
Planting time	No. of samples	treatment	0 - 7.5 cm	7.5–15 cm	15–30 cm
	1	0	15.2	0.08	NA
	3	30	4.07	0.08	NA
	3	39	4.89	0.06	NA
30 DAT	1	55	$0.05^2$	3.18 <sup>2</sup>	NA
30 DA1	1	69	5.54	0.06	NA
	1	89	3.17	0.04	NA
	1	111	4.66	0.04	NA
	1	148	3.50	0.04	NA
	1	0	15.9	0.06	NA
	3	120	4.34	0.06	0.04
	1	149	3.42	0.05	NA
120 DAT	1	151	3.51	0.04	NA
	1	158	4.02	0.03	NA
	1	195	3.12	0.17	NA
	1	239	2.51	0.03	NA
	1	0	21.1	0.05	NA
	3	365	3.00	2.37	1.10
365 DAT	2	395	2.93	0.24	NA
303 DA1	1	427	1.47	0.03	NA
	1	448	3.08	0.11	NA
	1	523	4.06	1.67	NA

DAT: days after treatment

NA: not analysed

Lettuces were picked at maturity 75 to 84 days after planting (DAP) by cutting them 2.5 to 5 cm above the surface of the soil, and radishes were sampled as whole plants at an immature stage between 29 and 39 DAP and separately as root and tops at the mature harvest between 48 and 63 DAP. Wheat was harvested at an immature stage between 25 and 31 DAP, and as grain and straw when mature between 118 and 130 DAP.

The total radioactive residue (TRR) in the plants was determined by combustion and quantification of the <sup>14</sup>C in a liquid-scintillation counter. Plant samples were then extracted with acetone/water (50:50), the acetone evaporated and the aqueous phase partitioned against dichloromethane. The radioactivity was determined in the total extract and the organosoluble, aqueous and insoluble phases (Table 19).

<sup>&</sup>lt;sup>1</sup> Number of samples averaged for each soil depth

<sup>&</sup>lt;sup>2</sup> Value suspect, possible switch during collection

The total radioactive residues were lower in every crop except wheat straw at 120- and 365-day planting times than at 30 days. The average extractable radioactivity was  $\le$ 64%, of which  $\le$ 11% was organosoluble, suggesting the absence of free carbaryl and free metabolites. Water-soluble residues ranged from 0.071 mg/kg in 120-day wheat straw to 0.009 mg/kg in 365-day immature radish. In most reverse-phase HPLC profiles of aqueous phases exceeding 0.05 mg/kg, a very early-eluting peak was observed, indicating very polar material. No metabolite was identified.

Table 19. Distribution of radioactivity in extracts of rotational crops.

Distriction of descent	TRR, mg/kg <sup>1</sup>	Acetone extra		Organos phas		Water-solul	ble phase	Inso	luble
Planting time and sample	mg/kg <sup>1</sup>	% of TRR	mg/kg <sup>1</sup>	% of TRR	mg/kg <sup>1</sup>	% of TRR	mg/kg <sup>1</sup>	% of TRR	mg/kg <sup>1</sup>
30 DAT									
Lettuce (average)	0.103	51	0.053	8	0.007	51	0.052	48	0.049
Immature radish	0.106	34	0.036	3	0.003	31	0.033	70	0.074
Radish tops	0.109	35	0.038	2	0.002	27	0.029	62	0.068
Radish roots (average)	0.062	41	0.026	4	0.002	32	0.020	65	0.040
Immature wheat	0.248	29	0.071	3	0.007	27	0.067	69	0.171
Wheat grain	0.095	21	0.020	0.3	0.000	16	0.015	68	0.065
Wheat straw (average)	0.155	36	0.056	4	0.006	31	0.048	64	0.099
120 DAT									
Lettuce	0.090	53	0.047	2	0.001	49	0.044	46	0.041
Immature radish	0.084	39	0.033	4	0.003	34	0.029	66	0.055
Radish tops	0.033	64	0.021	4	0.001	45	0.015	63	0.021
Radish roots	0.044	42	0.019	4	0.002	37	0.016	70	0.031
Immature wheat	0.078	37	0.029	3	0.002	34	0.026	52	0.040
Wheat grain	0.064	26	0.017	6	0.004	15	0.010	76	0.049
Wheat straw	0.155	28	0.044	7	0.011	46	0.071	93	0.144
365 DAT									
Lettuce	0.019	44	0.008	NA <sup>2</sup>	NA <sup>2</sup>	NA <sup>2</sup>	NA <sup>2</sup>	52	0.010
Immature radish	0.032	33	0.011	6	0.002	27	0.009	92	0.029
Radish tops	0.026	56	0.014	11	0.003	33	0.009	54	0.014
Radish Roots	0.022	38	0.008	NA <sup>2</sup>	NA <sup>2</sup>	NA <sup>2</sup>	NA <sup>2</sup>	87	0.019
Immature wheat	0.055	162 <sup>3</sup>	$0.089^3$	-	1	-	-	80	0.044
Wheat grain	0.043	46	0.020	2	0.001	41	0.018	62	0.027
Wheat straw	0.062	50	0.031	7	0.004	43	0.027	64	0.039

<sup>&</sup>lt;sup>1</sup> As carbaryl

Insoluble residues ranged from 0.010 to 0.171 mg/kg as carbaryl in all samples. Unextractable residues above 0.05 mg/kg carbaryl equivalents were refluxed with 1% aqueous NaCl for 24 hours, and hydrolyzed with protease and HCl. Generally only small quantities of radioactivity were removed (Table 20). Several of the HCl hydrolysates analysed by reverse-phase HPLC contained highly-polar, early-eluting unidentified component(s).

<sup>&</sup>lt;sup>2</sup> Not analysed owing to low levels of radioactivity in acetone/water extract

<sup>&</sup>lt;sup>3</sup> Apparent contamination of sample gave high recoveries; insufficient sample to re-extract and reanalyse.

Table 20. Characterization of insoluble residues. TRR is total radioactive residue; mg/kg values are in [14C]carbaryl equivalents.

	Insoluble		NaCl Ex	tract	Protease hy	drolysate 1	HCl hyd	rolysate 1	Final une	xtracted '	
Sample	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	
	30 days										
Lettuce 1	39	0.040	4	0.004	d	d	5	0.005	30 c	0.031 c	
Lettuce 2	56	0.058	d	d	d	d	13	0.013	37	0.038	
Immature radish	70	0.074	7	0.007	7	0.007	17	0.018	30	0.032	
Radish tops	62	0.068	11	0.012	6	0.006	d	d	11 e	0.012	
Radish root 1	58	0.036	d	d	d	d	23	0.014	31	0.019	
Radish root 2	71	0.044	13	0.008	d	d	d	d	58	0.036	
Immature wheat	69	0.172	4	0.010	12	0.030	17	0.042	41	0.102	
Wheat grain	68	0.065	f	f	d	d	58 f	0.055 f	13	0.012	
Wheat straw	55	0.085	7	0.011	1	0.002	11	0.017	35	0.054	
					120 days						
Immature radish	66	0.055	d	d	d	d	23	0.019	43 g	0.036 g	
Wheat grain	76	0.049	d	d	d	d	61	0.039	13 g	0.008g	
Wheat straw	54	0.083	d	d	d	d	11 h	0.017 h	35 g,h	0.054 g,h	

<sup>&</sup>lt;sup>1</sup> Protease and HCl hydrolyses were on separate samples so <sup>14</sup>C in these fractions cannot be summed to determine recoveries *c:* Calculated by difference, and representing residue after HCl hydrolysis when both HCl and protease hydrolyses were performed

### Environmental fate in water/sediment systems

Aqueous photolysis. The photodegradation of [1-naphthyl- $^{14}$ C]carbaryl solution at 10.1 mg/l exposed to artificial sunlight (513.8-507.2 W/m $^2$ ) for 360 hours of continuous irradiation was studied in sterile water buffered at pH 5 (Das, 1990b). The test vessels were positioned 23 cm beneath the light source in the photolysis chamber and the test solutions maintained at 25  $\pm$  1°C. Control samples treated in the same manner were incubated in the dark.

Test solutions were sampled at 0, 12, 36, 84, 168, 252 and 360 hours and immediately analysed by HPLC for the parent compound and degradation products. Under irradiation the parent concentrations gradually decreased from 97.0% of the applied radioactivity to 33.4% by the end of the period. The only major labelled product was 1-naphthol, 62.4% of the total radioactivity at the end of the study. The half-life for continuous irradiation was 10.3 days. The parent concentrations under the unirradiated conditions changed insignificantly (96.6%-97.6%). A half-life for a 12-h irradiation and

d: Step omitted

e: This number represents the remaining residue after treatment with NaCl

f: Owing to emulsion formation, the insoluble residue and NaCl extract were hydrolysed together with HCl in the next step, and not independently analysed

g: Calculated by difference

h: Average of two residues after HCl hydrolysis.

12-h darkness period of 21 days was calculated from the degradation rate constants under irradiated and unirradiated conditions.

Hydrolysis in water. A hydrolysis study with [1-naphthyl-<sup>14</sup>C]carbaryl was conducted at 25°C under dark and sterile conditions at pH 5, 7 and 9 (Carpenter, 1990). The initial nominal concentration was 10 mg/l in buffer solution. Samples of pH 5 and 7 solutions were analysed after 0, 1, 2, 3, 4, 5, 7, 14, 21 and 30 days. No evidence of hydrolytic degradation of carbaryl was detected in the pH 5 test samples (calculated half-life of 1277 days). Carbaryl was degraded to 1-naphthol in pH 7 buffer (2 samples), with an average half-live of 12 days.

Samples of pH 9 solutions were analysed after 0, 1, 2, 3, 4, 6, 8, 12, 24 and 48 hours. Carbaryl was degraded rapidly to 1-naphthol, with a half-life of 3.2 hours.

The only degradation product of significance was 1-naphthol, accounting for 77% of the radioactivity in pH 7 solutions after 30 days. After 24 hours 95% of the carbaryl in the pH 9 samples had been converted to 1-naphthol. No other degradation product accounted for more than 2% in any sample, and the mean recovery of <sup>14</sup>C was 99.8%.

Anaerobic degradation. The degradation of [1-naphthyl- $^{14}$ C]carbaryl under anaerobic conditions was studied in a mixture of pond sediment (sandy loam, pH 6.0 and 6.5% organic matter) and pond water (pH 6.6) at a soil to water ratio of approximately 1 to 2 (Miller, 1993b). The samples were treated with the [ $^{14}$ C]carbaryl at approximately 10 mg/l and the test vessels maintained in the dark at 25  $\pm$  1°C. The evolution of volatile organic  $^{14}$ C and  $^{14}$ CO<sub>2</sub> was monitored, and the vessels sampled at intervals for 126 days.

The water was separated from the sediment in each sample and partitioned with methylene chloride. The sediment samples were extracted with a series of solvents and partitioned into organic solvent. The average recovery of  $^{14}$ C was  $94.7 \pm 6.8\%$  (Table 21). Except at day 0, most of the radioactivity was found in the methanol/water extract of the sediment. Unextracted residues reached a maximum of 23.6% of the TRR by day 126 (Table 21). Base hydrolysis released an average of 45% of the unextracted residue.

Lable 71 Lotal radioact	ivity in water/sedime	ent system under anaerobic	conditions % of IRR

Intomol	Water		Sediment		Tatal		
Interval, days	MeCl	methanol: water (9:1)	0.5M phosphoric acid in acetone: water (7:3)	acetone	Total volatiles <sup>1</sup>	Unextractable	Total <sup>2</sup>
Day 0	81.4	6.7	1.7	NQ		1.2	91.0
Day 1	36.0	51.9	2.4		NQ	0.5	90.8
Day 3	16.0	64.9	13.1	0.3	NQ	1.1	95.4
Day 7	11.0	72.5	19.5	0.6	0.1	3.0	106.7
Day 14	5.4	69.9	20.0	1.0	0.3	5.3	101.9
Day 29	3.3	66.6	5.6	2.9	0.4	12.6	91.4
Day 63	2.9	49.4	14.4	3.7	5.3	13.7	89.4
Day 94	3.8	55.4	14.6	1.9	4.0	20.1	100.1
Day 126	3.0	32.8	11.0	2.5	12.4	23.6	85.3

NQ: not quantified

<sup>2</sup>4th extract of sediment <0.6% of TRR

<sup>1&</sup>gt;99% identified as <sup>14</sup>CO<sub>2</sub> by precipitation as barium carbonate

The organic extract of the sediment and the methylene chloride extract of the water were concentrated and analysed by HPLC. Carbaryl was degraded under anaerobic aquatic conditions with a calculated half-life of 72.2 days. 1-naphthol was the major degradation product in the extracts of sediment and water (Table 22). Approximately 12% of the applied radioactivity was recovered as <sup>14</sup>CO<sub>2</sub> by Day 126 (confirmed by barium carbonate precipitation). Several minor degradation products were also observed, none of which exceeded an average of 2.5% of the applied dose.

Table 22. Distribution of radioactivity in the combined organic extracts of water and sediment.

Compound	Radioactivity, %	Day 0	Day 1	Day 3	Day 7	Day 14	Day 29	Day 63	Day 94	Day 126
Carbaryl	in water in sediment, % of TRR	64.5 7.5	26.5 51.3	12.8 65.1	5.2 75.5	2.0 51.6	1.3 44.9	1.2 40.6	0.7 31.5	0.4 21.4
·	Total mg/kg as carbaryl	0.71	0.78	0.78	0.81	0.53	0.46	0.42	0.32	0.22
1-Naphthol	in water in sediment, % of TRR	12.5 NQ	8.0 0.8	2.2 10.1	1.0 13.7	0.4 24.8	0.3 23.4	NQ 19.8	NQ 26.3	NQ 13.2
	Total mg/kg	0.13	0.09	0.12	0.15	0.25	0.24	0.20	0.26	0.13
5-Hydroxy-1-naphthyl methylcarbamate	in water in sediment, % of TRR	NQ NQ	NQ 0.1	NQ NQ	NQ 0.1	NQ 0.1	NQ 0.4	NQ 0.6	NQ 1.1	NQ 0.6
	Total mg/kg as carbaryl	NQ	NQ	NQ	NQ	NQ	NQ	0.01	0.01	0.01
Hydroxymethylcarbaryl	in water in sediment, % of TRR	NQ NQ	NQ NQ	NQ 0.2	NQ 0.1	NQ NQ	NQ 0.5	NQ 0.6	NQ 1.6	NQ 1.1
	Total mg/kg as carbaryl	NQ	NQ	NQ	NQ	NQ	NQ	0.01	0.02	0.01
1,4-Naphthoquinone	in water in sediment, % of TRR	NQ NQ	NQ 0.1	NQ 0.4	NQ 0.2	NQ 0.5	NQ 1.1	NQ 1.2	NQ 2.5	NQ 0.7
1 1	Total mg/kg as carbaryl	NQ	NQ	NQ	NQ	NQ	0.01	0.01	0.03	0.01
Others	in water in sediment, % of TRR	NQ NQ	NQ NQ	NQ NQ	NQ NQ	NQ NQ	NQ 0.4	0.1 0.1	0.1 1.4	NQ 0.4
	Total mg/kg as carbaryl	NQ	NQ	NQ	NQ	NQ	NQ	NQ	0.01	NQ

NQ: not quantifiable

<u>Aerobic degradation</u>. [1-Naphthyl- $^{14}$ C]carbaryl was incubated at a concentration of 9.99 mg/l (equivalent to approximately 11.2 kg ai/ha) in a microbially viable pond sediment/water mixture at 25  $\pm$  1°C in the dark for 30 days (Misra, 1994). The sediment was a sandy loam with a pH of 7.1 and 0.7% organic matter and the water was at pH 7.5. Nine triplicate samples were taken at intervals for 30 days after dosing.

The recovery of  $^{14}$ C throughout was  $101.6 \pm 2.2\%$  and the radioactivity in the aqueous phase steadily decreased. Extractable  $^{14}$ C from the sediment reached a maximum on day 3 and about 65% of the TRR was bound to sediment at day 21 (Table 23). The sediment was fractionated into fulvic acid,

humic acid and humin which contained  $4.0 \pm 1.8\%$ ,  $18.9 \pm 9.2$  and  $24.3 \pm 7.1\%$  (n = 8) of the applied <sup>14</sup>C respectively. Volatile <sup>14</sup>C reached a plateau at about 1% of the applied radioactivity.

Table 23. Total	l radioactivity in	water/sediment system	under aerobic condition	s, % of TRR.

Sampling		Sediment			
time, days	Water	Extractable	Bound	Volatiles	Total recovered
0	77.9	24.6	0.35	-	103
1	59.2	29.4	15.3	0.11	104
2	37.3	36.2	26.8	0.11	100
3	17.6	38.7	45.8	0.27	102
5	15.0	34.0	51.3	0.40	101
7	9.9	33.9	61.0	0.51	105
14	5.3	33.7	60.3	0.83	100
21	4.0	30.7	65.0	0.79	100
30	2.6	30.6	63.8	0.88	97.9

1-Naphthol, the major primary metabolite, was detected in both water and sediment extracts (Table 24). Two polar compounds (Unkn 1 and Unkn 2) and Unkn 3, a composite of several peaks, were detected in water at a combined maximum of 5% of the TRR. The chromatographic behaviour of the polar Unkn 1 and Unkn 2 did not change after acid hydrolysis and enzyme digestion, indicating that they are not conjugates of dihydroxydihydronaphthalenes or hydroxylated carbaryl. Unkn 3 was mainly detected in sediment extracts, with each peak representing <3% of the applied <sup>14</sup>C. The chromatographic pattern of Unkn 3 did not change noticeably upon acid or enzyme treatment. 1,4-Naphthoquinone was detected but not quantified in sediment samples from days 3 and 14.

Table 24. Distribution of carbaryl and its degradation products in an aerobic water/sediment system.

				Sediment extracts, % of applied <sup>14</sup> C			
		Water, % of ap	oplied <sup>14</sup> C				
Day	Carbaryl	1-Naphthol	Unknowns 1+2 + 3	Carbaryl	1-Naphthol	Unknown 3	Extract 2 <sup>2</sup>
0	76.1	0.87	0.58 (only unkn2)	23.7	Nd	0.91	-
1	46.5	12.3	0.24 (only unkn1)	22.9	5.9	0.63	-
2	22.1	12.3	2.2	13.7	9.5	3.48	9.3
3	11.3	3.7	2.3	15.2	5.7	7.1	9.8
5	6.0	4.9	3.5	9.0	5.4	10.3	8.6
7	4.8	1.86	2.1	7.5	5.8	10.9	8.1
14	0.08	0.13	4.6	3.9	3.4	19.3	6.9
21	nd	0.22	3.4	2.1	2.0	8.5	16.1
30	nd	nd	2.5	1.2	1.3	7.3	18.7

 $<sup>^{1}</sup>$  extracted with 1N aqueous  $H_{3}PO_{4}$ /acetone/methanol (10:45:45), 1N aqueous  $H_{3}PO_{4}$ /acetone (20:80) and centrifuged  $^{2}$  residue from extract 1 was dissolved in dioxane.

The proposed degradation pathway of carbaryl under aerobic aquatic conditions via 1,4-naphthoquinone is shown in Figure 4. The half-life of carbaryl was estimated by first order regression analysis to be 4.9 days with a correlation coefficient (r<sup>2</sup>) of 0.91.

Figure 4. Proposed degradation pathway of carbaryl in water-sediment systems under aerobic conditions.

#### **RESIDUE ANALYSIS**

### **Analytical methods**

<u>Animal products</u>. In a method for the determination of carbaryl in chicken samples by reverse-phase HPLC (Cooper and Outram, 1989), the compound is extracted by maceration with methanol and the extract cleaned up by liquid-liquid partition followed by separation on a C-18 cartridge. A mean recovery of 80% (75-86%) was found in samples fortified in the range 0.02 to 1 mg/kg.

A method has been developed for the determination of free and conjugated carbaryl and 5,6-dihydro-5,6-dihydroxycarbaryl, and 5-methoxy-6-hydroxycarbaryl in raw milk, eggs, and cow and poultry tissues (Ibrahim, 1996, 1997) that involves extraction of the analytes with a combination of acetone, acetonitrile and water followed by evaporation of the organic solvents before the analytes are subjected to mild acid hydrolysis. This converts the conjugates to their free forms, and 5,6-dihydro-5,6-dihydroxycarbaryl to 5-hydroxycarbaryl. The reaction mixture is finally partitioned with dichloromethane. The dichloromethane is evaporated to dryness and the residue partitioned between acetonitrile and hexane. The acetonitrile layer is then evaporated and the residue dissolved in 40% methanol in water for analysis on a C-18 HPLC column equipped with a post column derivatization system and fluorescence detector. The hydrolysis reaction gave recoveries of 75.5 to 106.4% for all metabolites in all cases.

The average LOD (limit of detection) and LOQ (limit of quantification) for milk, eggs, cow liver, muscle, kidney and fat, and chicken muscle and fat were 0.005 and 0.020 mg/kg for each of the three analytes. The LOQ in chicken liver was 0.1 mg/kg. Average recoveries of analytes from samples fortified over a range from the LOQ to 0.1 or 5 mg/kg ranged from 75.9 to 107% for milk, eggs, cow and chicken muscle, chicken fat and chicken liver for all analytes (Table 25). Relative standard deviations were <20%.

		Carba	ryl		5,6-dihydro-5,6-dihydroxy carbaryl				5-meth	5-methoxy,6-hydroxy carbaryl			
Sample	0.02 1	ng/kg	0.10	mg/kg	0.02 mg/kg		0.10 1	ng/kg	0.02 1	0.02 mg/kg		0.10 mg/kg	
Cow muscle	79.7		89	9.7	77.5		83	3.2	80	0.0	87	'.2	
Cow fat	76.1		82	2.2	81.7 82.6		2.6	38.4		31.2			
Cow kidney	79.0		70	5.2	66.	7	68.8		44.9		64.2		
Chicken liver	-		82	2.3	-		81.0		-		94.6		
Chicken muscle	87	7.6	9	8.4	81.8 91.5		10	00	107				
Chicken fat	90	).8	9.	93.5 86.5		88	3.8	10	00	101			
	0.02 mg/kg	0.10 mg/kg	1.0 mg/k g	5.0 mg/k g	0.02 mg/kg	0.10 mg/kg	1.0 mg/kg	5.0 mg/kg	0.02 mg/kg	0.10 mg/kg	1.0 mg/kg	5.0 mg/kg	
Cow liver	57.8	70.8	93.2	72.4	71.0	73.1	79.1	76.1	82.7	83.0	98.1	88.5	
Milk	87.1	85.4	78.8	-	76.4	80.4	76.8	-	76.4	87.1	82.2	-	
Eggs	75.9	87.6	82.8	-	89.3	89.2	76.6	_	102	96.3	83.0	-	

Table 25. Average % recoveries (n=5) from animal samples fortified with carbaryl and metabolites.

Validation of this method by an independent laboratory (Curti and Keller, 1997) failed for 5,6-dihydro-5,6-dihydroxycarbaryl and 5-methoxy-6-hydroxycarbaryl in milk (recoveries <30%). A successful result (75% recovery at 0.02 mg/l) was achieved by introducing the use of a high-speed centrifuge for layer separation, evaporation of acetonitrile/water extracts to about 2 ml at approximately 40°C, and filtration of final extracts through 2 or 3 Acrodisk cartridges.

<u>Vegetable crops</u>. A method was developed in 1992 for the separate determination of carbaryl and 1-naphthol (Lee, 1992). Extraction is with dichloromethane and samples are then cleaned up on a Florisil column, with quantification by HPLC with post-column base hydrolysis at 100°C and fluorescence detection.

The method was validated later for some crops, with some changes in chromatography conditions to enhance sensitivity and resolution (Method CACR-0194, Lee, 1994; Thiem, 1995). Average recoveries of carbaryl from a variety of crops and processed products ranged from 32.6 to 88.8% (Table 26), and LOQs from 0.003 to 0.02 mg/kg.

A description of a residue method for carbaryl was provided by the government of Thailand. The sample is extracted with dichloromethane, cleaned up on an alumina column, using chloroform/hexane (1:1) and 100% chloroform as eluant, and residues are determined by GC-NPD. A recovery of 85% and a limit of quantification of 0.1 mg/kg was reported for this method.

Table 26. Average recoveries of carbaryl from raw and processed commodities by method CACR-0194.

Substrate	Fortification (mg/kg)	Average, % recovery	SD, %
Turnip tops	0.03	87.4	3.9
	0.15	85.1	0.9
	12.0	84.0	2.5
Turnip roots	0.01	75.6	7.5
	0.05	76.9*	3.6
	5.0	76.4	2.6
Soya bean hay	0.007	34.8	22.5
	0.04	69.1	3.9
	0.05	71.8	4.0
	0.25	81.0	2.7
	100.0	85.1	2.5
Soya bean forage	0.005	32.6	35.2
	0.025	60.6	5.0
	0.04	80.4	1.3
	0.2	87.6	2.8
	100.0	85.4	5.3
Soya bean seed	0.003	100.4	139.7
	0.015	64.3	14.7
	0.02	82.3	3.2
	0.1	77.8	3.0
	5.0	86.8	2.0
Soya bean hulls	0.02	64.8/76.1	9.4/Nr
	0.1	78.2	4.1
	100.0	87.8	4.3
Soya bean meal	0.02	58.8/78.2	21.1/Nr
	0.1	71.1	4.8
	0.5	84.2	4.5
	5.0	78.9	2.0
Soya bean crude oil	0.02 0.04	73.6 75.4	Nr 5.3
	0.2	86.4	2.2
	5.0	86.4	3.8
Carrot	0.02	84.0	Nr
Wheat grain	0.02	72.6	Nr
Wheat flour	0.02	82.9	Nr
Wheat shorts	0.02	85.3	Nr
Wheat bran	0.02	72.4	Nr
Wheat middlings	0.02	88.8	Nr
Field corn, grain	0.02	77.0	Nr

Substrate	Fortification (mg/kg)	Average, % recovery	SD, %
Field corn forage	0.02	83.9	Nr
Field corn silage	0.02	79.1	Nr
Field corn fodder	0.02	62.7	Nr
Lemon	0.02	82.1	Nr
Spinach	0.02	84.7	Nr
Strawberry	0.02	87.8	Nr

n = 5 unless otherwise stated

Nr: not reported.

Method CACR-0194, using ethyl acetate or dichloromethane as extraction solvents, was independently validated for mustard greens and potatoes (Humble and Herzig, 1995) and peanuts (Nandihali, 1996). Recoveries of carbaryl at levels of 10 and 50 mg/kg ranged from 78 to 95% for mustard greens and from 75 to 79% for potatoes. Recoveries of carbaryl from raw shelled peanut samples fortified at 5 and 10 mg/kg levels varied from 68.7 to 93.9%.

The method was tested for extraction efficiency with incurred [\frac{14}{C}]carbaryl residues on lettuce and radish leaves (Pittman, 1995). After two ethyl acetate extractions and clean-up on a 5% deactivated Florisil column (60-100 mesh), the final solution was analysed for total radioactivity and by HPLC on a C-8 column with basic post-column hydrolysis at 100°C and fluorescence detection. The average LSC recoveries from duplicate samples were 81.9% for lettuce and 104.1% for radish leaves. The average HPLC recoveries from labelled samples were 98.16% for lettuce and 119.5% for radish leaves and from unlabelled samples 114.3% and 90.77% respectively.

<u>Soil</u>. Carbaryl residues are extracted from soil with a mixture of acetone, water, and phosphoric acid. After filtration and dichloromethane partition, clean-up is on a Florisil column. Carbaryl is quantified as 1-naphthol by HPLC with post-column hydrolysis and fluorescence detection. An average recovery of 89.4% was obtained from soil samples fortified at levels ranging from 0.01 to 20 mg/kg, with an LOQ of 0.02 mg/kg (Davis and Thomas, 1985).

### Stability of residues in stored analytical samples

The storage stability of [14C]carbaryl in extracts of fortified hen tissues and eggs was determined. The extractable radioactivity, as % carbaryl, remained the same in egg yolks (88.1%), fat (94.8%), kidneys (83.7%), liver (122.7%) and muscle (77.7%) during the 18 months of the study at -20°C (Struble, 1994b).

The storage stabilities of carbaryl and its free and conjugated metabolites in animal commodities containing field-incurred residues or fortified with the analytes were assessed by Lee (1997a). Conjugated carbaryl seems to be degraded in milk and fat after 2 months, but remained stable for more than 6 months at  $-20^{\circ}$ C in the liver, kidney and muscle. Over 80% of the conjugated metabolite concentrations were found in all samples after a minimum of 4 months' storage (Table 27).

Table 27. Stability of incurred conjugated carbaryl and metabolite residues in cow tissues and milk.

	Storage period	Concentration (mg/kg) of conjugates of				
Sample		Carbaryl	5,6-dihydro-5,6- dihydroxy carbaryl	5-methoxy-6- hydroxy carbaryl		
Milk	0 day	0.25	3.85	3.68		

<sup>\*</sup> an outlier of 53.8% was rejected, n = 4

	Storage period		Concentration (mg/kg) of c	onjugates of
Sample		Carbaryl	5,6-dihydro-5,6- dihydroxy carbaryl	5-methoxy-6- hydroxy carbaryl
	2 months	0.15	3.49	2.90
	3 months	0.15	3.87	3.08
	4 months	0.12	3.90	2.95
	248 days	0.14	3.75	2.92
	% of initial level remaining on last day	56.0	97.4	79.3
Liver	0 day	1.04	1.35	0.12
	2 months	1.25	1.55	0.07
	3 months	1.39	1.71	0.19
	4 months	1.14	1.42	0.15
	173 days	1.29	1.42	0.12
	% of initial level remaining on last day	124	105	100
Kidney	0 day	2.76	4.46	1.03
	2 months	2.74	4.92	1.22
	3 months	2.26	3.82	1.09
	4 months	2.32	3.92	0.88
	196 days	2.55	4.37	0.97
	% of initial level remaining on last day	92.4	98.0	94.2
Muscle	0 day	0.05	1.97	< 0.02
	2 months	0.05	1.86	< 0.02
	3 months	0.05	1.86	< 0.02
	4 months	0.06	2.10	< 0.02
	158 days	0.04	2.02	< 0.02
	% of initial level remaining on last day	80.0	102	-
Fat	0 day	0.12	0.34	0.06
	2 months	0.07	0.36	0.06
	3 months	0.07	0.35	0.06
	4 months	0.07	0.32	0.04
	215 days	0.07	0.35	0.04
	% of initial level remaining on last day	58.3	103	66.7

Free carbaryl added to muscle and fat was stable for at least 5 months, but had degraded to about half the initial concentration in liver after 2 months. Free 5,6-dihydro-5,6-dihydroxy-carbaryl was stable in muscle, liver and fat for the period of the study, but 5-methoxy-6-hydroxy carbaryl was not stable in liver after 4 months or in muscle and fat after 2 months (Table 28).

Table 28. Average % of free carbaryl-related residues remaining in fortified cow tissues after storage.

	Storage	Average % remaining				
Sample		Carbaryl <sup>1</sup>	5,6-dihydro-5,6-dihydroxy carbaryl	5-methoxy-6-hydroxy carbaryl		
Muscle	0 day	96.8	77.9	85.7		
	2 months	92.8	71.2	26.6 (31.0)		
	3 months	93.9	73.2	21.4		
	4 months	95.0	70.8	14.8		
	5 months	93.0 (96.1)	72.3 ( <b>92.8</b> )	18.5 ( <b>21.6</b> )		
Liver	0 day	85.5	77.1	89.2		
	2 months	48.4 (56.6)	75.3	72.5		
	3 months	34.2	66.6	61.5		
	4 months	26.0	65.2	51.3		
	5.5 months	29.6 ( <b>34.6</b> )	74.5 ( <b>96.6</b> )	55.0 (61.7)		
Fat	0 day	78.3	77.0	82.2		
	2 months	86.9	80.5	28.5 (34.7)		
	3 months	87.1	77.7	32.7		
	4 months	89.8	84.4	41.1		
	6.3 months	98.8 (126)	87.6 (114)	23.7 (28.8)		

<sup>&</sup>lt;sup>1</sup> values in parenthesis are % of 0-day percentage remaining on final day

The storage stability of carbaryl residues on or in crop commodities has been studied by Thomas (1986). In one study on sugar beet roots fortified with 0.13 mg/kg carbaryl and stored at  $-10^{\circ}$ C, an average of 67.0 % of the residue remained after 287 days (n=6).

At a level of 10 mg/kg, carbaryl was stable for 12 months (>80% of the initial residue remaining) in most of the samples analysed except barley hulls, tomato dry pomace, wheat hay and pearl barley (Table 29).

Table 29. Stability of carbaryl in crops fortified at 10 mg/kg and stored at  $-20^{\circ}$ C (Shults and Kovtavy, 1995).

Crop	Sample	Storage, months	% remaining <sup>1</sup> (average of 2 fortified samples)		
		0	81.0		
	Hulls	3	38.4 (47.4)		
Barley		6	33.6		
		9	33.8		
		12	30.3		
	Pearl	0	81.4		
		3	70.7		
Barley		6	57.7		
		9	62.8		
		12	64.4 (79.5)		

		Storage, months	% remaining <sup>1</sup>		
Crop	Sample	Storage, menus	(average of 2 fortified samples)		
		0	84.9		
		3	75.8		
Barley	Flour	6	73.8		
		9	71.0		
		12	80.4 <b>(94.7</b> )		
		0	81.4		
		3	91.7		
Lettuce	Head lettuce	6	92.3		
		9	104		
		12	88.7 (109)		
Peanut	Hulls	0	61.0		
		3	49.0		
		6	42.7		
		9	63.0		
		12	49.6 (81.3)		
		0	85.2		
		3	83.8		
Potato	Tuber	6	88.4		
		9	101		
		12	94.8 (111)		
		0	95.6		
		3	97.6		
Tomato	Whole fruit	6	91.4		
		9	104		
		12	102 (107)		
		0	96.2		
		3	77.3		
Tomato	Wet pomace	6	77.0		
		9	88.6		
		12	91.1 ( <b>94.7</b> )		
		0	80.9		
		3	93.8		
Tomato	Dry pomace	6	52.8 (65.3)		
		9	51.4		
		12	50.9 (62.9)		

		Storage, months	% remaining <sup>1</sup> (average of 2 fortified samples)		
Crop	Sample				
		0 day	92.0		
		3	89.6		
Tomato	Purée	6	84.6		
		9	97.2		
		12	92.3 (100)		
		0 day	90.1		
		3	79.1		
Tomato	Paste	6	64.8		
		9	85.8		
		12	83.1 ( <b>92.2</b> )		
		0 day	95.0		
		3	94.0		
Tomato	Juice	6	78.3		
		9	78.6		
		12	90.8 (95.6)		
	Forage	0 day	77.6		
		3	92.3		
Wheat		6	82.2		
		9	81.1		
		12	84.0 <b>(108</b> )		
		0 day	89.8		
	Hay	3	83.0		
Wheat		6	67.2 ( <b>74.8</b> )		
		9	68.1		
		12	67.6 (75.3)		
		0 day	81.3		
		3	82.2		
Wheat	Straw	6	76.2		
		9	77.2		
		12	81.6 (100)		

<sup>&</sup>lt;sup>1</sup>·values in parenthesis are % of initial percentage remaining on final day

In a study with incurred carbaryl at levels from 0.08 to 55 mg/kg, residues were stable ( $\geq$ 80% of the initial residue level) for the periods tested, 6-15 months, in almonds, soya beans, hull, meal and oil, apples, juice and pomace, and grapes but not in raisins, or dry bean vines and hay (Table 30).

Table 30. Stability of incurred carbaryl residues at -20°C in various crop samples (Norris, 1996).

Crop	Sample	Time	C	arbaryl	
		0 day	mg/kg	0.08	
	Kernel	7.5 months	% remaining	86.2	
Almonds		10.5 months	% remaining	91.2	
Aimonds		0 day	mg/kg	26	
	Hulls	7.5 months	% remaining	101	
		10.5 months	% remaining	95.0	
		0 day	mg/kg	2.2	
	Seed	3 months	% remaining	86.4	
		6 months	% remaining	79.1	
		0 day	mg/kg	0.95	
	Hulls	3 months	% remaining	101	
Soya bean		6 months	% remaining	91.6	
Soya bean		0 day	mg/kg	0.49	
	Meal	3 months	% remaining	137	
		6 months	% remaining	126	
		0 day	mg/kg	5.6/6.2	
	Crude oil	3 months	% remaining	88.3/98.7	
		6 months	% remaining	115/103	
		0 day	mg/kg	5.4	
	Whole	11.5 months	% remaining	91.0	
		15 months	% remaining	98.5	
		0 day	mg/kg	1.9	
	Juice	11.5 months	% remaining	98.4	
Apple		15 months	% remaining	94.2	
Арріс		0 day	mg/kg	5.8	
	Wet pomace	12 months	% remaining	108	
		15 months	% remaining	106	
		0 day	mg/kg	16	
	Dry pomace	11.5 months % remaining		104	
		15 months	% remaining	104	
		0 day	mg/kg	8.5	
Grapes	Whole	10 months	% remaining	109	
		15 months	% remaining	86.1	
		0 day	mg/kg	51	
Raisins	Processed <sup>1</sup>	8 months	% remaining	60.0	
		15 months	% remaining	57.2	

Crop	Sample	Time	Carbaryl	
Dry bean	Vines	0 day	mg/kg	55
		6 months	% remaining	58.5
			% remaining	71.6
	Нау		mg/kg	54
		6 months	% remaining	82.0
		10.5 months	% remaining	60.7

<sup>&</sup>lt;sup>1</sup>Unprocessed raisins were dried and stored at 15°C to ambient temperature for 18 days. Storage at processing facility (before processing): 6.5 to 5.5°C for 27 days; -12.5 to -13.5°C for 38 days

In another study with samples fortified with carbaryl at 0.40 mg/kg, residues were stable for about 25 months in olive oil and apples and relatively unstable in olives (Table 31).

Table 31. Stability of carbaryl in samples fortified with 0.40 mg/kg carbaryl and stored at -20°C.

Sample	Storage period	Average % remaining <sup>1</sup>
	0 months	92.6
	6 months	71.8
Oli - C. it	9 months	69.9 (75.5)
Olive fruit	14 months	74.0
	19 months	76.5
	25 months	67.0 (75.4)
	0 months	78.4
	5 months	70.8
Olive oil	8 months	70.9
Olive oil	13 months	63.3
	18 months	78.2
	24 months	64.4 (82.1)
	0 months	90.5
	3 months	79.8
	9 months	102
Apple fruit	12 months	80.6
	16 months	74.6
	21 months	75.6
	26 months	84.2 <b>(93.2</b> )

<sup>&</sup>lt;sup>1</sup> % of initial percentage in parentheses

## **Definition of the residue**

In plants, carbaryl is the major residue (55-98% of the TRR), and no metabolite is present at a concentration exceeding 10% of the TRR.

The Meeting agreed that the definition of the residue for compliance with MRLs and for dietary intake estimation in plant commodities should be carbaryl.

Carbaryl accounted for <20% of the total radioactivity found in milk, and the metabolites 5,6-dihydro-5,6-dihydroxy-carbaryl, sulfate conjugates of 1-naphthol and 5-methoxy-6-hydroxy-carbaryl accounted for about 82% of the radioactivity. Carbaryl was the major residue in muscle (17% of the TRR), but was present at <10% of the TRR in other tissues, which contained mainly the metabolites 1-naphthol sulfate (27 to 30% of the TRR in kidney and lung) and 5,6-dihydro-5,6-dihydroxy-carbaryl (31 to 40% of the TRR in muscle and heart).

The Meeting acknowledged that carbaryl was not the major residue in animal products. However, the available method to determine the metabolites 5,6-dihydro-5,6-dihydroxy-carbaryl and 5-methoxy-6-hydroxy-carbaryl, are not straightforward, and it is not clear whether the standards for these metabolites can be made available to the laboratories for enforcement. Additionally, storage stability studies have shown that the metabolites have limited stability in some samples after 1 month of storage. Currently, no information is available to give assurance that these two metabolites are not of health concern.

The Meeting agreed that, for practical reasons, the definition of the residue for compliance with MRLs and for dietary intake estimation in animal commodities should also be carbaryl.

Carbaryl has a log  $P_{ow}$  of 1.85 to 2.36, and is not concentrated in the fat of animals dosed orally. The Meeting concluded that carbaryl is not fat-soluble.

#### **USE PATTERN**

Carbaryl is registered in many countries for the control of insect pests on fruit, vegetables, cereals, nuts, oilseeds and forage crops. The information available to the Meeting on registered uses in summarized in Table 32.

Table 32. Registered uses of carbaryl.

			Application				PHI
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
Apples	Argentina	WP 850 g/kg	Foliar	NS	0.085-0.127	NS	3 11 (Granny Smith)
	Australia	SC 500 g/l	Foliar	NS	0.08-0.10	NS	3
	Belgium	SC 480 g/l	Foliar	0.72-1.16	0.048-0.077	NS	4
	Brazil	SC 480 g/l	Foliar	NS	0.1728	NS	7
		WP 850 g/kg	Foliar	NS	0.153	NS	7
	Canada	SC 480 g/l	Foliar	1.37-2.76		NS	11
	Chile	SC 480 g/l	Foliar	NS	0.081	NS	1
		WP 850 g/kg	Foliar	NS	0.085	NS	1
	Ethiopia	WP 850 g/kg	Foliar	1.275	NS	NS	1
	France	WP 850 g/kg	Foliar		0.085	1-2	7
	India	WP 500 g/kg	Foliar	NS	0.1	NS	7
	Italy	SC 473 g/l	Foliar	NS	0.06-0.12	NS	7
	Japan	WP 850 g/kg	Foliar			4	30

				A	pplication		PHI
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
		WP 500 g/kg	Foliar			4	30
		EC 150 g/l	Foliar			4	30
	Korea	WP 500 g/kg	Foliar	NS	0.062	4	30
	Mexico	WP 800 g/kg	Foliar	NS	0.16-0.32	NS	1
		SC 480 g/l	Foliar	NS	0.168-0.288	NS	1
	New Zealand	SC 500 g/l	Foliar	NS	0.08-0.12	CP 1	1
	Peru	WP 850 g/kg	Foliar	2.55-3.4	0.51-0.68	NS	1
	South Africa	WP 850 g/kg	Foliar	3.2	0.106	2-3	7
	USA only, thinning	SC 480 g/l WP 800 g/kg	Foliar	2.4-6.73	0.06-0.72 (935-3740 l/ha)	1	Not relevant (application at blooming)
Almonds	Chile	SC 480 g/l	Foliar	NS	0.081	NS	1
		WP 850 g/kg	Foliar	NS	0.085	NS	1
	Italy	SC 473 g/l	Foliar	NS	0.071-0.118	NS	7
	USA	SC 480 g/l WP 800 g/kg	Foliar	2.24-5.6		4 (7-d interval)	14
Apricot	Australia	SC 500 g/l	Foliar	NS	0.08-0.10	NS	3
	Canada	SC 480 g/l	Foliar	3.0	0.3	NS	5
	Chile	SC 480 g/l	Foliar	NS	0.081	NS	1
		WP 850 g/kg	Foliar	NS	0.085	NS	1
	India	WP 500 g/kg	Foliar	NS	0.1	NS	7
	Italy	SC 473 g/l	Foliar	NS	0.071-0.118	NS	7
Asparagus	Canada	SC 480 g/l	Foliar	1.200-3.072	NS	1 or more	2
	Chile	SC 480 g/l	Foliar	1.248-1.680	0.25-0.42	NS	1
		WP 850 g/kg	Foliar	1.275-1.700	0.255-0.425	NS	1
	France	WP 850 g/kg	Foliar	0.85-1.53	NS	Up to 3 (8-10-d interval)	7
	USA	SC 480 g/l WP 800 g/kg	Foliar	1.122-4.488	0.249-0.997	5 (3-d interval)	1
Barley	Canada	SC 480 g/l	Foliar	1.2-2.52 (ground) 0.55-1.76 (aerial)	NS	1 to 2 (7 to 14 days interval)	28
Beetroot	Australia	SC 480 g/l	foliar	0.864-1.08	0.108	NS - CP 1	30
	Brazil	SC 480 g/l	Foliar	0.864-1.08	0.108	NS	30
	Colombia	WP 800 g/kg	Bait	0.52	Not applicable	NS	15
		WP 800 g/kg	Foliar	1.0-1.44	0.2-0.288	NS	15
	Canada	SC 480 g/l	Foliar	0.518-2.52	NS	CP 1	7
	USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-2.24	0.12-0.5	6 (7-d interval)	7

				A	pplication		PHI days
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
Brinjal	India	WP 500 g/kg	Foliar	1.0	NS	NS	7
	Malaysia	WP 850 g/kg	Foliar	1.275	0.013	7	7
	Sri Lanka	SC 480 g/l	Foliar	0.54	0.12	NS	7
		WP 850 g/kg	Foliar	2	0.255	NS	7
Carrot	Brazil	SC 480 g/l	Foliar	0.864-1.08	0.108	NS	30
	Canada	SC 480 g/l	Foliar	0.518-2.52	NS	CP 1	1
	Colombia	WP 800 g/kg	Bait	0.52	Not applicable	NS	15
		WP 800 g/kg	Foliar	1.0-1.44	0.2-0.288	NS	15
	Ethiopia	WP 850 g/kg	Foliar	1.275	NS	NS	0
	USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-2.24		6 (7-d interval)	7
Cashew nuts	Thailand	WP 850 g/kg	Foliar	NS	0.297	NS	NS
Cereals, general	Australia	SC 500 g/l	Foliar	0.9-1.1	0.08-0.10	NS	1 (graze)
Cereals	Argentina	SC 480 g/l	Foliar	0.77-1.4	0.384-0.7	1-2	3
except as otherwise		WP 850 g/kg	Foliar	1.19-1.53	0.595-0.765	1-2	3
specified	India	WP 500 g/kg	Foliar	1.5	NS	NS	7
Cherries	Argentina	SC 480 g/l	Foliar	1.73-2.5	0.086-0.125	1-2	11
		WP 850 g/kg	Foliar	1.7-2.55	0.085-0.127	1-2	11
	Canada	SC 480 g/l	Foliar	1.488-3.0	0.15-0.3	NS	2
	Chile	SC 480 g/l	Foliar	NS	0.081	NS	1
		WP 850 g/kg	Foliar	NS	0.085	NS	1
	Italy	SC 473 g/l	Foliar	NS	0.071-0.118	NS	7
Chestnuts	USA	SC 480 g/l WP 800 g/kg	Foliar	2.24-5.6		4 (7-d interval)	14
Citrus	Australia	SC 500 g/l	Foliar	NS	0.08-0.1	NS	3
	Belize	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Brazil	SC 480 g/l	Foliar	0.192-1.08	0.024-0.108	NS	7
	Brazil	WP 850 g/kg	Foliar	NS	0.01275	NS	14
	Colombia	WP 800 g/kg	Foliar	NS	0.24-0.48	NS	5
	Costa Rica	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Dominican Republic	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	El Salvador	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Ethiopia	WP 850 g/kg	Foliar	1.275	NS	NS	5
	Greece	DP 100 g/kg	Dusting	2-4	Not applicable	NS	7
		SC 479 g/l	Foliar	1.15-6.5	0.057-0.163	NS	7
		WP 850 g/kg	Foliar	1.19-6.8	0.059-0.17	NS	7
	Guatemala	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10

				A	pplication		PHI
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
	Honduras	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	India	WP 500 g/kg	Foliar	NS	0.2	NS	7
	Italy	SC 473 g/l	Foliar	NS	0.071-0.142	NS	7
	Myanmar	WP 850 g/kg	Foliar	NS	0.075-0.112	NS	5
	Nicaragua	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Panama	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Philippines	WP 850 g/kg	Foliar	NS	0.212-0.319	NS	5
	Spain	WP 850 g/kg	Foliar	0.85-1.6	0.085-0.16	NS	7
	Thailand	WP 850 g/kg	Foliar	NS	0.085	NS	NS
	USA	SC 480 g/l WP 800 g/kg	Foliar	2.42-8.4		8 (14-d interval) <sup>1</sup>	5
	USA, California <sup>2</sup>	SC 480 g/l WP 800 g/kg	Foliar	5.6-17.9		1-21 <sup>1,3</sup>	5
	Vietnam	SC 430 g/l	Foliar	0.86	0.27	NS	14
Corn	Argentina	SC 480 g/l	Foliar	1.2-2.25	0.6-1.3	1-2	7
See also		WP 850 g/kg	Foliar	1.19-2,295	0.595-1.15	1-2	7
maize	Brazil	SC 480 g/l	Foliar	0.912-1.08	NS	1 to 2	14
	Canada	SC 480 g/l	Foliar	0.6-1.92	NS	CP 1	1
	Greece	DP 100 g/kg	Dusting	1.5-2.5	Not applicable	NS	7
		SC 479 g/l	Foliar	0.61-2.44	0.122-0.244	NS	7
		WP 850 g/kg	Foliar	0.64-2.55	0.127-0.255	NS	7
	Indonesia	WP 850 g/kg	Foliar	1.275	0.32	NS	NS
	Italy	SC 473 g/l	Foliar	0.355-0.71	0.071-0.142	1-2	7
	Myanmar	WP 850 g/kg	Foliar	1.26-2.01	0.315-1.050	NS	0
	Philippines	WP 850 g/kg	Foliar	NS	0.212-0.319	NS	NS
	Spain	WP 850 g/kg	Foliar	0.425-0.85	0.085-0.170	CP 1-2	7
	Thailand	WP 850 g/kg	Foliar	NS	0.106-0.212	NS	NS
	USA	SC 480 g/l WP 800 g/kg	Foliar	1.121-2.242	1.121-2.242	4 (14-d interval)	48 (grain) 14 (grazing)
Cotton	Argentina	SC 480 g/l	Foliar	0.768-2.54	0.384-1.272	1-2	3
		WP 850 g/kg	Foliar	0.765-2.55	0.382-1.275	1-2	3
	Australia	SC 500 g/l	Foliar	1.1	0.1	NS	1 (graze)
	Belize	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Brazil	SC 480 g/l	Foliar	0.720-1.44	NS	NS	1
		WP 850 g/kg	Foliar	0.785-1.53	NS	NS	1
	Colombia	SC 440 g/l	Foliar	1.54	0.77-1.54	NS	Not applicable
		WP 800 g/kg	Foliar	2.0-2.4	NS	NS	Not applicable
	Costa Rica	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10

				A	pplication		PHI
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
	Dominican Republic	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Egypt	WP 850 g/kg	Foliar	3.06	NS	NS	15
	El Salvador	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Ethiopia	WP 850 g/kg	Foliar	1.275	NS	NS	7
	Greece	DP 100 g/kg	Dusting or bait	1.5-2.5	Not applicable	NS	7
		SC 479 g/l	Foliar	0.61-1.95	0.122-0.244	NS	7
		WP 850 g/kg	Foliar	0.64-2.0	0.127-0.255	NS	7
	Guatemala	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Honduras	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Indonesia	WP 850 g/kg	Foliar	1.7	0.34	NS	NS
	Mexico	WP 800 g/kg	Foliar	1.2-2.4	NS	NS	0
		SC 480 g/l	Foliar	1.2-2.4	NS	NS	0
	Myanmar	WP 850 g/kg	Foliar	1.47-2.10	0.367-1.050	NS	7
	Nicaragua	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Panama	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Peru	WP 850 g/kg	Foliar	1.7-2.125	0.34-0.425	NS	2
	Philippines	WP 850 g/kg	Foliar	NS	0.212-0.319	NS	NS
	South Africa	WP 850 g/kg	Foliar	1.275	0.012	3-4	7 (grazing)
	Spain	WP 850 g/kg	Foliar	0.425-0.85	0.085- 0.170	CP 2	7
	Thailand	WP 850 g/kg	Foliar	NS	0.170-0.255	NS	NS
	Turkey	WP 850 g/kg	Foliar	1.785	NS	CP 1	7
	Venezuela	WP 800 g/kg	Foliar	2.0-2.4	NS	NS	7
	Vietnam	WP 850 g/kg	Foliar	0.850	0.212	NS	14
	Zimbabwe	WP 850 g/kg	Foliar	0.998	0.066	NS	NS
Cucurbits,	Australia	SC 500 g/l	Foliar	NS	0.100	NS	3
general	India	WP 500 g/kg	Foliar	1.0	NS	NS	NS
	Philippines	WP 850 g/kg	Foliar	NS	0.212-0.319	NS	NS
	Sri Lanka	SC 480 g/l	Foliar	0.54	0.12	NS	7
		WP 850 g/kg	Foliar	1.1-2	0.212-0.364	NS	7

				Application					
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days		
Egg plant	Canada	SC 480 g/l	Foliar	0.6-3.07	NS	CP 1	2		
	France	850 WP	Foliar	1.275	NS		7		
	Italy	SC 473 g/l	Foliar	0.355-1.42	0.071-0.284	1-2	7		
	Mexico	WP 800 g/kg	Foliar	1.6-2	NS	NS	1		
		SC 480 g/l	Foliar	1.44-1.92	NS	NS	1		
	Sudan	WP 850 g/kg	Foliar	2.142	NS	NS	NS		
	USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-2.242	0.12-0.5	4 (6-8-d interval)	3		
Field corn, see Maize									
Fruit trees, general	Myanmar	WP 850 g/kg	Foliar	NS	0.112-0.150	NS	7		
	Spain	WP 850 g/kg	Foliar	1.275-2.55	0.085-0.170	CP 1-2	7		
	Turkey	WP 850 g/kg	Foliar	0.85-1.02	NS	CP 1	7		
Garden beets	USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-2.24		6 (7-d interval)	7		
Grapes	Australia	SC 500 g/l	Foliar	NS	0.08-0.10	NS	3		
	Canada	SC 480 g/l	Foliar	2.52-3.07	0.126-0.15	1	5		
	Chile	SC 480 g/l	Foliar	1.25-3.12	NS	NS	1		
		WP 850 g/kg	Foliar	1.275-3.4	NS	NS	1		
	Italy	SC 473 g/l	Foliar	0.71-0.95	0.071-0.095	1-2	7		
	Japan	WP 850 g/kg	Foliar			2	30		
		WP 500 g/kg	Foliar			2	30		
	Spain	WP 850 g/kg	Foliar	0.595-1.19	0.085-0.170	CP 1-2	7		
	USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-2.24	0.28-1.121	5 (7-d interval)	7		
Grapevine	Greece	SC 479 g/l	Foliar	0.28-1.83	0.057-0.122	NS	14		
		DP 100 g/kg	Dusting	2-4	Not applicable	NS	14		
		WP 850 g/kg	Foliar	0.297-1.9	0.059-0.127	NS	14		
	India	WP 500 g/kg	Foliar	NS	0.1	NS	7		
	South Africa	WP 850 g/kg	Foliar	2.65	0.106	2-3	14		
	Turkey	WP 850 g/kg	Foliar	1.02	NS	CP 1	7		
	Vietnam	SC 430 g/l	Foliar	0.86	0.27	NS	14		
	Zimbabwe	WP 850 g/kg	Foliar	0.204	0.102	NS	56		
Hazelnuts	Turkey	SC 480 g/l	Foliar	0.960	NS	CP 1	7		
	Turkey	WP 850 g/kg	Foliar	1.275	NS	CP 1	7		
	USA	SC 480 g/l WP 800 g/kg	Foliar	2.24-5.6		4 (7-d interval)	14		
Lemons	Japan	EC 150 g/l	Foliar			2	200		

				A	pplication		PHI
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
Lettuce	Canada	SC 480 g/l	Foliar	0.65-2.72	NS	NS	5
	USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-2.24	NS	5 (7-d interval)	14
Macadamia	Australia	SC 500 g/l	Foliar	1.1	0.1	1	3
nuts	South Africa	WP 850 g/kg	Foliar	NS	0.212	NS	NS
Maize,	Australia	SC 500 g/l	Foliar	0.6-0.7		NS	1 (graze)
see also corn	Chile	SC 480 g/l	Foliar	1.25-1.68	0.250-0,420	NS	NS
	Belize	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Colombia	GR 50 g/kg	Soil	0.9-1.0	Not applicable	NS	1
•		WP 800 g/kg	Foliar	0.8-2.0	NS	NS	1
		WP 800 g/kg	Bait	0.52	Not applicable	NS	1
	Costa Rica	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Dominican Republic	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Egypt	WP 850 g/kg	Foliar	3.06	NS	NS	NS
	El Salvador	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Guatemala	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Honduras	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Mexico	GR 50 g/kg	Foliar	0.3-0.5	NS	NS	0
•		WP 800 g/kg	Foliar	1.2-2.0	NS	NS	0
		SC 480 g/l	Foliar	1.2-1.92	NS	NS	0
	New Zealand	SC 500 g/l	Foliar	0.8-1.2	0.12	CP 1	3
•	Nicaragua	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Panama	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Peru	WP 800 g/kg	Foliar	1.6-2	NS	NS	2
		WP 850 g/kg	Foliar	1.275-2.125	0.255-0.425	NS	5
	South Africa	WP 850 g/kg	Foliar	0.531	0.106	NS	14
	Turkey	WP 850 g/kg	Foliar	1.44	NS	CP 1	7
	USA	SC 480g/l	Foliar	1.12-2.24		4	14 (forage) 48 (grain and fodder)
	Venezuela	WP 800 g/kg	Foliar	0.8-1.6	NS	NS	21
	Zimbabwe	WP 850 g/kg	Foliar	0.531	NS	NS	7
Nectarines	Australia	SC 500 g/l	Foliar	NS	0.08-0.10	NS	3
	Chile	SC 480 g/l	Foliar	NS	0.081	NS	1
		WP 850 g/kg	Foliar	NS	0.085	NS	1
	USA	SC 480 g/l WP 800 g/kg	Foliar	2.24-3.36		4 (7-d interval)	3

				A	pplication		PHI days
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	aays
	USA, California	SC 480 g/l WP 800 g/kg	Foliar	3.36-5.56		4 (14d-interval)	1
Oats	Canada	SC 480 g/l	Foliar	1.2-2.52	NS	1 to 2	14
Okra	Brazil	SC 480 g/l	Foliar	0.864-1.44	0.108-0.144	NS	3
(lady's finger)	India	WP 500 g/kg	Foliar	0.75-1.0	NS	NS	7
	Mexico	WP 800 g/kg	Foliar	1.2-1.6	NS	NS	0
		SC 480 g/l	Foliar	1.44-1.92	NS	NS	0
	Sri Lanka	SC 480 g/l	Foliar	0.54	0.12	NS	7
		WP 850 g/kg	Foliar	1.1-1.3	0.212-0.255	NS	7
	USA	SC 480 g/l WP 800 g/kg	Foliar	1.12-1.68	0.25-0.37	7 (7-d interval)	3
Oilseeds, general	Bangladesh	WP 850 g/kg	Foliar	1.445	NS	NS	7
Olives	Argentina	SC 480 g/l	Foliar	1.54-2.02	0.077-0.101	1-2	11
		WP 850 g/kg	Foliar	1.53-2.54	0.076-0.102	1-2	11
-	France	WP 850 g/kg	Foliar		0.127	1-2	7
	Greece	SC 479 g/l	Foliar		0.057-0.163	NS	7
		DP 100 g/kg	Dusting	2-4	-	NS	7
		WP 850 g/kg	Foliar	NS	0.059-0.17	NS	7
	Italy	SC 473 g/l	Foliar	NS	0.071-0.142	NS	7
	Spain	WP 850 g/kg	Foliar	0.85-1.7	0.085-0.170	NS	7
	USA	SC 480 g/l WP 800 g/kg	Foliar	5.6-8.4		2 (14-d interval)	14
Oranges	Japan	WP 850 g/kg	Foliar			4	21
		WP 500 g/kg	Foliar			4	21
		EC 150 g/l	Foliar			4	21
Peach	Argentina	SC 480 g/l	Foliar	1.73-2.5	0.086-0.125	1-2	11
		WP 850 g/kg	Foliar	1.7-2.55	0.085-0.127	1-2	11
	Australia	SC 500 g/l	Foliar	NS	0.08-0.10	NS	3
	Brazil	SC 480 g/l	Foliar	NS	0.1728	NS	7
	Canada	SC 480 g/l	Foliar	3.0	0.3	NS	1
	Chile	SC 480 g/l	Foliar	NS	0.081	NS	1
		WP 850 g/kg	Foliar	NS	0.085	NS	1
	Ethiopia	WP 850 g/kg	Foliar	1.275	NS	NS	1
	India	WP 500 g/kg	Foliar	NS	0.1	NS	7
	Italy	SC 473 g/l	Foliar	NS	0.071-0.118	NS	7
	Japan	WP 850 g/kg	Foliar			3	14
		WP 500 g/kg	Foliar			3	14
	Korea	WP 500 g/kg	Foliar	NS	0.05	3	14

				A	pplication		PHI days
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
	Mexico	WP 800 g/kg	Foliar	NS	0.16-0.32	NS	1
		SC 480 g/l	Foliar	NS	0.168-0.288	NS	1
Pears	Argentina	WP 850 g/kg	Foliar	NS	0.085-0.127	NS	3
	Australia	SC 500 g/l	Foliar	NS	0.08-0.10	NS	3
	Canada	SC 480 g/l	Foliar	1.37-2.76		NS	11
	Chile	SC 480 g/l	Foliar	NS	0.081	NS	1
		WP 850 g/kg	Foliar	NS	0.085	NS	1
	Ethiopia	WP 850 g/kg	Foliar	1.275	NS	NS	1
	India	WP 500 g/kg	Foliar	NS	0.1	NS	7
	Italy	SC 473 g/l	Foliar	NS	0.060-0.118	NS	7
	Japan	WP 850 g/kg	Foliar			3	45
	(001000010000100001000010000100001	WP 500 g/kg	Foliar			3	45
		EC 150 g/l	Foliar			4	14
	Korea	WP 500 g/kg	Foliar	NS	0.05	4	14
	Mexico	WP 800 g/kg	Foliar	NS	0.16-0.32	NS	1
		SC 480 g/l	Foliar	NS	0.168-0.288	NS	1
	New Zealand	SC 500 g/l	Foliar	NS	0.08-0.12	CP 1	1
	South Africa	WP 850 g/kg	Foliar	3.2	0.106	2-3	7
Pecans	Australia	SC 500 g/l	Foliar	1.1	0.1	1	3
	South Africa	WP 850 g/kg	Foliar	NS	0.212	NS	NS
	Mexico	WP 800 g/kg	Foliar	NS	0.2-0.28	NS	4
		SC 480 g/l	Foliar	NS	0.192-0.24	NS	4
	USA	SC 480 g/l WP 800 g/kg	Foliar	2.24-5.6		4 (7-d interval)	14
Peppers	Belgium	SC 480 g/l	Foliar	0.768	NS	NS	4 (F) 3 (G)
	Brazil	SC 480 g/l	Foliar	0.864-1.08	0.108	1-6	3
	Canada	SC 480 g/l	Foliar	0.6-3.07	NS	CP 1	2
	USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-2.24		7 (7-d interval)	3
Pistachio nuts	USA	SC 480 g/l WP 800 g/kg	Foliar	3.36-5.6		4 (7-d interval)	14
Plums	Argentina	SC 480 g/l	Foliar	1.73-2.5	0.086-0.125	1-2	11
		WP 850 g/kg	Foliar	1.7-2.55	0.085-0.127	1-2	11
	Australia	SC 500 g/l	Foliar	NS	0.08-0.10	NS	3
	Canada	SC 480 g/l	Foliar	1.488-3.0	0.15-0.3	NS	2
	Chile	SC 480 g/l	Foliar	NS	0.081	NS	1
	(001000010000100001000010000100001	WP 850 g/kg	Foliar	NS	0.085	NS	1
	Italy	SC 473 g/l	Foliar	NS	0.071-0.118	NS	7

Country  Greece  USA  Australia  Brazil  USA  USA, California  Brazil	SC 479 g/l DP 100 g/kg WP 850 g/kg SC 480 g/l WP 800 g/kg SC 480 g/l SC 480 g/l SC 480 g/l WP 800 g/kg SC 480 g/l WP 800 g/kg	Application method Foliar Dusting Foliar Foliar Foliar Foliar Foliar Foliar	Rate, kg ai/ha 0.57-2.44 2-4 0.59-2.55 0.56-3.36 NS	Spray conc., kg ai/hl  0.057-0122  Not applicable  0.059-0.127	No. of applications  NS  NS  NS  NS  Apple: 8 including thinning sprays (14-d interval)  Others: 8 (14-d interval)	7 7 7 3 3
Australia Brazil USA USA, California	DP 100 g/kg WP 850 g/kg SC 480 g/l WP 800 g/kg SC 500 g/l SC 480 g/l SC 480 g/l WP 800 g/kg SC 480 g/l	Dusting Foliar Foliar Foliar Foliar	2-4 0.59-2.55 0.56-3.36	Not applicable 0.059-0.127 0.08-0.10	NS  NS  Apple: 8 including thinning sprays (14-d interval)  Others: 8 (14-d interval)	7 7 3
Australia Brazil USA USA, California	WP 850 g/kg  SC 480 g/l WP 800 g/kg  SC 500 g/l  SC 480 g/l  SC 480 g/l WP 800 g/kg  SC 480 g/l	Foliar Foliar Foliar	0.59-2.55 0.56-3.36 NS	0.059-0.127	NS Apple: 8 including thinning sprays (14-d interval) Others: 8 (14-d interval)	7 3
Australia Brazil USA USA, California	SC 480 g/l WP 800 g/kg SC 500 g/l SC 480 g/l SC 480 g/l WP 800 g/kg SC 480 g/l	Foliar Foliar Foliar	0.56-3.36 NS	0.08-0.10	Apple: 8 including thinning sprays (14-d interval) Others: 8 (14-d interval)	3
Australia Brazil USA USA, California	SC 500 g/l SC 480 g/l SC 480 g/l WP 800 g/kg SC 480 g/l	Foliar Foliar	NS		thinning sprays (14-d interval) Others: 8 (14-d interval)	
Brazil USA USA, California	SC 480 g/l SC 480 g/l WP 800 g/kg SC 480 g/l	Foliar			NS	3
USA, USA, California	SC 480 g/l WP 800 g/kg SC 480 g/l		NS	0 1-1-		_
USA, California	WP 800 g/kg SC 480 g/l	Foliar		0.1728	NS	7
California			2.24-3.36		4 (7-d interval)	3
Brazil	WP 800 g/kg	Foliar	3.36-5.56		4 (14d-interval)	1
	SC 480 g/l	Foliar	0.864-1.08	0.108	NS	30
Canada	SC 480 g/l	Foliar	0.518-2.52	NS	CP 1	7
Italy	SC 473 g/l	Foliar	0.475	0.095	1-2	7
Japan	GB 50 g/kg	Soil	0.37-0.74	Not applicable	6	7
USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-2.24	0.12-0.5	6 (7-d interval)	7
Australia	SC 500 g/l	Foliar	1.1	NS	1-2 (14-d interval)	3
Bangladesh	WP 850 g/kg	Foliar	1.445	NS	NS	7
Belize	SC 480 g/l	Foliar	1.123-2.242	0.374-1.120	NS	14
	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
Brazil	SC 480 g/l	Foliar	0.912-1.08	NS	1 to 2	14
Colombia	GR 50 g/kg	Soil	0.9-1.8	N/a	NS	15
	SC 440 g/l	Foliar	1.76	0.88-1.76	NS	15
	WP 800 g/kg	Foliar	1.0-1.44	NS	NS	15
Costa Rica	SC 480 g/l	Foliar	1.123-2.242	0.374-1.120	NS	14
	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
Dominican Republic	SC 480 g/l	Foliar	1.123-2.242	0.374-1.120	NS	14
	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
El Salvador	SC 480 g/l	Foliar	1.123-2.242	0.374-1.120	NS	14
	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
Greece	DP 100 g/kg	Dusting	1.5-2.5	Not applicable	NS	7
	SC 479 g/l	Foliar	0.766-1.916	0.081-0.163	NS	7
Greece	WP 850 g/kg	Foliar	NS	0.085-0.17	NS	7
Guatemala	SC 480 g/l	Foliar	1.123-2.242	0.374-1.120	NS	14
J U / H H C III III III III III III III III I	Japan JSA  Australia Bangladesh Belize  Brazil Colombia  Costa Rica  Dominican Republic  El Salvador  Greece	Gapan GB 50 g/kg  JSA SC 480 g/l WP 800 g/kg  Australia SC 500 g/l  Bangladesh WP 850 g/kg  Belize SC 480 g/l WP 800 g/kg  Brazil SC 480 g/l Colombia GR 50 g/kg SC 440 g/l WP 800 g/kg  Costa Rica SC 480 g/l WP 800 g/kg  Costa Rica SC 480 g/l WP 800 g/kg  Cominican SC 480 g/l WP 800 g/kg  El Salvador SC 480 g/l WP 800 g/kg  El Salvador SC 480 g/l WP 800 g/kg  Greece DP 100 g/kg SC 479 g/l Greece WP 850 g/kg	GB 50 g/kg  SC 480 g/l WP 800 g/kg  Australia  SC 500 g/l  Foliar  Bangladesh WP 850 g/kg Foliar  Belize SC 480 g/l WP 800 g/kg Foliar  WP 800 g/kg Foliar  GR 50 g/kg Soil SC 480 g/l Foliar  Foliar  WP 800 g/kg Foliar  Foliar  WP 800 g/kg Foliar  Foliar  WP 800 g/kg Foliar  Foliar  WP 800 g/kg Foliar  Foliar	GB 50 g/kg   Soil   0.37-0.74     JSA	Section   Sect	Apan   GB 50 g/kg   Soil   0.37-0.74   Not applicable   6     JSA

				A	pplication		PHI
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
	Honduras	SC 480 g/l	Foliar	1.123-2.242	0.374-1.120	NS	14
		WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	India	WP 500 g/kg	Foliar	1.5	NS	NS	7
	Japan	EC 150 g/kg	Foliar			5	45
		WP 500 g/kg	Foliar			5	45
		WP 850 g/kg	Foliar			5	45
	Korea	WP 500 g/kg	Foliar	NS	0.062	6	3
	Malaysia	WP 850 g/kg	Foliar	1.275	0.013	3	14
	Mexico	WP 800 g/kg	Foliar	1.2-1.6	NS	NS	14
	Ì	SC 480 g/l	Foliar	1.2-1.44	NS	NS	14
	Myanmar	WP 850 g/kg	Foliar	1.47-2.10	0.367-1.050	NS	14
	Nicaragua	SC 480 g/l	Foliar	1.123-2.242	0.374-1.120	NS	14
		WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Panama	SC 480 g/l	Foliar	1.123-2.242	0.374-1.120	NS	14
		WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Philippines	WP 850 g/kg	Foliar	NS	0.212-0.319	NS	14
	Spain	WP 850 g/kg	Foliar	0.425-0.85	0.085-0.170	CP 1-2	7
	Sri Lanka	SC 480 g/l	Foliar	0.54	0.12	NS	7
		WP 850 g/kg	Foliar	0.5-0.7	0.094-0.134	NS	7
	Thailand	WP 850 g/kg	Foliar	NS	0.085	NS	NS
	USA	SC 480 g/l WP 800 g/kg	Foliar	1.12-1.68		2 (7-d interval)	14
	Venezuela	WP 800 g/kg	Foliar	1.2	NS	NS	NS
		SC 430 g/l	Foliar	0.86	0.27	NS	14
		WP 850 g/kg	Foliar	0.850	0.212	NS	14
	Vietnam	SC 430 g/l	Foliar	0.86	0.27	NS	14
		WP 850 g/kg	Foliar	0.850	0.212	NS	14
Root vegetables	Australia	SC 500 g/l	Foliar		0.150	NS	3
Rye	Canada	SC 480 g/l	Foliar	1.2-2.52 (ground) 0.55-1.76 (aerial)	NS	1 to 2 (7 to 14 days interval)	14
Sorghum	Argentina	SC 480 g/l	Foliar	1.2-2.25	0.6-1.3	1-2	7
		WP 850 g/kg	Foliar	1.19-2,295	0.595-1.15	1-2	7
	Australia	SC 500 g/l	Foliar	0.9-1.1	0.08-0.10	NS	1 (graze)
	Belize	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Colombia	GR 50 g/kg	Soil	0.9-1.0	Not applicable	NS	1
		WP 800 g/kg	Foliar	0.8-2.0	0.16-0.44	NS	1
		WP 800 g/kg	Bait	0.52	N/a	NS	1

				A	pplication		PHI
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
	Costa Rica	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Dominican Republic	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	El Salvador	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Ethiopia	WP 850 g/kg	Foliar	1.275	NS	NS	21
	Guatemala	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Honduras	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Mexico	GR 50 g/kg	Foliar	0.3-0.5	NS	NS	21
		SC 480 g/l	Foliar	1.2-1.92	NS	NS	21
	Myanmar	WP 850 g/kg	Foliar	1.26-2.01	0.315-1.050	NS	21
	Nicaragua	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Panama	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Peru	WP 850 g/kg	Foliar	1.275-2.125	0.255-0.425	NS	2
	Philippines	WP 850 g/kg	Foliar	NS	0.212-0.319	NS	NS
	Spain	WP 850 g/kg	Foliar	0.425-0.85	0.085-0.170	CP 1-2	7
	Sudan	WP 850 g/kg	Foliar	1.63	NS	NS	NS
	Thailand	WP 850 g/kg	Foliar	NS	0.212	NS	NS
	USA	SC 480 g/l WP 800 g/kg	Foliar	1.121-2.242		4 (7-d interval)	3 (fresh beans) 14 (grazing) 21 (grain)
	Venezuela	WP 800 g/kg	Foliar	0.8-1.6	NS	NS	21
Soya bean	Brazil	SC 480 g/l	Foliar	0.912-1.08	NS	NS	3
		WP 850 g/kg	Foliar	0.204	NS	NS	3
	Belize	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Colombia	WP 800 g/kg	Foliar	1.0-1.6	0.2-0.32	NS	15
	Costa Rica	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Dominican Republic	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Egypt	WP 850 g/kg	Foliar	3.06	NS	NS	NS
	El Salvador	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
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				A	pplication		PHI
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
	Guatemala	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Honduras	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Indonesia	WP 850 g/kg	Foliar	1.275	0.32	NS	NS
	Mexico	WP 800 g/kg	Foliar	0.48-2.4	NS	NS	0
		SC 480 g/l	Foliar	0.8-2.4	NS	NS	0
	Nicaragua	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Panama	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Philippines	WP 850 g/kg	Foliar	NS	0.212-0.319	NS	NS
	Thailand	WP 850 g/kg	Foliar	NS	0.212-0.255	NS	NS
	Turkey	WP 850 g/kg	Foliar	1.49	NS	CP 1	7
	USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-1.68		4 (7-d interval)	3 (fresh) 14 (forage) 21 (dried)
	Vietnam	SC 430 g/l	Foliar	0.86	0.27	NS	14
		WP 850 g/kg	Foliar	0.850	0.212	NS	14
Spinach	Canada	SC 480 g/l	Foliar	0.6-2.5	NS	NS	5
	USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-2.24	NS	5 (7-d interval)	14
Stone fruits,	Greece	SC 479 g/l	Foliar	0.86-3.05	0.057-0122	NS	7
general		DP 100 g/kg	Dusting	2-4	Not applicable	NS	7
		WP 850 g/kg	Foliar	0.89-3.19	0.059-0.127	NS	7
	New Zealand	SC 500 g/l	Foliar	NS	0.08-0.12	NS	1
Stone fruits: Apricot,	USA	SC 480 g/l WP 800 g/kg	Foliar	2.24-3.4		3 (7-d interval)	3
Cherries, Nectarines,	USA, California	SC 480 g/l WP 800 g/kg	Foliar	3.4-4.5		3 (14-d interval)	1
Peaches, Plums and Prunes	USA, California (dormant timing)	SC 480 g/l WP 800 g/kg	Foliar	4.5-5.6		1	Not relevant
Sugar beets	USA	SC 480 g/l	foliar	0.56-1.68		2	14
Sunflower	Australia	SC 500 g/l	Foliar	0.9-1.1	0.08-0.10	NS	3
	Myanmar	WP 850 g/kg	Foliar	1.26-2.01	0.315-1.050	NS	60
	USA	SC 480 g/l WP 800 g/kg	Foliar	1.12-1.68	0.56-0.84	2 (7-d interval)	30 (forage) 60 (seed)
Sweet corn	Australia	SC 500 g/l	Foliar	0.8-1.0	NS	NS	1 (graze)
	Canada	SC 480 g/l	Foliar	0.6-1.92	NS	CP 1	1
	New Zealand	SC 500 g/l	Foliar	0.8-1.2	0.12	CP 1	3
	USA	SC 480 g/l WP 800 g/kg	Foliar	1.121-2.242		8 (3-d interval)	2 (ears) 14 (forage) 48 (fodder)

				A	pplication		PHI
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
Sweet potato	USA	SC 480 g/l	Pre-planting dip + Foliar	0.56-2.24	0.96	Up to 8	7
Table grapes	South Africa	WP 850 g/kg	Foliar	2.65	0.106	2-3	28-42
Tomato	Australia	SC 500 g/l	Foliar	1.1	0.100	Not specified	3
	Belgium	SC 480 g/l	Foliar	0.768	Not specified	Not specified	4 (outdoor) 3 (glasshouse)
	Belize	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Brazil	SC 480 g/l	Foliar	0.864-1.08	0.108	1-6	3
		WP 850 g/kg	Foliar	1.02-1.275	0.01275	NS	3
	Canada	SC 480 g/l	Foliar	0.6-3.07	NS	CP 1	2
	Chile	WP 850 g/kg	Foliar	1.275-1.7	0.255-0.425	NS	0
	Colombia	WP 800 g/kg	Bait	0.52	Not applicable.	NS	15
		WP 800 g/kg	Foliar	1.0-1.44	0.2-0.288	NS	15
	Costa Rica	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Dominican Republic	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	El Salvador	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	France	WP 850 g/kg	Foliar	1.275	NS	1-2	7
	Guatemala	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Honduras	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	India	WP 500 g/kg	Foliar	1.0	NS	NS	7
	Italy	SC 473 g/l	Foliar	0.355-1.42	0.071-0.284	1-2	7
	Mexico	WP 800 g/kg	Foliar	0.8-2.0	NS	NS	0
		SC 480 g/l	Foliar	0.72-2.4	NS	NS	0
	Myanmar	WP 850 g/kg	Foliar	1.26-2.10	0.315-1.05	NS	60
	New Zealand	SC 500 g/l	Foliar	1.2-2.4	0.12	CP 1	0
	Nicaragua	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Panama	WP 800 g/kg	Foliar	0.56-1.12	NS	NS	10
	Philippines	WP 850 g/kg	Foliar	NS	0.212-0.319	NS	NS
	USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-2.242	0.12-0.5	7 (7-d interval)	3
Tree nuts,	Greece	SC 479 g/l	Foliar	0.57-2.44	0.057-0122	NS	7
general		DP 100 g/kg	Dusting	2-4	Not applicable	NS	7
		WP 850 g/kg	Foliar	0.595-2.55	0.059-0.127	NS	7
Turnip	Canada	SC 480 g/l	Foliar	0.6-2.52	NS	CP 1	7 (roots) 21 (tops)
	Italy	SC 473 g/l	Foliar	0.355-1.42	0.071-0.284	1-2	7

				A	pplication		PHI
Crop	Country	Formulation	Application method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	days
Vegetables,	Australia	SC 500 g/l	Foliar	0.9-1.1	0.08-0.1	NS - CP 1	3
general	Greece	DP 100 g/kg	Dusting or bait	1.5-2.5	Not applicable	NS	7
		SC 479 g/l	Foliar	0.28-1.22	0.057-0.122	NS	7
		WP 850 g/kg	Foliar	0.297-1.275	0.059-0.127	NS	7
	New Zealand	SC 500 g/l	Foliar	1.2-2.4	0.12	CP 1	1
	Spain	WP 850 g/kg	Foliar	0.425-0.85	0.085-0.170	CP 2	7
	Syria	WP 850 g/kg	Foliar	NS	0.1275-0.170	NS	No PHI
	Turkey	WP 850 g/kg	Foliar	1.7	NS	CP 1	7
	Vietnam	SC 430 g/l	Foliar	0.86	0.27	NS	14
	Zimbabwe	WP 850 g/kg	Foliar	NS	0.170	NS	7
Walnuts	Chile	SC 480 g/l	Foliar	NS	0.081	NS	1
		WP 850 g/kg	Foliar	NS	0.085	NS	1
	Mexico	WP 800 g/kg	Foliar	NS	0.2-0.28	NS	4
		SC 480 g/l	Foliar	NS	0.192-0.24	NS	4
	USA	SC 480 g/l WP 800 g/kg	Foliar	2.24-5.6		4 (7-d interval)	14
Wheat	Argentina	SC 480 g/l	Foliar	0.845	0.422	1-2	7
		WP 850 g/kg	Foliar	0.85	0.425	1-2	7
	Brazil	SC 480 g/l	Foliar	0.912-1.08	NS	1 to 2	30
	Canada	SC 480 g/l	Foliar	1.2-2.52 (ground) 0.55-1.76 (aerial)	NS	1 to 2 (7 to 14 days interval)	14
	India	WP 500 g/kg	Foliar	1.0	NS	NS	7
	Spain	WP 850 g/kg	Foliar	0.425-0.85	0.085-0.170	CP 1	7
	USA	SC 480 g/l WP 800 g/kg	Foliar	0.56-1.68		2 (14-d interval)	7 (forage) 21 (grain & straw)

NA: not applicable NR: not reported NS: not specified CP: commercial practice

#### RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Tables 33 to 64 show the supervised trials conducted on crops in the USA, Latin America and Europe. In all US trials residues are an average from replicate plots. When residues were not detected they are shown as below the LOQ (e.g. <0.02 mg/kg). Residues are rounded to two significant figures except those near the LOQ which are rounded to one significant figure. Underlined values are within GAP in each country (±30%) and double-underlined values are from trials according to maximum GAP

<sup>&</sup>lt;sup>1</sup> Do not apply more than 22.4 kg/ha per crop <sup>2</sup> Against California red scale and yellow scale

<sup>&</sup>lt;sup>3</sup> For California red scale not more than 1 application per season

 $(\pm 30\%)$  and have been considered for the estimation of maximum residue levels and STMRs: unless otherwise specified, all trials were in the field with foliar application.

Reports submitted according to GLP requirements include method validation, dates of analyses of samples, sprayers used and their calibration, plot and residue sample sizes and sampling methods. Trials before 1990 may not be completely according to GLP but were considered for the estimation. All trials included control plots. Unless so specified, residue data were not corrected for recoveries. Trials reported only as summary Tables were not evaluated.

The Meeting received information on the following.

#### **Fruits:**

Table 33. Citrus fruits Table 34. Pome fruits

Table 35. Apple (individual fruits)

Table 36. Peaches and plums

Table 37. Cherries Table 38. Grapes

Table 39. Olives

### **Vegetables:**

Table 40. Cabbage

Table 41. Fruiting vegetables

Table 42. Sweet corn

Table 43. Lettuce and spinach

Table 44. Garden beet tops and turnip greens

Table 45. Soya beans

Table 46. Root and tuber vegetables

Table 47. Sugar beets

Table 48. Asparagus

#### **Cereals:**

Table 49. Field corn Table 50. Barley

Table 51. Rice

Table 52. Rye

Table 53. Sorghum

Table 54. Wheat

#### **Nuts:**

Table 55. Almonds, pecans, pistachios and walnuts

#### Oil seeds:

Table 56. Sunflower

#### Animal feed:

Table 57. Soya bean forage and hay

Table 58. Corn forage and fodder

Table 59. Barley forage and straw

Table 60. Rice straw

Table 61. Rye forage and straw

Table 62. Sorghum forage, silage and fodder

Table 63. Wheat forage and straw

Table 64. Almond hulls

#### Citrus fruits

Supervised trials on citrus fruits were conducted in the USA (21 trials), Italy (4 trials), and Spain (4 trials) (Table 33).

Table 33. Residues of carbaryl in citrus fruits from supervised trials

Crop			Application			PHI,	Residues	
Location, year	Form.	kg ai/ha	Water, l/ha or kg ai/hl	No.	Sample	Days	mg/kg	References
GAP, USA: 480 SC	or 800 WP j	formulation; ma	x. of 8 application	ons at 2.4	12-8.4 kg ai/ha	a, max.	22.4 total kg	g ai/ha, 5 days
PHI In California 1 appl	lication at 5.	6-17.9 kg ai/ha						
GRAPEFRUIT								
CA, USA, 1994	480 SC	2.79-8.24	6900-7351	4	Fruit	5	3.5	Study No.
94-0081		Total: 22.7	(water, l/ha)					US94S08R
CA, USA, 1994	480 SC	2.81-8.26	6905-8470	4	Fruit	5	2.5	(Hovis, 1995)
940082		Total: 22.4						
CA, USA, 1995	480 SC	2.80-8.3	1398-1451	4	Fruit	5	6.8	
940083		Total: 22.1						
FL, USA, 1994	480 SC	2.78-8.16	2866-2962	4	Fruit	5	<u>0.59</u>	
940084		Total: 21.8						
FL, USA, 1994	480 SC	2.53-8.13	2655-2950	4	Fruit	5	<u>1.9</u>	
940085		Total: 21.7						
FL, USA, 1994	480 SC	2.63-8.48	3303-3735	4	Fruit	5	<u>2.8</u>	
940086		Total: 22.7						
LEMON								
CA, USA, 1994	480 SC	2.80-8.39	-	4	Fruit	5	5.5	
940087		Total: 22.4						
CA, USA, 1994	480 SC	2.80-8.30	7241-7340	4	Fruit	5	5.0	
940088		Total: 22.3						
AZ, USA, 1995	480 SC	2.77-8.37	1410-1569	4	Fruit	5	<u>4.8</u>	
940089		Total: 19.3						
AZ, USA, 1995	480 SC	2.58-8.30	1295-1662	4	Fruit	5	<u>5.1</u>	
940090		Total: 21.2						
ORANGES								
CA, USA, 1994	480 SC	2.92-7.86	7028-7856	4	Fruit	5	4.5	
940075		Total: 22.8						
CA, USA, 1993	480 SC	2.96-8.69	7173–7400	4	Fruit	5	6.5	
940076		Total: 23.1						
CA, USA, 1995	480 SC	2.86-8.54	1520-1548	4	Fruit	5	8.1	
940077		Total: 22.5						

Crop			Application			PHI,	Residues	
Location, year	Form.	kg ai/ha	Water, l/ha or kg ai/hl	No.	Sample	Days	mg/kg	References
CA, USA, 1995	480 SC	4.37	3516	2	Fruit	5	6.5	Study No.
950132		17.94	3760			10	3.8	US95S11R
CA, USA, 1995	480 SC	4.48	3918	2	Fruit	5	3.7	(Ely, 1997a)
950133		17.94	3909			10	3.2	
CA, USA, 1995	480 SC	4.48	3862	2	Fruit	5	4.6	
950134		18.05	3844			10	4.3	
CA, USA, 1995	480 SC	4.48	3825	2	Fruit	5	2.9	
950135		18.05	3937			10	3.1	
CA, AZ, 1995	480 SC	4.37	1365	2	Fruit	5	10	
950136		18.4	1300			10	6.7	
FL, USA, 1994	480 SC	2.68-8.04	2141–2323	4	Fruit	5	<u>5.7</u>	Study No.
940078		Total: 21.7						US94S08R
FL, USA, 1995	480 SC	2.80-8.36	1394–1422	4	Fruit	5	<u>4.2</u>	(Hovis, 1995)
940398		Total: 22.3						
FL, USA, 1995	480 SC	2.84-8.42	1403-1424	4	Fruit	5	<u>4.2</u>	1
940399		Total: 22.4						
GAP, Italy: 476 SC,	0.32-0.63 k	g ai/hl, 7 days P	PHI					
Italy, 1997 (South)	480 SC	2.94	0.147 (kg ai/hl)	1	Fruit	0 7 14 21 29	1.4 0.93 0.55 0.39 0.39	R&D- 9815892 (Maestracci, 1998a)
		2.93	0.147	1	Fruit	0 7 14 21 29	2.1 0.83 0.71 0.64 0.54	
Italy, 1998 <sup>1</sup> (South)	480 SC	2.86	0.143	1	Pulp Peel Fruit Pulp Peel Fruit	7 7 7 14 14 14	0.32 9.6 2.6 0.18 7.0 2.0 <sup>3</sup>	R&D- 9916493 (Yslan and Baudet, 1999a)
		2.89	0.144	1	Pulp Peel Fruit Pulp Peel Fruit	7 7 7 14 14 14	0.53 10 3.6 0.26 7.7 2.5	
GAP, SPAIN: WP 8	350 formulat	tion, 0.85-1.6 kg	ai/ha or 0.085-	0.16 kg ai/	hl, 7 days PI	II		
Spain, 1997	850 WP	4.27–4.00	0.14-0.16	2	Fruit	7	<u>0.82</u>	R&D- 9815785
			(kg ai/hl)			14	0.56	(Gatuaud and
		3.65–3.80	0.14	2	Fruit	7	<u>4.4</u>	Maestracci,
						14	2.0	1998)

Crop			Application			PHI,	Residues	
Location, year	Form.	kg ai/ha	Water, l/ha or kg ai/hl	No.	Sample	Days	mg/kg	References
Spain, 1998	850 WP	2.65–2.56	0.14	2	Fruit	0 7 14 21 29	4.0 2.8 <u>3.2</u> 1.1 1.2	R&D- 9916491 (Yslan and Baudet, 1999b)
Spain, 1999	850 WP	4.70–4.41	0.14	2	Fruit	0 7 14 21 29	3.2 3.2 <u>3.4</u> 2.3 2.2	

<sup>&</sup>lt;sup>1</sup> Total residues obtained by calculation from residues in peel and in pulp.

### **Pome Fruits**

Thirty one supervised trials on apples and pears were conducted in Argentina (1 trial), Canada (8 trials), France (8 trials), Italy (4 trials), the UK (1 trial) and the USA (8 trials) (Table 34).

Table 34. Residues of carbaryl in apples and pears from supervised trials.

Crop		Appl	ication		PHI,	Residues	
Country, year	Form.	kg ai/ha	kg ai/hl	No.	Days	mg/kg	References
APPLE							
GAP ARGENTINA	(apple and p	pear) WP 850	, at 0.076-0.127 I	kg ai/hl, 3	days PF	HI, 11 days Pl	HI for Granny Smith apple
Argentina (Granny Smith)	850 WP	1.02	0.102	1	0 3 7 14 21 28	3.8 3.9 2.5 1.2 1.1 0.87	Battla, 1993 (only summary table)
GAP CANADA: (a <sub>l</sub>			mulation at 1.34-	-2.75 kg a	i/ha, 11	-	
Canada	480 SC	0.5		2	7	0.32	Project 801R11
1985	480 SC	0.5		2	7	0.19	(Davis, 1986)
	570 W	0.5		2	7	0.14	
	50 W	0.5		2	7	0.17	
GAP USA (pome fr 16.8/crop), 3 days I		and 800 WP f	ormulations, Ma	x. 8 appl	ications (	at 0.56–3.36 k	g ai/ha (maximum of
USA, WA, 1995	480 SC	3.36		5	3	<u>9.6</u>	Study No.
	480 SC	3.43- 3.50		5	3	<u>10</u>	US95S06R
USA, PA, 1995	480 SC	3.34-3.48		5	3	<u>8.8</u>	(Mede, 1996)
GAP FRANCE (ap	ple): 850 WF	formulation,	max. 2 appl. at 0	0.042-0.08	35 kg ai/l	hl, 1000-2000	l/ha, 7 days PHI
France (North) 1997	850 WP	0.98 1.02 1.02	0.20 0.22 0.20	3	2 7 14 21 28	0.42 0.29 0.21 0.17 0.12	R&D-9815200 (Richard and Maestracci 1998a)

Crop		App	lication	i	PHI,	Residues	
Country, year	Form.	kg ai/ha	kg ai/hl	No.	Days	mg/kg	References
	850 WP	1.02	0.25	3	2 7 14 21 28	1.7 0.66 0.51 0.28 0.20	
France (South) 1997	850 WP	0.948 0.998 1.03	0.17	3	2 7 15 21 28	0.71 0.23 0.10 <0.05 <0.05	R&D-9815343 (Richard and Maestracci, 1998a)
	850 WP	1.01 1.09 1.09	0.126 0.11 0.10	3	2 7 15 21 28	0.34 0.40 0.36 0.30 0.30	
France (South), 1998 Trial 98540AV1	480 SC	0.97	0.11-0.23	3	7 14	0.52 0.34	R&D-9916086 (Yslan and Baudet,
France (North), 1998 Trial 98540AM1	480 SC	1.02	0.17	3	7 14	0.49 0.41	1999c)
France (South), 1998 Trial 98540AR1	480 SC	1.02	0.17	3	6 14	0.24 0.15	
France (South), 1998	480 SC	1.02	0.16	3	2 hrs 7 14 21 28	1.6 0.66 0.36 0.23 0.29	(Yslan and Baudet, 1999d)
GAP ITALY (apple at	nd pear): 4	73 SC formul	ation at 0.06-0.12	2 kg ai/hl	and 7 d	ays PHI	
Italy, 1997	480 SC	1.19-1.25	0.08	3	7 14	<u>0.22</u> 0.16	R&D-9815915 (Maestracci,
	480 SC	1.6-1.25	0.077-0.08	3	7 14	<u>0.57</u> 0.57	1998b)
Italy, 1989	480 SC	1.08	0.25	1	0 7 14 21	0.97 0.69 <0.2 <0.2	R&D-9015846 (Dupuis and Muller, 1990) <sup>1</sup>
Italy, 1988	480 SC	1.15-1.24	0.077-0.082	3	0 7 14 21 28	0.68 0.68 0.74 0.62 0.46	R&D-9916450 <sup>2</sup> (Yslan and Baudet, 1999e)
Italy, 1988	480 SC	1.16-1.23	0.078-0.82	3	7 14	<u>0.67</u> 0.54	R&D-9915543 (Yslan and Baudet, 1999f)
UK, 1998 (No GAP)	480 SC	1.03	0.103	3	7 14	0.15 0.06	R&D-9915436 (Yslan and Baudet, 1999g)
PEAR	<u> </u>	<u> </u>	<u> </u>	<u> </u>	1	<u> </u>	1
Argentina	850 WP	1.02	0.102	1	0 2 7 14	3.1 1.8 1.3 0.85	Battla, 1993 (only summary table)
Canada	480 SC	0.5		2	7	0.28	Project 801R11

Crop		Appl	lication		PHI,	Residues	
Country, year	Form.	kg ai/ha	kg ai/hl	No.	Days	mg/kg	References
1985	480 SC	0.5		2	7	0.05	(Davis, 1986a)
	50 W	0.5		2	7	0.12	
	50 W	0.5		2	7	0.06	
USA, WA, 1995	480 SC	3.25-3.42		5	3	<u>3.5</u>	Study No.
	480 SC	3.26-3.41		5	3	<u>2.9</u>	US95S06R
	480 SC	3.34-3.37		5	3	<u>4.0</u>	(Mede, 1996)
USA, CA, 1995	480 SC	3.63		5	3	<u>2.8</u>	
	480 SC	3.36-3.37		5	3	<u>0.98</u>	

In three trials conducted on apples in France (Yslan and Baudet, 1999c) and in one trial in Italy (Yslan and Baudet, 1999f) residues were determined in individual apples (Table 35).

Table 35. Residues of carbaryl in individual apples from supervised trials.

	AV1 (France uth)	Trial 98540AM	1 (France North)	Trial 98540A Sou		Trial 98706AGR1 (Italy)	
Sample	mg/kg	Sample	mg/kg	Sample	mg/kg	Sample	mg/kg
148469	0.50	156878	0.67	152612	0.22	147809	0.64
148471	0.54	156880	0.31	152610	0.26	147810	0.76
148712	0.43	156886	0.55	152620	0.13	147811	0.94
148714	0.31	156888	0.27	152622	0.17	147812	1.28
147960	0.56	156791	0.28	152660	0.64	147813	0.60
147962	0.39	156792	1.05	152661	0.03	147814	0.75
147963	0.49	156793	0.75	152662	0.22	147815	0.79
147964	0.58	156794	0.41	152663	0.15	147816	1.26
147965	0.49	156795	0.48	152664	0.10	147817	0.54
147966	0.37	156796	0.55	152665	0.48	147818	0.47
147967	0.33	156797	0.39	152666	0.27	147819	0.85
147968	0.71	156798	0.13	152667	0.07	147820	0.61
147969	0.32	156799	0.61	152668	0.01	147821	0.14
147970	0.21	156800	0.05	152669	0.16	147822	0.54
147971	0.32	156801	1.23	152670	0.28	147823	0.14
147972	0.09	156802	0.26	152671	0.13	147824	0.41
147972	0.09	156803	0.13	152672	0.28	147825	0.67
147973	0.55	156804	0.09	152673	0.15	147826	0.55

 $<sup>^{1}</sup>$  Colorimetric method  $^{2}$  except for the first sampling, residues were found in untreated samples, between 0.20 and 0.36 mg/kg;

	AV1 (France uth)	Trial 98540AM	1 (France North)	Trial 98540A Sout		Trial 98706AGR1 (Italy)	
Sample	mg/kg	Sample	mg/kg	Sample	mg/kg	Sample	mg/kg
147974	0.48	156805	0.15	152674	0.70	147827	0.51
147975	0.23	156806	0.18	152675	0.18	147828	0.70
147976	0.29	156807	0.53	152676	0.04	147829	0.52
147977	0.45	156808	0.07	152677	0.15	147830	0.68
147978	0.56	156809	0.06	152678	0.16	147831	0.39
147979	0.56	156810	0.48	152679	0.15	147832	0.34
147980	0.46	156811	0.11	152680	0.29	147874	1.70
147981	0.52	156812	0.45	152681	0.08	147875	0.91
147982	0.77	156813	0.21	152682	0.16	147876	0.87
147983	0.46	156814	0.32	152683	0.21	147877	1.69
147984	0.44					147878	0.46
148003	0.81					147879	0.90
148004	0.49					147880	0.69
148005	0.49					147881	0.57
148006	0.37					147882	0.64
148007	0.25					147883	0.51
148008	0.40					147884	1.55
148009	0.35					147885	0.59
148010	0.28					147886	0.75
148011	0.30					147887	0.40
148012	0.44					147888	0.44
148013	0.54					147889	0.42
148014	0.35					147890	0.49
148015	0.40					147891	0.82
148016	0.53					147892	0.72
148017	0.32					147893	0.36
148018	0.48					147894	0.52
148019	0.47					147895	0.34
148020	0.46					147896	0.64
148021	0.49					147897	0.87
148022	0.26					159042	0.83

	OAV1 (France outh)	Trial 98540AM1 (France North)		Trial 98540A Sout		Trial 98706AGR1 (Italy)		
Sample	mg/kg	Sample	mg/kg	Sample	mg/kg	Sample	mg/kg	
148023	0.56					159044	0.51	
148024	0.46					159050	0.59	
148025	0.40					159052	0.50	
148026	0.32							
N= 53 Average 0.43 mg/kg Std. deviation 0.14 mg/kg Highest 0.81 mg/kg		N= 28 Average 0.39 m Std. deviation 0 Highest 1.2 mg/	.29 mg/kg	N= 28 Average 0.21 n Std. deviation 0 Highest 0.70 mg	.16 mg/kg	N= 52 Average 0.68 I Std. deviation ( Highest 1.7 mg	0.33 mg/kg	

### **Stone fruit**

<u>Peaches and plums</u>. Ten supervised trials were conducted in the USA on peaches and ten on plums, and one on peaches in Italy (Table 36).

Table 36. Residues of carbaryl in peaches and plums from supervised trials.

		$\mathbf{A}_{i}$	pplication			PHI,	Residues	
Country, year	Form.	kg ai/ha	Water l/ha or kg ai/hl	No.	Interval, days	Days	mg/kg	References
			and 800 WP for 1.4-5.6 kg ai/ha, 1				olications at 2.24-3.4	kg ai/ha, 7 days
PEACHES								
CA, 1994	480 SC	3.59-3.70	1309-1393 water, l/ha	3	7	3	5.5	Study No.
	480 SC	3.59	1450	3	7	3	2.6	US94S17R
	480 SC	3.36-3.48	1384-1450	3	7	3	2.0	(Ely, 1995a)
GA, 1994	480 SC	3.36-3.70	524-533	3	7	3	<u>0.96</u>	
SC, 1994	480 SC	3.36	916-935	3	7	3	<u>3.6</u>	
	480 SC	3.36	926-945	3	7	3	<u>2.3</u>	
PA, 1994	480 SC	3.36	1047-1066	3	7	3	<u>3.0</u>	
CA, 1996	800 WP	4.38-4.48	697-709	3	14	1	<u>7.6</u>	Study No.
	800 WP	4.48-4.57	748-751	3	14	1	<u>4.8</u>	96S10562
	800 WP	4.48-4.57	1869-1907	3	14	1	<u>4.8</u>	(Macy, 1997))
GAP ITALY:	480 SC form	ulation, 0.071	-0.118 kg ai/hl, 7	days	PHI	•	•	
Italy, 1989	480 SC	2.15	0.25 <b>kg ai/hl</b>	1		0 7-21	0.62 <0.2	AG-9015837 <sup>1</sup> Dupuis and Muller, 1990a)

		Aj	pplication			PHI,	Residues		
Country, year	Form.	kg ai/ha	Water l/ha or kg ai/hl	No.	Interval, days	Days	mg/kg	References	
PLUMS (USA)									
CA, 1994	480 SC	3.25-3.48	963-1019 water, l/ha	3	7	3	0.05	Study No.	
	480 SC	3.36	1038-1160	3	7	3	0.06	US94S17R	
MI, 1994	480 SC	3.36	935	3	7	3	<u>2.1</u>	(Ely, 1995a)	
	480 SC	3.36	935	3	7	3	<u>1.4</u>		
0R, 1994	480 SC	3.36	935-1430	3	7	3	<u>0.37</u>		
	480 SC	3.36	935-1430	3	7	3	<u>1.6</u>		
CA, 1996	800 WP	4.47-4.52	743-751	3	14	1	<u>1.1</u>	Study No.	
	800 WP	4.50-4.55	751-758	3	14	1	<u>0.99</u>	96S10562	
CA, 1996	800 WP	4.47-4.64	751-778	3	14	1	<u>0.69</u>	(Macy, 1997)	
CA, 1996	800 WP	4.47-4.54	1864-1953	3	14	1	<u>1.1</u>		

<sup>&</sup>lt;sup>1</sup> Colorimetric method was used

Cherries. Nine trials were conducted on cherries in the USA in 1994 and 1996 (Table 37).

Table 37. Residues of carbaryl in cherries in the USA from supervised trials.

Crop			Application	n		PHI,	Residues			
Location, year	Form.	kg ai/ha	Water l/ha	No.	Interval, days	Days	mg/kg	References		
GAP, US: 480 SC and 800 WP formulations, max. 3 x 2.24–3.36 kg ai/ha, 7 days interval (max. 15.7 kg ai/ha/season); 3 days PHI. In California: 3.36-4.5 kg ai/ha, 14 days interval (max. 10.1 kg/ai/ha total), 1 day PH,I in production season; 4.5-5.6 kg ai/ha in dormant season										
CA, 1994	480 SC	3.36-3.48	1244-1281	3	7	3	6.3	Study No.		
CO, 1994	480 SC	3.48	486-514	3	7	3	<u>6.7</u>	US94S17R		
MI, 1994	480 SC	3.36	935	3	7	3	<u>2.4</u>	(Ely, 1995a)		
NY, 1994	480 SC	3.36	468	3	7	3	<u>3.4</u>			
OR, 1994	480 SC	3.36	935	3	7	3	<u>16</u>			
WA, 1994	480 SC	3.63	935	3	7	3	<u>4.7</u>			
WA, 1994	480 SC	3.48	1870-1908	3	7	3	<u>3.9</u>			
CA, 1996	800 WP	4.42-4.45	927-944	3	14	1	<u>4.7</u>	Study No.		
Sweet cherry	800 WP	4.44-4.46	934-938	3	14	1	<u>2.1</u>	96S10562 (Macy, 1997)		

### Berries and other small fruit

<u>Grapes</u>. Seventeen supervised field trials were conducted on grapes in the USA in 1988 and 1994 (Table 38).

Table 38. Residues of carbaryl in grapes in the USA from supervised trials.

Crop			Application			PHI,	Residues	
Location, year	Form.	kg ai/ha	Water l/ha	No.	Interval, days	Days	mg/kg	References
<b>GAP, US:</b> 480 S PHI	C and 800 V	VP formulation	on, max. 5 time	es at 1.1	2-2.24 kg ai/ha	(max. total	10.2), min 7 da	ys interval, 7 days
CA, 1988	800 WP	2.24	1122	2	15	0 3 7	5.5 4.0 <u>4.5</u>	Project No. 801R10 (Romine, 1989)
	800 WP	2.24	2338	2	15	0 3 7	1.0 0.40 <u>0.42</u>	
	800 WP	2.24	-	2	15	0 3 7	5.6 5.8 <u>3.8</u>	
	800 WP	2.24	1403	2	15	0 3 7	8.0 7.8 <u>6.5</u>	
NY, 1988	800 WP	2.24	468	2	15	0 3 7	3.8 4.2 <u>2.4</u>	
	800 WP	2.24	468	2	15	0 3 7	7.4 8.1 <u>4.9</u>	
	800 WP	2.24	-	2	15	0 3 7	11 5.9 <u>5.3</u>	
AZ, 1994	480 SC	2.22-2.29	1375-1431	5	7	7	<u>6.5</u>	Study No.
CA, 1994	480 SC	2.23 -2.31	936-982	5	7	7	<u>7.9</u>	US94S29R
	480 SC	2.18-3.36	720-786	5	7	7	<u>2.4</u>	(Tew and Mede,
	480 SC	2.24-2.39	1412-1505	5	7	7	<u>33</u>	1995)
	480 SC	2.24-2.32	468-486	5	7	7	<u>3.0</u>	
NY, 1994	480 SC	2.22-2.30	468-486	5	7	7	<u>7.2</u>	
	480 SC	2.20-2.26	468-477	5	7	7	<u>7.5</u>	
WA, 1994	480 SC	2.24	965	5	7	7	<u>2.4</u>	
	480 SC	2.12-2.14	888-916	5	7	7	<u>3.3</u>	
	480 SC	2.24	963	5	7	7	<u>6.2</u>	

# **Tropical fruits**

<u>Olives</u>. Fourteen supervised trials were conducted on olive trees in the USA, Spain, Greece and Italy (Table 39). The recommended PHI in the USA is 14 days and in Europe 7 days.

Table 39. Residues of carbaryl in olives from supervised trials.

Crop			Application	n		PHI,	Residues,	
Location, year	Form.	kg ai/ha	kg ai/hl	No.	Sample	days	mg/kg	References
GAP, Greece: 85	0 WP formu	lation at 0.0.	59–0.17 kg ai/h	l, 7 days I	PHI			
Greece 1998	850 WP	2.11	0.139	1	Fruit with stone Stoned fruit	14 7 14	$0.91^{2} \\ \frac{1.9}{1.3}$	R&D-9916451 (Yslan and Baudet, 1999h)
GAP, Italy: 480 S	SC formulati	on, at 0071	–0.142 kg ai/hl	, 7 days P	PHI			
Italy 1997	480 SC	2.15	0.142	1	1 Fruit with stone		7.9 <0.05 <sup>1</sup> 1.9 0.70 0.16 <sup>2</sup>	R&D-9916485 (Barrière and Gateaud, 1999)
					Stoned fruit	28	0.20	
					Fruit with stone  Stoned fruit	0 7 14 21 28 28	$ \begin{array}{c} 10 \\ \underline{7.9} \\ 2.1 \\ 1.0 \\ 0.20^2 \\ 0.28 \end{array} $	
Italy 1998	480 SC	2.15	0.143	1	Fruit with stone  Stoned fruit	0 7 14 21 28 28	9.4 <u>1.6</u> 1.2 0.10 0.21 <sup>2</sup> 0.28	R&D-9916501 (Yslan and Baudet, 1999i)
GAP, Spain: 850	WP formula	ution, 0.8-1.7	kg ai/ha or 0.0	)85–0.17 F	kg ai/hl, 7 days PHI			
Spain 1997	850 WP	1.75	0.140	2	Fruit with stone	7 14 7	0.07 0.05 0.08	R&D-9815730 (Richard and Maestracci, 1998c)
					fruit	14	< 0.05	Widestracei, 1996c)
1998	850 WP	2.13	0.142	2	Fruit with stone	0 7 14 21 28	20 <u>26</u> 2.7 1.3 1.2 <sup>2</sup>	R&D-9916462 (Yslan and Baudet, 1999j)
					Stoned fruit	28	1.7	
	850 WP	2.1	0.140	2	Fruit with stone Stoned fruit	0 7 14 21 28 28	$ \begin{array}{c} 20 \\ \underline{22} \\ 4.0 \\ 1.9 \\ 1.9^2 \\ 3.4 \end{array} $	
GAP, USA: 480 S	SC or 800 W	P formulatio	ns, max. 2 appi	lications a	at 5.6–8.4 kg ai/ha, I	4 days	PHI	
USA, CA, 1994	480 SC	5.61		2	Fruit with stone	14	<u>5.8</u>	Study No.
	480 SC	5.63		2	Fruit with stone	14	2.8	US94S09R
	480 SC	5.58		2	Fruit with stone	14	<u>5.5</u>	(Macy and Lee,
	480 SC	5.48		2	Fruit with stone	14	<u>1.4</u>	1995)

Crop		Application						
Location, year	Form.	kg ai/ha	kg ai/hl	No.	Sample	days	mg/kg	References
USA, CA, 1996	800 WP	8.52		2	Fruit with stone	14	<u>4.0</u>	Study No.
	800 WP	9.17		2	Fruit with stone	14	<u>3.3</u>	96S10561
	800 WP	8.49		2	Fruit with stone	14	<u>6.6</u>	(Mede, 1997)

<sup>&</sup>lt;sup>1</sup> unexplained result <sup>2</sup> by calculation

### Brassica vegetables

Cabbage. A summary Table was provided by the Government of Thailand with results of trials on Chinese cabbage and kale from 1995 to 1997 (Table 40).

Table 40. Carbaryl residues in cabbage and kale in Thailand.

		Residues, mg/kg, at PHI, days								
Dose (kg ai/ha)	0	1	3	5	7	10				
Cabbage										
1.28	18	7.1	0.25	0.04	< 0.01					
2.55	18	5.6	0.85	0.13	< 0.01					
2.56	27	20	1.6	0.14	0.07					
5.1	51	18	1.6	0.31	< 0.01					
		Kale								
2.34	121	72	3.5	1.7	1.2	0.32				
2.34	101	96	0.58	0.18	0.04	0.01				
4.67	370	225	2.6	0.42	0.09	0.66				

### **Fruiting vegetables**

Eighteen supervised trials were conducted from 1994 to 1996 on egg plants and tomatoes in France and sixteen on bell peppers and tomatoes in the USA. Four trials in chilli peppers were reported by the Government of Thailand (Table 41).

Table 41. Residues of carbaryl in fruiting vegetables from supervised trials.

		Applica	tion		PHI,	Residues			
Year	Form.	kg ai/ha	kg ai/hl	No.	Days	mg/kg	References		
GAP FRANCE (egg plant and tomato): 850 WP formulation at 1.275 kg ai/ha and 7 days PHI									
EGG PLANT				_	_				
1995	850WP	1.275	0.35	2	7	<u>&lt;0.2</u>	Study 95-563		
	850 WP	1.275	0.51	2	7	<u>&lt;0.2</u>	Richard and Maestracci, 1996		
North 1996	850 WP	1.275	0.51	2	2 h 2 4 7	0.21 0.26 0.17 <u>0.08</u>	R&D- 9616669 (Maestracci, 1997a; Yslan, 1999a,b)		

		Applica	ation		PHI,	Residues	
Year	Form.	kg ai/ha	kg ai/hl	No.	Days	mg/kg	References
South	850 WP	1.275	0.14	2	2 h 3 5 7	0.86 0.59 0.39 <u>0.16</u>	
1994	850 WP	1-1.5		3	15	< 0.20	R&D-9516427
	850 WP	0.97-1.53		3	20	< 0.20	(Richard and
	850 WP	1-1.5		3	9	<u>0.49</u>	Muller, 1995)
1995	850 WP	1.275	0.51	2	2 h 2 4 7	0.49 ≤0.2 <0.2 <u>&lt;0.20</u>	R&D-9615862 (Richard and Muller, 1996a)
	850 WP	1.275	0.2	2	2 h 2 4 7	1.6 0.56 0.45 <u>&lt;0.2</u>	
North	850 WP	1.275	0.51	2	8	<u>0.06</u>	R&D-9616675
1996							(Maestracci, 1997b; Yslan, 1999a)
BELL PEPPER	1		1	1	ı		
USA, CA, 1994	480 SC	2.15-2.23		4	3	<u>1.8</u>	Study No.
	480 SC	2.15-2.29		4	3	<u>2.0</u>	US94S14R
USA, FL, 1994	480 SC	2.31-2.33		4	3	<u>0.33</u>	(Ely, 1995b)
	480 SC	2.20-2.34		4	3	<u>3.8</u>	
USA, TX, 1994	480 SC	2.13-2.26		4	3	<u>0.61</u>	
CHILLI PEPPER-	-GAP THAI	LAND: 0.085-0	.127 kg ai/hl				
Thailand, 1995	NS	0.63		NS	0 1 3 5 7	5.4 1.4 0.73 0.39 0.85	Summary Table provided by the Government of Thailand
	NS	0.64		NS	0 1 3 5 7 10 15	1.2 1.1 0.72 0.62 0.55 0.25 0.04	
	NS	1.26		NS	0 1 3 5 7	8.7 5.7 0.77 0.95 2.74	

		Applica	ation		PHI,	Residues	
Year	Form.	kg ai/ha	kg ai/hl	No.	Days	mg/kg	References
	NS	1.28		NS	0 1 3 5 7 10 15	3.8 2.3 1.8 1.2 0.88 0.44 0.26	
ТОМАТО							
USA, CA, 1995	480 SC	2.24-2.31		4	3	<u>2.4</u>	Study No.
	480 SC	2.22-2.33		4	3	<u>1.4</u>	US95S05R
	480 SC	2.18-2.29		4	3	<u>0.52</u>	(Macy, 1996)
	480 SC	2.19-2.29		4	3	<u>1.1</u>	
	480 SC	2.24-2.35		4	3	0.08	
	480 SC	2.24-2.35		4	3	<u>2.2</u>	
	480 SC	2.13-2.35		4	3	<u>2.3</u>	
	480 SC	2.23-2.28		4	3	<u>0.85</u>	
	480 SC	2.21-2.24		4	3	<u>1.9</u>	
	480 SC	2.21-2.24		4	3	<u>0.67</u>	
USA, FL, 1995	480 SC	2.14-2.22		4	3	<u>0.47</u>	
France, 1995	850 WP	1.275		2	7	<u>0.22</u>	R&D-9616006
	850 WP	1.275		2	7	<u>&lt;0.20</u>	(Richard and
	850 WP	1.275		2	7	<u>0.21</u>	Muller, 1996b)
	850 WP	1.275		2	2 h 2 4 7	0.56 0.29 0.22 <u>≤0.2</u>	R&D-9615857 (Richard and Muller, 1996c)
	850 WP	1.275		2	2 h 2 4 7	0.93 0.73 0.69 <u>0.41</u>	
France, 1996	850 WP	1.275		2	2 h 2 4 7	0.85 0.34 0.14 <u>0.11</u>	R&D-9915143 (Maestracci, 1996a; Yslan, 1999d)
	850 WP	1.25-1.29		2	7	<u>0.06</u>	R&D-9816095
indoor	850 WP	1.21-1.27		3	7 14	$\frac{0.31}{0.08}$	(Maestracci, 1996b Yslan, 1999c)

NS: not specified

 $\underline{\text{Sweet corn}}.$  Six trials were conducted in the USA in 1995, and two trials in Thailand (Table 42).

Table 42. Residues of carbaryl in sweet corn treated 8 times with 480 SC in the USA in 1995 and in Thailand.

	Appli	cation		PHI,	Residues <sup>1</sup>	
Location	kg ai/ha	Water l/ha	Sample	Days	mg/kg	Referrences
GAP, USA: 480 SC	, 8 applicati	ons of 1.12-	-2.24 kg ai/ha,	28-187 l wat	er/ha, PHI: 2 d	lays for ears
USA, WA	2.25	215	Ears <sup>1</sup>	2	0.05	Project S86-024-R01
USA, ID	2.28	119	Ears	2	0.04	Kowite, 1996
	2.24	119	Ears	2	0.04	
USA, MN	2.26	192	Ears	2	< 0.02	
USA, WI	2.25	267	Ears	2	0.02	
	2.28	267	Ears	2	< 0.02	
GAP THAILAND	for corn–0.	1-0.2 kg ai/l	ıl			
Thailand, 1991	1.02		Kernels	0 1-7	0.01 <0.01	Summary Table provided by the
	2.04		Kernels	0 1 3-7	0.01 0.01 <0.01	Government of Thailand

<sup>&</sup>lt;sup>1</sup> kernels and cobs with husks removed

# Leafy vegetables

<u>Lettuce and spinach</u>. Twenty supervised trials were conducted on lettuce and spinach in Canada in 1984 (Table 43).

Table 43. Residues of carbaryl in lettuce and spinach from supervised trials in Canada in 1984 after 2 applications of 480 SC and 50 W formulations. Project 801R11 (Davis, 1986a).

	Application	PHI,	Residues							
Formulation	kg ai/ha	Days	mg/kg							
GAP, 480 SC, 0.6-2.5 kg	g ai/ha, 21 days PHI for	spinach; 5 d	ays lettuce							
Lettuce										
480 SC	2.63	13	3.8							
	2.0	14	3.2							
	2.0	14	0.07							
	2.0	14	1.0							
	2.0	14	5.5							
50W	2.25	13	0.79							
	2.0	14	1.7							
	2.0	14	0.05							
	2.0	14	1.6							
	2.0	14	3.6							
Spinach										
480 SC	2.63	13	0.39							
	2.0	14	4.3							

	Application	PHI,	Residues
Formulation	kg ai/ha	Days	mg/kg
	2.0	14	0.03
	2.0	14	11
	2.0	14	6.2
50W	2.25	13	0.06
	2.0	14	0.32
	2.0	14	< 0.02
	2.0	14	9.1
	2.0	14	2.4

<u>Garden tops and turnip greens</u>. Eight trials were conducted on garden beet tops and eleven on turnip greens in the USA (Table 44).

Table 44. Residues of carbaryl in garden beet tops and turnip greens in trials in the USA in 1994 with 3 applications of 480 SC formulation and 7 days PHI. Study No. US94503R (Kowite, 1995a).

	Application	Residues					
State	kg ai/ha	mg/kg					
GAP (carrots and garden beets): 480 SC formulation, max. 6 applications of 0.56–2.24 kg ai/ha, maximum of 6.7 kg ai/ha; and 7 days PHI							
GARDEN BEETS TOPS							
CA	2.46	<u>4.2</u>					
NY	2.21-2.26	<u>17</u>					
	2.24-2.28	<u>8.9</u>					
OR	2.22-2.24	<u>30</u>					
	2.20	<u>3.7</u>					
TX	2.24	<u>3.3</u>					
WI	2.24	<u>1.8</u>					
	2.21-2.23	<u>9.3</u>					
	TURNIP GREENS (no GAP in USA) GAP in Canada: 0.6-2.5 kg ai/ha, 21 days PHI						
AL	2.38-2.40	8.4					
AZ	2.21-2.25	59					
	2.22-2.30	5.6					

	Application	Residues
State	kg ai/ha	mg/kg
CA	2.43-2.57	8.6
	2.24-2.28	48
GA	2.34-2.43	13
MI	2.26-2.37	9.6
	2.24-2.35	1.2
NJ	2.22-2.29	1.7
PA	2.24-2.24	6.0
TX	2.24	2.6

### **Pulses**

Soya beans. Eight trials were conducted in the USA and 4 in Thailand (Table 45).

Table 45. Residues of carbaryl in dry soya beans.

		Application	No. of	PHI,	Residues		
Location, year	Formulation	kg ai/ha	applications	Days	mg/kg	References	
GAP, USA, 480 SC, max. 4 x 0.56–1.68 kg ai/ha, 14 days PHI for forage, 21 for dried beans							
USA, AR	480 SC	1.67-1.69	4	21	<u>0.05</u>	Study No.	
USA, IA	480 SC	1.67-1.72	4	21	<u>0.05</u>	US94S41R	
	480 SC	1.63-1.73	4	21	<u>0.03</u>	Robinson, 1995f	
USA, IL	480 SC	1.68-1.72	4	20	<u>0.15</u>		
	480 SC	1.63-1.72	4	21	<u>0.11</u>		
USA, OH	480 SC	1.61-1.67	4	21	<u>0.12</u>		
USA, MN	480 SC	1.67-1.70	4	21	<u>0.04</u>		
USA, IN	480 SC	1.65-1.76	4	22	<u>0.09</u>		
USA, MO	480 SC	1.66-1.70	4	21	< <u>0.02</u>		
GAP THAILAN	D: 0.21-0.255 I	kg ai/hl					
Thailand, 1993	NS	1.17	NS	38	< 0.01	Summary Table	
1992	NS	1.17	NS	34	< 0.01	provided by the Government of	
1993	NS	2.34	NS	38	< 0.01	Thailand	
1992	NS	2.34	NS	34	< 0.01		

# Root and tuber vegetables

Supervised trials were conducted in the USA on carrots (7 trials), garden beets (8 trials), sweet potatoes (7 trials), turnips (9 trials) and sugar beets (13 trials). Tables 46 and 47.

Table 46. Residues of carbaryl in roots of root and tuber vegetables in the USA in 1994 with 480 SC formulation and 7 days PHI.

<u> </u>			
	Application		Residues
Location	kg ai/ha	No.	mg/kg
GAP (carrots and garden beets and 7 days PHI Sweet potato: pre-plant dip at a			56–2.24 kg ai/ha, max. total 6.7 kg ai/ha; max. total 9 kg ai/ha
CARROTS Study No. US94S031	R (Kowite, 1995a)		
CA	2.16-2.53	3	<u>0.25</u>
	2.16-2.30	3	<u>0.31</u>
FL	2.22-2.26	3	<u>&lt;0.02</u>
MI	2.23-2.32	3	<u>&lt;0.02</u>
	2.18-2.37	3	<u>&lt;0.02</u>
TX	2.28	3	<u>&lt;0.02</u>
WA	2.16-2.28	3	0.03
GARDEN BEETS Study No. US	94S03R (Kowite, 1995	a)	
CA	2.46	3	<u>0.02</u>
NY	2.21-2.26	3	<u>0.06</u>
	2.24-2.28	3	0.03
OR	2.22-2.24	3	<u>&lt;0.02</u>
	2.20	3	<u>&lt;0.02</u>
TX	2.24	3	<u>&lt;0.02</u>
WI	2.24	3	0.05
	2.21-2.23	3	0.03
SWEET POTATO . Pre-plant di	ip at 1.2 kg ai/hl solutio	on. Study No. US94S16R (F	Kowite, 1995b)
CA	2.20-2.28	4	<u>&lt;0.02</u>
	2.21-2.42	4	<u>&lt;0.02</u>
LA, USA 1994	2.23-2.24	4	<u>&lt;0.02</u>
	2.19-2.29	4	<u>&lt;0.02</u>
NC, USA, 1994	2.13-2.14	4	<u>&lt;0.02</u>
	2.26	4	<u>&lt;0.02</u>
	2.23-2.30	4	<u>&lt;0.02</u>
TURNIPS (no GAP in USA) Stu GAP in Canada: 0.6-2.5 kg ai/h		wite, 1995a)	
AL	2.38-2.40	3	<u>0.02</u>
AZ	2.21-2.25	3	<u>0.10</u>
	2.22-2.30	3	<u>&lt;0.02</u>
CA	2.43-2.57	3	0.89

	Application		Residues
Location	kg ai/ha	No.	mg/kg
	2.24-2.28	3	<u>&lt;0.02</u>
GA	2.34-2.43	3	<u>0.03</u>
NJ	2.22-2.29	3	<u>&lt;0.02</u>
PA	2.24-2.24	3	<u>&lt;0.02</u>
TX	2.24	3	<u>&lt;0.02</u>

Table 47. Residues of carbaryl in sugar beet roots with 3 applications at 0.81 kg ai/ha of 480 SC formulation, 14 days PHI, in the USA in 1985. Project No 801R11 (Thomas, 1986).

Location	Residues, mg/kg			
GAP, USA 2 applications of	of 0.56–1.6814 kg ai/ha; max. 3.36 kg ai/ha and 14 days PHI			
CA	<u>&lt;0.05</u>			
	<u>≤0.05</u>			
СО	<u>≤0.05</u>			
ID	<u>≤0.05</u>			
	<u>≤0.05</u>			
MN	<u>≤0.05</u>			
	<u>≤0.05</u>			
	<u>0.06</u>			
MI	<u>0.45</u>			
ND	<u>0.05</u>			
	<u>≤0.05</u>			
NE	<u>≤0.05</u>			
TX	<u>≤0.05</u>			

# Stalk and stem vegetables

Asparagus. Six trials were conducted in the USA in 1994 (Table 48).

Table 48. Residues of carbaryl in asparagus treated 3 times with 480 SC formulation, 1-day PHI, in the USA in 1994. Study No. US94S20R (Chancey, 1995).

State	Application, kg ai/ha	Residues mg/kg
GAP, USA 480 SC, n	nax. 3 applications at 1.12–2.24	4 kg ai/ha
CA	2.22-2.30	<u>9.0</u>
	2.22-2.24	<u>10</u>
MI	2.24	<u>7.2</u>
	2.24	<u>10</u>
WA	2.38-2.42	<u>2.0</u>
	2.38-2.41	<u>2.2</u>

# **Cereal grains**

<u>Maize (Field corn)</u>. Eight trials were conducted in the USA in 1995 yielding residues in grain of <0.02 mg/kg (Table 51).

Table 50. Residues of carbaryl in grain from maize treated 4 times with 480 SC formulation in the USA. Study No. US95S01R (Chancey, 1996b).

Location	Appli	cation	PHI,	Residues		
	kg ai/ha	Water l/ha	Days	mg/kg		
GAP, USA: 480 SC and 800 WP; max. 4 applications 1.12–2.24 kg ai/ha; min. 187 l water/ha; days						
IL	2.18-2.53	140	48	<u>&lt;0.02</u>		
IA	2.28-2.35	199	48	<u>&lt;0.02</u>		
ОН	2.22-2.32	178	49	<u>&lt;0.02</u>		
	2.23-2.29	178	49	<u>&lt;0.02</u>		
MN	2.23-2.29	187	48	<u>&lt;0.02</u>		
WI	2.19-2.33	262	48	<u>&lt;0.02</u>		
NB	2.23-2.28	187	62	<u>&lt;0.02</u>		
	2.23-2.25	187	48	<u>&lt;0.02</u>		

Barley. Thirty trials were conducted on barley in Canada and the USA in 1985 and 1986 (Table 50).

Table 50. Residues of carbaryl in barley grain.

Country, year	Application			Interval days	PHI, days	Residues	Reference
	Formulation	Rate, kg ai/ha	No			mg/kg <sup>1</sup>	
GAP CANADA: 4 days PHI	480 SC, max. 2 t	imes 1.2–2.5	i2 kg ai/ha	a (ground); 0.55-1	1.76 kg ai/ha (d	uerial),7 to 14 day	vs interval, 28
Canada, 1986	480 SC	2.0	2		14	12	Project
	480 SC	2.0	2		14	7.4	801R11,
	480 SC	2.0	2		14	19	Davis, 1986a
	480 SC	2.0	2		14	0.28	
	480 SC	2.63	2		14	1.2	
	50W	2.0	2		14	5.9	
	50W	2.0	2		14	5.3	
	50W	2.0	2		14	18	
	50W	2.0	2		14	0.20	
	50W	2.25	2		14	0.99	
USA, CA, 1985	480SC	1.68	2	14	14 21	7.4 2.3	Project No 801R11
	480SC	1.68	4	14	14 21	11 3.9	(Davis and Thomas,

Country, year	Ap	plication		Interval days	PHI, days	Residues	Reference
	Formulation	Rate, kg ai/ha	No			mg/kg <sup>1</sup>	
ID, 1985	480SC	1.68	2	25	14 22	2.4 1.1	1987)
	480SC	1.68	4	25	14 22	3.9 1.4	
KS, 1985	480SC	1.68	2	12	14 21	< 0.15 <u>0.15</u>	
			4	6-18	14 22	< 0.15 < 0.15	
ND, 1985	480SC	1.68	2	09	14 24	0.35 < 0.10	
	480SC	1.68	4	10-20	14 24	0.24 <0.10	
CA, 1986	480SC	1.68	2	16	7 14 21	13 9.6 <u>7.6</u>	
	480SC	1.68	2	32	7 14 21	24 10 9.6	
	480SC	1.68	2 Aerial	32	7 14 21	22 16 9.4	
CA, 1986	480SC	1.68	2	22	7 14 21	4.3 4.8 3.3	
ID, 1986	480SC	1.68	2	19	8 15 22	11 9.3 <u>8.5</u>	
MN, 1986	480SC	1.68	2	7	7 15 22	7.6 2.9 <u>0.36</u>	
	480SC	1.68	2 Aerial	7	7 15 22	2.1 1.0 <u>0.52</u>	
MT, 1986	480SC	1.68	2	13	7 14 21	6.3 4.3 <u>3.0</u>	
NC, 1986	480SC	1.68	2	14	7 14 21	12 4.0 <u>0.51</u> <sup>2</sup>	
ND, 1986	480SC	1.68	2	14	7 14 21	2.5 2.3 <u>0.79</u>	
	480SC	1.68	2 Aerial	14	7 14 21	1.5 0.41 <u>0.33</u>	
Washington, 1986	480SC	1.68	2	16	7	<u>8.6</u>	

<sup>&</sup>lt;sup>1</sup> results corrected for method recovery <sup>2</sup> lowest value, others were 20 and 7.1 mg/kg

Rice. Nine trials were conducted in the USA in 1994 (Table 51).

Table 51. Residues of carbaryl in grain of rice treated twice with 480 SC formulation in the USA in 1994. Study No US94S24R (Mede, 1995).

Crop	Application	PHI,	Residues				
location, year	kg ai/ha	Days	mg/kg				
GAP, USA: 480 SC and 800 WP, 2 applications 1.12–1.68 kg ai/ha, max. total 4.48 kg ai/ha, 7-day interval, 14 days Pl							
AR	2.23	13	<u>8.4</u>				
	2.23	14	<u>7.1</u>				
	2.23	14	<u>11</u>				
	2.26	14	<u>2.8</u>				
CA	2.20	14	<u>10</u>				
	2.20	14	<u>46</u>				
LA	2.16	14	<u>6.0</u>				
MS	2.27	14	<u>11</u>				
	2.21	14	<u>3.1</u>				

Rye. Five trials were conducted in the USA in 1986 (Table 52).

Table 52. Residues of carbaryl in grain of rye treated twice with 1.68 kg ai/ha with 480 SC formulation in the USA in 1986. Project No. S86-024-R01 (Lee, 1990b).

State	Water	Interval,	PHI,	Residues			
	l/ha	days	days	mg/kg			
GAP CANADA: 480 SC, max. 2 times 1.2–2.52 kg ai/ha (ground); 0.55-1.76 kg ai/ha (aerial),7 to 14 days interval 14 days PHI							
GA	187	14	7 14 21	2.0 <u>0.36</u> 0.32			
MI	608	15	8 15 21	3.8 <u>2.6</u> 2.0			
SD	-	19	7 14 21	0.62 <u>0.98</u> 0.85			
MN	47	29	7	9.4			
		aerial	7	6.2			

Sorghum. Nine trials were conducted in the USA (Table 53).

Table 53. Residues of carbaryl in grain of sorghum treated 3 times with 480 SC formulation in the USA in 1994. Study No US94S42R (Cappy and Robinson, 1995).

	App	lication	PHI,	Residues				
State	kg ai/ha	Water l/ha	days	mg/kg				
GAP, USA: 480 SC or 800 WP formulation, max. 4 applications at 1.12–2.24 kg ai/ha, max. total 6.72 kg ai/ha, 21 days PHI								
KS	2.37	140	15	7.1				
	2.40	140	15	5.4				
	2.23	164	13	0.07				
MO	2.26	200	14	0.32				
	2.26	201	15	< 0.02				
NB	2.25	187	14	0.55				
	2.25	187	14	6.1				
OK	2.24	138	13	2.5				
TX	2.24	158	14	0.72				

Wheat. Twenty three trials were conducted in Canada and the USA in 1986 and 1996 (Table 54).

Table 54. Residues of carbaryl in wheat grain.

		Application		PHI,	Residues	Reference	
Location, year	Formulation	Rate, kg ai/ha	No	Days	mg/kg		
GAP CANADA:	480 SC, max. 2 t	imes 0.55–2.52 kg a	ui./ha, 14 d	lays PHI			
Canada, 1986	480 SC	2.0	2	12	<u>1.3</u>	Project	
	480 SC	2.63	2	14	<u>0.33</u>	801R11,	
	480 SC	2.63	2	14	<u>0.49</u>	Davis, 1986a	
	480 SC	2.0	2	14	<u>1.6</u>		
	480 SC	2.0	2	14	<u>1.1</u>		
	480 SC	2.63	2	14	0.22		
	50W	2.0	2	14	<u>1.2</u>		
	50W	2.25	2	14	0.23		
	50W	2.25	2	14	<u>0.28</u>		
	50W	2.0	2	14	<u>1.6</u>		
	50W	2.0	2	14	<u>1.6</u>		
	50W	2.25	2	14	<u>0.26</u>		
GAP, USA: 480 S	SC and 800 WP,	max. 2 applications	s at 0.56–1	.68 kg ai/ha, w	vith 14 days i	nterval; 21 days PHI	
USA, KS, 1986	480SC	1.68	2	7 14 21	0.06 <0.02 <0.02	Project No. S86-054-02 (Lee, 1990a)	
USA, MS, 1986	480SC	1.68	2	7 14 21	0.28 0.15 < <u>0.02</u>		
USA, MT, 1986	480SC	1.68	2	7 14 21	0.12 0.25 <u>0.07</u>		

		Application		PHI,	Residues	Reference
Location, year	Formulation	Rate, kg ai/ha	No	Days	mg/kg	
Aerial application	480SC	1.68	2	7 14 21	0.64 0.36 <u>0.12</u>	
USA, NC, 1986	480SC	1.68	2	7 14 21	0.60 2.7 <u>1.4</u>	
USA, ND, 1986	480SC	1.68	2	7 14 21	0.49 0.05 <u>0.19</u>	
USA, ND, 1995	480SC	1.68	2	21	<u>&lt;0.02</u>	Study No.
USA, OK, 1995	480SC	1.68	2	21	<u>&lt;0.02</u>	US95S10R
USA, ND, 1996	480SC	1.68	2	21	<u>&lt;0.02</u>	(Ely, 1997b)
USA, KS, 1996	480SC	1.68	2	21	<u>&lt;0.02</u>	
	480SC	1.68	2	21	<u>&lt;0.02</u>	
USA, ID, 1996	480SC	1.68	2	21	<u>0.27</u>	

# Tree nuts

Twenty trials were conducted in the USA in 1994 (Table 55).

Table 55. Residues in almonds, pecans, pistachios and walnuts after 3 applications of 480 SC and 14 days PHI in the USA in 1994.

	Applic	ation		Residues						
State	kg ai/ha	Water l/hl	Sample	mg/kg	References					
GAP, USA: 480 SC and 800 WP formulation, max. 4 applications of 2.24-5.6 kg ai/ha, total of 16.8 kg ai/ha, 14 days PHI										
Almonds										
CA	5.57	1029	Kernel	<u>0.07</u>	Study No.					
	5.58	888	Kernel	<u>0.09</u>	US94S19R					
	5.57	870	Kernel	<u>0.04</u>	(Macy and					
	5.90	1496	Kernel	0.08	Chism, 1995a)					
	5.67	476	Kernel	<u>0.03</u>						
Pecans	•			•						
AL	5.57	926	Pecan	<u>&lt;0.02</u>	Study No.					
GA	5.57	561	Pecan	<u>&lt;0.02</u>	US94S32R					
	5.57	467	Pecan	<u>&lt;0.02</u>	(Macy, 1995a)					
NM	5.77	627	Pecan	<u>0.05</u>						
	5.77	678	Pecan	<u>0.03</u>						
TX	5.59	429	Pecan	0.02						
Pistachios										
CA	5.61	973	Kernel	<u>0.03</u>	Study No.					
	5.61	1113	Kernel	<u>&lt;0.02</u>	US94S23R					

	Applic	ation		Residues	
State	kg ai/ha	Water l/hl	Sample	mg/kg	References
	5.72	1431	Kernel	<u>0.09</u>	(Macy, 1995b)
	5.48	468	Kernel	<u>&lt;0.02</u>	
Walnuts					
CA	5.82	766	Walnuts	<u>0.09</u>	Study No.
	5.57	1066	Walnuts	<u>0.04</u>	US94S31R
	5.62	612	Walnuts	<u>0.77</u>	(Macy and
	5.64	611	Walnuts	<u>0.44</u>	Chism, 1995b)
	6.00	1066	Walnuts	<u>0.02</u>	

#### Oilseed

Sunflower. Five trials were conducted in the USA in 1994 (Table 56).

Table 56. Residues of carbaryl in seed from sunflower treated twice with 480 SC formulation in the USA in 1994. Study No. US94S44R (Robinson, 1995a).

	Application kg ai/ha Water l/ha		PHI,	Residues					
State			Days	mg/kg					
GAP, USA: 480 SC and 800 WP; max. 2 applications of 1.12–1.68 kg ai/ha; 60 days PHI									
ND	1.75–1.67	187	61	<u>0.03</u>					
	1.65-1.68	187	62	<u>&lt;0.02</u>					
	1.70-1.64	187	62	<u>&lt;0.02</u>					
SD	1.66-1.67	187	60	<u>0.07</u>					
	1.67-1.66	187	60	<u>0.08</u>					

### Animal feed

Supervised trials were conducted on animal feed forage, fodder, silage and/or straw in the USA (Tables 57-62).

Table 57. Residues of carbaryl in soya bean forage and hay in the USA in 1994 from 4 applications of 480 SC formulation. Study No. US94S41R (Robinson, 1995f).

	Application	PHI,		Residues
State	kg ai/ha	Days	Sample	mg/kg
GAP, USA	, 480 SC, Max. 4, 0.56–1.	.68, 14 days	for forage, 21 for d	ried beans and hay
AR	1.64-1.69	15	Forage	<u>1.3</u>
	1.67-1.69	21	Hay	<u>4.0</u>
IA	1.63-1.70	14	Forage	<u>8.5</u>
	1.67-1.72	21	Hay	<u>8.0</u>
	1.67-1.72	14	Forage	<u>3.6</u>
	1.63-1.73	21	Hay	<u>9.6</u>

	Application	PHI,		Residues
State	kg ai/ha	Days	Sample	mg/kg
IL	1.61-1.72	13	Forage	<u>1.8</u>
	1.68-1.72	20	Hay	<u>6.4</u>
	1.65-1.78	14	Forage	<u>1.4</u>
	1.63-1.72	21	Hay	<u>8.4</u>
IN	1.72-1.74	14	Forage	<u>4.6</u>
	1.65-1.76	22	Hay	<u>2.6</u>
MN	1.68-1.70	13	Forage	<u>3.8</u>
	1.67-1.70	21	Hay	<u>6.41</u>
MO	1.66-1.70	21	Hay	<u>&lt;0.02</u>
ОН	1.65-1.76	14	Forage	<u>1.9</u>
	1.61-1.67	21	Hay	<u>6.3</u>

Table 58. Residues of carbaryl in sweet and field corn forage and fodder with 480 SC in the USA in 1995.

State		Application			PHI,	Residues	Reference
	kg ai/ha	Water l/ha	No.	Sample	Days	mg/kg	
GAP, USA sweet ai/ha, 28-187 l w					veet corn)	application	s of 1.12–2.24 kg
Sweet corn							
WA	2.25	215	8	Forage Fodder	14 48	3.8 1.5	Project S86-024- R01
ID	2.28	119	8	Forage Fodder	14 48	163 184	(Kowite, 1996)
	2.24	119	8	Forage Fodder	14 48	<u>124</u> <u>68</u>	
MN	2.26	192	8	Forage Fodder	14 48	<u>1.8</u> <u>0.62</u>	
WI	2.25	267	8	Forage Fodder	14 48	<u>12</u> <u>1.5</u>	
	2.28	267	8	Forage Fodder	14 48	<u>1.8</u> <u>0.24</u>	
Field corn							
IL	2.18-2.53	140	4	Forage Fodder	14 48	<u>24</u> <u>7.6</u>	Study No. US95S01R
IA	2.28-2.35	199	4	Forage Fodder	14 48	<u>1.2</u> <u>0.38</u>	(Chancey, 1996b)
ОН	2.22-2.32	178	4	Forage Fodder	14 49	<u>2.0</u> <u>0.06</u>	
	2.23-2.29	178	4	Forage Fodder	14 49	<u>24</u> <u>0.46</u>	
MN	2.23-2.29	187	4	Forage Fodder	14 48	<u>16</u> <u>2.4</u>	
WI	2.19-2.33	262	4	Forage Fodder	14 48	<u>7.7</u> <u>0.71</u>	

State	Application			PHI,	Residues	Reference	
	kg ai/ha	Water l/ha	No.	Sample	Days	mg/kg	
NB	2.23-2.28	187	4	Forage Fodder	14 62	<u>10</u> <u>0.14</u>	
	2.23-2.25	187	4	Forage Fodder	14 48	<u>4.1</u> <u>0.70</u>	

Table 59. Residues of carbaryl in barley forage and straw in the USA. Ground applications unless otherwise stated.

Location, year	Арр	olication		Interval	Sample	PHI, Days	Residues	Reference
	Formulation	Rate, kg ai/ha	No	Days			mg/kg <sup>1</sup>	
GAP CANADA 28 days PHI	l: 480 SC, max.	2 x 1.2–2	52 kg ai/h	na (ground	d); 0.55-1.76	kg ai/ha (ae	rial),7 to 14	days interval,
CA, 1985	480SC	1.68	2	14	Straw	14 21	21 14	Project No 801R11
	480SC	1.68	4	14	Forage Straw	0 3 14 21	40 30 33 7.3	(Davis and Thomas, 1987)
ID, 1985	480SC	1.68	2	25	Straw	14 22	14 11	
	480SC	1.68	4	25	Forage Straw	0 3 14 22	37 24 16 16	
KS, 1985	480SC	1.68	2	12	Straw	14 21	25 4.4	
			4	6-18	Forage Straw	0 3 14 22	41 9.6 4.4 2.4	
ND, 1985	480SC	1.68	2	19	Straw	14 24	0.96 0.82	
	480SC	1.68	4	10-20	Forage Straw	0 3 14 24	50 19 0.52 0.82	
CA, 1986	480SC	1.68	2	14	Forage	0 3	58 52	
	480SC	1.68	2	16	Straw	7 14 21	85 113 82	
	480SC	1.68	2	32	Straw	7 14 21	78 31 35	
	480SC	1.68	2 aerial	32	Straw	7 14 21	102 59 25	

Location, year	Арр	olication		Interval	Sample	PHI, Days	Residues	Reference
	Formulation	Rate, kg ai/ha	No	Days			mg/kg <sup>1</sup>	
CA, 1986	480SC	1.68	2	14	Forage	0 3	66 39	
				22	Straw	7 14 21	43 35 30	
ID, 1986	480SC	1.68	2	14	Forage	0 3	34 30	
				19	Straw	8 15 22	30 38 <u>24</u>	
MN, 1986	480SC	1.68	2	8	Forage	0 3	28 1.9	
	480SC	1.68	2 aerial	8	Forage	0 3	43 8.0	
	480SC	1.68	2	16	Forage	0 3	23 2.1	
	480SC	1.68	2 aerial	16	Forage	0 3	12 0.86	
	480SC	1.68	2	7	Straw	7 15 22	26 4.3 1.9	
	480SC	1.68	2 aerial	7	Straw	7 15 22	4.3 1.7 <u>2.4</u>	
MT, 1986	480SC	1.68	2	04	Forage	0 3	34 25	
NC, 1986	480SC	1.68	2	14	Forage	0 3	45 24	
	480SC	1.68	2	14	Straw	7 14 21	147 86 35	
ND, 1986	480SC	1.68	1 ground	14	Forage ground	0 3	61 29	
			1 aerial		Forage aerial	0 3	33 13	
	480SC	1.68	2	14	Straw	7 14 21	2.3 0.47 <0.2	
	480SC	1.68	2 aerial	14	Straw	7 14 21	2.5 0.49 < <u>0.2</u>	
Washington, 1986	480SC	1.68	2	14	Forage	0 3	54 3.7 <sup>2</sup>	
	480SC	1.68	2	16	Straw	7 14	14 8.3	

<sup>&</sup>lt;sup>1</sup> results are corrected for method recovery <sup>2</sup> lowest value, the others 18 and 34 mg/kg

Table 60. Residues of carbaryl in straw of rice treated twice with 480 SC formulation in the USA in 1994. Study No US94S24R (Mede, 1995).

	Application	PHI,	Residues					
Country, year	kg ai/ha	Days	mg/kg					
GAP, USA: 480 SC ar	GAP, USA: 480 SC and 800 WP, 2 applications 1.12–1.68, 14 days PHI							
AR	2.23	13	<u>23</u>					
	2.23	14	<u>7.5</u>					
	2.23	14	<u>9.4</u>					
	2.26	14	<u>47</u>					
CA	2.20	14	<u>23</u>					
	2.20	14	<u>102</u>					
LA	2.16	14	<u>48</u>					
MS	2.27	14	<u>26</u>					
	2.21	14	<u>14</u>					

Table 61. Residues of carbaryl in the forage and straw of rye treated twice with 1.68~kg ai/ha with 480~SC formulation in the USA in 1986. Project No. S86-024-R01 (Lee, 1990b).

State	Water	Interval		PHI,	Residues
	l/ha	Days	Sample	Days	mg/kg
No US GAP for days interval; P				0 WP, max. 2 application	ns at 0.56–1.68 kg ai/ha, with 14
GA	187	14	Straw	7 14 21	33 14 10
MI	608	15	Forage Straw	0 4 8 15 21	19 16 35 9.6 13
SD	-	19	Straw	7 14 21	2.9 2.7 2.0
MN	47	14	Forage	0 3	67 0.84
		aerial	Forage	0 3	81 4.0
	47	29	Straw	7 14 21	4.2 0.40 0.24
		aerial	Straw	7 14 21	2.8 1.4 0.36

Table 62. Residues of carbaryl in sorghum treated 3 times with 480 SC formulation in the USA in 1994. Study No US94S42R (Cappy and Robinson, 1995).

	Applio	cation	Sample	PHI,	Residues
Sample	kg ai/ha	Water l/ha		Days	mg/kg
GAP, USA: 480 SC 14 days (forage; sil		rmulation, mo	ax. 4 applicatio	ns at 1.12–2	.24 kg ai/ha, PHI 21 days (fodder) and
KS	2.37	140	Forage Silage Fodder	14 14 15	$\frac{4.1}{4.9}$ $\frac{4.3}{4.3}$
	2.40	140	Forage Silage Fodder	14 14 15	1.0 5.4 7.8
	2.23	164	Forage Silage Fodder	14 14 13	0.08 0.38 0.54
M0	2.26	200	Forage Silage Fodder	14 14 14	$   \begin{array}{r}                                     $
	2.26	201	Forage Silage Fodder	14 14 15	$\frac{7.3}{4.3}$ $0.04$
AK	2.25	193	Forage Silage	14 14	<u>14</u> <u>6.2</u>
NB	2.25	187	Forage Silage Fodder	14 14 14	<u>0.41</u> <u>2.9</u> 1.8
	2.25	187	Forage Silage Fodder	14 14 14	2.0 1.5 2.2
OK	2.24	138	Forage Silage Fodder	14 15 13	<u>0.60</u> <u>1.8</u> 22
TX	2.24	158	Forage Silage Fodder	14 14 14	0.85 0.40 0.75

Table 63. Residues of carbaryl in wheat forage and straw.

Location,	Application			Sample	PHI,	Residues	Reference
year	Formulation	Rate, kg ai/ha	No		days	mg/kg	
GAP, USA: 480 SC and 800 WP, max. 2 applications at 0.56–1.68 mg/kg, with 14 days interval; PHI: 21 days (straw) and 7 days (forage)							
KS, 1986	480SC	1.68	2	Forage straw	0 3 7 14 21	62 5.2 3.6 0.51 <u>0.92</u>	Project No. S86-054-02 (Lee, 1990a)
MT, 1986	480SC	1.68	2	Forage straw	0 3 7 14 21	46 4.2 7.3 9.4 <u>8.2</u>	

Location,	Application Sample				Reference		
year	Formulation	Rate, kg ai/ha	No		days	mg/kg	
Aerial application	480SC	1.68	2	Forage Straw	0 3 7 14 21	29 8.8 16 11 <u>11</u>	
NC, 1986	480SC	1.68	2	Forage Straw	0 3 7 14 21	26 31 6.3 <sup>1</sup> 54 <u>22</u>	
ND, 1986	480SC	1.68	2	Forage Straw	0 3 7 14 21	78 5.2 25 9.6 <u>5.2</u>	

<sup>&</sup>lt;sup>1</sup> mean of two values, the third was 81 mg/kg

Table 64. Residues in almond hulls after 3 applications of 480 SC and 14 days PHI in the USA in 1994.

	Application			Residues		
State	kg ai/ha	Water l/hl	Sample	mg/kg	References	
GAP, USA: 480 SC and 800 WP formulation, max. 4 applications of 2.24- 5.6 kg ai/ha, 14 days PHI						
CA	5.57	1029	Hull	<u>5</u>	Study No.	
	5.58	888	Hull	<u>16</u>	US94S19R	
	5.57	870	Hull	<u>36</u>	(Macy and	
	5.90	1496	Hull	<u>39</u>	Chism, 1995a)	
	5.67	476	Hull	<u>27</u>		

### FATE OF RESIDUES IN STORAGE AND PROCESSING

## In storage

No information was reported.

### In processing

Four studies were conducted on citrus fruits, one on grapefruit, one on lemons and two on oranges. In all trials except the second one on oranges (Davis, 1986b) fruits were treated 2 or 3 times at 22.4 kg carbaryl/ha with a 1-day PHI, and in the second orange trial (Robinson, 1995b) oranges were treated 4 times at 8.4 to 17 kg carbaryl/ha and harvested after a 5-day PHI. Although the studies were 10 years apart, processing procedures were similar and simulated commercial practices. Samples were rinsed with water in an FMC Fruit Cleaner and passed over a foam rubber eliminator and horsehair brushes. An FMC In-Line Juice Extractor was used to separate juice, which was "finished" with an FMC finisher. The oil/water/peel-frit emulsion was centrifuged to produce the oil and peel-membrane-seed fractions. This latter peel residue was chopped in a Fitzpatric comminuting machine to yield wet pulp, and 0.3% dehydrated lime was added as a liquid slurry to produce limed wet pulp, which was passed

though a continuous press and separated into cake and liquor. The press cake was dried in a triple-pass direct-fired oven to produce dried pulp of 8-10% moisture, and the liquor heated under vacuum and concentrated to  $\sim 50^{\circ}$  Brix to produce molasses. The residues were concentrated in the peel and oil, and also in the molasses from grapefruit and lemons (Table 65).

Table 65. Processing studies on citrus fruits.

	Residue	s, mg/kg	Processir	ng factor		
Grapefruit Whole fruit, RAC	10	).7				
Whole fruit, washed	4	55	0.4	13		
Juice	0.0	05	0.0	05		
Peel	12	2.1	1.	1		
Wet pulp	3.	10	0.2	29		
Dried pulp	3.0	02	0.2	28		
Oil	23	39	22	.3		
Molasses	14	1.5	1.	4		
Lemon Whole fruit, RAC	12	2.0				
Whole fruit, washed	10	).9	0.	9		
Juice	0.	88	0.07			
Peel	13	3.9	1.16			
Wet pulp	3.9	95	0.33			
Dried pulp	1.	71	0.14			
Oil	53	30	44			
Molasses	14	1.5	1.2		Average PF	
Orange Whole fruit, RAC	11.5	17.6 <sup>1</sup>			Orange	Citrus
Whole fruit, washed	5.3	3.1	0.46	0.18	0.32	0.49 (n=4)
Juice	0.10	0.20	0.01	0.01	0.01	0.02 (n=4)
Peel	14.6	-	1.3	-	1.3	1.2 (n=3)
Wet pulp	9.0	-	0.78	-	0.78	0.47 (n=3)
Dried pulp	3.0	4.5	0.26	0.26	0.26	0.24 (n=4)
Oil	297	42.6	25.8	2.42	14.1	23.6 (n=4)
Molasses	4.0	1.45	0.34	0.08	0.21	0.76 (n=4)

<sup>&</sup>lt;sup>1</sup> Robinson, 1995b

In one processing study simulating commercial practice, field-treated apples were washed and processed into juice, wet and dry pomace (Davis and Thomas, 1986a); residues were concentrated in dry pomace with a processing factor of 2.48 (Table 67). In another study in 1994 in the USA, in which apples were treated 5 times at 10 kg ai/ha, harvested at a 3-day PHI and processed according to simulated commercial practice except by batch instead of continuously (Cappy, 1995c) residues were also concentrated in dry pomace by a factor of 3.7 (Table 67). In a third study in France apples treated with 1 kg carbaryl/ha, were harvested at a 40-days PHI, steam-peeled, hand-crushed, cooked at 100°C for 10 min and heated at 80-105°C to produce refined pulp and compote (Maestracci, 1998c). In all fractions residues decreased to half the value in whole fruit (Table 66).

Table 68. Processing studies on apples.

	Average residues	Processing factor	
Davis and Thomas, 1986a			
Whole fruit	6.85		
Whole fruit, washed	3.97	0.58	
Juice	2.52	0.36	
Wet pomace	6.59	0.96	
Dry pomace	17	2.48	
Сарру, 1995с			
Whole fruit	4.20		Average PF
Juice	1.67	0.4	0.38 (n=2)
Wet Pomace	5.28	1.3	1.1 (n=2)
Dry Pomace	15.5	3.7	3.1, n=2)
Maestracci, 1998c			
Whole fruit	0.06		
Whole fruit, washed	0.03	0.5	0.54 (n=2)
Peeled apple	0.03	0.5	0.5
Refined pulp from peeled apple	0.03	0.5	0.5
Compote from peeled apple	0.03	0.5	0.5
Refined pulp from unpeeled apple	0.03	0.5	0.5
Compote from unpeeled apple	0.03	0.5	0.5

Four processing studies were conducted on grapes in the USA. In the first by Davis (1986c) grapes were treated twice at 4.49 kg carbaryl/ha and processed by a close approximation to commercial practice. Residues were concentrated in the juice and wet and dry pomace (Table 67). In a second study by Davis (1986l) grapes were treated twice with 2.24 kg carbaryl/ha, harvested at 0-day PHI, and processed to raisins in a manner close to commercial practice except amounts were smaller. Residues were reduced in raisins and concentrated in the waste (Table 67).

In two other studies in 1994 grapes were treated 5 times at a nominal rate of 11.2 kg carbaryl/ha and either processed to juice and pomace (Robinson, 1995c) simulating commercial practice, or to raisins (Robinson, 1995d). In the latter study, with 3 trials, harvested grapes were placed on paper trays for 19 to 28 days to produce unprocessed raisins. Stems, cap stems and small raisins were separated to become part of the waste fraction. The raisins were washed in the processing line and visually inspected to remove pieces and low-grade raisins. Residues were concentrated in the pomace, unprocessed and washed raisins and in the waste with processing factors varying from 1.1 to 6 (Table 67).

Table 69. Processing studies on grapes.

	Carbaryl residues (mg/kg)	Processing factor
Davis, 1986c		
Whole fruit	9.74	
Juice	10.8	1.1

	Carbaryl residues (mg/kg)	Processing factor	
Wet pomace	14.0	1.4	
Dry pomace	11.5	1.2	
Davis, 1986l			
Whole fruit	17/11/14		
Raisins	3.3/2.4/2.1	0.19/0.22/0.15	
Raisin waste	18/16/21	1.1/1.4/1.5	
Robinson, 1995c		5 (1.45)	Average PF
Whole fruit	36.4		
Juice	8.73	0.24	0.67 (n=2)
Wet pomace	49.7	1.4	1.4 (n=2)
Dry pomace	140	3.8	2.5 (n=2)
Robinson, 1995d			
Grapes	28.7/47.2/38.4		
Unprocessed raisins	79.3/97.5/71	2.8/2.1/1.8	1.2 (n=6)
Washed raisins	47.9/59.4/49.5	1.7/1.3/1.3	1.4 (n=3)
Raisin waste	104.7/284/169	3.6/6.0/4.4	3.0 (n=6)

Prunes treated twice with 6.72 kg carbaryl/ha were commercially processed to dried prunes. The residues in the fruit (4.1 mg/kg) decreased to 1.06 mg/kg after washing (PF 0.26) and to 0.62 mg/kg (PF 0.15) after drying (Davis, 1986c).

In a processing study on olives in 1994 by Robinson (1995e) olives treated with 2 x 17.4 kg carbaryl/ha were washed over a screen and leaves and twigs removed by hand, ground in a Rietz type mill, and crushed in a hydraulic press in cloth bags to allow only the liquid to flow from the press. The oil was separated from the vegetable fluid by centrifugation and filtered. Average residues in replicate samples were 81.1 mg/kg in olives (52.2, 73.1 and 118 mg/kg) and 66.2 mg/kg in the oil (44.3, 94.1, 60.1 mg/kg), with an average processing factor (ratio of the mean residues) of 0.82.

Two processing studies were conducted on tomatoes in the USA. In the first in 1985 tomatoes treated at twice the label rate were harvested at a 0-day PHI, washed with tap water and crushed with a stainless steel hammer mill (Davis and Thomas, 1986b). The mixture was heated to 60°C and run through a pulper to separate the juice. Part of the wet pulp was dried at 70-75°C for 4 hours. A portion of the juice was heated in a steam-jacketed kettle until it reached 8.5% Brix. In the more recent second study tomatoes treated 4 times at 12 kg ai/ha and harvested at a 3-day PHI were processed using normal commercial practices with scaled-down equipment (Lee, 1995a). The results of the two studies are shown in Table 68.

Table 68. Processing studies on tomatoes.

	Residues ( mg/kg)	Processing factor
Davis and Thomas, 1986b		
Whole fruit	2.93	
Whole fruit, washed	1.75	0.6
Juice	1.58	0.5

	Residues ( mg/kg)	Processing factor	
Wet pomace	7.15	2.4	
Dry pomace	1.52	0.5	
Purée	1.91	0.7	
Lee, 1995a			Average PF (n=2, except paste)
Whole fruit	3.73		
Juice	1.92	0.5	0.5
Wet pomace	6.49	1.7	2.1
Dry pomace	10.8	2.9	1.7
Purée	4.71	1.3	1.0
Paste	7.49	2.0	2.0

Sweet corn was treated 4 times at 7.3 kg carbaryl/ha and 4 samples harvested at a 0-day PHI were processed to cannery waste (Davis, 1986g). Husks were removed from the ears and kernels from cobs. The cannery waste was produced by blending 1/3 of the husks with 1/3 of the cobs using a food processor. Carbaryl residues in kernels plus cobs with husks removed were 0.11, 0.29, 0.25 and 1.48 mg/kg, and in cannery waste 16.5, 17.2, 18.2 and 21.4 mg/kg, giving an average of the four processing factors of 74 (14.5 to 150, n=4).

Soya beans treated with carbaryl at twice the label rate and harvested at a 0-day PHI were processed into hulls, meal, crude and refined oil and soapstock (Davis and Thomas, 1986c). The beans containing 1.5 mg/kg carbaryl were first dried to 10% moisture in a Proctor-Schwartz forced-air oven for 10 min at 75°C, the seeds cracked into 4-6 pieces and the hulls air-aspired. The kernels were flaked and the meal extracted with hexane. The crude oil in hexane was concentrated at 85°C, treated with 14% NaOH, neutralized and filtered, giving the refined oil and soapstock fractions. Residues of carbaryl were 2.0 mg/kg in hulls (PF 1.3), 0.04 mg/kg in meal (PF 0.03) and 1.4 mg/kg in crude oil (PF 0.9). No residues (<0.02 mg/kg) were detected in refined oil or in soapstock.

Two processing studies were conducted on potatoes. In the one in 1985 potatoes treated twice with 9 kg carbaryl/ha and harvested at a 0-day PHI were peeled using a combination of abrasive and hand peeling, cut into slabs, and pre-cooked for 20 min at 70-75°C (Davis, 1986k). After cooling, the slabs were cooked for 50 min in 95°C flowing atmospheric steam, put through a hand-ricing machine, and the resulting potato mash mixture dehydrated on a single drum dryer at 5.6 kg/cm² steam pressure for 20 seconds to give potato flakes. To produce French fries, peeled potatoes were cut into appropriate strips, the strips blanched at 70°C for 12 min followed by 82°C for 3 min, dried and deepfat fried in vegetable oil for 60 seconds at 177°C. For chips, the potatoes were lightly abrasively peeled, which leaves a certain amount of peel in the product, hand-sliced, washed twice in cold water, soaked for 3 minutes in 55°C water, fried at 165°C for 45-60 seconds, and dried at 50°C to about 3% moisture. The residues in the unwashed tubers were 0.67 mg/kg, in the washed tubers 0.50 mg/kg (PF 0.75); 0.3 mg/kg in fries (PF 0.4) and 0.02 mg/kg in chips and flakes (PF 0.03).

In the second study in 1994 potatoes treated 3 times with 11.2 kg carbaryl/ha and harvested at 7 days PHI, were processed simulating commercial operations as closely as possible. Residues in potato tubers and wet peel were 0.03 mg/kg, and were below the limit of quantification (<0.02 mg/kg) in chips, flakes, and dry peel (Robinson, 1995).

When sugar beets treated at 4 times the label rate were harvested at a 0-day PHI, the carbaryl residues in the roots were 0.23 mg/kg. The roots were processed in a pilot plant representative of actual processing and manufacturing conditions. Residues in the wet and dry pulp, molasses and refined sugar were <0.02 mg/kg, resulting in processing factors of <0.09 (Davis, 1986e).

In a processing study on field corn treated at 14.6 kg carbaryl/ha and harvested at a 0-day PHI, grain was dry-milled using a Ripple impact mill to crack the kernels, the corn stock dried for 30 min at 66-71°C, and the endosperm, hull and germ fractions separated in a Great Western sieve (Davis, 1986f). The germ fraction was dried to about 3% moisture before pressing to presscake and crude oil. The residual oil in the presscake was extracted with hexane. In the wet milling process, grain was steeped for 72 h at 52°C in a 0.25% sulfur dioxide/water solution. A burr mill was used to grind the grain and release the starch (endosperm) and germ. Both procedures are similar to those used in commercial practice. Carbaryl residues in the fractions are shown in Table 69.

In another study conducted in 1994/1995 (Cappy, 1995e), field corn treated 4 times with 11.2 kg carbaryl/ha was processed to produce fractions from dry and wet milling, using procedures that simulated industrial practices as closely as possible. Residues were concentrated in crude oil in both milling processes (Table 69).

Table 69. Processing studies on field corn.	Table 69.	Processing	studies or	n field corn.
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	Davis	s, 1986f	Сарру		
Fraction	Residues (mg/kg)	Processing factor	Residues (mg/kg)	Processing factor	Average PF
Grain (dry milling)	1.35		0.04		
Meal	1.74	1.3	< 0.02	<0.5	<0.9 (n=2)
Flour	2.31	1.7	< 0.02	<0.5	<1.1 (n=2)
Grits	0.41	0.3	< 0.02	<0.5	<0.4 (n=2)
Expeller crude oil	3.18	2.4	-	-	2.4
Solvent extracted crude oil	1.58	1.2	-	-	1.2
Crude oil			0.09	2.2	
Refined oil	0.26	0.2	< 0.02	< 0.5	
Grain (wet milling)	2.00		0.04		
Germ	3.59	1.8	-		1.8
Starch	< 0.02	< 0.01	< 0.02	< 0.5	<0.5
Crude oil	-	-	0.18	4.5	3.4 (n=2)
Refined oil	-	-	< 0.02	< 0.5	<0.4 (n=3)

In a US study in 1985 Davis (1986h) rice treated twice at 4.48 kg carbaryl/ha and harvested at day 0 was dried, cleaned, hulled and polished in a laboratory-scale process close to commercial practice. Residues of carbaryl were concentrated in the hulls by a factor of 4.2 (Table 70). In a second more recent study (Macy and Mede, 1995) rice treated twice at 11.2 kg carbaryl/ha was harvested at 14 days PHI and processed using a forced-air rice dryer, a Rimac Rice Huller and a Cecoco Rice Mill to simulate commercial processing. Residues of carbaryl were also concentrated in the hulls (Table 70).

Table 70. Processing studies on rice.

	Residues, mg/kg	Processing factor	
Davis, 1986h			
Grain	150		
Dried Grain	143	0.95	
Hulls	628	4.2	
Bran	142	0.95	
Polished Rice	1.4	0.01	
Macy and Mede, 19	995		Average PF (n=2)
Grain	39.6		
Hulls	94	2.4	3.3
Bran	14.3	0.4	0.68
Polished rice	1.38	0.03	0.02

Rye was treated twice at 1.68 kg carbaryl/ha and grain samples were processed into bran, flour, middlings and shorts (Ely, 1990). Residues in the grain (1.0 mg/kg) were concentrated in the bran, flour and shorts with processing factors of 1.4,1.1 and 1.7 and reduced in middlings by a factor of 0.8. No details of processing procedures were reported.

In a study on sorghum treated twice at 4.48 kg carbaryl/ha the grain harvested at 3 days PH was dry-milled using two different procedures and wet-milled by a third method (Davis, 1986i). The first dry method involved milling the samples with a Quadromat Sr. flour mill simulating commercial procedures, and in the second an IDRC decorticator was used to produce bran and grits in a similar method to commercial processes. In the wet milling grain was steeped for 48 hours at 52°C in a solution containing 0.1% sulfur dioxide and 0.5% lactic acid, ground in a blender, sieved to separate bran and germ from starch and gluten, and the starch separated, washed and dried. The results are shown in Table 71.

In another study in 1994/1995 sorghum treated 3 times at 11.2 kg carbaryl/ha was harvested at 14 days PHI (Cappy, 1995d). The grain was processed to flour simulating industrial practice as closely as possible, but with the samples processed by batch rather then continuously (Table 71).

Table 71. Processing studies on sorghum.

Fraction	Residues, mg/kg	Processing factor	Average PF
Grain (Davis, 1986h)	1.11		
Bran (Dry milling A)	2.37	2.1	
Bran (Dry milling B)	2.65	2.4	2.3 (n=2)
Shorts (Dry milling A)	0.73	0.7	0.7
Flour (Dry milling A)	0.32	0.3	
Grits (Dry milling B)	0.24	0.2	0.2
Starch (Wet milling)	< 0.02	-	
Grain (Cappy, 1995d)	59		
Flour	2.1	0.04	0.16 (n=2)

In a study by Davis (1986j) sweet sorghum treated twice at 4.48 kg carbaryl/ha was harvested at a 0-day PHI and a sample of the stalks crushed in a standard roll mill to produce crushed stalks (bagasse) and juice. The juice was boiled for 10 hours, until the solids reached 60-70% (syrup). Residues of carbaryl in the stalks were 1.28 mg/kg, in juice 13.0 mg/kg (PF 10.2), in bagasse 9.95 mg/kg (PF 7.8) and in syrup 2.1 mg/kg (PF 1.6).

In a processing study in the USA wheat treated twice at 8.4 kg carbaryl/ha and harvested at 19 days PHI was processed into germ, bran, middlings, shorts and flour under simulated commercial practices (Cappy, 1995a). Residues in grain were 0.28 mg/kg, almost the same in bran (PF 1.03) and lower in low-grade flour (PF 0.08), patent flour (PF 0.10) and wheat germ (PF 0.49). The patent flour is made from the finer and whiter flour streams, with lower bran content and higher endosperm content, while the low-grade flour is coarser and lighter coloured.

In a study on peanuts in 1995 in the USA Chancey (1996a) treated plants 4 times with 6.7-11.2 kg carbaryl/ha. The peanuts were dug 14 days after the last application, and the nuts collected after 7 days drying in the field, for processing to meal and refined oil according to procedures outlined by GLP Processing Program, Texas A and M University SOP. The residues in the kernels were 0.04 mg/kg and were not detected in meal or oil.

Cotton was treated with 4 applications of 8.4 kg carbaryl/ha, harvested at 38 days PHI and processed using Texas A and M's Standard Operation Procedure (Lee, 1995b). This procedure duplicates normal commercial practice using similar equipment scaled down from commercial operation to produce hulls, meal, and crude and refined oil. Three treated cotton samples were processed, and residues were concentrated in crude oil only (Table 72).

Table 72. P	occessing study on cotton.

Sample	Residues, mg/kg			Processing factor			Average PF
Grain	0.34	0.60	0.44				
Hulls	0.15	0.19	0.12	0.44	0.32	0.27	0.34 (n=3)
Meal	0.10	0.45	0.30	0.29	0.75	0.68	0.58 (n=3)
Crude oil	1.0	-	1.6	2.9	-	3.6	3.3 (n=2)
Refined oil	< 0.02	< 0.02	< 0.02	< 0.06	< 0.03	< 0.04	<0.04 (n=3)

In a US study in 1994 three seed samples from a sunflower field plot treated twice with 8.4 kg carbaryl/ha were harvested at 60 days PHI and processed to hulls, meal and oil (Robinson, 1995g). The sunflower seeds were dried, cleaned and fed into a mill to crack the hulls and free the kernels. The moisture of the kernels was adjusted to 12% and the kernels heated and pressed to free part of the crude oil. The presscake was extracted twice with hexane under heat to give crude oil. Both crude oil fractions were filtered and the solvent removed. Refined oil was prepared by adding sodium hydroxide to the crude oil and the mixture heated to neutralize the oil. The procedure simulates industrial practice, except that the samples were processed by batch rather than continuously. Residues were reduced in all processed fractions (Table 73).

Table 73. Processing study on sunflowers.

	Residues, mg/kg			Processing factor			Average PF
Seed	0.32	0.33	0.33				
Hulls	0.11	0.10	0.27	0.34	0.30	0.82	0.48
Meal	< 0.02	< 0.02	< 0.02	< 0.06	< 0.06	< 0.06	< 0.06

	Residues, mg/kg			Processing factor			Average PF
Crude oil	0.06	0.05	0.07	0.19	0.15	0.21	0.18
Refined oil	< 0.02	< 0.02	< 0.02	< 0.06	< 0.06	< 0.06	< 0.06

# **RESIDUES IN ANIMAL COMMODITIES**

## Farm animal feeding studies

Ruminants. Dairy cattle were dosed daily with carbaryl in gelatine capsules by oral bolus after morning milking for 28 days (Lee, 1997a,b). There were three cows per group and four groups including the control group (I). Group II was dosed at the equivalent of 114 ppm carbaryl in dry feed; group III at 342 ppm and group IV at 1140 ppm, changed to 570 ppm at day 5 because of toxic effects presumably owing to the high dose. Group I animals were administered a placebo containing cellulose powder. Cows were milked twice daily throughout and samples collected for analyses approximately twice each week. A day's sample consisted of a proportional mix of the pm milk and the following morning's am milk. The pm milk was refrigerated overnight until it was pooled with the am milk. The cows were slaughtered within 7 hours of the last dose, and muscle, fat, liver, and kidney analysed for the parent compound and the metabolites 5,6-dihydro-5,6-dihydroxy-carbaryl and 5-methoxy-6-hydroxy-carbaryl by HPLC with post-column fluorescence detection. The LOQ was 0.02 mg/kg. Residues in the milk were mostly the metabolites, with the average carbaryl concentration during the 28 days <0.1 mg/kg in the dosed groups, increasing with the dose (Table 74).

Table 74. Residues in milk and milk fat of cattle dosed with carbaryl for 28 days.

Day		Resi	dues, mg/kg	
	Carbaryl	5,6-dihydro-5,6-dihydroxy- carbaryl	5-methoxy-6-hydroxy-carbaryl	Total residues
Group II–11	4 ppm			
01	<0.02, <0.02, <0.02	0.14, 0.12, 0.15	0.10, 0.09, 0.10	0.26; 0.23, 0.26
	(<0.02)	(0.14)	(0.10)	(0.25)
04	<0.02, <0.02, <0.02	0.17, 0.12, 0.14	0.09, 0.07, 0.08	0.27, 0.20, 0.23
	(<0.02)	(0.14)	(0.08)	(0.23)
08	0.02. 0.02, 0.02	0.18, 0.10, 0.15	0.10, 0.08, 0.09	0.30, 0.21, 0.27
	(0.02)	(0.14)	(0.09)	(0.26)
11	<0.02, 0.02, 0.02	0.18, 0.10, 0.17	0.10, 0.07, 0.10	0.30, 0.19, 0.29
	(0.02)	(0.15)	(0.09)	(0.26)
15	<0.02, <0.02, <0.02	0.20, 0.09, 0.14	0.08, 0.08, 0.10	0.25, 0.17, 0.26
	(<0.02)	(0.14)	(0.09)	(0.23)
18	<0.02, <0.02, <0.02	0.20, 0.16, 0.13	0.12, 0.11, 0.10	0.34, 0.28, 0.25
	(<0.02)	(0.16)	(0.11)	(0.29)
22	0.03, 0.02, 0.02	0.20, 0.15, 0.18	0.12, 0.09, 0.10	0.36, 0.26, 0.30
	(0.02)	(0.18)	(0.10)	(0.31)
25	0.04, 0.02 (2)	0.30, 0.07, 0.18	0.10, 0.09, 0.12	0.44, 0.15, 0.31
	(0.02)	(0.18)	0.10)	(0.30)
28	<0.02, <0.02, <0.02	0.19, 0.10, 0.15	0.12, 0.11, 0.13	0.32, 0.20, 0.28
	(<0.02)	(0.15)	(0.12)	(0.27)
Average	0.02	0.15	0.10	0.26

Day		Resi	Residues, mg/kg				
	Carbaryl	5,6-dihydro-5,6-dihydroxy- carbaryl	5-methoxy-6-hydroxy-carbaryl	Total residues			
Group III-342	2 ppm						
01	0.04, 0.07, 0.04	0.42, 0.72, 0.50	0.20, 0.29, 0.26	0.66, 1.2, 0.81			
	(0.05)	(0.55)	(0.25)	(0.89)			
04	0.03, 0.06, 0.04	0.47, 0.74, 0.45	0.30, 0.36, 0.28	0.80, 1.2, 0.77			
	(0.04)	(0.55	(0.31)	(0.92)			
08	0.04, 0.05, 0.03	0.42, 0.56, 0.36	0.30, 0.32, 0.22	0.76, 0.93, 0.61			
	(0.04)	(0.45)	(0.28)	(0.77)			
11	0.04, 0.04, 0.04	0.46, 0.56, 0.46	0.28, 0.30, 0.30	0.79, 0.91, 0.80			
	(0.04)	(0.49)	(0.29)	(0.83)			
15	0.04, 0.03, 0.04	0.30, 0.22, 0.34	0.24, 0.21, 0.32	0.58, 0.46, 0.71			
	(0.04)	(0.29)	(0.26)	(0.58)			
18	0.03, 0.04, 0.03	0.49, 0.67, 0.48	0.23, 0.37, 0.27	0.75, 1.1, 0.78			
	(0.03)	(0.55)	(0.29)	(0.88)			
22	0.02, 0.03, 0.02	0.40, 0.41, 0.46	0.28, 0.36, 0.27	0.71, 1.0, 0.76			
	(0.02)	(0.42)	(0.30)	(0.82)			
25	0.04, 0.03, <0.02	0.40, 0.50, 0.20	0.22, 0.27, 0.12	0.66, 0.80, 0.34			
	(0.03)	(0.37)	(0.20)	(0.60)			
28	0.03, 0.05, 0.03	0.42, 0.64, 0.36	0.24, 0.30, 0.24	0.69, 0.99, 0.64			
	(0.04)	(0.47)	(0.26)	(0.77)			
Average	0.04	0.46	0.27	0.78			
Group IV-114	40 ppm, 570 ppm fro	m day 5					
1	0.08, 0.14, 0.09	1.1, 1.4, 2.1	0.83, 2.0, 1.2	2.5, 3.6, 3.4			
	(0.10)	(1.5)	(1.3)	(3.2)			
4	0.14, 0.25, 0.16	2.1, 3.8, 4.5	1.7, 3.7, 1.2	4.0, 7.8, 5.9			
	(0.18)	(3.5)	(2.2)	(5.9)			
8	0.03, 0.02, 0.04	0.84, 0.30, 0.49	0.56, 0.46, 0.50	1.4, 0.80, 1.0			
	(0.03)	(0.54)	(0.51)	(1.1)			
11	0.02, 0.03, 0.03	0.43, 0.36, 0.36	0.25, 0.36, 0.29	0.70, 0.75, 0.68			
	(0.03)	(0.38)	(0.30)	(0.71)			
15	0.03, 0.04, 0.04	0.70, 0.77, 0.58	0.56, 0.78, 0.42	1.3, 1.6, 1.0			
	(0.04)	(0.68)	(0.59)	(1.3)			
18	0.02, 0.05, 0.04	0.56, 0.83, 1.1	0.56, 0.74, 0.42	1.2, 1.6, 1.6			
	(0.04)	(0.83)	(0.57)	(1.5)			
22	0.03, 0.06, 0.04	0.85, 1.1, 1.0	0.59, 0.86, 0.67	1.4, 2.0, 1.8			
	(0.04)	(0.98)	(0.71)	(1.7)			
22	0.03, 0.04, 0.04	0.60, 0.76, 0.71	0.36, 0.56, 0.40	0.99, 1.4, 1.2			
(fat)	(0.04)	(0.69)	(0.44)	(1.2)			
25	0.03, 0.05, 0.03	0.73, 0.98, 0.74	0.52, 0.90, 0.44	1.3, 1.9, 1.2			
	(0.04)	(0.82)	(0.62)	(1.5)			
27	0.02, 0.02, 0.02	0.37, 0.50, 0.25	0.24, 0.40, 0.18	0.64, 0.92, 0.44			
(fat)	(0.02)	(0.37)	(0.27)	(0.67)			
28	0.03, 0.06, 0.03	0.70, 1.1, 0.52	0.54, 0.88, 0.40	1.3, 2.0, 0.95			
	(0.04)	(0.77)	(0.61)	(1.4)			
Average	0.06	1.1	0.82	2.0			
whole milk	0.04 (d 22–28)	0.86 (d 22–28)	0.65 (d 22–28)	1.5 (d 22–28)			
Average fat	0.03	0.53	0.36	0.94			

Residues of carbaryl and its metabolites were detected in muscle, liver, kidney, and fat at all dose levels, increasing with increased dose. The highest residue concentrations were found in the kidneys, 6.9 mg/kg of total residues at the highest dose (Table 75).

Table 75. Residues of carbaryl in cattle tissues after dosing for 28 days.

Group No.	Sample	Carbaryl	5,6-dihydro-5,6- dihydroxy-carbaryl	5-methoxy-6- hydroxy-carbaryl	Total residue, as carbaryl
II	Muscle	<0.02, <0.02, <0.02 (<0.02)	0.36, 0.25, 0.32 (0.31)	<0.02, <0.02, <0.02 (<0.02)	0.39, 0.27, 0.35 (0.34)
	Liver	0.54, 0.28, 0.66 (0.49)	0.30, 0.16, 0.12 (0.19)	<0.02, <0.02, 0.02 (0.02)	0.86, 0.46, 0.94 (0.75)
	Kidney	0.67, 0.56, 0.85 (0.69)	0.69, 0.47, 0.64 (0.60)		
	Fat	0.04, <0.02, <0.02 (0.02)	0.12, 0.03, 0.04 (0.06)	0.17, 0.05, 0.06 (0.09)	0.29, 0.09, 0.11 (0.16)
III	Muscle	0.03, 0.05, 0.03 (0.04)	0.92, 1.2, 0.78 (0.97)	<0.02, <0.02, <0.02 (<0.02)	0.97, 1.2, 0.82 (1.0)
	Liver	0.74, 1.0, 1.0 (0.91)	0.51, 0.79, 0.43 (0.58)	0.04, 0.08, 0.03 (0.05)	1.3, 1.9, 1.5 (1.6)
	Kidney	2.3, 2.1, 1.9 (2.1)	1.8, 2.5, 1.6 (2.0)	0.38, 0.46, 0.50 (0.45)	4.5, 4.9, 4.0 (4.5)
	Fat	0.03, 0.12, 0.02 (0.06)	0.12, 0.34, 0.08 (0.18)	<0.02, 0.06, <0.02 (0.03)	0.17, 0.51, 0.11 (0.26)
IV	Muscle	0.04, 0.05, 0.04 (0.04)	1.6, 1.8, 1.9 (1.8)	<0.02, <0.02, <0.02 (<0.02)	1.7, 1.9, 2.0 (1.9)
	Liver	0.95, 1.0, 1.4 (1.1)	0.98, 1.4, 1.2 (1.2)	0.09, 0.12, 0.06 (0.09)	2.0, 2.5, 2.7 (2.4)
	Kidney	1.3, 2.9, 2.8 (2.3)	2.6, 4.1, 4.4 (3.7)	0.72, 0.75, 1.1 (0.86)	4.7, 7.8, 8.2 (6.9)
	Fat	0.05, 0.07, 0.03 (0.05)	0.14, 0.22, 0.18 (0.18)	0.02, 0.02, <0.02 (0.02)	0.21, 0.31, 0.22 (0.25)

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Data from the US Department of Agriculture Pesticide Data Program (PDP) from 1994 to 1998 were submitted. The minimum number of samples collected for a crop in one year was 47, and the average 530 samples per year. The residues of carbaryl ranged from 0.004 mg/kg to 4.8 mg/kg, with 98% of the residues  $\leq 1$  mg/kg (Table 76).

Table 76. Residues of carbaryl found in the US Pesticide Data Program (1994-1998).

Crop	Samples	in 1994	Samples	in 1995	Samples	in 1996	Samples	in 1997	Samples	s in 1998
	Analysed	Detected <sup>1,2</sup>	Analysed	Detected	Analysed	Detected	Analysed	Detected	Analysed	Detected
Apple juice					177	57 (≤0.1)	683	68(≤0.1)	694	229(≤0. 1)
Apple	686	144 (≤2)	693	76 (≤1)	530	65 (≤1)				
Carrots	687	0	701	0	500	0				

Crop	Samples	s in 1994	Samples	in 1995	Samples	in 1996	Samples	in 1997	Samples	s in 1998
	Analysed	Detected <sup>1,2</sup>	Analysed	Detected	Analysed	Detected	Analysed	Detected	Analysed	Detected
Grape juice									665	245 (≤0.05)
Grapes	669	12 (≤1)	690	19 (≤0.5)	525	34 (≤1)				
Milk					570	0	727	0	595	0
Orange juice							692	24 (≤0.05)	700	6 (≤0.02)
Orange	683	52 (≤0.2)	691	71 (≤0.2)	518	61 (≤0.2)				
Peaches	396	62 (≤5)	367	54 (≤5)	325	52 (≤2)				
Peaches, canned							739	80 (≤0.5)		
Pears							708	42 (≤1)	712	18 (≤0.5)
Processed sweet corn	462	0	671	0	173	0				
Soya bean grain							159	0	590	0
Sweet potatoes					507	1 (≤1)	695	2 (≤0.5)	357	0
Tomatoes					174	0	722	2 (≤0.2)	717	2 (≤0.02)
Wheat grain					600	3 (≤0.02)	340	1 (≤0.02)	623	

 $<sup>^{1} &</sup>gt; 0.005 \text{ mg/kg}$ 

The Carbamate Market Basket Survey Task Force (CMBSTF) sponsored a study to determine the level of certain methylcarbamate insecticide residues in single-serving samples of fresh fruits and vegetables available for US consumption (Study No. TCI-99-001). Carbaryl was one of the insecticides considered and the selected crops were apples, tomatoes, head lettuce, grapes, peaches, broccoli, oranges and bananas.

Sampling took place during 1999 and 2000, although most samples were gathered in 1999. There was a minimum of 393 samples of all crops except peaches (285 samples). Residues of carbaryl were <0.00033 mg/kg in all broccoli and <0.001 mg/kg in all tomato samples, and were found in apples, lettuce, grapes, peaches, bananas, and oranges at levels from ND to a maximum of 1.2 mg/kg (in peaches). Table 77 summarizes the data from the market basket survey of single-serving commodities. Of approximately 400 samples analysed 80%–100% had no residues above the LOQ of 0.001 mg/kg, and the highest were 0.21 mg/kg in apples, <0.001 mg/kg in tomatoes, 0.78 mg/kg in grapes, 1.2 mg/kg in peaches, and 0.043 mg/kg in oranges.

<sup>&</sup>lt;sup>2</sup> in parenthesis, the highest residues, mg/kg.

Table 77. Carbaryl residues in the Carbamate Market Basket Survey in the USA (1999-2000).

Crop	No. of	Samples with residu	ies, ≥0.001 mg/kg	Residue range, mg/kg
	analyses	No.	%	
Apple	400	32	8	ND-0.21
Tomato	399	0	0	ND-<0.001
Head lettuce	399	1	<1	ND-0.0014
Grape	393	31	8	ND-0.78
Peach	285	58	20	ND-1.2
Broccoli	395	0	0	ND
Orange	399	11	3	ND-0.043
Banana	400	5	1	ND-0.0020

ND: <0.00033 mg/kg

The Government of Australia reported results from monitoring programmes conducted in Queensland and Victoria under the Agricultural and Veterinary Chemical Control Act requirements. In the Queensland programme from June 1996 to July 2000 no residues were detected (<0.02 mg/kg) in 24 samples of strawberries, rambutans, shallots, broccoli, Brussels sprouts, rockmelon, lettuce and egg plants analysed. Data from Victoria from 1987 to 2001 showed that of the 79 samples of apricots, grapes, nectarines, peaches and tomatoes analysed, only three had detectable residues of carbaryl (LOQ of 0.05 mg/kg) ranging from 0.17 to 0.63 mg/kg.

### NATIONAL MAXIMUM RESIDUE LIMITS

Carbaryl is registered for use in a number of countries, where national MRLs have been set for a variety of commodities. These are shown in Table 78 (Food commodities) and Table 79 (Animal Feed commodities).

Table 78. National MRLs-Food commodities.

Crop	Country	MRL
Almonds	USA	1
Almonds (hulls)	USA	40
Apple	Argentina	3
	Australia	5
	Brazil	5
	Canada	5
	Chile	5
	Europe	3
	France	3
	Italy	3
	Peru	5
	Spain	3
	Turkey	3
Apricot	Australia	10
	Belgium	3
	Canada	10
	Chile	5
	Italy	3
	Spain	3
	USA (Pre and post-harvest)	10

Crop	Country	MRL
Asparagus	Australia	10
	Canada	10
	Chile	10
	Europe	1
	France	1
	Spain	3
	USA (post-harvest.)	10
Avocados	Australia	10
	Cuba	1
	USA	10
Banana	Australia (in the pulp)	5
	Brazil	5
	Canada	10
	Chile	5
	Costa Rica	10
	Ecuador	5
	Nicaragua	10
	Panama	10
	USA(Pre and post-harvest)	10
Barley	Canada	2
	USA	0
Beans	Brazil	5
	Chile (immature green)	5
	Cuba	0.5
	Cuba (string)	0.5
	Peru	10
	USA	10
	Venezuela	10
Beet	Brazil	2
	Canada	5
	USA (garden)	5
Blackberries	Australia	10
	Canada	10
	Chile	10
	USA (post-harvest)	12
Blueberries	Australia	7
	Canada	7
	Chile	7
	USA (post-harvest)	10
Brassica, general	Belgium	3
-	Italy	3
Broccoli	USA	10
Brussels sprouts	USA	10
Cabbage	Brazil	10
-	Canada	5
	Chile	5
	Cuba	2
	USA	10
Cabbage (Chinese)	USA	10
Cacao	Cuba	1

Crop	Country	MRL
Carrots	Brazil	2
	Canada	5
	Chile	2
	Cuba	0.5
	USA	10
Cassava	Cuba	1
Cauliflower	USA	10
Cereals	Argentina (other)	0.1
	Australia (grain)	5
	Belgium (other)	0.5
	Italy (other)	0.5
	Turkey	0.5
Cherries	Argentina	3
	Australia	5
	Canada	10
	Chile	5
	Spain	3
	USA	10
Chestnuts	USA (post-harvest)	1
Citrus	Argentina	3
	Australia	7
	Brazil	7
	Canada	10
	Chile	7
	Cuba	0.5
	Nicaragua	10
	Spain	5
	USA	10
Coffee	Cuba	1
Corn	Argentina	1
(see also Maize)	Cuba	0.5
	USA	5
	Venezuela	5
Cotton	Argentina	1
	Australia	1
	Brazil	1
	Italy	1
	Nicaragua	5
	Peru	5 (seed)
	USA	5
	Venezuela	5
Cranberries	USA	10
Cucumber	Brazil	3
	Chile	3
	Cuba	1
	USA	10
Cucurbits	Australia	3
Dewberries	Australia	10
Democritics	USA	12
Edible offal	Australia	5
Edible Offar	Australia	J J

Crop	Country	MRL
Egg plant	Chile	5
	Cuba	1
	Europe	1
	France	1
	Germany	0.05
	Netherlands	3
	USA	10
Eggs	Australia	0.2
	Chile	0.5
	USA	0.5
Endive (escarole)	USA	10
Fat, cattle	USA (pre-spray, min. 14	0.1
	days)	
Fat, meat	USA	5
Fat of meat, goat	USA	0.1
Fat of meat, hogs	USA	0.1
Fat, horse	USA	0.1
Fat, sheep	USA	0.1
Fruits, fresh	Cuba	1
Fruits, other	Belgium	1
	Italy	1
	Turkey	2
Garlic	Cuba	1
Grapes	Australia	5
	Belgium	3
	Europe	3
	Italy	3
	USA (post-harvest)	10
Grapefruit	Chile	50
Hazelnuts	Turkey	0.5
	USA	1
Kohlrabi	USA	10
Kale	USA	12
Kidney, cattle	USA	1
Kidney, goat	USA	1
Kidney, hogs	USA	1
Kidney, horse	USA	1
Kidney, sheep	USA	1
Kiwifruit	Australia	10
	Chile	10
Large berries (unless specified)	Australia	0.1
Leafy Vegetables	Argentina	3
	Australia	10
	Canada	10
	Chile	10
	Italy	3
Lentils	USA	10
Lettuce	USA	10
Lettuce and similar	Belgium	3
Liver, cattle	USA (pre-spray, min. 14	1
	days)	
Liver, goat	USA	1
Liver, hogs	USA	1

Crop	Country	MRL
Liver, horse	USA	1
Maize	Peru	5
(see also Corn)	Spain	0.5
	Turkey	0.5
Meat	Australia	0.5
	Turkey	0.2
	USA (pre-spray)	5
Meat (mammalian)	Australia	0.2
Meat, cattle	Chile	0.2
	USA	0.1
Meat, goat	USA	0.1
Meat, hogs	USA	0.1
Meat, horse	USA (pre-spray)	0.1
Meat, sheep	USA (pre-spray)	0.1
Meat by-products	Turkey	0.2
Meat by-products, cattle	USA	0.1
Meat by-products, goat	USA	0.1
Meat by-products, hogs	USA	0.1
Meat by-products, horse	USA	0.1
Meat by-products, sheep	USA	0.1
Melon	Brazil	3
	Canada	3
	Chile	3
	Turkey	3
	USA	10
Milk	Australia	0.05
	Chile	0.1
	Turkey	0.2
	USA	0.3
Milky products	Chile	0.1
Nectarines	Australia	10
rectariles	USA	10
Nuts	Argentina	1
Titalo	Canada	10
Nuts, Tree	Australia	1
11465, 1100	Australia (whole in shell)	10
Oats	Canada	2
- Cum	USA	0
Okra	Australia	10
Ontu	Canada	10
	USA	10
Olives	Argentina	3
Onves	Argentina Australia	10
	Australia (processed)	+
	Canada	10
		10
	Chile	10
	Europe	1
	France	1
	Spain	1
	Turkey	0.5
	USA	10

Crop	Country	MRL
Onions	Cuba	1
Passion fruit	Australia	5
	Brazil	5
Peach	Chile	5
	Argentina	3
	Australia	10
	Belgium	3
	Brazil	5
	Canada	10
	Chile	5
	Europe	3
	Italy	3
	Spain	3
	USA (pre and post-harvest)	10
Peanuts	Brazil	5
	USA (post-harvest)	5
Pears	Argentina	3
	Chile	5
	Italy	3
	Spain	3
	Turkey	3
Peas	Brazil	5
	Canada	5
	Chile	100
	USA	10
Pecans (post-harvest)	USA	1
Pepper	Brazil	5
**	Canada	5
	Cuba	3
	USA (post-harvest)	10
Pineapple	Brazil	5
•	Costa Rica	2
	Ecuador	2
	USA	2
Pistachio nuts	USA	1
Plums	Argentina	3
	Australia (including prunes)	5
	Belgium	3
	Brazil	5
	Canada	10
	Chile	5
	Europe	3
	Italy	3
	Spain	3
	USA	10
Pome fruits	Belgium	3
	USA	10
Potato	Australia	0.2
	Belgium	0.1
	Brazil	0.2
	Canada	0.2
	Chile	0.2

Crop	Country	MRL
	France	1
	Italy	0.2
	Peru	0.2
	Spain	0.1
	Turkey	0.2
	USA	0.2
Pumpkin	Brazil	3
	Canada	10
	USA	10
Radish	Brazil	2
	Canada	5
Radish (without tops)	USA	5
Raspberries	Australia	10
•	Canada	10
	Chile	10
	Spain	3
	USA (post-harvest)	12
Rice	Belgium	1
	Chile	5
	Chile (peeled)	5
	Cuba	0.5
	Italy	1
	Nicaragua	5
	Panama	5
	Spain	1
	USA (pre and post-harvest)	5
	Venezuela	5
Rye	Canada	2
Teye	USA	0
Small berries (unless specified)	Australia	0.1
Strawberries	Australia	7
Stawberries	Canada	7
	Chile	7
	Cuba	0.5
	USA (post-harvest)	10
Soya bean	Argentina Argentina	1
Soyu ocuir	Brazil	5
	Cuba	1
	Italy	1
	USA (post-harvest)	5
Spinach	USA (post-narvest)	12
Squash, Summer	Canada	3
Squasii, Suiiiillei	USA	10
Sorghum	Argentina	0.1
Sorgium	Peru	10
		0.5
	Spain	
	USA	10
Commanda	Venezuela	10
Sugar beet	Italy	1
	Spain	0.1
Sugar cane	Australia	0.05

Crop	Country	MRL
Sunflower	Australia	1
	Italy	1
	Turkey	0.5
	USA (post-harvest)	1
Sweet corn	Australia	1
	USA	5
Swiss chard	USA	12
Tomato	Brazil	5
	Canada	5
	Chile	5
	Cuba	1
	Europe	1
	France	1
	Netherlands	3
	USA	10
Turnip	USA	5
Vegetables	Turkey	1
Vegetables (unless specified)	Australia	5
	Belgium	1
Vegetables, other	Italy	1
Vegetables, tuber	Argentina	0.2
Yams	USA	0.2
Walnuts	Chile	10
Walnuts (meat, shell removed)	USA	1
Wheat	Argentina	0.1
	Brazil	0.01
	Canada	2
	Spain	0.05
	USA (post-harvest)	3
Wheat (flour)	Chile	0.2

Table 79. National MRLs-Animal feed commodities.

Crop	Country	MRL
Alfalfa	Peru	100
	USA	100
Alfalfa (hay)	USA	100
Barley (fodder)	USA	100
Barley (straw)	USA	100
Beans (forage)	USA	100
Beans (hay vines)	USA	100
Clover	USA	100
Clover (hay)	USA	100
Corn (forage)	Peru	100
Corn (fodder, stover)	USA	100
Corn (forage, green)	USA	100
Corn (hay)	USA	100
Cowpeas (vines)	USA	100
Forage of cereal grain	Australia	100
Grass (straw)	USA	100

Crop	Country	MRL
Grasses	USA	100
Leguminous fodder	Italy	1
Oats (fodder)	USA	100
Oats (straw)	USA	100
Peanuts (hay)	USA	100
Peas (vines)	USA	100
Peas, cowpeas (hay)	USA	100
Rice (straw)	USA	100
Rye (fodder, green)	USA	100
Rye (straw)	USA	100
Sorghum (forage, green)	USA	100
Soya beans (forage)	USA	100
Soya beans (hay)	USA	100
Sugar beet (tops)	USA	100
Trefoil, Birdsfoot (forage)	USA	100
Trefoil, Birdsfoot (hay)	USA	100
Wheat (fodder, green)	USA	100
Wheat (straw)	USA	100

#### **APPRAISAL**

Carbaryl (1-naphthyl methylcarbamate) has been re-evaluated several times after its first evaluation in 1965. The 2001 Toxicological evaluation established an ADI of 0.008 mg/kg body weight/day and an acute reference dose of 0.2 mg/kg body weight. The compound is schedule for periodic review at this Meeting. The Meeting received from the manufacturer data on metabolism in laboratory and farm animals, metabolism in plants, environmental fate in soil and water, bioaccumulation in fish, analytical methods, use pattern, residues in food in commerce or at consumption and national MRLs. Supervised trials were submitted on citrus fruit, pome fruits, stone fruits, grapes, olives, eggplant, tomato, sweet corn, pepper, lettuce, spinach, soybeans, carrots, beets, turnips, sweet potato, asparagus, field corn, rice, sorghum, wheat, and sunflower. Processing studies in various crops and a cattle feeding study were also submitted.

The Government of Australia submitted GAP information and residues in food in commerce or at consumption. The Government of Thailand submitted GAP information, summary of analytical method and summarized supervised trials residue data on cabbage, chili pepper, sweet corn, kale and soybean dry. The Government of the Netherlands submitted GAP information and national MRLs.

# Metabolism in animals

In a <u>rat</u> metabolism study of 1-naphthyl-<sup>14</sup>C Carbaryl, animals received single intravenous (IV) 1.02 mg/kg dose (Group A); single oral dose of 1.21 mg/kg (Group B); 14 daily non-radiolabeled doses of 1.0 mg/kg followed by a single radiolabeled dose of 1.21 mg/kg on the 15th day (Group C) and single oral dose of 48.0 mg/kg (Group D). The majority of the radioactivity (70-80%) was eliminated within 12 hours for the low dose groups and within 24 hours for the high dose group. Urine was the primary elimination route in all dosing groups, with 84.5% to 95.0% of the administered dose recovered, followed by the faeces, with 7 to 12.5% of the radioactivity. A maximum of 0.02% of the dose was recovered from tissues, and from 0.10 to 0.90 % TRR in carcass. The metabolism of <sup>14</sup>C-carbaryl was similar regardless of the route of administration, dose level or sex. The major metabolite identified in the faeces was 5,6-dihydro-5,6-dihydroxycarbaryl. In urine, free carbaryl accounted for 0.2 % TRR

and the main metabolites were free and conjugated 1-naphthol (14.5% TRR), 5-hydroxycarbaryl (12.8% TRR), and 5,6-dihydroxycarbaryl (8.2% TRR).

A summary of studies conducted in dairy <u>cattle</u> fed carbaryl was submitted. In two studies with 450 ppm in the diet for 2 weeks, carbaryl, 1-naphthol or conjugates of 1-naphthol residues were not found in the milk and one study did not detected carbaryl in tissues of cattle fed with 200 ppm carbaryl for 27 days. In another study, milk of a treated goat was shown to consist mainly of conjugated carbamate metabolites and 5,6-dihydro-5,6-dihydroxycarbaryl.

In the 4th study, carbaryl was fed to lactating cows at levels up to 100 ppm in the feed for 14 days. At each feeding level, approximately 0.2% of the dose was secreted in the milk, and the major metabolites in the milk were 5,6-dihydro-5,6-dihydroxy-carbaryl (34% of total radioactivity in milk), 1-naphthyl sulphate (26%) and the sulphate conjugate of 5-methoxy-6-hydroxy-carbaryl (23%). 1-naphthyl sulphate was the major metabolite in kidney (29.3%) and lung (27.3%) and 5,6-dihydro-5,6-dihydroxy-carbaryl the major metabolite in muscle and heart (38.6 and 31.3 % of total radioactivity, respectively)

The metabolism of carbaryl in <u>hens</u> was studied after oral administration of 1-naphthyl- $^{14}$ C Carbaryl to laying hens treated twice a day, for 7 consecutive days at 8.8 ppm and 10.5 ppm of carbaryl in the diet. In average, 97.7 % of the radioactivity was recovered in the excreta. Tissues contained only 0.17% of the administered dose, mostly concentrated in kidney (0.268 µg/g  $^{14}$ C-carbaryl eq) and liver (0.187 µg/g  $^{14}$ C-carbaryl eq). Egg york contained up to 0.176 µg/g  $^{14}$ C-carbaryl eq, being 1-naphtol sulfate the major metabolite (0.078 µg/g  $^{14}$ C-carbaryl eq). Desmethyl carbaryl was the major metabolite in liver (0.017 µg/g  $^{14}$ C-carbaryl eq), and 1-naphthol the major in abdominal fat (39.1 µg/g  $^{14}$ C-carbaryl eq). The highest concentration of free carbaryl was found in fat (26.9% TRR, 0.004 µg/g  $^{14}$ C-carbaryl eq q).

The metabolic pathway of carbaryl in <u>animals</u> involves hydroxylation of the N-methyl group, hydrolysis of the carbamate ester, and hydroxylation of the naphthalene ring through epoxide formation. The main metabolites formed are 1-naphtol, 4-hydroxycarbaryl, 5-hydroxycarbaryl, 3,4-dihydro-3,4-dihydroxycarbaryl, 5,6-dihydro-5,6-dihydroxy-carbaryl and 5-methoxy-6-hydroxycarbaryl (cow). The metabolites are subsequently conjugated to form water-soluble glucuronides and or sulphates. Metabolism through GSH conjugation forms 5,6-dihydro-5-(S-cysteinyl)-6-hydroxycarbaryl or a positional isomer.

### Metabolism in plant

Greenhouse-grown radishes were treated five times with  $^{14}$ C-carbaryl at 2.0 kg/ha and harvested at 7 days PHI. In the tops, most of the radioactivity was found in the acetone:water (50:50) rinse (37.9% TRR) and in the internal organic extracts (42.8% TRR), with carbaryl representing the single radioactive component (total of 121  $\mu$ g/g  $^{14}$ C-carbaryl eq). In roots, 36.34 %TRR remained in the organosoluble extract, all being carbaryl (1.34  $\mu$ g/g  $^{14}$ C-carbaryl eq). The major identified metabolites in aqueous extracts of radishes were 5,6-dihydro-5-(S-cysteinyl)-6-hydroxycarbaryl or a positional isomer (<3% TRR - 1.51  $\mu$ g/g  $^{14}$ C-carbaryl eq in the tops and 0.076  $\mu$ g/g  $^{14}$ C-carbaryl eq in the roots) and 4-hydroxycarbaryl glycoside (1.97  $\mu$ g/g  $^{14}$ C-carbaryl eq in the tops). Nonextractable residues (8.3% TRR in the tops and 43.4% TRR in roots) were separated into cellulose and lignin fractions after buffer extractions and enzyme treatments. The radioactivity in the cellulose fraction was the largest portion in the root (0.694 mg/g  $^{14}$ C-carbaryl eq), and in tops accounted for 3.27  $\mu$ g/g  $^{14}$ C-carbaryl eq.

Leaf lettuce was treated with four applications of 1-naphthyl-<sup>14</sup>C carbaryl at 1.96 kg/ha and harvested at 8 days PHI. Most of the radioactivity was found in the rinse (64.0% TRR, 23.53 µg/g

 $^{14}$ C-carbaryl eq) and organosoluble extract (29.9% TRR, 10.3 μg/g  $^{14}$ C-carbaryl eq), and showed to be unchanged carbaryl. Glycoside conjugates of 1-naphthol, hydroxycarbaryl and hydroxymethylcarbaryl were the main metabolites found in the aqueous extract (0.13 to 0.25 μg/g  $^{14}$ C-carbaryl eq).

Soybean plants were treated four times with at 1.7-2.1 kg/ha  $^{14}$ C-carbaryl and soybean forage and mature plants (seed and hay) were harvested at 7 and 47 days PHI. Carbaryl accounted for 96.2% of the radioactivity in the external rinse and organosoluble extract of forage (156.3  $\mu$ g/g  $^{14}$ C-carbaryl eq), 94.5% of organic extracts of hay (149.9  $\mu$ g/g  $^{14}$ C-carbaryl eq) and 85.4% TRR in bean (0.9  $\mu$ g/g  $^{14}$ C-carbaryl eq). In beans, most of the radioactivity was in the aqueous phase (83.2% TRR, 18.25  $\mu$ g/g  $^{14}$ C-carbaryl eq). Hydroxymethyl carbaryl hexose conjugate was the main metabolite in forage (7.3% TRR, 20.4  $\mu$ g/g  $^{14}$ C-carbaryl eq) and hay (12.2% TRR, 50.3  $\mu$ g/g  $^{14}$ C-carbaryl eq). In bean, the main metabolite was tentatively assigned as 1-naphthyl malonyl glucoside (26.1 % TRR, 5.72  $\mu$ g/g  $^{14}$ C-carbaryl eq). The radioactivity remained in the nonextractable residues ranged from 14.5 to 25.6 %TRR in all matrices, mostly in the cellulose fraction (3.1 to 7.8% TRR). Protease was the most effective enzyme treatment in removing radioactivity (1.7 to 5.0% TRR).

Apple fruit on the tree was painted once or twice with 50:50 acetone:water solution containing radiolabeled carbaryl (specific activity of 6.56 mCi/mM), at 10  $\mu$ Ci/apple and harvested at 28 or 53 days after treatment. The surface residues of samples from all treatments is mainly carbaryl (93.6% TRR in average) and traces of 1-naphthol, (hydroxymethyl)carbaryl and one minor unidentified material. Carbaryl was also the main internal residues in the fruit internal extracts, with concentration in the pulp approximately 2 times higher (20.1 to 45.8% TRR) than in the peel (9.5 to 21.9% TRR). Conjugates of 1-Naphthol, 4-hydroxycarbaryl and 5-hydroxycarbaryl were the major metabolites, with TRR ranging from 1.9 to 8.3% in peel and pulp.

The metabolic pathway for carbaryl in plants includes methyl and ring hydroxylation, carbamate ester hydrolysis, N-demethylation, followed by conjugation to form water-soluble glycosides. The main metabolites are free and or conjugated 1-naphthol, 4-hydroxycarbaryl, 5-hydroxycarbaryl, 7-hydroxycarbaryl, 5,6-dihydro-5,6-dihydroxycarbaryl, 5,6-dihydroxy-1-naphthol, desmethylcarbaryl, and (hydroxymethyl)carbaryl, in addition to 5,6-dihydro-5-(S-cysteinyl)-6-hydroxycarbaryl, or a positional isomer.

### **Environmental fate**

## Soil

The photolytic degradation of 1-naphthyl- $^{14}$ C carbaryl following surface application to a 1-mm layer of sandy loam soil at  $9.8 \pm 0.3$  mg/kg (equivalent to  $\sim 11.2$  kg a.i./ha.) was studied. The soil plates were exposed to artificial sunlight regime of approximately 12 hr light and 12 hr dark per day for 30 days, at  $25 \pm 1$ °C. Carbaryl concentration declined from 97.5% to 58.5% by the end of the 30-day period, with a calculated half-life of 41 days. Non-extracted  $^{14}$ C-residues represented 32.4% of the applied dose at 30 days and contained a mixture of not identified highly polar materials.

Carbaryl rapidly degraded under aerobic conditions in sandy loam soil treated with 11.2 mg/kg 1-naphthyl- $^{14}\text{C}$ , with a calculated half-life of 4.0 days. Total volatiles ( $^{14}\text{CO}_2$ ) ranged from 0.1 % at day 1 to 59.7% TRR at the end of the study (day 14). 1-naphtol was the only major degradation product identified in the extractable fraction, reaching a maximum of 34.5% TRR at day 1 (0.35  $\mu$ g/g  $^{14}\text{C}$ -carbaryl eq), dropping to 2.8 % TRR by day 2 (0.03  $\mu$ g/g  $^{14}\text{C}$ -carbaryl eq) Unextractable residues reached 17.7 % TRR by day 14.

The adsorption/desorption characteristics of carbaryl were determined in four soils and one sediment at 0.27, 1.02, 2.51, 5.01 and 10.0 ppm of 1-naphthyl-<sup>14</sup>C Carbaryl in 0.01 M calcium chloride. Each soil system was shaken in a water bath for 4 hours at 24-26 °C (adsorption phase) after what the remained solution was removed, 0.01 M CaC1<sub>2</sub> solution added to the soil pellet and the system treated as before (desorption phase). No measurable adsorption occurred with the loamy sand soil (0.05% organic matter, OM). The Freundlich adsorption coefficients (K values) correlated well (r<sup>2</sup> = 0.95) with the organic matter, being 1.74 in sandy loam soil (1.43% OM), 2.04 in clay loam sediment (1.4% OM), 3.0 in silty loam soil (2.4% OM), and 3.52 in silty clay loam soil (3.38 % OM). Freundlich K values for desorption ranged from 6.72 in sandy loam to 7.66 in silty clay loam (7.01 average). K<sub>oc</sub> were 385 and 485 in silty soils (medium mobility) increasing to 800 and 827 in sandy loam soil and clay loam sediment, respectively, showing a higher mobility in these soils.

Another carbaryl adsorption study was conducted on sand, sandy loam, silt loam, and silty clay loam soils as well as an aquatic sediment using 1-naphthyl-<sup>14</sup>C Carbaryl at 1, 2, 3, 4 and 5 mg/kg concentration. Carbaryl adsorption to soils and aquatic sediment increased with the carbaryl concentration in solution and with the organic matter content of the soil.

The organic matter content did not affect the carbaryl\_mobility in soil thin-layer. Rfs were 0.11 in silty loam soil (5.3% OM), 0.17 in sandy loam and loam soils (OM 0.8 - 3.0%), and 0.23 in silty clay loam soil (3.6% OM). This last soil had the pH of 6.3, while the pH in the other soils ranged from 5.0 to 5.8.

In an aged-residues column leaching study, <sup>14</sup>C-labeled and non-labelled carbaryl (equivalent to 3 kg/ha) was placed on top of a 30 cm length glass column (5.2 cm i.d.) packed with moist sandy loam and allowed to age for 30 days. Only 0.61% of the applied radioactivity eluted with the leachate after 46 days and 28.7% of TRR remained in the soil at this time, being 18.9% in the top 5 cm of the column. The remaining <sup>14</sup>C (70.7%) was probably lost as volatile degradation products.

### Water and water/sediment

The photodegradation of 1-naphthyl- $^{14}$ C Carbaryl at 10.1 mg/l exposed to artificial sunlight for 360 hours of continuous irradiation was studied in sterile water buffered at pH 5 and 25  $\pm$  1°C. Carbaryl concentrations declined from 97.0% to 33.4% TRR, with a half-life of 10.3 days. 1-naphthol was the only darkness period was calculated using the degradation rate constants under irradiated and no-irradiated conditions.

The hydrolysis of 1-naphthyl- $^{14}$ C Carbaryl (10 mg/l) was studied at 25°C under dark and sterile conditions at pH 5, 7, and 9. No evidence of hydrolytic degradation of carbaryl was detected in the pH 5 test samples (calculated  $t_{1/2}$  of 1277 days). Carbaryl degraded to 1-naphthol in pH 7 with a half-live of 12 days, and at pH 9 with a half-life of 3.2 hours. No other individual degradation product accounted for more than 2% of the radioactivity

The degradation of carbaryl was studied under anaerobic conditions in a pond water/sediment system treated with the  $^{14}$ C-carbaryl at 10 mg/l and maintained in the dark at  $25 \pm 1^{\circ}$ C up to 126 days. Total radioactivity in methylene chloride water extracts ranged from 81.4 % at day 0 declining to 5.4% at day 14, after what maintained from 2.9 to 5.4 % up to 26 days. Radioactivity in the sediment methanol : water extracts increased from 6.7 % at day 0 to 51.9% at day 1, declining to 32.8% at the end of the study. Unextracted residues reached a maximum of 23.6% of the applied radioactivity by Day 126. The calculated half-life was 72.2 days, with 1-naphthol being the major degradation product present, reaching an average maximum concentration of 26.3% TRR in sediment at day 94 (0.26  $\mu$ g/g  $^{14}$ C-carbaryl eq). None of other metabolites detected exceeded 2.5% of the applied dose.

An aerobic pond sediment/water degradation study of 1-naphthyl- $^{14}$ C Carbaryl at 10 mg/l was conducted for 30 days at  $25 \pm 1^{\circ}$ C in the dark .The radioactivity in aqueous phase steadily decreased from 77.9% on day 0 to 2.6% on day 30. Extractable  $^{14}$ C from the sediment ranged from 24.6% TRR at day 0 to 30.6 on day 30. Residues bound to sediment reached a maximum at day 21 (65% TRR), and it was fractionated to fulvic acid, humic acid and humin (average of 4.0, 18.9 and 24.3 % TRR, respectively). The half-life of carbaryl under the experimental conditions was estimated to be 4.9 days. 1-Naphthol was the major primary metabolite detected, reaching a maximum concentration at day 2 of 12.3 % TRR in water and in of 9.5% TRR in sediment. Several other degradation products were detected in water at levels from 2.2 to 4.6% TRR after 2 days, and in sediment extract, up to 19.3% TRR at day 14. None of the degradation products were identified, but the pathway is proposed to involve 1,4-naphthoquinone as an intermediate.

In summary, carbaryl undergo photodegradation during a 12 h artificial light/12 h dark regime in soil and water with half lives of 41 and 21 days, respectively, and of 10.3 days in water under continuous light. Under natural sunlight, the calculated half life in soil was 4 hours. Carbaryl is rapidly hydrolysed under basic condition (t1/2 = 3.2 hours), much slower at pH 7 (12 days) and is very stable under pH 5. In a water/sediment system, carbaryl degrades with a half-life of 72.2 days under anaerobic conditions and of 4.9 days under aerobic conditions. The main metabolite formed in all systems studied was 1-naphthol, which can degrade to 1,4-naphthoquinone under aerobic conditions. The compound adsorption capacity in soil increases with the organic matter content, and can be insignificant in soils with <0.1% organic matter. Carbaryl can be classified as having a medium to high mobility in soil.

#### **Accumulation in confined rotational crops**

A confined rotational crop study was conducted with 1-naphthyl- $^{14}$ C Carbaryl applied to a sandy loam soil at exaggerated rates of 17.3 - 18.0 kg a.i./ha. The plots were aged for 30, 120, or 365 days and subsequent planted with lettuce, radish, and wheat. The soil layer up to 7.5 cm showed the highest level of  $^{14}$ C-residues, with 15 to 21 ppm of carbaryl equivalents at day 0, which decreased to  $<6~\mu g/g$   $^{14}$ C-carbaryl eq in subsequent days. Residues in the 7.5 to 15 cm layer were  $<0.1~\mu g/g$   $^{14}$ C-carbaryl eq in the 30 and 120 days plot and up to 2.37  $\mu g/g$   $^{14}$ C-carbaryl eq in the 365 days plot.

Total radioactive residue revels decreased in every crop at subsequent planting intervals, except wheat straw. Lettuce harvested at maturity had 0.103 ppm  $^{14}$ carbaryl equivalents in the 30 DAT plot, 0.09 ppm at the 120 DAT plot and 0.019  $\mu g/g$   $^{14}$ C-carbaryl eq at the 365 DAT plot, being most of the radioactivity present in aqueous soluble fraction and as insoluble residues. Total radioactivity in radish matrices harvested at immature (whole plant) and mature stage (tops and root) varied from 0.022 to 0.109  $\mu g/g$   $^{14}$ C-carbaryl eq. Total residues in wheat grain and straw ranged from 0.043 to 0.155  $\mu g/g$   $^{14}$ C-carbaryl eq, most of it being insoluble residues. Organosoluble residues accounted for 2 to 11% TRR in all crops. No compound was identified in any radioactive fraction. Radioactivity removed after protease and acid hydrolysis ranged from 0.055 to 0.002  $\mu g/g$   $^{14}$ C-carbaryl eq.

In summary, carbaryl concentration in crops planted in aged soils can be considered insignificant, which is supported by its limited mobility and rapid degradation in soil.

## Methods of residue analysis

Single residues methods for carbaryl in animal, vegetal and soil matrices were provided. No multiresidue method was submitted.

In a method for the determination of carbaryl in chicken, using reverse phase HPLC equipped with a post column hydrolysis system and fluorescent detector, the compound is extracted by maceration with methanol, the extract is cleaned up by liquid-liquid partition followed by C18 cartridge. A mean recovery of 80% (75-86%) was found in fortified samples at the range of 0.02 to 1 mg/kg.

A method has been developed for the determination of carbaryl, ant the free and conjugated metabolites 5,6-dihydro-5,6-dihydroxycarbaryl and 5-methoxy-6-hydroxy carbaryl in milk, egg, and cow and poultry tissues. This method involves extraction of the analytes with a combination of acetone, acetonitrile and water followed by mild acid hydrolysis reaction to convert the conjugates to This procedure also converts 5,6-dihydro-5,6-dihydroxycarbaryl forms. 5-hydroxycarbaryl. The reaction mixture is partitioned with dichloromethane and acetonitrile /hexane. The acetonitrile phase was analysed in a C18 column HPLC equipped with a post column hydrolysis system and fluorescent detector. The hydrolysis reaction gave recoveries of 75.5 to 106.4% for all metabolites in all cases. LOD (limit of detection) and LOQ (limit of quantification) for milk, egg, cow liver, cow muscle, cow kidney, cow fat, chicken muscle, chicken liver and chicken fat were 0.005 and 0.020 mg/kg, respectively. The LOO of carbaryl and 5-methoxy-6-hydroxy carbaryl in chicken liver was 0.10 mg/kg. Average recoveries of fortified samples from the LOQ to 5 mg/Kg, ranged from 72.4 to 107.4% for milk, egg, cow muscle, chicken muscle, chicken fat and chicken liver for all analytes. A modification of this method introduced mainly a high-speed centrifuge for layer separation and filtration of final extracts through 2 or 3 Acrodisk cartridges.

A method has been developed in 1992 for the determination of Carbaryl and 1-naphthol separately in vegetal crops after extraction with dichloromethane, clean-up with florisil column and quantification by HPLC with the basic post-column hydrolysis at  $100^{\circ}$ C and fluorescence detection. This method was validated in turnips, carrot, wheat, lemon, spinach and strawberry, at levels from 0.003 to 100 mg/kg, with recoveries ranging from 58.8 to 100.4%, and in mustard green, potato and peanut at levels from 5 to 50 mg/kg with recoveries from 75 to 95%. The extraction efficiency of the method was tested with grown-in  $^{14}$ C-carbaryl residues on lettuce and radish leaves, showing average LSC recoveries ranging from 82 to 104%.

Carbaryl residues can be extracted from soil with a mixture of acetone, water, and phosphoric acid. After filtration, dichloromethane partition and clean using florisil column, carbaryl is quantified as 1-naphthol by HPLC with a post-column hydrolysis / fluorescence detection system. Fortified samples at levels ranging 0.01 to 20 mg/kg had average recovery of 89.4%, and a LOQ of 0.02 mg/kg.

## Stability of residues in stored analytical samples

The percent of radioactivity in extracts of <sup>14</sup>C-carbaryl fortified hen tissues remained constant in egg yolk, fat, kidney, liver and muscle over 18 months of storage at -20 °C.

The stability of incurred residues of carbaryl and conjugated 5,6-dihydro-5,6-dihydro-carbaryl (5,6 DDC) and 5-methoxy-6-hydroxy carbaryl (5,6 MHC) in animal commodities was studied. The compounds were stable in liver (100 to 124 % remained after 173 days), kidney (91.4 to 98% remaining after 196 days) and muscle (80 to 102% remaining after 158 days). 5,6 DDC was also stable in milk and fat (97 to 103% remaining after 215 to 248 days), but only 56 to 79.3% of carbaryl and 5,6 MHC remained in these matrices during the same period.

Another study was conducted in samples fortified with carbaryl and the free 5,6 DDC and 5,6 MHC metabolites. 5,6 MHC was unstable under storage condition in fortified samples of muscle and

fat after 2 months (31.0 and 34.7% remaining) and of liver after 5.5 months (57.5% remaining). Carbaryl was stable in fortified samples of muscle and fat (96.1 and 126% remained) after 5 to 6.3 months, but not in liver after 2 months (56.7% remained). 5,6 DDC was stable in all three matrices, with 92.8 to 114% of the residues remaining after 5 to 6.3 months.

Carbaryl was relatively unstable (67% remained) in sugar beet roots fortified at 0.13 mg/kg level and stored at -10 °C for 287 days. At level of 10 mg/kg, carbaryl was relatively stable for 12 months (>80% of the initial residue) in barley flour, lettuce peanut, potato, tomato, tomato wet pomace, pure, paste and juice and wheat straw), but not in barley hulls and barley pearled (47% and 70.9% of the initial residue after 3 months), tomato dry pomace and wheat hay (65-75% of the initial residue after 6 months). Carbaryl fortified samples at 0.40 mg/kg, were stable up to 25 months in olive oil and apple (82.1 and 93.2 % remained) and relatively unstable in olive fruit after 6 months of storage at -20 °C (75.9% of the residues remained).

In a study with incurred carbaryl at levels from 0.08 to 55 mg/kg, residues were stable (>80% of the initial residue level) up to 15 months in almonds, soybeans, apples and grapes. Residues dropped to  $\le 60\%$  after 8 months in raisins, after 6 months in dry bean vines and after 10.5 months in dry bean hay.

### **Residue definition**

In plants, carbaryl represents the major residue (55-98% of the total radioactivity, TRR), and no metabolite is present at concentration >10% TRR.

The Meeting agreed that the residue definition for compliance with MRL and for dietary intake estimation in plant commodities is carbaryl.

Carbaryl accounted for <20% of the total radioactivity found in milk, and the metabolites 5,6-dihydro-5,6-dihydroxy-carbaryl, sulphate conjugates of 1-naphthyl and 5-methoxy-6-hydroxy-carbaryl and accounted for  $\sim\!82\%$  of the radioactivity. Carbaryl was the major metabolite in muscle (17% TRR), but was present at <10 % TRR in other tissues, which had mainly the metabolites 1-naphthyl sulphate (27 to 30 % TRR in kidney and lung) and 5,6-dihydro-5,6-dihydroxy-carbaryl (31 to 40% TRR in muscle and heart).

The Meeting acknowledge that carbaryl is not the major metabolite in animal products. However, the available methodology to analyse the metabolites 5,6-dihydro-5,6-dihydroxy-carbaryl and 5-methoxy-6-hydroxy-carbaryl, is not trivial, and it is not clear whether the standards for these metabolites can be made available to the laboratories for enforcement. Additionally, storage stability studies have shown that the metabolites have limited stability in some matrices after 1 month of storage. Currently, no information is available to assure that these two metabolites are not of health concern.

Furthermore, the Meeting agreed that, for practice purposes, the residue definition for compliance with MRL and for dietary intake estimation in animal commodities is carbaryl.

Carbaryl has a log  $P_{ow}$  of 1.85 to 2.36, and is not concentrated in fat of animals dosed orally. The Meeting concluded that carbaryl is not fat soluble.

### **Results of supervised trials**

The Meeting did not receive any information on residues on alfalfa forage, banana, bean forage, blackberries, clover, cotton seed, common bean, cranberry, cowpea (dry), cucumber, dewberries, eggs, hay or fodder of grasses, kiwifruit, melons, milk products, oats, okra, parsnip, peas, pea vines, peanut, peanut fodder, potato, poultry meat, poultry skin, pumpkins, raspberries, rice, husked, strawberry, swede, winter and summer squash and radish. The Meeting agreed to recommend to withdraw the current MRLs for these crops/commodities.

<u>Citrus fruit</u>. Supervised trials on citrus fruits were conducted in the United States (21 trials), Italy (4 trials), and Spain (4 trials). The GAP in USA for citrus is up to 8 applications of 2.42 to 8.4 kg a.i./ha, with a maximum of 22.4 kg a.i./ha per season and 5 days PHI. Additionally, in California, a rate of 5.6-17.9 kg a.i./ha, can be applied once against red scale and up two times against yellow scale (max. 22.4 kg a.i./ha). GAP in Italy recommends 0.071-0.142 kg a.i/hl and 7 days PHI. In Spain, the recommend GAP rate is 0.85 – 1.7 kg a.i./ha or 0.085-0.16 kg a.i./hl and 7 days PHI.

In six trials conducted in grapefruit in Florida and California within maximum GAP, residues were 0.59, 1.9, 2.5, 2.8, 3.5 and 6.8 mg/kg. In 4 trials conducted at the same rates in lemon in Arizona and California, residues were 4.8, 5.0, 5.1 and 5.5 mg/kg. Eleven trials conducted in Florida and California in orange within maximum GAP gave residues of 3.1, 3.7, 4.2 (2), 4.5, 4.6, 5.7, 6.5 (2), 8.1 and 10 mg/kg.

Four trials conducted in orange Italy at maximum GAP, residues in fruit at 7 days PHI were 0.83, 0.93, 2.6 and 3.6 mg/kg. In two declining trials, residues after 29 days represented  $\sim$ 27% of the initial levels. In the other two studies, the ration of residues pulp/residues in whole fruit averaged 0.12 (0.09, 0.10, 0.12 and 0.15). In four trials conducted in Spain at GAP rate, residues in orange fruit were 0.82, 3.2, 3.4 and 4.4 mg/kg at 7 days PHI.

The Meeting agreed that residues from trials conducted according to GAP in orange in USA, Italy and Spain belong to the same population (Mann-Whitney U-test, FAO Manual, 2002) and can be combined as follow: 0.82, 0.83, 0.93, 2.6, 3.1, 3.2, 3.4, 3.6, 3.7, 4.2 (2), 4.4, 4.5, 4.6, 5.7, 6.5 (2), 8.1 and 10 mg/kg. The orange residue population is in the same range as the residues in lemon (4.8, 5.0, 5.1 and 5.5 mg/kg) and grapefruit (0.59, 1.9, 2.5, 2.8, 3.5 and 6.8 mg/kg) and can be combined as a citrus residue population as follow: 0.59, 0.82, 0.83, 0.93, 1.9, 2.5, 2.6, 2.8, 3.1, 3.2, 3.4, 3.5, 3.6, 3.7, 4.2 (2), 4.4, 4.5, 4.6, 4.8, 5.0, 5.1, 5.5, 5.7, 6.5 (2), 6.8, 8.1 and 10 mg/kg.

The Meeting agreed to withdraw the Codex MRL of 7 mg/kg and recommends a maximum residue level of 15 mg/kg for carbaryl in citrus fruit.

Applying the ratio of residues in pulp/whole fruit to the median (4.2 mg/kg) and the highest residue (10 mg/kg) in the citrus residue population, the Meeting recommends an STMR of 0.487 mg/kg and an HR 1.16 mg/kg for carbaryl in citrus fruit, edible portion.

<u>Apple</u>. Supervised trials on apples were conducted in Argentina, Canada, France, Italy, the United Kingdom and the United States. One trial conducted in Argentina according to GAP could not be evaluated as only a summary table was provided.

In three supervised trials conducted in USA within maximum GAP for pome fruit (up to 8 applications of 0.56-3.36 kg a.i./ha, max. of 16.8 kg a.i./ha, and 3 days PHI), residues were 8.8, 9.6 and 10 mg/kg. In four trials conducted in Italy according to GAP (0.06-0.12 kg a.i./hl and 7 days PHI), residues were 0.22, 0.57, 0.67 and 0.68 mg/kg. In one trial conducted in France according to

Italian GAP, residues were 0.40 mg/kg. Thirteen trials were conducted in Canada, France, Italy and UK (against French GAP) at higher GAP rates and/or lower PHI and could not be used.

In one trial conducted in South France at higher GAP, 53 apple units were analysed, giving an average residue of 0.43 mg/kg, a standard deviation of 0.14 mg/kg and a highest residue of 0.81 mg/kg. In two French trials (North and South) conducted at higher GAP with 28 apple units analysed in each, average residues were 0.39 and 0.21 mg/kg, standard deviation of 0.29 and 0.16 mg/kg and highest residues of 1.2 and 0.70 mg/kg, respectively. In one trial conducted ion Italy at GAP, average residues of 52 apple units was 0.68 mg/kg, with a standard deviation of 0.33 mg/kg and a highest residue of 1.7 mg/kg.

Residues in apples from trials conducted in USA (8.8, 9.6 and 10 mg/kg) represent a distinct residue population from trials conducted in apple in Italy and France (0.22, 0.40, 0.57, 0.67 and 0.68 mg/kg) and cannot be combined.

The Meeting agreed that insufficient number of supervised trials were conducted according to the critical GAP (USA data), and recommends the withdrawal of the MRL of 5 mg/kg (T) for carbaryl in apple.

<u>Pear</u>. Ten trials were conducted in pears in America. One trial conducted in Argentina at lower GAP could not be evaluated as only summary table was provided. Four trials were conducted in Canada at lower GAP and could not be used. In five trials conducted in USA within maximum GAP for pome fruit residues, were 0.98, 2.8, 2.9, 3.5 and 4.0 mg/kg.

The Meeting agreed that insufficient number of supervised trials were conducted according to GAP and recommends the withdrawal of the MRL of 5 mg/kg (T) for carbaryl in pears.

Stone fruits. Ten supervised trials were conducted in the United States in peaches (GAP for stone fruits is up to 4 applications of 2.24 – 3.4 kg a.i./ha and 3 days PHI in all states, except for California were the rate is 3.4-4.5 kg a.i./ha, and 1 day PHI). One trial conducted in Italy (GAP is 0.071-0.118 kg a.i./hl) at higher GAP could not be used.

In seven trials conducted in peaches in Georgia, South Caroline and Pennsylvania according to maximum USA GAP rate, residues in fruit were 0.96, 2.3, 3.0 and 3.6\_mg/kg at 3 days PHI. In three trials conducted in California according to California maximum GAP, residues at 1day PHI were 4.8 (2) and 7.8 mg/kg. In three other trials conducted in California at maximum USA GAP residues were 2.0, 2.6 and 5.5 mg/kg. Trials conducted in California at different rates and in the other USA states yield residues which belong to the same population (Mann-Whitney U-test, FAO Manual, Chapter 6) and can be combined as, in rank order, 0.96, 2.0, 2.3, 2.6, 3.0, 3.6, 4.8 (2), 5.5 and 7.8 mg/kg.

In four trials conducted in plums in Michigan and Oregon according to maximum USA GAP, residues in fruit were 0.37, 1.4, 1.6 and 2.1 mg/kg at 3 days PHI. In four trials conducted in California at maximum GAP for this state (3.4-4.5 kg a.i./ha and 1 day PHI), residues were 0.69, 0.99 and 1.1 (2) mg/kg. These trials gave residues within the same range and can be combined as 0.37, 0.69, 0.99, 1.1 (2), 1.4, 1.6 and 2.1 mg/kg. Two trials conducted in California at maximum USA GAP gave residues of 0.05 and 0.06 mg/kg, which are in a lower range and can not be combined with the previous residue data set.

The Meeting agreed that the residues in peaches and plums comprise a single residue population (Mann-Whitney U-test) and can be combined as a residue population for stone fruits, in

rank order, 0.37, 0.69, 0.96, 0.99, 1.1 (2), 1.4, 1.6, <u>2.0, 2.1</u>, 2.3, 2.6, 3.0, 3.6, 4.8 (2), 5.5 and 7.8 mg/kg.

The Meeting agreed to withdraw the current MRL of of 10 mg/kg (T) for plums (including prunes), apricot and nectarine and recommends a maximum residue level of 10 mg/kg, an STMR of 2.05 mg/kg and an HR of 7.8 mg/kg for carbaryl in stone fruits, except cherries.

<u>Cherries</u>. Nine trials were conducted in cherries in USA (same GAP as for peaches and plums). In six trials conducted according to maximum GAP in Colorado, Michigan, New York, Oregon and Washington., residues were 2.4, 3.4, 3.9, 4.7, 6.7 and 16 mg/kg. In two trials conducted in California at maximum GAP for this state or at maximum USA GAP, residues at 1 or 3 days PHI were 2.1, 4.7 and 6.3 mg/kg. The trials conducted in USA gave residues in the same range which can be combined as 2.1, 2.4, 3.4, 3.9, 4.7 (2), 6.3, 6.7 and 16 mg/kg.

The Meeting agreed to withdraw the Codex MRL of 10 mg/kg (T) and recommends a maximum residue level of 20 mg/kg, an STMR of 4.3 mg/kg and an HR of 16 mg/kg for carbaryl in cherries.

<u>Grapes</u>. Seventeen trials were conducted in USA in California, Arizona, New York and Washington. In ten trials conducted in 1994 within maximum GAP rate (5 times 1.12-2.24 kg a.i./ha), residues at 7 days PHI were 2.4 (2), 3.0, 3.3, 6.2, 6.5, 7.2, 7.5, 7.9 and 33 mg/kg. In seven trials conducted in 1988 using 2 applications of the same rate, gave residues of 0.42, 2.4, 3.8, 4.5, 4.9, 5.3 and 6.5 mg/kg.

The trials conducted in 1994 at maximum GAP (5 applications) and in 1988 with 2 applications gave residues in the same range and will be combined as follow, in rank order, 0.42, 2.4 (3), 3.0, 3.3, 3.8, 4.5, 4.9, 5.3, 6.2, 6.5 (2), 7.2, 7.5, 7.9 and 33 mg/kg.

The Meeting recommends a maximum residue level of 40 mg/kg, an STMR of 4.9 mg/kg and an HR of 33 mg/kg for carbaryl in grapes

Olives. Fourteen trials were conducted in olive in Greece, Italy, Spain and USA from 1994 to 1998. In one trial conducted in Greece within maximum GAP rate (0.17 kg a.i/hl), residues were 1.9 mg/kg at 7 days PHI. In three trials conducted in Italy at maximum GAP rate (0.142 kg a.i/hl), residues at 7 days PHI were 1.6, 1.9 and 7.9 mg/kg. In three trials conducted in Spain within maximum GAP rate (2 applications of 0.17 kg a.i./ha), residues in fruit at 7 days PHI were 0.07, 22 and 26 mg/kg, and dropped to <0.05 - 4.0 mg/kg after 14 days.

In three trials conducted in California at maximum GAP rate (up to 2 applications at 8.4 kg a.i/ha), residues in fruit were 3.3, 4.0 and 6.6 mg/kg at 14 days PHI. Four other trials were conducted at a lower rate range (5.48-5.63 kg a.i/ha), and could not be used.

Residues from trials conducted according to GAP were 1.9 mg/kg in Greece, 1.6, 1.9 and 7.9 mg/kg in Italy, 0.07, 22 and 26 mg/kg in Spain, and 3.3, 4.0 and 6.6 mg/kg in USA.

The Meeting agreed that the residue data from trials conducted in USA and in Europe are not distinct and can be combined as, in rank order, 0.07, 1.6, 1.9 (2), 3.3, 4.0, 6.6, 7.9, 22 and 26 mg/kg.

The Meeting agreed to withdraw the Codex MRL of 10~mg/kg (T) and recommends a maximum residue level of 30~mg/kg for carbaryl in olives.

In the 7 trials from Spain, Greece and Italy, stoned fruit/fruit ratio were 1.14, 1.4 (3), 1.7, 1.25 and 1.33 and averaged 1.4. This ratio can be applied to the median residue level (3.65 mg/kg) and the highest residue (26 mg/kg) in the residue data set and the Meeting recommends an STMR of 5.1 mg/kg and an HR of 36.4 mg/kg for carbaryl in olive, edible portion

<u>Cabbage and kale</u>. The Government of Thailand provided data of 4 trials in Chinese cabbage and 3 trials in kale conducted from 1995 to 1997. There is no GAP for carbaryl in cabbage and kale in Thailand, and as only summary tables were provided, it was not possible to evaluate the trials.

The Meeting agreed to withdraw the current MRL of 5 mg/kg (T) for cabbage.

Eggplant. In eight trials conducted in France in eggplant with 2 applications at the maximum GAP rate (1.275 kg a.i/ha), residues at 7 days PHI were 0.06, 0.08, 0.16, 0.16, 0.16, and 0.49 mg/kg. Two other trials were conducted at higher PHI, and were not used.

The Meeting agreed to recommend a maximum residue level of 1 mg/kg, an STMR of 0.18 mg/kg and an HR of 0.49 mg/kg for carbaryl in eggplant.

<u>Pepper</u>. Five trials were conducted in USA in pepper at maximum GAP rate for pepper and tomato (up to 7 times at 2.24 kg a.i./ha, total of 9 kg a.i./ha) yielding residues at 3 days PHI of 0.33, 0.61, <u>1.8</u>, 2.0 and 3.8 mg/kg. Four trials were conducted in chilli pepper in Thailand and submitted as summary table by the Government could not be evaluated.

The Meeting agreed to confirm the current MRL of 5 mg/kg and recommends an STMR of 1.8 mg/kg and an HR of 3.8 mg/kg for carbaryl in pepper.

Tomato. In eleven trials conducted in tomato in California and Florida at the maximum GAP rate (up to 7 times at 2.24 kg a.i./ha, total of 9 kg a.i./ha), residues were 0.08, 0.47, 0.52, 0.67, 0.85, 1.1, 1.4, 1.9, 2.2, 2.3 and 2.4 mg/kg. In eight trials conducted in France at maximum GAP rate (1.275 kg a.i./ha), residues at 7 days PHI were 0.06, 0.11, <0.2 (2), 0.21, 0.22, 0.31 and 0.41 mg/kg. The residue population of carbaryl in tomato from trials conducted in USA and France can be combined (Mann-Whitney U-test) as, in rank order, 0.06, 0.08, 0.11, <0.2 (2), 0.21, 0.22, 0.31, 0.41, 0.47, 0.52, 0.67, 0.85, 1.1, 1.4, 1.9, 2.2, 2.3 and 2.4 mg/kg.

The Meeting agreed to confirm the current MRL of 5 mg/kg and recommend an STMR of 0.47 mg/kg and an HR of 2.4 mg/kg for carbaryl in tomato.

Sweet corn. In four trials conducted in USA in 1995 at maximum GAP rate (up to 8 times at 1.12-2.24 kg a.i./ha, min 187 l/ha), residues in ears (kernels and cobs with husks removed) at 2 days PHI were <0.02 (2), 0.02 and 0.05 mg/kg. In two trials conducted at the same rate but using lower water volume (119 l/ha) gave residues of 0.04 (2) mg/kg. This trial, although at higher GAP, can be used to support the data according to GAP.

Two trials were submitted by the Government of Thailand, but as only a summary report was provided, it was not possible to evaluate them.

The Meeting agreed to withdraw the current MRL of 1 mg/kg and recommends a maximum residue level of 0.1 mg/kg, an STMR of 0.02 mg/kg and an HR of 0.05 mg/kg for carbaryl in sweet corn (corn on the cob).

<u>Leafy vegetables</u>. Ten trials were conducted with carbaryl in lettuce and 10 in spinach in 1984 in Canada at maximum GAP rate, but samples were harvested before or after the recommended PHI (5 days for lettuce and 21 days for spinach). Eleven trials conducted in turnip greens at the same conditions in USA (no GAP) were evaluated against the Canadian GAP rate, however the samples were harvested before the recommended 21 days PHI and the trials could not be used.

As no trials according to GAP were submitted in lettuce, spinach and turnip greens, the Meeting agreed to withdraw the current MRL for leafy vegetables.

Soybeans. In nine trials conducted in soybean seeds in USA in 1994 using the maximum GAP rate (4 times 1.68 kg a.i./ha), residues in dry beans were <0.02, 0.03, 0.04, 0.05 (2), 0.09, 0.11, 0.12 and 0.15 mg/kg. Four trials conducted in Thailand were submitted only as summary table by the Government and could not be used.

The Meeting agreed to withdraw the current MRL of 1 mg/kg (T) and recommends a maximum residue level of 0.2 mg/kg, an STMR of 0.05 mg/kg and an HR of 0.15 mg/kg for carbaryl in dry soybeans.

<u>Carrots</u>. Seven trials were conducted in carrots in USA at the maximum GAP rate (2.24 kg a.i./ha, total of 6.72 kg a.i./ha). Residues at 7 days PHI in carrots were <0.02 (4), 0.03, 0.25 and 0.31 mg/kg.

The Meeting agreed to withdraw the current MRL of 2 mg/kg (T) and recommends a maximum residue level of 0.5 mg/kg, an STMR of 0.02 mg/kg and an HR of 0.31 mg/kg for carbaryl in carrots

<u>Garden beets</u>. Eight trials were conducted in USA in in garden beets root within maximum GAP (2.24 kg a.i./ha, total of 6.72 kg a.i./ha) and residues at 7 days PHI were <0.02 (3), <u>0.02</u>, <u>0.03</u> (2), 0.05 and 0.06 mg/kg.

The Meeting recommends a maximum residue level of 0.1 mg/kg, an STMR of 0.025 mg/kg and an HR of 0.06 mg/kg for carbaryl in garden beets.

<u>Sugar beet</u>. Thirteen trials were conducted in sugar beets in USA in 1985 at lower GAP and could not be used.

As no trials according to GAP were submitted, the Meeting agreed to withdraw the current recommendation of 0.2 mg/kg (T) for carbaryl in sugar beet.

Sweet potato. In seven trials conducted in sweet potato in USA at the maximum GAP rate (préplanting dip at 0.96 kg a.i./hl, followed by up to 8 applications at 0.56-2.24 kg a.i./ha or a total of 9 kg a.i./ha), residues at 7 days PHI were <0.02 (7) mg/kg.

The Meeting recommends a maximum residue level of 0.02\* mg/kg, an STMR and an HR of 0.02 mg/kg for carbaryl in sweet potato.

<u>Turnips</u>. In nine trials conducted in turnips using 3 applications at 2.2-2.4 kg a.i./ha, residues in root were < 0.02 (5), 0.02, 0.03, 0.10 and 0.89 mg/kg. There is no GAP for turnips in USA, but the trials can be evaluated against the maximum GAP rate recommended in Canada (0.6-2.5 kg a.i./ha, 7 days PHI).

The Meeting recommends a maximum residue level of 1 mg/kg, an STMR of 0.02 mg/kg and an HR of 0.89 mg/kg for carbaryl in turnip root.

Asparagus. In six trials conducted in asparagus in USA in 1994 at maximum GAP rate (3 times at 1.12-2.24 kg a.i/ha), residues at 1 day PHI were 2.0, 2.2, 7.2, 9.0 and 10 (2) mg/kg.

The Meeting agreed to withdraw the current MRL of 10~mg/kg (T) and recommends a maximum residue level of 15~mg/kg, an STMR of 8.1~mg/kg and an HR of 10~mg/kg for carbaryl in asparagus.

Maize. In eight trials conducted in USA in 1995 at maximum GAP rate (4 applications at 1.12-2.24kg a.i./ha), residues in grain were <0.02 mg/kg (8).

The Meeting recommends a maximum residue level of 0.02\* mg/kg, an STMR and an HR of 0.02 mg/kg for carbaryl in maize

<u>Barley</u>. In ten trials conducted in barley in Canada in 1986 at maximum GAP rate plants were harvested at 14 days (PHI is 28 days) and the results could not be used.

Twenty trials were conducted in USA, where there is no approved use in barley. Two trials using aerial application matched maximum GAP in Canada (2 x 1.76 kg a.i./ha, 7 to 14 days interval, 28 days PHI) and gave residues of 0.52 and 0.33 mg/kg.

As no sufficient number of trials according to GAP were submitted, the Meeting agreed to withdraw the current MRL of 5 mg/kg (Po, T) for carbaryl in barley.

<u>Rice</u>. In nine trials conducted in rice in USA in 1994 at maximum GAP rate (total of 4.48 kg.ai./ha), residues in grain within 14 days PHI were 2.8, 3.1, 6.0, 7.1, <u>8.4</u>, 10, 11 (2) and 46 mg/kg.

The Meeting recommends a maximum residue level of 50~mg/kg, an STMR of 8.4~mg/kg and an HR of 46~mg/kg for carbaryl in rice.

Rye. Three trials conducted in rye in USA, where there is no approved use, within Canada GAP (1.1-2.3 kg a.i./ha), had residues in grain at 14 days PHI of 0.36, 0.98 and 2.6 mg/kg, which dropped to 0.32, 0.85 and 2.0 mg/kg after 21 days. In two other trials, residues after 7 days were 9.4 and 6.2 mg/kg.

As insufficient number of supervised trials was conducted according to approved GAP, the Meeting agreed to withdraw the current MRL of 5 mg/kg (Po, T) for carbaryl in rye.

Sorghum. In nine trials conducted in sorghum in USA in 1994 at maximum GAP rate (up to 4 times at 1.12-2.24 kg a.i/ha, total of 6.7 kg a.i./ha), residues in grain after 14 days of the last application ranged from <0.02 to 7.1 mg/kg. The recommended PHI is 21 days.

As no supervised trials was conducted according to approved GAP, the Meeting agreed to withdraw the current MRL of 10 mg/kg (Po, T) for carbaryl in sorghum.

Wheat. Twenty-four trials were conducted in Canada and USA from 1986 to 1996. In twelve trials conducted in Canada at maximum GAP rate (2.3 kg a.i/ha), residues in grain were 0.22, 0.23, 0.26, 0.28, 0.33, 0.49, 1.1, 1.2, 1.3 and 1.6 (3) mg/kg within 14 days PHI. In twelve trials conducted in USA at maximum GAP (1.68 kg a.i./ha), residues in grain at 21 days PHI were <0.02 (7), 0.07, 0.12, 0.19, 0.27 and 1.4 mg/kg.

Residue populations from Canada and the USA were tested (Mann-Whitney U-test) and found to represent similar populations, and furthermore can be combined, in rank order, as <0.02 (7), 0.07, 0.12, 0.19, 0.22, 0.23, 0.26, 0.27, 0.28, 0.33, 0.49, 1.1, 1.2, 1.3, 1.4, and 1.6 (3) mg/kg.

The Meeting agreed to withdraw the current MRL of 5 mg/kg (Po, T) and recommends a maximum residue level of 2 mg/kg, an STMR of 0.245 mg/kg and an HR of 1.6 mg/kg for carbaryl in wheat.

<u>Nuts.</u> Twenty trials were conducted in nuts in USA in 1994 at maximum GAP rate (up to 4 applications at 5.56 kg a.i./ha and 14 days PHI). Residues in almonds nut meat were 0.03, 0.04, 0.07, 0.08 and 0.09 mg/kg.

Residues in pecans were <0.02 (3), 0.02, 0.03 and 0.05 mg/kg. Residues in pistachio nut meals were <0.02 (2), 0.03 and 0.09 mg/kg. Residues in walnuts were 0.02, 0.04, 0.09, 0.44 and 0.77 mg/kg.

The residue population in almonds, pecans, pistachio and walnuts can be combined as, in rank order, <0.02 (5), 0.02 (2), 0.03 (3), 0.04 (2), 0.05, 0.07, 0.08, 0.09 (3), 0.44 and 0.77 mg/kg.

The Meeting agreed to confirm the current MRL of 1 mg/kg (T) and recommend an STMR of 0.035 mg/kg and an HR of 0.77 mg/kg for carbaryl in tree nuts.

The Meeting also agreed to withdraw the current MRL of 10 mg/kg (T) for nuts (whole shell).

<u>Sunflower</u>. In five trials conducted in USA in 1994 in sunflower at maximum GAP (2 applications at 1.12-1.68 kg a.i./ha, 60 days PHI), residues in seeds were <0.02 (2), 0.03, 0.07 and 0.08 mg/kg.

The Meeting recommends a maximum residue level of 0.2~mg/kg, an STMR of 0.03~mg/kg and an HR of 0.08~mg/kg for carbaryl in sunflower seed.

#### **Animal feed commodities**

# Soybean forage and hay

Eight trials were conducted in soybeans forage in USA at maximum GAP. Residues in forage within 14 days PHI, on a fresh weight basis, were 1.3, 1.4, 1.8, <u>1.9, 3.6</u>, 3.8, 4.6 and 8.5 mg/kg. . Allowing the standard 35% dry matter content (DM) in soybean forage (FAO Manual, 2002), the media and the highest residues in forage, on a dried basis, are 7.86 mg/kg [2.75/0.35] and 24.3 mg/kg (8.5/0.35), respectively.

The Meeting agreed to withdraw the current MRL of 100 mg/kg (T fresh weight) and recommends a maximum residue level of 30 mg/kg and a STMR of 7.86 for carbaryl in soybean forage green, dried basis.

Residues from 9 trials conducted at maximum GAP in USA in soybean hay, residues within 21 days PHI were <0.02, 2.6, 4.0, 6.3,  $\underline{6.4}$  (2), 8.0, 8.4 and 9.6 mg/kg. Allowing a 85% DM, the median and the highest level, on a dried basis, are 7.5 mg/kg (6.4/0.85) and 11.3 mg/kg (9.6/0.85), respectively.

The Meeting recommends a maximum residue level of 15 mg/kg and an STMR of 7.5 mg/kg for carbaryl in soybean hay.

## Maize fodder and forage

Six trials were conducted in sweet corn in USA in 1995 at maximum GAP giving residues in forage at 14 days PHI, on a fresh weight base, of 1.8 (2), 3.8, 12, 124 and 163 mg/kg. Eight trials were conducted in field corn in USA at maximum GAP giving residues, on a fresh weight basis, in forage of 1.2, 2.0, 4.1, 7.7, 10, 16 and 24 (2) mg/kg.

The forage residue populations coming from trial conducted in sweet and field corn were found to represent similar populations which can be combined (Mann-Whitney U-test), as , in rank order, 1.2, 1.8 (2), 2.0, 3.8, 4.1, 7.7, 10, 12, 16, 24 (2), 124 and 163 mg/kg. Allowing for 44% dry matter content (DM) in corn forage (average between %DM of sweet corn forage and field corn forage, FAO Manual, 2002), the medium and the highest residues of carbaryl in maize forage, on a dried base, is  $20 \text{ mg/kg} \left[ (7.7+10)/2 * 0.44 \right]$  and  $370 \text{ mg/kg} \left( 163/0.44 \right)$ , respectively.

The Meeting agreed to withdraw the current MRL of 100~mg/kg (T, fresh weight) and recommends a maximum residue level of 400~mg/kg and an STMR of 20~mg/kg for carbaryl in maize forage, dried basis.

Residues in maize fodder from six trials conducted in sweet corn in USA at maximum GAP were 0.24, 0.62, 1.5 (2), 68 and 184 mg/kg at 48 days PHI, fresh weight. Eight trials conducted in field corn in USA at maximum GAP gave residues in fodder, on a fresh weight basis, of 0.06, 0.14, 0.38, 0.46, 0.70, 0.71, 2.4 and 7.6 mg/kg. The fodder residue populations coming from trials conducted in sweet and field corn were found to represent similar populations which can be combined (Mann-Whitney U-test) as, in rank order, 0.06, 0.14, 0.24, 0.38, 0.46, 0.62, 0.70, 0.71, 1.5 (2), 2.4, 7.6, 68 and 184 mg/kg. Allowing for 83% dry matter content (DM) in corn fodder (stover, FAO Manual, 2002), the median and the highest residues of carbaryl in maize fodder, on a dried base, is 0.85 mg/kg 0.705/0.83] and 221 mg/kg (184/0.83), respectively.

The Meeting recommends a maximum residue level of 250 mg/kg and an STMR of 0.85 mg/kg for carbaryl in maize fodder, dry basis.

#### Barley forage and straw

Thirty two trials were conducted in barley forage and straw in USA, where there is no approved used for barley. Two trials conducted according to Canadian GAP of aerial application gave residues of <0.2 mg/kg and 0.4 mg/kg in straw.

As no sufficient number of trials were conducted, the Meeting agreed not to recommend a maximum residue level for carbaryl in barley straw and forage

#### Rice straw

In nine trials conducted in rice straw in USA according to maximum GAP, residues were 7.5, 9.4, 14, 23 (2), 26, 47, 48 and 102 mg/kg. Allowing for 90% DM (FAO Manual, 2002), the medium and the highest residue in rice straw are 25.6 (23/0.9] and 113 mg/kg (102/0.9), respectively.

The Meeting recommends a maximum residue level of 120 mg/kg and an STMR of 25.6 mg/kg for carbaryl in rice straw.

# Rye forage and straw

In seven trials conducted in rye forage and straw with 2 applications at 1.68 kg a.i./ha, residues in forage at PHI from 0 to 4 days varied from 4 to 81 mg/kg (3 trials). Residues in rye straw from 5 trials ranged from 0.24 to 35 mg/kg at PHI from 7 to 21 days. There is no approved GAP for rye in USA.

As no supervised trials was conducted according to approved GAP, the Meeting agreed not to recommend a maximum residue level for carbaryl in rye forage and rye straw.

## Sorghum forage and fodder

Ten trials were conducted in sorghum forage and silage in USA in 1994 at maximum GAP rate and 14 days PHI. In nine trials conducted in fodder, samples were collected before the recommended 21 days PHI, and residues ranged from 0.04 to 22 mg/kg. Residues in forage were 0.08, 0.41, 0.60, 0.85, 1.0, 2.0, 4.1, 7.3, 12 and 14 mg and in silage varied from 0.38 to 6.2 mg/kg. Allowing for 35% DM (FAO Manual, 2002), the median and the highest residue in sorghum forage are 4.3 mg/kg (1.5/0.35] and 40 mg/kg (14/0.35), respectively, on a dried base.

The Meeting agreed to withdraw the current MRL of 100 mg/kg (T, fresh weight) and recommends a maximum residue level of 50 mg/kg, an STMR of 4.3 mg/kg for carbaryl in sorghum forage, dried basis.

### Wheat straw and forage

In five trials conducted in wheat forage, samples were harvested before the recommended PHI of 7 days, and the results could not be used.

Five trials were conducted in wheat straw in USA at maximum GAP, yielding residues of 0.92, 5.2, <u>8.2</u>, 11 and 22 mg/kg at 21 days PHI. Allowing for 88% DM (FAO Manual, 2002), the mediam and the highest residue are 9.3 mg/kg (8.2/0.88] and 25 mg/kg (22/0.88), respectively, on a dried base.

The Meeting recommends a maximum residue level of 30 mg/kg and an STMR of 9.3 mg/kg for carbaryl in wheat straw.

## Almond hulls

In five trials conducted in almond hulls in USA at maximum GAP rate, residues were 5, 16, <u>27</u>, 36 and 39 mg/kg aft 14 days PHI. Allowing for 90% DM (FAO Manual, 2002), the medium and the highest residue in almond hulls are 30 mg/kg (27/0.9] and 43.3 mg/kg (39/0.9), respectively, on a dried base.

The Meeting recommends a maximum residue level of 50 mg/kg and an STMR of 30 mg/kg for carbaryl in almond hulls.

### Fate of residues in processing

Four processing studies were conducted in USA in 1985 and 1994 in citrus fruit, one in grapefruit, one in lemon and two in oranges. The fruits were treated with carbaryl and processed using procedures similar to commercial practices. Residues concentrated in oil in all fruits, with processing factors (PF) of 22.3 in grapefruit, 44 in lemon and 25.8 and 2.4 in orange (average of 23.6 for citrus, n=4). In molasses, the residues concentrated in grapefruit and lemon (PF of 3.2 and 1.2, respectively), but not in orange (0.34 and 0.08, average 0.21). Residues were 10-30% higher in peel (average PF of 1.2 in citrus, n=3), and reduced in juice (average PF of 0.03 for citrus, n=4, and of 0.01 for orange, n=2), wet pulp (average PF of 0.46 in citrus, n=3) and in dried pulp (average PF of 0.24 for citrus, n=4). Washing the fruits removed the residues with a PF of 0.43 (grapefruit), 0.9 (lemon), 0.46 and 0.18 (orange), with an average PF of 0.49 in citrus fruit.

Field-treated apples under various field conditions were processed simulating commercial practice in three studies conducted in USA in 1985 and 1994 and in France in 1997. After washing, residues in apples were reduced with a PF of 0.54 (n=2). Residues concentrated in wet and dry pomace with an average PF of 1.1 (n=2) and 3.1 (n=2), respectively. Residues reduced in juice (PF of 0.38, n=2), and in peeled apple, refined pulp and compote from either peeled or unpeeled apple, with a PF of 0.5.

Four studies were conducted in USA from 1985 to 1994 with grapes treated and processed in a close approximation of commercial practice. In average, residues reduced in juice (PF of 0.65, n=2), but concentrated in wet and dry pomace (PF of 1.4 and 2.0, respectively, n=2), raisins (PF of 1.2, n=6), washed raisins (PF of 1.4, n=3) and raisin waste (PF of 3.0, n=6).

Prunes treated and commercially processed, had the residues reduced after washing and dried (PF of 0.26 and 0.15, respectively).

In one processing study in treated olive conducted in 1994, three samples of washed and cleaned fruits were ground in a Rietz type mill and crushed with a hydraulic press. The oil obtained by centrifugation and filtration had lower residues than the olives, with an average processing factor of 0.82 (n=3).

Two processing studies were conducted in tomato in USA in 1985 and 1994. Tomato were treated at exaggerated rates and processed using comparable procedures, simulating normal commercial practices. Residues were reduced in juice by a PF of 0.5 and did not change in puree, but concentrated in wet and dry pomace and paste, with PF of 2.1, 1.7 and 2.0, respectively (n=2).

In one study conducted in treated sweet corn the cannery waste produced by blending 1/3 of the husks with 1/3 of the cobs. Residues in cannery waste were higher than in sweet corn (kernel plus cob with the husks removed) by an average processing factor of 74 (n=4).

Soybeans treated with carbaryl at 2 times the label rate were processed into hulls, meal, crude oil, refined oil and soapstock. Residues of carbaryl increased in hulls (PF= 1.3), but reduced in meal (PF= 0.03), crude oil (PF = 0.9), refined oil and in soapstock (PF<0.01).

In one study conducted in 1985, potatoes treated with carbaryl were processed into fries, chips and flakes. Residues reduced in washed tubers with a PF of 0.75, in fries with a PF of 0.04, and in chips and flakes with a PF of 0.03. In a second study, conducted in 1994, potatoes treated 3 times with 11.2 kg carbaryl/ha and harvested at 7 days PHI contained 0.03 mg/kg of carbaryl. After being processed, no residues were detected (<0.02 mg/kg) in potato chips, flakes, and dry peel.

In a study conducted in sugar beets treated at 4 times the label rate a roots were processed in a pilot plant representative of actual conditions. Residues in the processing commodities wet pulp, dry pulp, molasses and refined sugar were <0.02 mg/kg. A processing factor for these commodities can be estimated to be <0.09.

In two processing studies conducted in field corn treated with carbaryl, grains were processed to produce fractions from dry and wet milling, using procedures that simulated industrial practices. While in one study conducted in 1985 residues increased in meal and flour produced by dry milling by PFs of 1.3 and 1.7, respectively, in the other conducted in 1994, they were reduced by a factor of <0.05. Residues in grits reduced in both studies by an average PF of <0.4 and in crude oil produced by dry and wet milling it increased by an average PF of 3.3 (n=2). Residues increase in germ by a factor of 1.8 and reduced in starch with PF <0.5 (n=2) and in refined oil (PF<0.4, n=3).

Two studies were conducted in USA in 1985 and 1994 in rice treated and processed in a lab-scale procedure close to commercial practice. Residues of carbaryl concentrated in hulls by a PF of 3.3 and reduced in bran and polished rice by PFs of 0.68 and 0.02, respectively (n=2).

In one study conducted in rye treated with carbaryl, residues in grain concentrated in bran, flour and shorts with processing factors of 1.4, 1.1 and 1.7 and reduced in middlings by a factor of 0.8. No detail of the processing procedure was giving in the report.

In one study conducted in treated sorghum, grain samples were dry and wet milled using simulating commercial procedures. Residues in bran increased by a factor of 2.3 (n=2), and reduced in flour by a PF of 0.16 (n=2), in shorts (PF=0.7) and in grits (PF=0.2).

In one study conducted in sweet sorghum, stalks treated at the same rate were crushed in a standard roll mill to produce the crushed stalks (bagasse) and juice, which was heated until 60-70% solid concentration (syrup). Residues of carbaryl increased in bagasse and syrup, with processing factors of 7.8) and 1.6, respectively.

In a processing study conducted in USA, treated wheat grain samples were processed simulating commercial practices. Residues of carbaryl remained almost the same in wheat bran (PF= 1.03) and reduced in low grade flour (PF=0.08), patent flour (PF=0.10) and wheat germ (PF=0.49). The patent flower is made from the finer and whiter flour streams, with lower bran content and higher endosperm content.

One study was conducted in 1995 in USA in peanuts treated with carbaryl and processed by procedures close to commercial practices. Residues in nutmeat were 0.04 mg/kg and were not detected (<0.02mg/kg) in meal and oil (PF<0.5).

Three treated <u>cotton</u> samples were processed in a procedure which duplicate normal commercial practices. Residues concentrated in crude oil (PF of 3.4, n=2) and reduced in hulls, meal and refined oil, with PFs of 0.35, 0.59 and <0.04 (n=3).

In one study conducted in 1994 in USA, three seed samples from a sunflower field plot treated with carbaryl were processed in a procedure which simulates industrial practice. Residues reduced in hulls, meal, crude and refined oil by processing factors of 0.48, <0.06, 0.18 and <0.06 (n=3).

#### Residues in processed commodities

Residues in processed commodities will be derived by multiplying the residues (maximum residue level, STMR and HR) in the raw commodity estimated from the supervised trials conducted according to GAP and the processing factors (PF) found in the processing studies conducted in the commodity. Estimations will only be derived for commodities of human consumption, for commodities of animal consumption, which can be used to estimate the animal dietary burden, or for commodities with a Codex code.

Based on a processing factor of 0.03 for citrus juice, of 0.24 for dried citrus pulp and the estimations for citrus fruit (maximum residue level of 15 mg/kg and an STMR of 4.2 mg/kg), the Meeting agreed to recommend a maximum residue level of 0.5 mg/kg and an STMR-P of 0.13 mg/kg for carbaryl in citrus juice, and a maximum residue level of 4 mg/kg and an STMR-P of 1.0 mg/kg for citrus pulp, dried.

Based on a processing factor of 2 for grape pomace, dry, of 1.2 for raisins and of 0.65 for grape juice, the estimations for grape (maximum residue level of 40 mg/kg, STMR of 4.9 mg/kg and an HR of 33 mg/kg), the Meeting recommends a maximum residue level of 80 mg/kg and an STMR-P of 9.8 mg/kg for carbaryl in grape pomace, dry; a maximum residue level of 50 mg/kg, an STMR-P of 5.9 mg/kg and an HR of 39.6 mg/kg for carbaryl in raisins; and a maximum residue level of 30 mg/kg and an STMR-P of 3.2 mg/kg for carbaryl in grape juice.

Based on a processing factor of 0.82 from olive to olive oil, and the estimations for olive (maximum residue level of 30 mg/kg and an STMR of 3.65 mg/kg ), The Meeting recommends a maximum residue level of 25 mg/kg and an STMR-P of 2.99 mg/kg for carbaryl in olive oil.

Based on a processing factor of 0.5 from tomato to tomato juice\_and of 2 from tomato to tomato\_paste and the estimations for tomato (maximum residue level of 5 mg/kg and STMR of 0.47 mg/kg), the Meeting recommends a maximum residue level of 3 mg/kg, and an STMR-P of 0.24 mg/kg g for carbaryl in tomato juice and a maximum residue level of 10 mg/kg and an STMR-P of 0.94 for carbaryl in tomato paste.

Based on the processing factor of 74 for sweet corn cannery waste and the estimations for sweet corn (maximum residue level of 0.1~mg/kg and STMR of 0.02~mg/kg), The Meeting estimates a maximum residue level of 7.4~mg/kg and an STMR-P of 1.48~mg/kg for carbaryl in sweet corn cannery waste.

Based on processing factors of 1.3 and 0.9 from soybeans to hulls and crude oil, respectively, and the estimations for soybeans (maximum residue level of 0.2 mg/kg and STMR of 0.05 mg/kg), the Meeting recommends a maximum residue level of 0.3 mg/kg, a STMR-P of 0.065 mg/kg for carbaryl in soybeans hulls, and a maximum residue level of 0.2 mg/kg and a STMR-P of 0.045 mg/kg for carbaryl in soybeans oil, crude. As the PF for soybean meal is low (0.03), it is unlikely that residues of carbaryl will remain in this fraction and no estimations will be performed in soybean meal.

As the estimations for carbaryl in sugar beet were at the LOQ (0.05 mg/kg) and the processing factors for pulp, molasses and sugar were <0.09, the Meeting agreed not proceed on the estimations for processed commodities of sugar beet.

The estimations for carbaryl in field corn were at the LOQ (0.02 mg/kg). Processing factors for grits and refined oil were <0.4, and the results from two studies in meal and

flour varied significantly (0.05 and 1.5). Based on a PF of crude oil of 3.3, the Meeting recommends a maximum residue level of 0.1 mg/kg and an STMR-P of 0.066 mg/kg for carbaryl in maize oil, edible.

Based on processing factors of 3.3, 0.68 and 0.02 from rice to hulls, bran and polished rice and the estimations for rice (maximum residue level of 50 mg/kg, STMR of 8.4 mg/kg and HR of 46 mg/kg), the Meeting recommends a maximum residue level of 170 mg/kg and an STMR-P of 27.7 mg/kg for carbaryl in rice hulls; a maximum residue level of 35 mg/kg, an STMR-P of 5.7 mg/kg for carbaryl in rice bran; and a maximum residue level of 1 mg/kg, an STMR-P of 0.168 mg/kg and an HR-P of 0.92 mg/kg for carbaryl in polished rice.

Based on processing factors of 1, 0.09 and 0.49 from wheat to bran, flour and germ and the estimations for wheat (maximum residue level of 2 mg/kg, and STMR of 0.26 mg/kg), the Meeting recommends a maximum residue level of 2 mg/kg and an STMR-P of 0.26 mg/kg for carbaryl in wheat bran; and a maximum residue level of 0.2 mg/kg, and an STMR-P of 0.2 mg/kg for carbaryl in wheat flour; a maximum residue level of 1 mg/kg and an STMR-P of 0.13 mg/kg for carbaryl in wheat germ.

Based on processing factors of 0.18 from sunflower to crude oil and the estimations for sunflower seed (maximum residue level of 0.2 mg/kg and STMR of 0.03 mg/kg), the Meeting recommends a maximum residue level of 0.05 mg/kg and an STMR-P of 0 mg/kg for carbaryl in sunflower seed crude oil. As the PF factor for meal is <0.06, no estimations will be conducted for this processed commodity.

### Animal dietary burden

The Meeting estimated the dietary burden of carbaryl in cow on the basis of the diets listed in Appendix IX of the FAO Manual (FAO, 2002) and the MRL and STMR estimated at this Meeting.

## Maximum farm dietary burden estimation

						% of diet			Resi	Residue contribution, mg/kg		
Commodity	_	Residues mg/kg	Basis		Residues, in dry basis, mg/kg	Beef	Dairy	Poultry	Beef	Dairy	Poultry	
Citrus pulp, dried	AB	1.0	STMR-P	91	1.1	20	20	-	0.22	0.22	-	
Almond hulls	AM	50	MRL	90	45	10	10	-	4.5	4.5	-	
Rice hulls		25.7	STMR-P	90	28.5	10	10	15	2.85	2.85	4.3	
Sweet corn cannery waste		2.22	STPM-P	30	7.4	35	20	-	2.59	1.48	-	
Maize forage	AF	400	MRL	100	400	40	50	-	160	250	_	
Sorghum forage	AF	50	MRL	100	50	40	50	-	10	25	-	
Soybean hay	AL	15	MRL	100	15	30	30	-	4.5	4.5	_	
Soybean forage	AL	30	MRL	100	30	30	25	-	9.0	7.5	-	
Rice straw	AS	120	MRL	100	120	10	10	-	12	12	-	
Maize fodder (stover)	AS	250	MRL	100	250	10	05		25	12.5	-	
Wheat straw	AS	30	MRL	100	30	10	10	_	3	3	_	

						% of diet			Residue contribution, mg/kg		
Commodity		Residues mg/kg	Basis		Residues, in dry basis, mg/kg	Beef	Dairy	Poultry	Beef	Dairy	Poultry
Rice	GC	50	MRL	88	56.8	10	10	60	5.7	5.7	34.1
Maize	GC	0.02	MRL	88	0.023	80	40	40	0.018	0.009	0.018
Wheat grain	GC	2	MRL	89	2.24	50	40	80	1.12	0.90	1.8
Soybean seed	VD	0.2	MRL	89	0.22	15	15	20	0.03	0.03	0.04
TOTAL	ГОТАL						100	100	208.6	279.6	34.3

# STMR farm animal dietary burden estimation

							% of diet			Residue contribution, mg/kg		
Commodity	Group	Residues mg/kg	Basis	% dry matter	Residues, in dry basis, mg/kg	Beef	Dairy	Poultry	Beef	Dairy	Poultry	
Citrus pulp, dried	AB	1.0	STMR-P	91	1.1	20	20	_	0.22	0.22		
Almond hulls	AM	30	STMR-P	90	33.3	10	10	_	3.33	3.33		
Rice hulls		27.7	STMR-P	90	30.8	10	10	15	3.1	3.1		
Sweet corn cannery waste		2.22	STPM-P	30	7.4	35	20	-	2.59	1.48		
Maize forage	AF	20	STMR	100	20	40	50	-	8.0	10		
Sorghum forage	AF	1.5	STMR	35	4.3	40	50	_	1.7	2.15		
Soybean hay	AL	7.5	STMR	100	7.5	30	30	_	2.25	2.25		
Soybean forage	AL	7.9	STMR	100	7.9	30	20	_	2.4	1.58		
Rice straw	AS	25.6	STMR	100	25.6	10	10	_	2.56	2.56		
Maize fodder (stover)	AS	0.85	STMR	100	0.85	25	15		0.21	0.13		
Wheat straw	AS	9.3	STMR	100	9.3	10	10	_	0.93	0.93		
Rice	GC	8.4	STMR	88	9.3	10	10	60	0.93	0.93	6.6	
Maize	GC	0.02	STMR	88	0.23	80	40	40	0.18	0.09	0.09	
Wheat grain	GC	0.26	STMR	89	0.29	50	40	80	0.15	0.12		
Soybean seed	VD	0.05	STMR	89	0.056	15	15	20	0.01	0.01		
TOTAL						100	100	100	17.3	17.3	6.7	

# **Animal feeding studies**

Dairy cattle were orally dosed daily with carbaryl for a period of 28 days at 114 ppm (Group II), 342 ppm (Group III) and 1140 ppm, changed to 570 ppm at day 5 (Group IV). Cows were milked twice daily and a day's sample consists of a proportional mix of PM milk and the following morning's AM milk. Test animals were terminated within 7 hours after receiving the final dose and samples, of muscle, fat, liver, and kidney were collected for analysis. Milk (days 1, 4, 8, 11, 15, 18, 22, 25 and 28), milk fat (days 22 and 27 of Group IV) and tissue samples were analysed for carbaryl and the metabolites 5,6-dihydro-5,6 dihydroxy carbaryl and 5-methoxy-6-hydroxy carbaryl.

Average residues of carbaryl in milk analysed throughout the study in each group increased with the dose rate, with 0.02, 0.04 and 0.06 mg/kg for the groups II, III and IV, respectively. 5,6-dihydro-5,6 dihydroxy carbaryl was the major residue in all dosing groups, with average residues of

0.15, 0.46 and 1.1 mg/kg in milk from cows from groups II, III and IV, respectively. Average 5-methoxy-6-hydroxy carbaryl residues were 0.11, 0.18 and 0.21 mg/kg.

Carbaryl and 5,6-dihydro-5,6 dihydroxy carbaryl was the main compounds found in kidney and liver and 5,6-dihydro-5,6 dihydroxy carbaryl was the main compound found in muscle. In kidney, average carbaryl concentrations were 0.69, 2.1 and 2.3 mg/kg and in liver 0.49, 0.93 and 1.1 mg/kg, in groups II, III and IV, respectively. 5,6-dihydro-5,6 dihydroxy carbaryl residues in kidney were 0.60, 2.0 and 3.7 mg/kg and in liver 0.21, 0.58 and 1.2 mg/kg. Muscle tissue contained 0.31, 0.97 and 1.9 mg/kg of 5,6-dihydro-5,6 dihydroxy carbaryl, and residues of carbaryl ranged from <0.02 to 0.04 mg/kg. In fat, carbaryl levels ranged from 0.02 to 0.06 mg/kg and of 5,6-dihydro-5,6 dihydroxy carbaryl from 0.06 to 0.18 mg/kg. 5-methoxy-6-hydroxy carbaryl was mostly present in kidney, at concentrations of 0.07, 0.45 and 0.86 mg/kg. In liver and fat, residues ranged from 0.02 to 0.09 mg/kg and it was not detected in muscle.

### **Animal commodities residue levels**

### Cattle

As the maximum dietary burden of beef and dairy cattle estimated by the Meeting were 208.6 and 279.6 mg/kg feed, respectively, the highest value (279.6 mg/kg feed) will be used for calculation of the residues. The levels will be derived from the interpolation between the levels found in animals from group II (114 ppm) and group III (342 ppm). For the STMR estimation, the residue levels at 17.3 mg/kg feed (dietary burden for both beef and dairy cattle), will be derived by applying the transfer factor (residue level in milk or tissue/residue level in diet) at the lowest feeding level (114 ppm) to the dietary burden.

Residue levels of carbaryl reached a maximum in milk at day 4 four in cows from the feeding groups III and IV, and the levels dropped to 85 and 24% of the maximum at day 28, respectively. Levels at milk from the lowest feeding group increased up to 24% of the initial value between days 18 to 25. The Meeting agreed that the maximum residue levels in tissues will be derived from the levels found at the maximum dietary burden, using the highest residue level. The STMRs will be derived from the STMR dietary burden and the mean residue levels. For milk, the mean residue at the plateau level from the relevant feeding group will be used to estimate both the maximum residue level and the STMR.

Dose (ppm)	Carbaryl concentration (mg/kg)									
(Interpolated)	Milk	Liver		Kidney		M	uscle	Fat		
[actual]	(mean)	Highest	Mean	Highest	Mean	Highest	Mean	Highest	Mean	
MRL										
(279.6)	(0.034)	(0.907)		(1.90)		(<0.042)		(0.062)		
[114/	[0.02/	[0.66/		[0.85/		[<0.02/		[0.04/		
342]	0.04]	1.0]		2.3]		0.05]		0.12]		
STMR										
(17.3)	(0.003)		(0.085)		(0.119)		(<0.003)		(0.003)	
[114]	[0.02]		[0.49]		[0.69]		[<0.02]		[0.02]	

The Meeting agreed to withdraw the current MRL of 0.1 mg/kg \* (T) and recommends a maximum residue level of 0.05 mg/kg and an STMR of 0.003 mg/kg for carbaryl in milks.

The Meeting recommends a maximum residue level of 1 mg/kg, an STMR of 0.085 mg/kg and an HR of 0.907 mg/kg for carbaryl in liver of cattle, goats, pigs and sheep.

The Meeting recommends a maximum residue level of 3 mg/kg, an STMR of 0.119 mg/kg and an HR of 1.9 mg/kg for carbaryl in kidney of cattle, goats, pigs and sheep.

For the purpose of dietary intake calculation, the Meeting also estimates an STMR of 0.003 mg/kg and an HR of 0.062 mg/kg for carbaryl in fat from mammals other than marine mammals.

The Meeting agreed to withdraw the current MRLs of 0.2 mg/kg (T) for cattle meat, goat meat and sheep meat and recommends a maximum residue level of 0.05 mg/kg, an STMR of 0.02 mg/kg and an HR of 0.05 mg/kg for carbaryl in meat (from mammals other than marine mammals).

#### **Poultry**

For poultry, the maximum and the STMR estimated dietary burden were 34.4 and 6.4 mg/kg feed, respectively. Metabolism studies on hens conducted at 8.8 and 10.5 mg/kg feed (7 consecutive days orally dosed) showed detectable residue of carbaryl in egg yolks, liver and abdominal fat (0.001 to 0.004 mg/kg <sup>14</sup> carbaryl eq.). The Meeting agreed that this study is not adequate to estimate maximum residue levels of carbaryl in poultry.

#### **FUTHER WORK OR INFORMATION**

1. Feeding study on poultry

### RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels shown below are suitable for establishing maximum residue limits and for dietary intake assessment.

Definition of residue for compliance with MRLs and for estimation of dietary intake in plant and animal commodities: carbaryl

	Commodity	MRI	., mg/kg	STMR or STMR-P	HR or HR-P
CCN	Name	New	Previous	mg/kg	mg/kg
AL 1021	Alfalfa forage (green)	W	100 T		
AM 0660	Almond hulls	50		30	
FP 0226	Apple	W	5 T		
FS 0240	Apricot	W	10 T		
VS 0621	Asparagus	15	10 T	8.1	10
FI 0327	Banana	W	5 T		
GC 0640	Barley	W	5 PoT		
AL 1030	Bean forage (green)	W	100 T		
VR 0574	Beetroot	0.1	2 T	0.025	0.06
FB 0264	Blackberries	W	10 T		
FB 0020	Blueberries	W	7 T		
VB 0041	Cabbages, Head	W	5 T		
VR 0577	Carrot	0.5	2 T	0.02	0.31
MM 0812	Cattle meat	W	0.2 T		
FS 0013	Cherries	20	10 T	4.3	16
FC 0001	Citrus fruit	15	7 T		

	Commodity	MRI	., mg/kg	STMR or STMR-P	HR or HR-P
CCN	Name	New	Previous	mg/kg	mg/kg
	Citrus fruit, edible portion			0.487	1.16
JF 0001	Citrus juice	0.5		0.13	
AB 0001	Citrus pulp, dry	4		1.0	
AL 1023	Clover	W	100 T		
VP 0526	Common bean (pods and/or immature seeds)	W	5 T		
SO 0691	Cotton seed	W	1 T		
VD 0527	Cowpea (dry)	W	1 T		
FB 0265	Cranberry	W	7 T		
VC 0424	Cucumber	W	3 T		
FB 0266	Dewberries (including Boysenberry and Loganberry)	W	10 T		
DF 0269	Dried grapes	50		5.9	39.6
VO 0440	Egg plant	1	5 T	0.18	0.49
PE 0112	Eggs	W	0.5 T		
MM 0814	Goat meat	W	0.2 T		
JF 0269	Grape juice	30		3.2	
AB 0269	Grape pomace, dry	80		9.8	
FB 0269	Grapes	40	5 T	4.9	33
AS 0162	Hay or fodder (dry) of grasses	W	100 T		
MO 0098	Kidney of cattle, goats, pigs and sheep	3		0.119	1.9
FI 0341	Kiwifruit	W	10 T		
VL 0053	Leafy vegetables	W	10 T		
MO 0099	Liver of cattle, goats, pigs and sheep	1		0.085	0.907
GC 0645	Maize	0.02 (*)		0.02	0.02
AS 0645	Maize fodder	250, dry		0.85	
AF 0645	Maize forage,	400, dry	100 T, fresh	20	
OC 0645	Maize oil, crude	0.1		0.066	
MF 0100	Mammalian fats (except milk fats)			0.003	0.062
MM 0095	Meat (from mammals other than marine mammals)	0.05		0.02	
VC 0046	Melons, except Watermelon	W	3 T		
AO3 0001	Milk products	W	0.1 (*)T		
ML 0106	Milks	0.05	0.1 (*) T	0.03	
FS 0245	Nectarine	W	10 T		
AO5 1900	Nuts (whole shell)	W	10 T		
GC 0647	Oats	W	5 PoT		
VO 0442	Okra	W	10 T		
OC 0305	Olive oil, virgin	25		2.99	
FT 0305	Olives	30	10 T		
	Olives, edible portion			5.1	36.4
DM 0305	Olives, processed	W	1 T		
VR 0588	Parsnip	W	2 T		
AL 0528	Pea vines (green)	W	100 T		
AL 0697	Peanut fodder	W	100 T		
SO 0703	Peanut, whole	W	2 T		
FP 0230	Pear	W	5 T		
VP 0063	Peas (pods and succulent = immature seeds)	W	5 T		
VO 0445	Peppers, sweet	5	5 T Peppers	1.8	3.8
FS 0014	Plums (including prunes)	W	10 T		

	Commodity	MRI	., mg/kg	STMR or STMR-P	HR or HR-P
CCN	Name	New	Previous	mg/kg	mg/kg
VR 0589	Potato	W	0.2 T		
PM 0110	Poultry meat	W	0.5 T (1)		
PO 0113	Poultry skin	W	5 T (1)		
VC 0429	Pumpkins	W	3 T		
VR 0494	Radish	W	2 T		
FB 0272	Raspberries, Red, Black	W	10 T		
GC 0649	Rice	50	5 PoT	8.4	46
CM 1206	Rice bran, unprocessed	170		5.7	
	Rice hulls	50		25.7	
AS 0649	Rice straw and fodder, dry	120		25.6	
CM 0649	Rice, husked	W	5 PoPT		
CM1205	Rice, polished	1		0.168	0.92
GC 0650	Rye	W	5 PoT		
MM 0822	Sheep meat	W	0.2 T		
GC 0651	Sorghum	W	10 PoT		
	Sorghum forage, dry	50	-5101	4.3	
AF 0651	Sorghum forage, green	20	100 T	1.5	
VD 541	Soya bean (dry)	0.2	1 T	0.05	0.15
AL 0541	Soya bean fodder	15	11	7.5	0.15
AL 1265	Soya bean forage, green	30, dry	100 T, fresh		
OC 0541	Soya bean oil, crude	0.2	100 1, 110011	0.045	
00 00 .1	Soya bean, hulls	0.3		0.065	
VC 0431	Squash, Summer	W	3 T	0.002	
FS 0012	Stone fruits	10	0.1	2.05	7.8
FB 0275	Strawberry	W	7 T		,,,,
VR 0596	Sugar beet	W	0.2 T		
AV 0596	Sugar beet leaves or tops	W	100 T		
	Sunflower forage	5		1.9	
SO 0702	Sunflower seed	0.2		0.03	0.08
OC 0702	Sunflower seed oil, crude	0.05		0	
VR 0497	Swede	W	2 T	Ü	
VO 0447	Sweet corn (corn-on-the-cob)	0.1	1 T	0.02	0.05
100117	Sweet corn cannery waste	7.4		1.48	0.00
VR 0508	Sweet potato	0.02 (*)		0.02	0.02
VO 0448	Tomato	5	5 T	0.47	2.4
JF 0448	Tomato juice	3	J 1	0.24	2.1
	Tomato paste	10		0.94	
TN 0085	Tree nuts	1	1 T	0.035	0.77
VR 0506	Turnips, Garden	1	1.1	0.02	0.89
GC 0654	Wheat	2	5 PoT	0.245	1.6
CM 0654	Wheat bran, unprocessed	2	20	0.17	1.0
CF 1211	Wheat flour	0.2	0.2 PoPT	0.02	
CF 1211	Wheat germ	1	0.21011	0.12	
AS 0654	Wheat straw	30		9.3	
	Wheat wholemeal	W	2 PoPT	7.3	
CF 1212			. /. LUE L		

<sup>(1)</sup> accommodates external animal application

### DIETARY RISK ASSESSMENT

# Long-term intake

The ADI for carbaryl is 0.008 mg/kg body weight/day. International estimated daily intake (IEDI) was calculated for commodities of human consumption which STMRs were estimated in this evaluation. The results are shown in Anex III.

International Estimated Daily Intakes for the five GEMS/Food regional diets, based on estimated STMRs, ranged from 10 to 60 % of the ADI. The Meeting concluded that the intake of residues of carbaryl resulting from its uses that have been considered by the JMPR is unlikely to present a public heath concern.

### Short- term intake

The acute RfD for carbaryl is 0.2 mg/kg body weight. The international estimate of short term intake (IESTI) for carbaryl was calculated for food commodities for which maximum residue levels, STMR values and/or HR values were established at this Meeting. The results are shown in Annex IV.

The IESTI for grapes was 420% of the acute RfD for the adult population and 1100% of the acute RfD for the children. The IESTI for apricot, cherries, peaches and plums were 130%, 130%, 170% and 140% of the acute RfD for children, respectively. The information provided to the Meeting precludes an estimate that the dietary acute intake of grapes by children and adults and of apricot, cherries, peaches and plums by children would be below the acute reference dose.

For all the other commodities considered the % of the acute RfD varied from 8-80%. The Meeting concluded that short-term intake of residues of carbaryl in these commodities, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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